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OBJECTIVES AND FOCUS FOR FY1995

NASA's Microgravity Science and Applications Division (MSAD) sponsors a program that seeks to focus on the use of space as a laboratory by undertaking the study of important physical, chemical, and biochemical processes on orbit that cannot easily be studied in the terrestrial gravity environment. However, since flight opportunities are rare and flight hardware is very expensive, a strong ground-based research program from which only the best experiments evolve is the cornerstone of the program.

The microgravity environment affords unique characteristics that allow the investigation of phenomena and processes that are difficult or impossible to study on Earth. Significant reductions in critical characteristics, such as buoyancy driven forces, convection, sedimentation, and hydrostatic pressures, make it possible to isolate and control gravity-related phenomena and make measurements that have significantly greater accuracy than can be achieved in normal gravity. Space flight gives scientists the opportunity to study the fundamental states of physical matter—solids, liquids, and gasses—and the forces that affect those states. Because microgravity tends to allow the treatment of gravity as a variable, research in microgravity leads to a greater fundamental understanding of the influence of gravity on the world around us. With appropriate emphasis, the results of space experiments lead to both knowledge and technological advances that have direct applications on Earth. Microgravity research also provides the practical knowledge essential to the development of future space systems.

The Office of Life and Microgravity Sciences and Applications (OLMSA) is responsible for planning and executing research stimulated by the Agency's broad scientific goals. OLMSA's Microgravity Science and Applications Division (MSAD) is responsible for guiding and focusing a comprehensive program, and currently manages its research and development tasks by dividing them into five major scientific disciplines: benchmark science, biotechnology, combustion science, fluid physics, and materials science.

FY 1995 was an important year for MSAD. Each of the major disciplines released new NRAs in FY 1995. The principal investigators chosen from those NRAs will form the core of the MSAD microgravity research program at the beginning of the space station era. The Space Shuttle made two historic linkups with the Mir space station in June and November of 1995 enabling NASA to conduct long-term microgravity research. Data from microgravity equipment placed on Mir are already being received and evaluated by NASA scientists. Planning for the international Space Station facilities is also continuing with respect to the furnace, fluids and combustion facility, and a newly planned low-temperature microgravity physics facility. On-orbit research carried out on the Space Shuttle USML-2 (October, 1994) mission provided valuable insight into each of the five discipline fields. The processing and evaluation of these results have provided a solid basis for the planning of future microgravity missions.

This document, NASA Technical Memorandum 4735 [1996], The Microgravity Science and Applications Program Tasks and Bibliography for Fiscal Year 1995 (October 1994 – September 1995), includes research projects funded by the Office of Life and Microgravity Sciences and Applications, Microgravity Science and Applications Division, during that year. This document is published annually and is sent to scientists in the microgravity field, both foreign and domestic. The information provided in the Task Book is used in reports to the NASA Associate Administrator, the Office of Management and Budget, and to the United States Congress.

The Microgravity Science and Applications Division wishes to thank Information Dynamics, Inc., and in particular recognize Mr. Bill Wilcox (task book review process and publications manager) and Mr. Duke Reiber (data system development) for their lead efforts in the development, compilation and publishing of this report. Gratitude is also expressed for significant data processing support at the responsible Centers for MSAD task management, recognizing: Dr. Donald Strayer and Angela Belcastro, JPL; Phyllis Golden, JSC; Mary Malone, LeRC; and Charles Walker, MSFC.
I. MSAD Program Tasks & Bibliography — FY1995

Objective & Metrics

FY1995 PROGRAM RESEARCH TASK SUMMARY:
Overview Information and Statistics

Total Number of Principal Investigators: ................................................................. 290
Total Number of Co-Investigators: ......................................................................... 286

Total Number of Bibliographic Listings: ................................................................. 1,200
- Proceedings Papers: ............................................................................................ 140
- Journal Articles: .................................................................................................... 526
- NASA Tech Brief Articles: .................................................................................... 11
- Science/Technical Presentations: ......................................................................... 509
- Books/Chapters: .................................................................................................. 14

Total Number of Patents Applied for or Awarded: .................................................. 1

Number of Graduate Students Funded: ................................................................. 534
Number of Graduate Degrees Granted
Based on MSAD-funded Research: ........................................................................ 178

Number of States with Funded Research (including District of Columbia): .......... 34

FY1995 Microgravity Science & Applications Budget: .......................................... $163.5 Million

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II. MSAD Program Tasks — Flight Research

Discipline: Biotechnology

Protein Crystal Growth Vapor-Diffusion Flight Hardware and Facility

Principal Investigator: Dr. Daniel C. Carter
NASA Marshall Space Flight Center (MSFC)

Co-Investigators:
Ho, J. (X. He)
NASA Marshall Space Flight Center (MSFC)
Miller, T.
NASA Marshall Space Flight Center (MSFC)

Task Objective:
The major objectives of this research are to provide a user-friendly interface between ground-based and flight protein crystal growth hardware, increased (common) availability of flight hardware, elimination of late access requirement, and individual loading by the principal investigator.

Task Description:
Initially, these experiments will be conducted with a small hand-held unit using human serum albumin. Subsequent tests will involve the growth of Fab 3D6, a human anti-HIV antibody. When the necessary tests and procedures have been completed with the hand-held unit, facility flight hardware will be constructed. The microgravity experiments will be conducted in two stages. In the first stage, the hand-held unit will be flown to test the overall concept, refine the hardware if necessary, and establish protocols for later scale-ups with multi-user hardware. The design would then be configured to accommodate several trays and interface directly with the existing refrigerator/incubator module (R/IM) and thermal environment system (TES) temperature control h wavelengths without modification. In addition, a new plastic tray will be developed to provide additional advantages in optical, storage, and hardware interfaces with a potential increase in the number of experiments in each tray assembly (VDT). In the early periods of the second stage, the facility hardware will utilize cryogenics only for improvements in experiment pre-loading efficiency. In order to proceed with the cryogenic aspects of the second stage, a sub-zero freezer will have to be developed. When flight freezers are utilized, the hardware is left in the activation configuration, and protein crystallization proceeds after the experiments are withdrawn from the freezer and placed in the temperature control units.

Task Significance:
This research is concerned with the development of a protein crystal growth system for microgravity which provides for rapid, convenient access to the microgravity environment and a greater number of samples, and eliminates numerous problems associated with logistics and handling of the current flight hardware.

Progress During FY 1995:
PCAM facility ground testing was successfully completed and the hardware approved for flight. A co-investigator group was established by previously described procedures. Subsequently, PCAM was flown on STS-63, STS-67, and is currently installed on STS-73 (USML-2). The hardware performed well on the first two missions, growing representative crystals of all proteins as compared with the ground-based controls. Several advantages of the hardware have been clearly demonstrated. Altogether, STS-63 and STS-67 included eight co-investigators and twelve proteins for a total of 756 individual experiments. Flight experiment results were particularly good for Co-Investigator Dr. Mark Wardell of the Department of Hematology at Cambridge University. Crystals produced of human antithrombin III were the best ever obtained and have allowed for the complete construction and refinement of the atomic model. Two manuscripts describing this result have been submitted for publication. Our research group obtained outstanding results using hen egg white lysozyme as a model system. This structure was refined to yield the highest resolution detail of this structure to date. The refined structure and other important physical properties of these flight crystals have been determined and at least one manuscript describing the results has been...
II. MSAD Program Tasks — Flight Research

submitted for publication. Details of other promising results await the analysis of additional crystalline flight samples. To this end USML-2 PCAM experiments include eleven co-investigators from industry, academics and government laboratories and approval for eighteen different proteins.

The Diffusion-Controlled Crystallization Apparatus for Microgravity (DCAM) was successfully developed and constructed. The hardware has passed all safety and flight tests and is now installed on USML-2 as a demonstration experiment to assess its future performance on Space Station for which it is currently manifested. USML-2 carries 81 DCAM's in a Single Thermal Enclosure System (STES) in the Spacelab. The total number of experiments flown in the Protein Crystallization Apparatus for Microgravity (PCAM) and DCAM on USML-2 is greater than 1000, yielding a total number of protein experiments for 1995 which exceeds the number flown since the program inception in 1985 (based on available data).

STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 5/93  EXPIRATION: 11/97
PROJECT IDENTIFICATION: 963-45-08
RESPONSIBLE CENTER: MSFC
II. MSAD Program Tasks — Flight Research

Protein Crystal Growth in Microgravity

Principal Investigator: Dr. Lawrence J. DeLucas

University of Alabama, Birmingham

Co-Investigators:
Noted Guest Investigators

See List, Appendix B

Task Objective:
The objectives of this research are: to develop improved protein crystal growth flight hardware to produce larger, high quality protein crystals in microgravity for use in the determination of protein molecular structures for applications in medicine, drug design, agriculture, and the biological sciences; and to understand the dynamics/process of protein crystal growth.

Task Description:
A breadboard system will be developed that utilizes light scattering to detect the onset of crystal nucleation. This optical monitoring system will be developed for both vapor diffusion and temperature-induced crystallization techniques. This hardware will be used to grow crystals with dynamic control of the appropriate crystallization parameter (i.e., temperature or vapor diffusion). The crystals will be evaluated microscopically and, from this evaluation, the best crystals will be used for x-ray data collection using the facilities available within the CMC. The data will then be compared with the best data obtained from ground-based crystals, and an evaluation of the usefulness of these dynamically-controlled systems will be made.

Task Significance:
Larger, high quality protein crystals may be used in molecular structure determinations for applications in medicine, drug design, agriculture, and the biological sciences.

Progress During FY 1995:
Automated dynamic control of protein crystal growth using controlled vapor diffusion has been used to investigate the effect of varying the evaporation rate on the crystals obtained for a given solution condition. The system incorporates a static laser light scattering subsystem consisting of a laser, photodetector, and fiber optics which allows the evaporation profile to be modified in response to nucleation events. This system has shown that detection of nucleation and modification of the evaporation profile while the experiment is in progress can improve the crystal growth results.

DCPCG-Temperature: Automated dynamic control of protein crystal growth using temperature has been used to show the effects of varying ramp rates, set points, and nucleation detection levels on the crystal growth results for a given solution condition. A static laser light scattering subsystem is also included as a non-invasive probe for detection of aggregation events. This allows the nucleation and growth phases of crystal growth to be separated in an attempt to optimize them. Several versions of this system have been used to demonstrate the effects of various temperature profiles and led to the development of strategies for growing small populations of large crystals.

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Project Identification: 963-45-06

NASA Contract No.: NAS8-39762

Responsible Center: MSFC

Printed: 27 FEB 1996
Electrophoretic Separation of Cells and Particles from Rat Pituitary

Principal Investigator: Dr. Wesley C. Hymer

Pennsylvania State University

Co-Investigators:

Mastro, A.
Grindeland, R.
Snyder, R.

Pennsylvania State University
NASA Ames Research Center (ARC)
NASA Marshall Space Flight Center (MSFC)

Task Objective:
The objectives are to separate (1) pituitary cells and (2) hormone-containing granules by free flow electrophoresis using the Japanese Free-Flow Electrophoresis Unit (FFEU) on Earth and in space.

Task Description:
To accomplish these objectives it is necessary to: (1) optimize conditions for maintaining live pituitary cells in Japanese cell culture kits (CCK) for 21 days; (2) remove cells from the CCK in space and fractionate them by electrophoresis; and, (3) break open the cells in space and fractionate the lysate by electrophoresis to obtain hormone-containing granules. These procedures must be done in such a way as to be executable in flight. It is also necessary to modify existing technologies in order to analyze different hormone forms of growth hormone and prolactin in these fractions. Both hormones will be assayed by both immune and biological assays.

Because FY94 was the flight year for this experiment, the logistics associated with conducting pre-flight, flight, and post-flight operations was also required.

Task Significance:
(1) Pituitary growth hormone and prolactin are required for proper function of the bone, muscle, and immune systems. Because these systems are modified by spaceflight, and because the results from four previous space experiments show that the biological activities of growth hormone and prolactin are diminished during and after spaceflight, this experiment is intended to probe the mechanism(s) by which these changes occur.

(2) Biotechnology research on earth routinely utilizes coupled technologies to meet focused objectives. Coupled technologies are difficult to accomplish in a low gravity environment; yet these will be routine on Space Station Alpha. This experiment serves as a prototype for such activities.

Progress During FY 1995:
Two manuscripts describing the results of our experiment done on the IML-2 mission have submitted to the Journal of Biotechnology and are currently in review. Key findings:

(a) The frequencies of changing cell culture medium in microgravity affected the amount and activity of some, but not all, of the six pituitary hormones released into the medium.

(b) Extensive cell clumping occurred in low gravity cultures without media change.

(c) Fresh enzyme solutions prepared in microgravity successfully detached cells from their substrate; these reattached and retained function in low gravity.

(d) Processing of a pituitary cell lysate, coupled to separation of its growth hormone containing particles by free flow electrophoresis, was successful.
(e) Post-flight electrophoresis trials of microgravity-exposed pituitary cells showed that cell feeding frequency affected their net surface charge. This result was unique to low gravity.

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals


Growth, Metabolism, and Differentiation of MIP-101 Carcinoma Cells

Principal Investigator: Dr. J. M. Jessup

Harvard Medical School

Co-Investigators:

No Co-I's Assigned to this Task

Task Objective:

Our objective is to determine whether the metabolism and differentiation of human colon carcinoma cells in actual microgravity is similar to that exhibited in simulated microgravity.

Task Description:

MIP-101 is a poorly differentiated human colon carcinoma that was initially established from the ascites of a patient with widely metastatic cancer. Previous work has found that these tumor cells do not produce carcinoembryonic antigen (CEA) or related molecules, are not metastatic, and grow as isolated cells in monolayer culture. However, growth in vivo in athymic nude mice induces the expression of CEA and similar proteins as well as increasing the metastatic potential of MIP-101 cells. Growth in simulated microgravity in the RWV induces the expression of CEA and related proteins and may have an effect on metastatic potential. Our task is to assess whether growth of MIP-101 cells in the Biotechnology Specimen Container (BSTC) in actual microgravity also upregulates the expression of CEA and related proteins and changes the metabolic or cell cycle parameters of the cells.

Task Significance:

This work is important because it will define the effects of microgravity on the growth, metabolism, and differentiation of a human colon carcinoma cell line that is useful for microgravity research. Our preliminary ground-based experiments suggest that metabolism of MIP-101 is significantly increased in simulated microgravity and strains the capacity of the RWV to support its growth. This cell line will assess how aggressively cells consume nutrients in microgravity when three-dimensional culture is attempted under non-perfused conditions and will test the metabolic requirements of space-based bioreactors. As a result, there is a need to know whether microcarrier cultures produce three-dimensional cultures in space at the metabolic rate observed in the RWV. The MIP-101 cells are important for this test because they are the only cells which display differentiation (production of CEA and CEA-related proteins) as monocultures in the RWV. Finally, we may begin to realize the potential of microgravity to foster three-dimensional cultures and to produce growth and regulatory factors.

Progress During FY 1995:

During the first three months of this grant our laboratory has begun to standardize its approach to the analysis of CEA and CEA family members as well as the analysis of the cell cycle. This was facilitated by the experience with microgravity culture in EDU-1 on Shuttle Mission STS-70. During that flight it was recognized that cultivation of MIP-101 cells was similar to what occurs on Earth in the NASA Bioreactor in terms of the number of cells produced, the gross metabolism of cells (e.g., the amount of acid and dissolved carbon dioxide produced or oxygen consumed), and the general morphological appearance of cells. However, it turns out that within these gross measures, there are a number of minor differences that may have profound consequences.

First, the STS-70 actual microgravity cultures have a normal distribution of cells within the cell cycle. Normally, MIP-101 cells have 15 - 30% of cells in G1-G0 phase, 10 - 20% in S phase, and approximately 30% in G2 + M phase. However, in the Bioreactor within 6 days of growth under simulated microgravity conditions, there is an increase in the number of cells in G2 + M to 45 - 50%. We have found that these cells proceed through another S phase and then undergo apoptosis. This is accompanied by increases in the amount of cyclin D1, ERK1 and ERK2, and tubulin expressed in these MIP-101 cells. Further, the MIP-101 cells in monolayer or static 3-D culture on poly-HEMA coated surfaces do not develop these characteristics which are the large cells seen in the MIP-101

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cultures in the Bioreactor runs. STS-70 samples do not have these large cells, have a normal distribution of cells in the cell cycle, and lack the apoptosis that was seen in the ground controls. This finding alone supports the growth of cells in microgravity and demonstrates that the results seen in the Bioreactor are a special problem in the simulation of microgravity.

Second, the question of the regulation of CEA is still a major problem. Preliminary results suggest that 3-D growth upregulates the expression of 18 - 200 kDa CEA as well as another family member which is the 55 or 90 kDa NCA which shares 70% homology with CEA and is a differentially glycosylated shorter peptide. We have finally begun to accurately quantitate the relative amounts of these two gene products produced under different conditions. CEA and some NCA was produced by the MIP-101 cells cultured in microgravity but not by the cells in the ground controls. Further, we have found that the amount of the gene product produced in static 3-D culture is greater on a per cell basis than in the monolayer cultures. This conclusion, which seems relatively simple to achieve, has been difficult to achieve because the amount of mRNA for the CEA gene product is minimal while the amount of NCA gene product is increased 40-fold in 3-D growth. Thus, increases in CEA gene product may be achieved by increased stability of the cEA gene transcript in 3-D culture. We will be testing these hypotheses in the future.

Thus, from the first three months of this new grant we have interesting insights into gene regulation and the cell cycle. We hope to follow these over the next few months. We also hope that the findings observed on STS-70 will be confirmed by a re-flight. Without this confirmation, we are not in a position to be able to publish these results in a credible scientific journal.
Membrane Transport Phenomena

Principal Investigator: Mr. Larry Mason

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
The Membrane Transport Phenomena (MTP) investigation addresses how gravity influences fluid boundary layers that are associated with membranes, and how the related membrane mediated mass transport processes are affected. The primary variable in the proposed experiments is the convective environment within the test solutions, of which there are three modes: induced convection (+1g), inhibited convection (-1g), and forced convection (externally applied stirring). All of the experiments will be performed for each of these three convective modes. Kinetic differences between gravimetric orientations will indicate experimental conditions where microgravity effects are likely.

Task Description:
A primary objective of the MTP study is the development of instrumentation to measure membrane mediated transport phenomena in microgravity. There are three areas of emphasis in this study: (1) development of an experimental apparatus; (2) experimental research using the apparatus; and, (3) analytical model development. The apparatus development involves fabrication of a Membrane Transport Apparatus (MTA) to contain experimental fluids. The experimental research involves experimental measurement of transport phenomena (i.e., osmosis) within the apparatus and model development to reconcile the experimental data and enable computational predictions.

Task Significance:
The Membrane Transport Phenomena (MTP) investigation will elucidate the role of gravity in membrane mediated mass transport. Prior microgravity experiments using individual cells in the Biorack (Shuttle D-1 mission), Biosatellite II, Salyut, Cosmos, and additional Shuttle missions have clearly shown that important cellular functions are altered in microgravity. Some of the alterations include cytoplasmic streaming (protozoa), membrane properties and proliferation (bacteria), intracellular composition (slime molds), photosynthesis, growth rate, and overall morphology (plant cells), aggregation and morphology (red blood cells), and cellular differentiation and gene expression (lymphocytes). The broad range of changes seen may be explained by an alteration in a fundamental mechanism that is common to all systems, and suggests a common, non-specific cause. Many of these observed cellular changes can be explained as consequences of altered mass transport due to increased thickness of fluid boundary layers associated with membranes. Membranes are present in many different systems: biological, physiological, chemical, and biotechnological. Thicker boundary layers can alter the associated membrane transport processes by many orders of magnitude.

Progress During FY 1995:
The MTP project received authorization to commence work on 13 November 1995. LMA Internal Research and Development (IRAD), and Capital Equipment funds are being used to supplement the MTP contract funding. The MTP activities to date are summarized below:

Generated initial design of the Membrane Transport Apparatus (MTA).
Ordered materials and supplies for MTA development and ground experiments.
Coordinated LMA Capital Equipment procurement activities for MTP video system, sensor array, and fluid manipulation pumps.
Generated detailed design of the Membrane Transport Apparatus (MTA), and ordered materials for optical refractive index visualization system. The design is based on commercially available 47 mm diameter membranes.

Generated design and ordered materials for the MTP Microsensor Array interface electronics. The microsensor array is not scheduled to be fabricated until MTP year 2, but the interface will be developed this year (LMA Capital funding).

Development of volumetric flow sensors under LMA Internal Research & Development (IRAD) funding. The design characteristics were parametrically analyzed, and a prototype version developed. The interface electronics were also fabricated.

**STUDENTS FUNDED UNDER RESEARCH:**

**TASK INITIATION:** 10/95  **EXPIRATION:** 11/98

**PROJECT IDENTIFICATION:** 963-45-10

**NASA CONTRACT NO.:** NAS8-40633

**RESPONSIBLE CENTER:** MSFC
An Observable Protein Crystal Growth Flight Apparatus

Principal Investigator: Dr. Alexander McPherson, Jr. University of California, Riverside

Co-Investigators:

Koszelak, S. University of California, Riverside
Malkin, A. University of California, Riverside
Kuznetsov, Y. University of California, Riverside
Kathman, A. Teledyne Brown Engineering
Dodds, A. University of California, Riverside
Garavito, M. University of Chicago

Task Objective:
The task objective is to initiate research and development efforts in the area of macromolecular crystal growth, and specifically focused on the direct observation of the relevant phenomena as they pertain to the design and ultimate flight of an observable protein crystal growth apparatus (OPCGA).

Task Description:
The experiment objective included the identification and characterization of candidate biochemical systems that included proteins, nucleic acids, and viruses. It further included the construction of an optical platform that would be suitable for detailed interferometric analysis of protein crystal growth experiments and the visualization of concentration fields, the time lapse microphotography of macromolecular crystals, and the further characterization of the mechanisms and fundamental parameters that determine the features of macromolecular crystal growth.

Task Significance:
The need for structural information on biological macromolecules is of paramount importance to the emerging field of biotechnology. Such information provides the basis for the rational design of pharmaceuticals, the determination of enzymatic mechanisms and the engineering of proteins to enhance or modify their function. The value of x-ray crystallography to provide structural information at atomic resolution is unsurpassed. This technique does, however, depend on the availability of crystals for the macromolecule under study, and furthermore, which possess a high degree of internal order and suitable size and shape to allow the accurate collection of x-ray diffraction data.

Typically, crystals grown in the earth's gravitational field suffer from one or more types of flaws which decrease the structural information that can be derived from them. These imperfections include the relatively simple, but nonetheless adverse, problem of intergrowth. In a 1-g field several crystals, which nucleate independently, sediment to the bottom of the growth chamber where they intergrow into a mass not suitable for x-ray diffraction analysis. Such intergrowth or the appearance of satellite crystals, which were earlier thought to occur by nucleation at the sides of defects in pre-existing crystals, have been shown to be almost exclusively due to the effects of sedimentation and intergrowth. The value of a microgravity environment for the elimination of sedimentation in the preparation of these crystals is obvious. Early experiments conducted on board the Space Shuttle clearly indicated such benefits. They also provided the impetus for other researchers to design experiments and procedures for mimicking the effect of microgravity in ground-based experiments. The development of methods for successfully growing protein crystals in gels is an example of such activities.

Progress During FY 1995:
During FY95 a number of crystallization experiments were carried out on board the U.S. Space Shuttle and on board the Russian Space Station Mir. These included a 20 reactor cell experiment in the ESA-designed and built Advanced
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Discipline: Biotechnology

Protein Crystallization Facility (APCF) flown on the Second International Microgravity Laboratory (IML-2), the first flight of the Hand Held Diffusion Test Cell (HHDTC) with video observation, Spacehab-3 (STS-63), and the first investigation of protein crystal growth on Mir by American investigators (Mir-1). This last experiment utilized the GN-2 Dewar and carried over 180 flash frozen batch and liquid-liquid diffusion samples. The results of the APCF experiment have been published (Koszelak, et al. 1995) and include some of the most definitive results obtained from protein crystallization experiments in space.

A series of breadboard instruments were utilized to evaluate which techniques would be most appropriate for the Observable Protein Crystal Growth Apparatus (OPCGA) currently under development. These included Michaelson interferometry, phase-shift and conventional Mach-Zehnder interferometry, atomic force microscopy, and time lapse video microscopy. From these studies time lapse video microscopy and phase shift Mach-Zehnder interferometry were selected for inclusion. A Science Concept Review (SCR) was held and approval to continue development of OPCGA toward RDR was granted. The most crucial component of the OPCGA, a phase shift Mach-Zehnder interferometer based on solid optics components was built and its performance verified. This prototype will serve as the basis for development of the flight instrument.

Using interferometric methods we established the fundamental kinetic and thermodynamic parameters for several macromolecular crystal growth systems, and with phase shift methods demonstrated the existence of depletion zones around crystals growing in the laboratory. Study of such depletion zones is a major objective of the OPCGA. Using atomic force microscopy, we have established the mechanistic basis for macromolecular crystal growth and begun to define the nature and effects of incorporated impurities, an essential prerequisite for the study of macromolecular crystal growth processes in microgravity.

Work has now begun on a full up prototype of the OPCGA that includes crystallization cells, activation mechanisms, optical components, and control services. From this prototype, following testing and verification, the flight instrument will be developed. Development has also begun on an Advanced Dewar-based apparatus that will include temperature control and monitoring.

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II. MSAD Program Tasks — Flight Research

Discipline: Biotechnology


Proceedings


Books

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McPherson, A. "Studies on macromolecular crystals grown in space." Tohoku University, Sendai, Japan, November 2, 1994.

Enhanced Dewar Program

Principal Investigator: Dr. Alexander McPherson, Jr. University of California, Riverside

Co-Investigators: Koszelak, S. University of California, Riverside
Malkin, A. University of California, Riverside

Task Objective:
The objective of the research is to develop a microgravity suitable system for screening protein crystallization conditions using the batch and liquid-liquid crystallization method.

Task Description:
Initially, a relatively simple system composed of a Dewar flask filled with small crystallization samples will be frozen on Earth and allowed to passively thaw in microgravity. Samples will be housed in the Passive GN₁ Freezer, a Dewar previously used in other microgravity applications. In addition to the crystallization experiments, plans are outlined for examination of crystals by standard light microscopy and also electron microscopy and atomic force microscopy, and detailed documentation of results, structure determination and high resolution refinement, and detailed analyses of structures obtained from crystals grown on Earth and in microgravity.

Task Significance:
These experiments represent an exciting and important new opportunity for protein crystal growth studies in microgravity. The proteins and protein solutions that will tolerate the freeze-thaw mechanism used to initiate these experiments will increase as the trend away from exclusion of glycerol from protein solutions used in crystallization continues. Shuttle experiments, furthermore, suggest that most orbiting platforms have some variations in the acceleration environment, and some protein crystallizations are more tolerant of these variations than others. Many of the difficulties that have affected most previous microgravity experiments can be circumvented in the Dewar system and long duration flights making possible crystallization experiments that cover much longer equilibration times.

Progress During FY 1995:
This task was funded only recently and is in its initial start-up stages. First mission initiated in July for the Mir-1 mission. Began design and development of temperature control and monitoring system.

Students Funded Under Research:

Task Initiation: 3/95 Expiration: 3/99
Project Identification: 963-45-07
Responsible Center: MSFC
II. MSAD Program Tasks — Flight Research

**Electrophoresis Technology**

**Principal Investigator:** Dr. Robert S. Snyder  
**NASA Marshall Space Flight Center (MSFC)**

**Co-Investigators:**
- Rhodes, P.  
  **NASA Marshall Space Flight Center (MSFC)**
- Miller, T.  
  **NASA Marshall Space Flight Center (MSFC)**
- Roberts, G.  
  **Roberts Associates, Inc.**

**Task Objective:**
The task objectives are to study the effects of sample concentration and dielectric constant on sample stream distortion and the limits of the electrohydrodynamic stability of the sample stream in the absence of shear flow.

**Task Description:**
The electrophoresis separation process can be considered to be simple in concept, but flows local to the sample filament produced by applied electric field have not been considered. These electrohydrodynamical flows, formulated by O.I. Taylor in 1965 for drops suspended in various liquids, distort the sample stream and limit the separation. In addition, electroosmosis and viscous flow, which are inherent in the continuous-flow electrophoresis device, combine to disturb further the process. Electroosmosis causes a flow in the chamber cross section which directly distorts the sample stream, which viscous flow causes a parabolic profile to develop in the flow plane. These flows distort the electrophoretic migration of samples by causing a varying residence time across the thickness of the chamber. Thus, sample constituents at the center plan will be in the electric field a different length of time and hence move more or less than comparable constituents closer to the chamber wall.

Both horizontal and vertical laboratory electrophoresis test chambers have been built to test the basic premise of continuous-flow electrophoresis that removal of buoyancy-induced thermal convection caused by axial and lateral temperature gradients will result in improved performance of these instruments in space. These gravity-dependent phenomena disturb the rectilinear flow in the separation chamber when high-voltage gradients and/or thick chambers are used, but distortion of the injected sample stream due to electrohydrodynamic effects causes major broadening of the separated bands observed in these chambers.

The initial part of the proposed space experiment was planned to be done in the French electrophoresis hardware (RAMSES) on the Second International Microgravity Laboratory (IML-2). This hardware has the capability of applying the required voltage at 1,000 Hz which can permit the dielectric dependence to be determined. Two different frequencies were planned to vary the dielectric constant of the samples and the cross-section illuminator used to show the sample filament cross section, and recorded photographically. This experiment was not done on IML-2 because of a hardware failure on orbit.

The experiment can be accommodated on a later RAMSES flight, or available TEXUS electrophoresis hardware, with its cross-section illuminator, can be supplied with the required high-frequency power supply. These measurements can then be completed during a short-duration rocket flight. Additional opportunities are being evaluated.

**Task Significance:**
Since the Continuous Flow Electrophoresis System (CFES) built by the McDonnell Douglas Astronautics Company achieved results in space on seven shuttle missions that were influenced by electrohydrodynamics, these scientific phenomena are a critical part of electrophoresis in space. The severity of sample distortion due to dielectric constant variations is poorly known in the laboratory because of the concurrent sample concentration effect.
Progress During FY 1995:
The activities during FY95 were directed into efforts to resolve discrepancies between measured dielectric spectra and those predicted by classical electrokinetic theory. In general, measured dielectric constant values are an order of magnitude larger than corresponding theoretical values. Experiments using our miniature electrohydrodynamic test chamber show greatly reduced values in the dielectric constant compared to measured values in the literature. However, problems in dynamically measuring the sample and buffer conductivity have prevented firm conclusions from being made.

A paper has been submitted which shows experimentally the role of dielectric constant in the deformation of polystyrene latex sample streams. These observations are believed to be the first experimental evidence of the dielectric constant effect in conducting fluids to appear in the literature.

STUDENTS FUNDED UNDER RESEARCH:

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

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Rhodes, P.H., Snyder, R.S., and Roberts, G.O. Role of dielectric constant in electrohydrodynamics of conducting fluid. J. Colloid Interface Sci., (accepted 1995).

Proceedings
Assignment of Protein Crystal Growth Mechanisms in Microgravity

Principal Investigator: Dr. Keith B. Ward

Co-Investigators:
Zuk, Dr. W.M.

Task Objective:
The objective of this program is to conduct flight experiments aimed at gathering fundamental data about how the processes of protein crystallization differ in a microgravity environment compared to unit gravity. The goals of these experiments therefore differ from those conducted by previous investigators who have limited their experiments to the preparation of large crystals suitable for x-ray diffraction analyses. The experiments performed under this project will be conducted in identical apparatuses both in ground-based and flight-based experiments. They will provide invaluable data about how solubility curves, nucleation kinetics, and crystal growth rates differ between microgravity and unit gravity. This information will then be used in the same apparatus to optimize conditions so as to favor the preparation of large, well ordered, single crystals. These results will prove crucial for the design of future generations of protein crystallization facilities that will be flown on a variety of space platforms.

Task Description:
The experiments will be conducted using the Canadian-American Telerobotic Crystallization Hardware (CATCH), a modified version of an apparatus developed under the auspices of the Canadian Space Agency. This equipment provides either liquid-liquid diffusion or mixed batch protocols, and incorporates a unique mixing mechanism that overcomes the difficulties encountered by current flight-based crystallization devices. The system also provides for monitoring light scattering signals from automated growth cells, allows video observation of the cells during the course of the experiments, and provides for precisely adjusting the growth cell temperature as a means of controlling protein supersaturation.

The automation system, which includes the flight computer and hardware interfaces of CATCH and the software necessary to control the apparatus, will be developed in the PI's laboratory as part of this project. Because of its ability to control the temperature and to monitor the progress of crystallization, CATCH will provide investigators the opportunity to devise experiments using dynamic control in which crystallization parameters are changed during the course of the experiments, either through predetermined experimental protocols or via commands provided by ground-based investigators. This unique ability to dynamically control the crystallization conditions is not afforded by any current microgravity crystallization device.

Task Significance:
Using CATCH, we will initiate a new series of flight experiments not limited to the preparation of single protein crystals, but which will also include the acquisition of fundamental data required to address the question of how microgravity may best be used to advantage in the preparation of such crystals. CATCH will allow investigators to monitor the results of crystallization trials, and more importantly, to reinitiate experiments based upon the nucleation and crystallization behavior observed in growth cells during the same flight. This use of telerobotic control inherent in CATCH will provide investigators an unparalleled level of efficiency in conducting microgravity crystallization experiments.

Progress During FY 1995:
We have begun development of a simple remote control system in our laboratory which can demonstrate an ability to operate crystallization experiments telerobotically. The laboratory-based system will initially be used to monitor remotely the progress of a crystallization experiment, and will provide users on the Internet the ability to control the
monitoring camera. The first version of the control software is run on the World Wide Web (WWW) and allows users to interact with driver software located on our Web Server computer. This month we have extended the Web software to access control electronics and software that we have developed to drive a set of stepper motors. These motors are used to position a video camera above a crystallization plate. We are currently waiting for Windows NT drivers which will allow us to use a video capture board installed in our Web server to acquire video images of individual wells. A user will be able to move the stepper motors to position the camera above a crystallization well, and then capture an image of the well, which will be posted to the Web page.

We have also begun conducting dynamic light scattering experiments to examine the states of aggregation of protein in bulk solution and around various faces of a protein crystal. This information may be useful in determining how proteins attach to a crystal face.

**STUDENTS FUNDED UNDER RESEARCH:**

**TASK INITIATION:** 7/95  **EXPIRATION:** 7/98

**PROJECT IDENTIFICATION:** 963-45-09  
**RESPONSIBLE CENTER:** MSFC
II. MSAD Program Tasks — Flight Research  

Low-Velocity, Opposed-Flow Flame Spread in a Transport-Controlled, Microgravity Environment

Principal Investigator: Prof. Robert A. Altenkirch  
Washington State University

Co-Investigators:
Bhattachanjee, S.  
San Diego State University
Olson, S.L.  
NASA Lewis Research Center (LeRC)

Task Objective:
The overall objectives of the proposed work are to uncover the underlying physics and increase the fundamental understanding of the mechanisms that cause flames to propagate over solid fuels against a low velocity flow of oxidizer in the low-gravity environment. Although the work is fundamental in nature, it has clear applications to fire safety in space and on Earth. Specific objectives are:

1. To analyze experimentally observed flame shapes, measured gas-phase field variables, spread rates, radiative characteristics, and solid-phase regression rates for comparison with theoretical prediction capability previously developed that will be continually extended.

2. To investigate the transition from ignition to either flame propagation or extinction in order to determine the characteristics of those environments that lead to flame evolution.

Task Description:
To meet the objectives of the research program, a series of experiments has been developed to exercise several of the dimensional, controllable variables that affect the flame spread process in microgravity. Those variables that will be changed from experiment to experiment are the opposing flow velocity (1-20 cm/s), the external radiant flux directed to the fuel surface (0-2 W/m²), and the oxygen concentration of the environment (35-70%). An experiment matrix is used that minimizes the number of experiments to be conducted in order to obtain the information needed to meet the scientific objectives of the effort.

Because the amount of data to be collected is limited, modelling is necessary to interpret the results and to sort out the important physics of the phenomenon. The modelling effort that will support the experimental program is numerical in nature and includes the capability to solve model flame spread problems over both thermally thin and thick fuels posed as steady-state, eigenvalue problems for spread rate or as unsteady problems from ignition through flame spread. In each approach, the two-dimensional continuity, momentum, species, and energy equations in the gas and the continuity and energy equations in the solid are solved using the SIMPLER algorithm. Gas radiation is included in the model to assist in the interpretation of species-specific emission data obtained with band pass-filtered video cameras in the experiments.

Task Significance:
Radiative heat transfer is critical to these and many other microgravity flame spread experiments, and so radiant heating will be imposed, and radiant heat loss will be measured. These are the first attempts at such experimental control and measurement in microgravity, and the experimental results and numerical modelling will allow the role of radiation, as well as diffusive transport, in these flames to be delineated.

Progress During FY 1995:
The flight hardware for DARTFire was assembled and an extensive series of DC-9 low gravity tests are being conducted to finalize the test matrix and match like intensity flames for the two simultaneous sounding rocket experiments. The sounding rocket is currently scheduled to fly during the 2nd quarter of FY96.
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Unsteady computational results for a full channel flow were obtained for comparison to the half channel results obtained earlier. Gas-phase radiation and surface reradiation loss are considered together. The momentum equations are solved in order to obtain a steady channel flow. After 14 seconds, the u velocity attains a steady value. Most results from the full and half channel agree reasonably well, with the exception perhaps of the velocity fields.

We continue to develop a simplified theory that builds on the de Ris formula for the prediction of flame spread rate over thick fuels. The biggest discrepancy between the de Ris formula and the spread rates computed from a comprehensive numerical model stems from the simplification introduced by the Oseen approximation. Five different geometrical configurations are considered for flame spread over a flat plate (a) with a free flow zone upstream (b) without any free flow upstream (c) inside a fully developed channel flow (d) inside a developing channel flow, and (e) case (d) with a free flow ahead of the channel. The difference in the hydrodynamics in all these configurations results in a different numerical spread rates for each situation, all other environmental and fuel parameters fixed. Configurations (c) and (d) are important for the DARTFire geometry. The spread rate formula developed contains two constants, evaluated numerically, and handles all these five configurations. The flame structure is also reasonably predicted by the closed-form formulas.

Additionally, variable gas-phase specific heat and thermal conductivity have been thoroughly integrated into the computational routines. These quantities are now functions of local mass fraction of species and temperature. We are also evaluating the effect of radiation on the Emmons's problem in microgravity. The Simplified Theory for the thermal regime of opposed-flow flame spread over thick fuel into the microgravity regime is being extended because accurate modeling of hydrodynamics is a must since the spread rate is most sensitive to the velocity field near the leading edge.

In the microgravity regime, if a uniform velocity field hits the channel entrance at x=0, there are two distinct phenomena that characterize the entrance flow development: i) the centerline velocity increases from u_inf at x=0 to 1.5u_inf at x=x_e, x_e is the entrance length obtainable from standard correlations, ii) the flow near the wall shows a velocity overshoot, i.e., the velocity profile reaches a u_max_wall that is higher than u_centerline. At first this second phenomenon was believed to be a numerical artifact, but computations could not get rid of this, especially at low Reynolds numbers that are common in microgravity. Experimental confirmation of this observation was made from researchers at NASA, Lewis so this appears to be a real phenomenon. A velocity overshoot correction is being incorporated into the spread rate expression of the thermal regime.

The effect of radiation on the Emmons problem in microgravity is being examined. The Emmons solution as been obtained within computational accuracy. A comprehensive model has been developed, where the major assumptions of Emmons have been removed, to do a parametric study of the problem and compared results with that of Emmons. The preliminary results indicate that at high opposing velocity Emmons prediction of the mass burning rate is reasonably accurate. However, in the microgravity regime the difference between the two models is large and can be mostly attributed to radiation.

Predictions of steady and unsteady codes are being compared to establish the pseudo-steadiness of the flame spread problem. The effect of flame size and changing boundary layer thickness have an unsteady effect on the spread rate. By solving the problem in a quiescent microgravity environment and in an opposing-flow environment it is possible to isolate the two sources of unsteadiness. The radiation code is also being upgraded to improve its predictive capabilities: i) The computational domain is being divided into n boxes, and n different a_p's are calculated for the field. In the old model n=1, that seems to lead to relatively higher losses from a flame making the flame temperature too low sometimes. ii) The discrete transfer method of Lockwood (Imperial College group of England) is being considered for incorporation into the radiation code.

Additionally, modelling efforts have concentrated on incorporating full radiation coupling, i.e., feedback from the flame to the fuel surface as well as surface reradiation and gas-phase radiation loss, and to experiment with "multi-box" radiation models in which the computational domain is divided into more than one region, and the radiative properties of each region, i.e., the Planck mean absorption coefficient, are allowed to differ as compared to a single Planck mean absorption coefficient for the entire computational domain as we have been using thus far.

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Comparisons with the steady computations were made for 50% oxygen, 1 atm, and an opposing flow of 1 cm/s, one of the DARTFire matrix points. Results show that the Planck mean absorption coefficient increase rapidly in the beginning, overshoots the steady value only slightly, and reaches the steady value of around 4.1 l/m within about 25 s. The fraction of radiation fed back to the surface increases rapidly, peaks at about 20% around 25 s, and then decreases to the steady value of about 17% at the steady flame location. From the unsteady computation, the spread rate peaks at around 25 s at approximately 0.011 cm/s and then decreases toward the steady value of 0.0087 cm/s to reach 0.0077 cm/s. The gas-phase temperature profiles from each computation are similar, but in the solid phase, the steady results show a much larger heated region downstream of the flame leading edge than do the unsteady results.

The gas phase radiation model has been improved. A multi-box radiation routine was used to divide the global control volume into a number of subdomains to determine the individual Planck mean absorption coefficient for each subdomain. This approach produces a distribution of $a_p$. However, the flame temperature with this approach does not show a significant change from that using the original global model. During the period of this report, effort has been spent improving the radiation routine in order to handle three-dimensional property fields. The reformulation of the code will allow us to use models such as Discrete Transfer without much recoding effort. The modified radiation code has been tested thoroughly. The $a_p$ values predicted for limiting situations agree with the results of Tien (C.L. Tien, Advances in Heat Transfer, P. 315, 1968).

The modified radiation code was used in the computation of the DARTFire/Learjet Series which was conducted aboard the NASA Lewis Research Center Learjet (Olson et. al.). Computed spread rates for 50% oxygen and 2.0 atm at 0.01 g is 0.032 cm/s. This is quite comparable to the measured values: 0.0321 cm/s. The computed temperatures do not match with measured values: the computed surface temperature $T_s$ is higher and the in-depth temperature lower than the measured value. Predicted flame appearance agrees well with experimental one: flame is very thin, close to the fuel surface, and flame is curved. There is a problem that remains to be solved: the theoretical surface temperature seems to be higher and the embedded temperature to be lower than those seen in the experiment, independent of the radiation model used. This raises questions about the pyrolysis model which was developed originally for flames spreading in normal-gravity environment.

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**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Journals**


**Presentations**

II. MSAD Program Tasks — Flight Research

Discipline: Combustion Science

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II. MSAD Program Tasks — Flight Research

Refight of the Solid Surface Combustion Experiment with Emphasis on Flame Radiation Near Extinction

Principal Investigator: Prof. Robert A. Altenkirch
Washington State University

Co-Investigators:
Bhattacharjee, Prof. S.
Sacksteder, K.R.
Delichatsios, Dr. M.
San Diego State University
NASA Lewis Research Center (LeRC)
Factory Mutual Research

Task Objective:
The objective of this flight experiment is to determine the mechanisms of flame spreading over solid fuels in the absence of buoyant or externally imposed, gas-phase flows. This experiment is an extension of the Solid Surface Combustion Experiment with the purpose of observing flame spread with radiative losses near the flammability limit.

Task Description:
The Solid Surface Combustion Experiment (SSCE) is to be reflown to perform additional tests and to obtain quiescent flame spread data for cylindrical, thermally thick fuels. In these tests, measurements of the fuel and flame temperature are to be made and recorded, and the flames will be photographed using motion picture film. The numerical model, developed as part of the SSCE project, is to be further extended to predict flame spread behavior over cylindrical samples in which the geometry of the gas-phase radiative interactions are simplified computationally. A detailed quantitative comparison of the experimental and computational results is to be performed, including comparisons of spread rate, temperature field, heat transfer rates, and flame structure.

Task Significance:
The spreading of flames over solid fuels is a fundamental combustion problem that has practical significance in the prevention and control of fires. Flame spreading in normal gravity is usually dominated by buoyant air flow that introduces a significant complexity into fundamental theoretical models. Experiments conducted in the microgravity environment nearly eliminate this complexity, providing a more fundamental scenario for the development of flame spreading theory.

Progress During FY 1995:
This is a continuation of the work in Solid Surface Combustion, which originated in December, 1984 under Task ID No: 963-22-05-01. All FY95 publications and presentations are listed under the original SSCE program.

1) Following the final flight of the Solid Surface Combustion Experiment in February, 1995, the baseline tests for the SSCE Reflight was determined. Two flights are planned, the first to essentially refly the existing SSCE-PMMA configuration changing only the atmospheric content/pressure in order to approach the flammability limit. This test is expected to exhibit the unsteady behavior of flame spread over thick, flat samples more clearly than the previously flight tests in which this unsteadiness was first observed. The second flight is to test a cylindrical configuration for the first time in a purely quiescent environment in which the radiative interaction between the flame, fuel surface and the surroundings is simplified from a theoretical point of view.

2) A series of 5.2 Second Drop Tower tests was conducted to evaluate the test conditions for the near extinction flight test and the ignition behavior of the cylindrical samples for the second flight test. The flat samples seem to be unignitable in the available test time at atmospheric conditions of 40% oxygen/60% nitrogen at 1 atmosphere, but are clearly ignitable in 50% oxygen at the same pressure. Several configurations of the cylindrical samples

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were tested, including varying ignitor wire path configurations, and sample mounting schemes, leading to a design of the flight samples that is readily ignited in the drop tower test time.

3) Some analytical work has been completed, extending the early work of deRis, in which it has been shown that the flat, thick samples are fundamentally unsteady in their flame spread behavior, while thin samples may spread at a steady rate. Discrimination between thick and thin are dependent upon the fuel property values, atmospheric conditions and the presence and strength of a flow field about the flame.

4) The numerical modeling work has been used to estimate the necessary test conditions for the flat-sample, near-limit case. The numerical results agree with the drop tower tests indicating that the flight test conditions should lie between 40% oxygen and 50% oxygen (with the balance of nitrogen) at 1 atmosphere, or at 50% oxygen at a reduced pressure. The modeling also indicates that tests in this regime should exhibit clear unsteadiness in the existing sample length of 2.5 cm.

5) The numerical modeling work has also been used to estimate the necessary geometry of the cylindrical sample. The proposed sample will be in two separate pieces, one being 2mm in diameter and 20mm long; and the second 6.4mm in diameter and 40mm long. The second sample will be one-half hollow (with a 1mm wall thickness) and one-half solid. This collection of three geometrical configurations are predicted to show independent variations in flame behavior based on surface radius and fuel thickness.

6) At year’s end the project was preparing for the MSAD Hardware Reflight Review, based upon the analytical, numerical and test results summarized here.

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BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals
II. MSAD Program Tasks — Flight Research

Scientific Support for an Orbiter Middeck Experiment on Solid Surface Combustion

Principal Investigator: Prof. Robert A. Altenkirch
Washington State University

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
The objective of this flight experiment is to determine the controlling mechanisms of flames spreading over solid fuels in the absence of buoyant or externally imposed, gas-phase flows. Ground-based testing of flame spreading in quiescent microgravity environments has identified the qualitative importance of radiative heat losses in determining flame spread rates, but these tests are too short in duration to establish spreading flames without residual effects of the ignition process.

Task Description:
The Solid Surface Combustion Experiment (SSCE) was built to perform a minimum of eight flight experiments. The experiments are to consist of five flame spreading tests using a thin fuel, varying the atmospheric composition and pressure, then three additional tests using a thick fuel with fewer variations of the same parameters. In these tests, measurements of the fuel and flame temperature are to be made and recorded, and the flames are to be photographed, using motion picture film. A parallel effort is to be made to develop a complex numerical model of the opposed-flow flame spread problem, including the important effects of surface and gas-phase radiative losses. Finally, a detailed, quantitative comparison of experimental and computed results for flame spreading over thin and thick fuels in various oxidizer and pressure environments is to be performed including comparisons of spread rate, temperature and heat transfer fields and the structure of the flame.

Task Significance:
The spreading of flames over solid fuels is a fundamental combustion problem that has practical significance in the prevention and control of fires. Flame spreading in normal gravity is usually dominated by buoyant air flow that introduces a significant complexity into the fundamental models of the phenomena. Experiments conducted in the microgravity environment nearly eliminate this complexity, providing a more fundamental scenario for the development of flame spreading theory.

Progress During FY 1995:
1) The eighth and final flight of the SSCE program was completed during February, 1995 with the burning of the third PMMA test condition of 50% oxygen 50% nitrogen in 1.0 atmospheres of pressure. Both samples ignited promptly and burned until the motion picture film was consumed, approximately 270 seconds, at which time the quenching mechanism was used to stop combustion and preserve the samples for later analysis. Based on the experience of the first two PMMA flights, the motion picture cameras were operated differently for the second test such that the start-up of the cameras was delayed for 60 seconds.

2) The flame images were analyzed using an automated computer image digitizer and flame tracking algorithm developed for the SSCE project. The principal result of this analysis was an additional demonstration of the repeatability of the test until the flame reaches the vicinity of the gas phase thermocouple. While near the thermocouple, the glow of the thermocouple obscures the flame leading edge. Both flames were recovered by the tracking algorithm after passing the thermocouple.

3) Analysis of the results of the three PMMA flight tests of the SSCE have led the investigator team to conclude the important result that the flame spreading observed in each case is not a steady process, but gradually slowing. Results of the unsteady numerical model, originally developed to account for the influence of the ignition process on flame spreading, was being compared at years end to the flight test results.
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4) During this year much of the focus of the SSCE program has focused on the preparations for the two additional reflight opportunities, which are reported in a related task, 963-15-OE.

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BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals

Presentations


**II. MSAD Program Tasks — Flight Research**  
**Discipline: Combustion Science**

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**Gravitational Effects On Laminar, Transitional, and Turbulent Gas-Jet Diffusion Flames**

**Principal Investigator:** Dr. M. Y. Bahadori  
Science Applications International Corporation

**Co-Investigators:**

Hegde, Dr. U.G.  
NYMA, Inc.

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**Task Objective:**

The overall objective of this program is to improve our fundamental understanding of microgravity gas jet diffusion flames in the entire laminar, transitional, and turbulent regimes. Specifically, tests are to be conducted in microgravity with the purpose of (i) determining the effects of buoyancy on these flames, (ii) determining the relative importance of buoyancy-induced turbulence on flame characteristics, (iii) revealing phenomena which may be masked by buoyant convection, and (iv) characterizing the processes of vortex/flame interaction for a better understanding of transition to turbulence of gas jet diffusion flames.

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**Task Description:**

In order to achieve these objectives, the program pursues two distinct but complementary paths, as follows:

(a) Investigating the effects of fuel type, flow rate, Reynolds number, nozzle size, and gravity on (i) global flame characteristics (such as flame shape, height, radiation and temperature), (ii) the extent of the transition regime, (iii) turbulent flame features, and (iv) stand-off characteristics and blow-off conditions. These tests provide data on flames in microgravity (using the Drop Tower and Zero-Gravity Facility, in addition to the KC-135 aircraft) and normal-gravity environments. The measurements include temperature, thermal radiation, pressure, species concentration, acceleration, and flame imaging. The data will be used to validate detailed analytical and numerical models of these flames.

(b) Identifying the mechanisms involved in the generation and interaction of observed large-scale structures which directly influence the flame characteristics noted under (a). This part involves an investigation to be carried out in space, which utilizes a controlled, well-defined set of disturbances to reveal the mechanisms that govern the dynamics of large-scale structures interacting with flame fronts under microgravity conditions. This will further our understanding of the naturally occurring disturbances that are an inherent part of the transitional and turbulent flames of part (a). As in part (a), a combined analytical and numerical modeling effort will be an integral component of this phase of the program.

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**Task Significance:**

Most practical combustion is turbulent because enhanced mixing is essential for increasing combustion efficiency. Despite this technical importance, turbulent flames are not well understood because their random, transient nature greatly complicates the interaction of processes occurring in flames and their associated analysis. This complication is overcome in a microgravity experiment through the use of a steady laminar flame and induced vortices. Controlled, repeating vortices are produced by an iris mechanism that opens and partially closes around the base of the flame. The operation of the mechanism creates sinusoidal variations in the air flow entrained by the fuel jet, at a single controlled frequency. The experiment cannot be conducted in normal-gravity because buoyant instabilities induce uncontrolled vortices at other frequencies and amplitudes, complicating the analysis. The results of this experiment will help in improving our understanding of turbulent flames with direct applications to problems of combustor efficiency and pollutant emission.

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**Progress During FY 1995:**

Contract NAS3-25982 has been in place since November 1991. It involves a comprehensive study of laminar, transitional and turbulent gas jet diffusion flames in microgravity. The following researchers participated in these...
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Discipline: Combustion Science

Efforts: 1) M.Y. Bahadori, Principal Investigator (SAIC); 2) J.F. Small, Jr., Research Scientist (SAIC); 3) U.G. Hegde, Co-Investigator (NYMA, Inc.); 4) L. Zhou, Research Scientist (Analex, Inc.); 5) D.P. Stocker, Project Scientist (NASA Lewis); and 6) F. Vergili, Project Manager (NASA Lewis).

An extensive series of tests in the 5.18-Second Zero-Gravity Facility at NASA Lewis was conducted for the flight test matrix of pulsed gas-jet diffusion flames. These tests were conducted for characterization of the axisymmetric-perturbation mechanism, flame behavior under the influence of oscillation parameters, visualization characteristics, radiometer (both global and slice) verification, and thermocouple-rake operation. Obtained data included:

(i) Time-resolved images of the flame under the influence of the imposed disturbances. The convection velocity of the pulsations along the flame was obtained, and it was found that the average convection velocity was approximately 20% of the injection velocity. In addition, structure wavelengths were shown to decrease with downstream distance. These findings are consistent with theoretical predictions made by the investigators.

(ii) Temperature measurements by means of fine-wire thermocouples at various axial and radial locations in the flame. Sampling rates in excess of 500 Hz were employed. The obtained thermocouple signals were corrected for the time constants which were also measured during the reduced-gravity tests. Amplitudes of the temperature oscillations were found to decrease axially. The decay in the amplitudes was frequency dependent. The disturbances displayed near-unity coherence over the flame region.

(iii) Flame radiation measurements by both global and slice radiometers as a function of both imposed pulsation frequency and radiometer location. Time-averaged global radiometer signals compared extremely well with theoretically predicted values. The sampling rate of the signals was greater than 500 Hz and the obtained signals were corrected for the radiometer time constants. The correspondence between temperature and radiation signals is currently under investigation.

Other significant findings also resulted from these tests. For unpulsed flames, it is found that: (a) Temperatures near the flame tip are still increasing at the end of the drop; (b) Global- and slice-radiometer signals are increasing at the end of the drop; (c) Optimum location of the global radiometer is consistent with numerical calculations; (d) Temperature drop along the centerline of the microgravity flames is verified both experimentally and numerically; and (e) Microgravity times longer than five seconds are needed to approach steady conditions in the near-field of the flame.

The results of the pulsed microgravity flame studies can be summarized as follows: (a) Iris pulsing has led to sinusoidal temperature and radiation oscillations; (b) Disturbances are damped downstream as centerline temperature amplitude decreases with axial distance; (c) Near-perfect coherence between both temperature and radiation oscillations are observed over the flame length (this is not observed in normal gravity); (d) Time-averaged data do not reach near-steady state in 5 seconds of microgravity; (e) Temperature oscillations near the centerline are 180 degrees out of phase with oscillations near the flame sheet, which is consistent with vortex dynamics; and (f) Five seconds of microgravity does not provide enough time to obtain statistically stationary data for flames under the influence of imposed disturbances.

The iris arrangement utilized to impose the periodic and repeatable pulsations on the investigated flames was characterized by LDV measurements. An air-into-air jet was utilized for this purpose and all three components of the velocity, i.e., axial, radial and tangential, were measured. It was found that the axial velocity dominated the radial and tangential velocities under both steady and oscillatory conditions. The axisymmetric nature of the imposed disturbances was confirmed. Experimental and numerical results were in good agreement for the flowfield.

Fabrication of the flight experiment package is in progress, and the planned completion date is early 1996. Currently, safety documents, the Product Assurance Plan, drawings, and other documentation are being prepared.
The modeling effort consists of analytical and numerical simulations of gas jet diffusion flames under the influence of imposed disturbances. To date, laminar base-flame calculations for flight experiment have been conducted, and a number of comparisons such as optimum radiometer location and temperature field of the flame are made with experimental data. Detailed numerical computations of a time-dependent, Navier-Stokes model are in progress. Disturbed flames are currently under investigation. Numerical simulation of the flame transient development under the experiment condition is obtained. Detailed modeling of iris-induced flowfield in jets was completed, and the results have shown good agreement with the LDV measurements.

Milestones:
(i) The project successfully passed the Requirements Definition Review (RDR) which was conducted in February.
(ii) Authority to proceed to flight was received from NASA Headquarters in March.
(iii) The final Science Requirements Document (SRD) was delivered to the Project Scientist in April.
(iv) The Project Plan and Hardware Capabilities documents were submitted to the Program Manager and Program Scientist in May.
(v) The Payload Accommodations Requirements (PAR) document was submitted to Goddard Space Flight Center in August.

STUDENTS FUNDED UNDER RESEARCH:

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BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals

Presentations

II. MSAD Program Tasks — Flight Research  
**Discipline: Combustion Science**

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**Sooting Effects in Reduced Gravity Droplet Combustion (SEDC)**

**Principal Investigator:** Prof. Mun Y. Choi  
**University of Illinois, Chicago**

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**Co-Investigators:**  
No Co-I's Assigned to this Task

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**Task Objective:**  
To determine the effects of sooting on droplet combustion characteristics using optical and intrusive techniques. The parameters to be studied include burning rate, flame dynamics, extinction, disruption and soot particle dynamics.

**Task Description:**  
The sooting behavior will be studied using expanded beam line-of-sight extinction and subsequent 3-point Abel deconvolution to determine the soot volume fraction distribution; two-wavelength optical pyrometry to determine the soot temperature within the region between the flame and the droplet; and thermophoretic sampling/transmission electron microscopy to determine the soot morphology (radius of gyration, primary particle size, fractal geometry, etc). Feasibility studies will also be performed using Laser-Induced-Incandescence techniques to determine droplet soot volume fraction.

These experiments will be performed for a wide range of conditions to vary the sooting propensity of the droplet using various fuels, pressure, oxygen indices, droplet dimension and inerts as parameters. In addition, computational modeling of the soot particle dynamics using the balance between thermophoresis and Stefan drag will be compared with the experimental measurements. The modeling efforts will be advanced interactively with the experimental measurements of the soot volume fractions and the soot particle dimensions.

**Task Significance:**  
The combustion of a pure, single-component liquid droplet provides an ideal problem from which to obtain valuable information for both basic and applied scientific purposes. The importance of the isolated droplet burning process has promoted extensive experimental and theoretical investigations for more than 40 years. In terms of the practical relevance, the knowledge of individual droplet burning characteristics provides insights into some of the more complex mechanisms involved in spray combustion. Since estimated energy production through spray combustion processes accounts for more than 25% of the world's output, droplet combustion remains a viable field for continued research.

Since the pioneering microgravity droplet combustion experiments of Kumagai and coworkers back in 1957, there have been numerous theoretical, computational and experimental studies analyzing the burning characteristics of isolated droplets. However, sooting effects have typically been neglected due to the complexities involved (both experimental and computational/theoretical). However, recent microgravity experiments indicate that soot/sootshell formation affects all four of the important measurable parameters involved in droplet combustion: burning rate, flame diameter, extinction and disruption. Thus, our understanding of the burning characteristics of droplets can only be complete by considering a detailed study of the sooting behavior for a wide range of characteristic times and dimensions.

In all previous microgravity studies, the degree of sooting was estimated by visual observations of the sootshell. This study will focus on the effects of sooting on the droplet burning rate, flame dimensions, extinction and disruption by accurately measuring the soot volume fraction, temperature and soot morphology and dynamics. These measurement of the temperature and soot volume fractions will also prove beneficial in assessing the importance of radiative heat transfer.
Progress During FY 1995:

The major accomplishment this year was the completion of a drop tower/aircraft rig for examining fiber-supported droplet combustion. The basic design was based on that of DCE (Droplet Combustion Experiment), with two significant, additional diagnostic features. These were full-field soot volume fraction measurements via laser light extinction, and gas phase temperature measurements using two-wavelength pyrometry. Many successful drops were performed.

In choosing a chamber to use for this experiment, our initial goal was to permit multiple optical paths and maximize chamber volume. This led us to consider a rectangular chamber, which would most efficiently utilize the space available on a standard drop package. This also would enable the placement of windows for viewing on multiple orthogonal paths. Unfortunately, such a chamber would have to be very thick and heavy to tolerate the corner stresses which would be induced when pressurized/evacuated. Therefore, the decision was made to use the DCE-style cylindrical chamber, with a modified window layout to accommodate the required optical paths.

The droplet deployment and ignition hardware was built using the successful design of DCE. However, a fiber support for tethering the droplet was designed and installed to keep the droplet from drifting out of the field of view of the optics. A thermophoretic soot sampler was designed, but not yet incorporated into the chamber.

The challenge for this drop rig buildup was to insert a substantial volume of additional diagnostics in the limited space available, while not exceeding the allowable weight limit for the drop tower. Therefore, early in the design stages, the specifications and weights of the optical components (and drawings of custom-made component holders) including the beam-expanding subsection, two-wavelength pyrometry subsection and extinction measurement subsection were determined. (A laser beam-expanding system not requiring the use of a spatial filter was chosen, significantly reducing the degree of alignment needed prior to the experiment.)

The calibration factors for the gray-level intensity conversion for several possible data acquisition and analysis systems were determined. A computer imaging code was written to measure droplet surface area, volume and diameter regression for use in normal-gravity and microgravity experiments.

The graduate student performing the drops duplicated the video analysis system used back at the university in the drop tower at LeRC. This system included a computer equipped with a frame grabber and a separate high-resolution monitor. All of the algorithms needed to deconvolute the light extinction and pyrometry measurements were available. The student, who spent a third of the year at LeRC, assisted NASA engineers in placing all of the optical components and holders on the drop rig. He also calibrated the two-wavelength pyrometry cameras using a high-temperature black body and calibrated the laser detection camera using various neutral density filters.

The first successful drop was performed on 9/22/95, producing ignition for a decane droplet in air. (The sooting region appeared smaller than expected, enabling a magnification of 2.5 times for subsequent experiments.) Heptane and decane droplets of different sizes have also been burned in air.

For most of the year (while the drop rig was being built), there was substantial 1-g work performed to determine settings for the optical diagnostics, establish numerical algorithms, and obtain baseline flame data. Soot volume fraction experiments were conducted using toluene, benzene, decane, heptane, and various toluene/methanol mixtures at 1, 0.5 and 0.25 atm. Gravimetric sampling of the emitted soot from the open-tip flames of toluene determined whether the optical measurements of soot volume fraction could provide information regarding total soot emission from the very sooty fuels. (This is important because most heavily sooting fuels have open-tipped flames and the sooting region is usually much larger than the area that can be measured using conventional optical techniques.)

Thermophoretic sampling experiments in toluene and heptane flames, and subsequent transmission electron micrographs measured primary particle size and radius of gyration. Then, the optical measurements from various sections of the sooting region were used to determine if a scaling law existed to predict the emitted soot. As initial mass of fuel used to generate the droplet decreased, so did the droplet size (and therefore the flame size), however, it was noted that despite the change in the initial diameter, the conversion of fuel to emitted soot remained constant.
Using ferrocene as an additive, normal gravity droplet soot volume fraction measurements were conducted. (Ferrocene may be used with toluene to reduce sooting characteristics.) Without additives, toluene combustion in microgravity will likely produce conditions that will violate the optically-thin assumptions used in light extinction techniques.

Soot volume fraction measurements in an ethylene/air diffusion flame (the Santoro flame) were compared to single line of sight results to assess the accuracy of the light extinction and deconvolution techniques. Two-wavelength temperature measurements using the Santoro burner were compared with thermocouple measurements to calibrate the full-field two-wavelength pyrometry technique.

TEM/radius of gyration measurements were made for various fuels that produce soot with similar optical properties as droplet flame soot. More than 500 soot aggregates from each flame of acetylene, ethylene and propane were analyzed using a non-subjective technique to determine the number of aggregates required to determine the sample threshold that would produce meaningful averages. This study, in conjunction with the gravimetric sampling/light extinction experiments that are being performed as part of a NIST effort, is important in determining the correct light extinction constant to be used in the experiments.

Laser-induced-incandescence experiments on droplets and gaseous diffusion flames were conducted. These gave an independent check on the soot concentration field.

Depending on the temperature and soot volume distributions, the emission intensity can change as a function of fuel-type, pressure, oxygen concentration, and various other environmental factors. To ensure that the use of a single neutral density filter (placed in front of the light extinction camera) can discriminate flame emission while maximizing the laser intensity transmission, a novel technique was designed in which the laser light is chopped. In this manner, successive frames can be used for light extinction plus flame emission, and flame emission alone. By subtracting the flame emission signal, a corrected attenuation frame can be obtained.

**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

Presentations


Candle Flames in Microgravity

Principal Investigator: Dr. Daniel L. Dietrich
NASA Lewis Research Center (LeRC)

Co-Investigators:
Ross, Dr. H.
T'ien, Prof. J.S.
NASA Lewis Research Center (LeRC)
Case Western Reserve University

Task Objective:
The goal of this work is to further investigate some of the features of candle flame combustion observed during a recent small scale candle flame experiment conducted on board the space shuttle. These are specifically as follows:
1. To observe whether a steady flame can exist in a purely diffusive environment;
2. To understand from a fundamental aspect the observed near-extinction flame oscillations;
3. To examine the nature of the interactions between two candle flames.

As a secondary objective, we will use the candle as a model system (non-propagating, steady-state diffusion (non-convective) flame) to investigate buoyant scaling arguments for diffusion flames. Testing and scaling analysis to date shows that utilizing reduced pressure, enhanced oxygen environments do not produce a buoyancy-free environment, as currently published scaling arguments suggest.

Task Description:
These objectives will be accomplished by a program consisting of the development of a comprehensive numerical model of the candle flame, ground-based testing in normal-gravity laboratories and drop towers, and another small-scale glovebox experiment.

The development of a comprehensive numerical model represents the most significant addition to the proposed program. The gas-phase model will be a modification of an existing two-dimensional code developed for flame spread over solid fuels under the guidance of one of the investigators. The initial model for the wick/liquid phase has already been developed by the principal investigator.

The ground based testing will utilize existing drop rigs and other existing experimental equipment to fully characterize the candle flame in a range of ambient conditions (i.e., atmospheric to sub-atmospheric pressures, nitrogen-oxygen, helium-oxygen atmospheres). The data will be imaging of the flame, thermocouple measurements, LDV and possibly PIV, all of which the investigators have experience using. The use of more advanced diagnostics will also be explored during the later phases of the program.

The space experiment will be a glovebox experiment that is similar to the existing Candle Flame in Microgravity glovebox experiment (same investigators). The minor proposed changes will be: a larger free volume, the performance of the thermocouple measurements (a capability that existed but was not used on the last experiment because scheduling conflicts), the ability to maintain a large candle separation distance and to simultaneously ignite the two candles.

Task Significance:
The candle flame in microgravity is a unique/model system (non-propagating, steady-state diffusion (non-convective) flame). The data from this work is expected to improve our understanding of diffusion flames on a fundamental level.
Progress During FY 1995:

The initial formulation of the computational model is complete. The initial formulation solves the steady-state droplet combustion problem with gas-phase radiative loss and single-step chemistry. The formulation for the more complex non-spherically candle flame is underway. The candle will be treated as a saturated porous material at a constant temperature which greatly simplifies the wick and coupling of the wick and gas-phase. The full Navier-Stokes equations must be solved for this formulation.

The candle flames project was selected for manifest as a MIR glovebox experiment. This experiment will be an enhancement of the USML-1 experiment with the following features: 1) a larger candle box with a greater free area which should translate into a longer flame lifetime, 2) more candles to develop statistics of the combustion process, 3) temperature measurements using fine wire thermocouples, 4) point measurements of the flame luminosity, 5) point measurements of the far-field oxygen concentration, 6) automatic ignition and simultaneous ignition of two candles, and 7) candles with different wick diameters and lengths and different candle diameters. The flight hardware was designed and constructed and will be delivered this year. Everything is on schedule for a December Priroda launch date.

A new style of candle was designed and constructed. The design of the candle is a liquid fuel and a porous metal wick (similar to an alcohol burner). Initial testing was completed using heptane, decane, isopropanol and methanol at air, 1atm (normal gravity) with different wick diameters and exposed wick lengths. Initial testing is extremely promising. The flame size and shape and burning rate are very reproducible. The flame size, shape and burning rate are also easily controlled from the wick length and diameter. This may prove to be an excellent model candle since the fuel properties are well known and the properties of the porous metal wick are known and can be controlled. An insert was manufactured for the Droplet Array 2.2 Second Drop Tower rig so that it can be used to test the liquid candles. Testing will start in August 1995.

STUDENTS FUNDED UNDER RESEARCH:

| BS Students: | 1 |
| MS Students: | 1 |
| PhD Students: | 0 |

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Presentations
II. MSAD Program Tasks — Flight Research

Discipline: Combustion Science

Investigation of Laminar Jet Diffusion Flames in Microgravity: A Paradigm for Soot Processes in Turbulent Flames

Principal Investigator: Prof. Gerard M. Faeth
University of Michigan

Co-Investigators:
No Co-Is Assigned to this Task

Task Objective:
The flight project is an experimental and theoretical investigation of the mechanisms of soot formation in laminar jet diffusion flames under conditions of low buoyancy. The flight experiment is in the Combustion Experiment Module.

Task Description:
Several types of experiments have been conducted. The majority have been in normal gravity, studying the influence of gravity by varying the ambient pressure. In addition, experiments have been conducted on NASA's low gravity aircraft, (the KC-135 at JSC). The work has focused on mapping soot volume fraction, temperature, soot particle size and gas species in a variety of hydrocarbon flames. The flight experiment will be limited to measurement of soot volume fraction, soot particle size and some temperatures.

Task Significance:
In most flames of practical interest, soot radiation is the dominant mode of heat transport to combustor components and a dominant mechanism for flame spread and growth. The experimental results, combined with theoretical modelling will confirm or deny the applicability of the conserved scalar formalism to soot properties in diffusion flames. These results, when combined with those of ground-based low- and normal gravity experiments will increase the current fundamental understanding of soot formation processes in both laminar and turbulent flames and increase our ability to model them. Consequently this will have a significant impact on our understanding of systems such as the spread of unwanted fires, design of jet engines and large scale boilers.

Progress During FY 1995:

October:
Data on various hydrocarbon air flames have been rechecked to find soot nucleation and growth behavior; the results indicate that nucleation is associated with a first-order acetylene reaction while growth can be explained by parallel first order reactions involving both ethylene and acetylene. Measurements of soot temperature distributions within the premixed ethylene/air flames are nearing completion; measurements for these flames will turn to velocities and soot structure during the next report period. Analysis of imaging measurements of soot volume fractions obtained at NASA-LeRC was completed satisfactorily for propylene/air flames and is in progress for ethylene/air and propane/air flames; this work will continue throughout the next report period.

November:
The two papers describing soot nucleation and growth for acetylene/air and other hydrocarbon/air flames were completed and submitted to Combustion and Flame. Measurements of soot temperature distributions within the premixed ethylene/air flames were completed; at the present time laser velocimetry measurements of velocities in these flames are in progress and will be completed during the next report period. Analysis of imaging measurements of soot volume fractions obtained at NASA-LeRC was completed satisfactorily for ethylene/air flames and is nearing completion for propane/air flames; this work should be completed, and analysis of multiline temperature measurements initiated, during the next report period.
II. MSAD Program Tasks — Flight Research

Discipline: Combustion Science

December:
A paper describing effects of hydrodynamics on soot nucleation and growth was completed for the upcoming Aerospace Sciences Meeting; this paper also has been submitted to the Journal of Propulsion and Power. P.B. Sunderland completed the written version of his Ph.D. thesis; the thesis currently is being evaluated and reproduced. Several issues concerning the flight experiment were resolved: the soot volume fraction measurement was demonstrated, agreements concerning success criteria and repeat tests were completed, and an agreement about an improved optical system for the soot volume fraction measurements was completed; work continues on testing the soot temperature measurement system. Measurements of velocities were completed in the premixed flame apparatus, they increase significantly with distance from the burner exit Q a property that was not considered during earlier studies of soot in these flames.

January:
The papers by Sunderland and Faeth, and Lin et al., were presented at the Aerospace Sciences meeting; the thesis by Sunderland has been approved; and a paper by Xu et al., has been submitted to the Spring Technical Meeting of the Canadian Section of the Combustion Institute. Work still is in progress concerning evaluation of the soot temperature measurement system for the flight experiment. Measurements of velocities in the premixed flame apparatus were repeated successfully. Current measurements on this apparatus are concentrating on soot structure and gas species concentrations.

February:
The paper by Sunderland et al., has appeared in Combustion and Flame; the paper by Xu et al., has been accepted for presentation at the Spring Technical Meeting of the Canadian Section of the Combustion Institute; and an invited paper by Faeth and Koylu was completed and submitted for the 8th ISTPC. Measurements of soot structure and gas species concentrations in the premixed flames are in progress and will continue during the next report period. Measurements of flame lengths at low gravity (KC-135 tests) were completed while evaluation of the soot temperature measurement system for the flight experiment is still in progress.

March:
The paper by Farias et al., has appeared in the Journal of Heat Transfer. Measurements of soot structure were completed for all three flames allowing computations of soot nucleation and growth rates. These calculations have highlighted a few problems with the data base where measurements are currently being repeated; these measurements and updated rate determinations will be completed during the next report period. Calibration of the gas chromatography revealed some problems about separating some species and obtaining adequate repeatability for other species. We have been working with the manufacturer to resolve the problems and plan to continue gas composition measurements throughout the next report period. Routine evaluation of test information from NASA-LeRC, for the LSP experiment, continued throughout the report period.

April:
Measurements of soot structure were completed for the three premixed flames and the data was reduced to yield soot nucleation and growth rates. Initial growth rates were comparable to earlier observations of Harris and Weiner (1993) and decreased with increasing residence time as Harris and Weiner observed as well; this behavior is thought to be caused by reduced flame temperatures due to radiative heat losses, which reduces the radical pool needed to activate the carbon surface for soot growth reactions. We also observed reduced nucleation rates, which supports parallel behavior for reactions of heavy hydrocarbons toward observable soot particles. Earlier problems about gas concentration measurements have been resolved and these measurements will continue throughout the next report period. Evaluation of test information from NASA-LeRC for the LSP experiment continued throughout the report period.

May:
Measurements of species concentrations were completed in the three flames and all data was reduced to find fundamental reaction rate parameters. Acetylene is the dominant species available for soot formation, growth rates decrease as temperature decreases and nucleation rates decrease consistent with their known temperature dependence. Measurements have now turned to condensable hydrocarbon species (CHS), this work, and routine evaluation of LSP results from NASA-LeRC work.

Printed: 27 FEB 1996
June:
The sampling system for condensable hydrocarbon species (CHS) and for gravimetric measurements of soot concentrations in turbulent premixed flames was developed successfully. Measurements with this arrangement are in progress but results thus far indicate relatively low concentrations of CHS and good agreement between earlier optical and present gravimetric measurements of soot concentrations; these measurements will continue throughout the next report period. Work has begun to develop a detailed model of soot formation in the premixed flames.

July:
Measurements of the concentrations of condensable hydrocarbon species, and gravimetric measurements of soot concentrations, continued throughout the report period, with results completed at C/O ratios of 0.79 and 0.89; similar measurements at C/O=0.99 are in progress and should be completed during the next report period. Findings continue to show good agreement between optical and gravimetric measurements of soot volume fractions. Initial calculations of premixed flame properties using a simplified model were completed satisfactorily; work during the next report period will involve calculations with a more complete model. Work on the write-up of experimental procedures, and the brochure write-up for the LSP experiment continued during the report period.

August:
Measurements of the concentrations of hydrocarbon species and gravimetric measurements of soot concentrations were attempted at a C/O ratio of 0.99, using both the original and new burners. Unfortunately, flame structure conditions to correspond to our earlier measurements could not be repeated; therefore, we are moving on to consider gas species concentrations for intermediate conditions that are less affected by burner age. Predictions of gas species concentrations for these conditions are very promising using the mechanism of Leung and Lindstedt, current work is considering other conditions and the mechanism of Frenklach and coworkers. Correlations of soot growth rates with predictions of radical concentrations look very promising; this work will continue throughout the next report period. Writeup of experimental procedures is still progress.

September:
Work continued on setting up intermediate premixed flames and measuring stable species concentrations and other properties within them; this work should be nearing completion by the end of the next report period. Work continues on preparing for calculations using the mechanism of Frenklach and coworkers; data has been entered and properties checked thus far, with calculations to follow during the next report period. It is found that the surface growth mechanism continues to look promising with quite reasonable collision efficiencies found thus far, we hope to complete this development during the next report period. Interactions with NASA-LeRC about LSP and writeup of experimental procedures continued.

**STUDENTS FUNDED UNDER RESEARCH:**

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**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Journals**


Proceedings


Presentations


II. MSAD Program Tasks — Flight Research

Unsteady Diffusion Flames: Ignition, Travel, and Burnout

Principal Investigator: Dr. Frank Fendell
TRW

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
Investigate the adequacy of frequently adopted, simplified mathematical models to describe the behavior of laminar flames, primarily diffusion flames, by comparison with definitive experimental data, conveniently available from burning in a microgravity environment.

Task Description:
A combined experimental and theoretical approach is used, in which a high degree of spatial symmetry is achieved by experimental design, as a simplification for both data collection and modeling. Parametric variation of stoichiometric proportion and reactant dilution at ignition is to provide sufficient data to test models.

Task Significance:
The adequacy of frequently used models to predict diffusion-flame behavior can be tested. This project will facilitate comparison of the accuracy of alternative diffusion-flame models, from the relatively simple to the highly inclusive. Clarifying what minimal level of inclusiveness in formulation suffices to meet a user's requirements is practically advantageous. The work is particularly pertinent for flames with gaseous fuels, such as hydrogen, natural gas and propane, and near-cold-wall quench layers. The approach may be extended for the study of other gaseous chemically reacting systems.

Progress During FY 1995:
The contract NAS3-27264 was signed by J. M. Smink of TRW on September 2, 1994 and by E. C. Mensurati of LeRC on September 6, 1994. It was received by TRW on September 13, 1994. The effective starting date of the contract is July 11, 1994.

The plan is to initially segregate hydrogen diluted with argon, and oxygen diluted with helium, into two equal half-volumes within a squat rectangular-solid-configured container with isothermal, noncatalytic, impervious walls. The contents of the two half volumes are each to be at atmospheric pressure and temperature, and to have equal density, so that the "average molecular weight" for each half volume is the same. The overall contents are to be sufficiently fuel-deficient such that, if (conceptually) the initial, segregated contents of the container were mixed to form a perfectly homogeneous gaseous mixture, the diabatic flame temperature (equilibrium burned-gas temperature) would be consistent with a vigorous diffusion flame, but not so hot that either dissociation of product species (mainly water vapor) or gaseous radiative heat transfer or the physical integrity of the container is of concern.

In practice, a thin metallic film seems a candidate material to serve as the impervious initial interface between the half volumes; it is to be removed, on command, by rapid lateral translation in its own plane, much in the manner of the roll-up of a window shade. Ignition, probably by spark, is to occur after a short interval for reactant interdiffusion. Aside from possible exceptions at early times, the diffusion flame is anticipated to travel into the hydrogen-containing half volume. The key objective is to measure the flame temperature and flame position as a function of time until depletion of the deficient reactant (hydrogen) results in extinction of the planar diffusion flame. The fairly hot, soot-free, barely visible nature of a vigorous hydrogen-air flame poses a challenge for diagnostics.
LeRC has provided information to TRW about H2O spectral emission characteristics, spark and hot wire ignition electrical circuitry and power requirements on the space shuttle and station.

The QFD brainstorming sessions identified separator design, ignitor design, gas fill system design and 2-D measurement of flame temperature and position as potential breadboards. We determined alternative approaches for the solution of each problem. We also selected the most promising approaches for each problem, to start building the breadboards. The Breadboard Management meetings of the Lewis team, which started after the QFD process, are continuing once a week for two and a half hours each. Conference calls are being held at least once a week with the PI and his experimentalists at TRW to keep them involved in the process. The bimonthly Combustion Advanced Development Review meeting was held on June 27, 1995 to discuss the status of the project with the Division management.

Experimentally, the design of the test chamber is nearly completed. The chamber is to be created specifically for testing in December in the NASA 2-second drop tower; this testing is to observe the adequacy of techniques adopted for thin-film removal, subsequent gas ignition, and planar-flame establishment. TRW and LeRC are designing a rapid-removal-and-storage mechanism for the thin-film separator, probably involving the use of an inflatable seal. The objective is to permit redeployment of the separator (for another experiment in the 2 Second Drop Tower) without breaking the pressure seal of the container. This would eliminate the need for a new leak-test before every experiment.

A major current activity is assuring that the chamber is designed to meet NASA requirements for pressure vessels. For that purpose the results obtained earlier by the Project Scientist, Suleyman Gokoglu, are being utilized. He ran the NASA CEC program extensively for adiabatic flame temperature calculations, under different fuel-to-oxidant ratios and for different dilutions, to clarify safety requirements, as well as to guide the choice of operating conditions. The calculations were done for both constant pressure and constant volume conditions, with the additional constraint that the initial density of the hydrogen + argon mixture is the same as that of oxygen + helium mixture. The calculations show that chamber pressure is not expected to exceed 10 atmospheres. The mixture ratios which result in adiabatic flame temperatures between 2100 - 2300 K are identified. The chamber material and wall thickness, and the locations and dimensions of the optical windows for diagnostics, are being selected on the basis of the constraints and requirements for science and engineering.

A set of experiments are being planned to determine the suitable ignition parameters, i.e. minimum and nominal ignition energy, temporal interval for energy deposition and electrode separation distance. These experiments will also provide assurance for safety that conditions which may lead to detonation are being avoided. TRW is sending a candidate "home-grown" spark-ignition system to LeRC for testing. For temperature measurement, LeRC is currently designing an experiment to properly calibrate the two candidate techniques selected via the LeRC QFD process: 1) band-ratio technique from the infrared-emission of the water-vapor product (LeRC's current first choice) and 2) ceramic fiber mission technique (LeRC's current second choice). TRW is currently exploring options for a minimally intrusive flame-temperature-measurement technique that is suited to the planar-flame geometry (and possible minor departures therefrom).

The current concept for parametric investigation in extended microgravity is for repeated use of one unit, initially evacuated and sealed, with a thin film in place to divide the contents into two equal subvolumes. Soon before test execution, one control is to initiate and terminate the gradual, simultaneous filling of the half volumes, each half volume being connected to its own 'feed' bottle. This procedure maintains negligible pressure differential across the thin film, yet avoids the problem of hydrogen permeability that would arise if long storage of a gas-filled unit were required. Upon command, chemical "freezing" of the container contents by expansion into a large volume is planned as an option during testing.

TRW favors composing the chamber for spaceflight of stainless steel, and forming it of two top-hat-configured parts, with a retractable/restorable foil fixed between the two halves by an inflatable seal. TRW favors the use of sapphire needle valves in backfilling the test chamber, to meet the requirement of maintaining "equal" pressure on
each side of the thin-film separator as the gaseous compositions are introduced. The maximum allowable pressure difference between the two half volumes after the fill but before the separator is removed is specified. The main concern in the repeated use of one chamber for parametric testing is the accumulation of the heat from combustion, in the absence of convective cooling in microgravity. However, the heat from any one test is modest, and is distributed over an apparatus with large thermal inertia.

A manuscript entitled "Unsteady Planar Diffusion Flames: Ignition, Travel, Burnout" prepared by F. Wu and F. Fendell, was presented as Paper 95-0147 in the Microgravity Combustion Session (Dr. Merrill K. King, chairperson) on January 9, 1995 at the AIAA 33rd Aerospace Sciences Meeting in Reno, NV. A six-page summary was prepared by the PI for, and presented by him at, the 3rd International Microgravity Combustion Conference, held on April 11-13, 1995 in Cleveland. Issues briefly discussed include: (1) thin-flame modeling for differing diffusivities (including peripheral calculation of when the sufficiency condition for extinction, derived from a global second-order irreversible Arrhenius-type model of the finite-rate chemical kinetics, is met); (2) flame quenching and possible water-vapor condensation, in proximity to the cold lateral walls of the container; and (3) assignment of values to the dimensionless parameters arising in the parabolic boundary/initial-value problem describing the travel of a planar diffusion flame. These parameters include the Lewis-Semenov numbers for fuel vapor (hydrogen) and gaseous oxidizer (oxygen), the first and second Damkohler numbers, the isentropic coefficient, the dimensionless Arrhenius activation temperature, and the stoichiometrically adjusted mass fractions for fuel vapor and gaseous oxidizer fractions for fuel vapor and gaseous oxidizer.

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals


Presentations

II. MSAD Program Tasks — Flight Research

Discipline: Combustion Science

Fundamental Study of Smoldering Combustion in Microgravity

Principal Investigator: Prof. A. C. Fernandez-Pello
University of California, Berkeley

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
The overall project objective is to increase the fundamental understanding and the prediction of smoldering combustion under normal and microgravity conditions. This in turn will help the prevention and control of smolder originated fires in both normal gravity, and in space-based environments. The specific objectives are to determine the smolder characteristics of a polymeric porous combustible material (polyurethane foam), in a quiescent or convective oxidizing environment, both at normal and micro-gravity.

Task Description:
The project objectives are accomplished by conducting experiments on ground-based and space-based facilities, and by developing theoretical models of the process. The experiments are conducted with polyurethane foam as fuel with mixtures of oxygen/nitrogen as an oxidizer. Temperature at several locations of the sample are measured with thermocouples; the resulting temperature histories are used to obtain the smolder propagation velocity and smolder reaction temperature as a function of the oxidizer flow velocity and oxygen concentration. The experimental results are used to verify and improve the theoretical models of smolder combustion. Experiments are also conducted to observe the potential transition from smoldering to gas flaming, and to determine the conditions and mechanisms leading to this transition.

Task Significance:
Smolder is important as both a fundamental combustion problem, i.e., the propagation of a heterogeneous, non-flaming, surface combustion reaction through a porous combustible material, and as a fire safety problem, i.e., the production of toxic compounds and the potential initiation of a fire through the transition to flaming. There is a need for further understanding of the problem for better prediction and control of smolder in both normal gravity and microgravity. Furthermore, microgravity introduces additional questions about the transport of mass to and from the reaction, and the transfer of heat from the reaction that must be resolved in order to predict smolder behavior in a space-based environment.

Progress During FY 1995:
The following is a monthly step-by-step progress report:

Task 1: Ignition Study of Opposed Smoldering
October
Experiments comparing char insulation v. FiberFrax insulation have been run. We are presently leaning toward the use of the char, for it seems to retain its consistency fairly well. All instrumentation has been recalibrated (data acquisition board, mass flow controllers) to ensure accurate experimentation after a change in personnel. TGA and DSC have been acquired from the Lawrence-Livermore Labs and are being set up.

November
The igniter wire for the heater broke during a run and has been rebuilt and has been tested to satisfaction.

December
We reviewed the NASA tapes for smoldering glovebox experiments aboard the space shuttle and plotted five different runs. We are analyzing and discussing some interesting things such as breathing behaviors. More tests

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have been conducted to expand the database and they have continued to show consistent and repeatable results. As part of this test set, we are monitoring additional parameters: the weights of char, foam before, lost weight, and unburnt foam.

January
The MSC post processor (provided by the MSC project engineering team) has been tested and performs acceptably. Further runs have been conducted; the variable being studied is voltage effects.

February
Several opposed flow smolder runs have been completed en route to the compilation of a flow regime chart. This chart shows three regimes: burn to completion, melting, and no light. The melt regime appears to lie between the low energy, no light zone and the high energy, burn to completion zone. The material behind the heater has been studied. Replacing the FiberFrax blanket after one or two runs shows the best reproducibility. Using char left from previous runs undergoes a reaction for the first test condition, but appears to be unreactive after this initial reaction. When the opposed reaction is allowed to continue until completion, the data show the return of the reaction to a forward smolder condition and the oxygen limitation of this secondary reaction.

March
Further runs are commencing to better define the burn regimes for the opposed flow smolder. Data from previous SCM runs 1-4 have been compiled into a MS Excel Workbook entitled SCM_1-4.xls. Data and plots comparing experimental geometries and gravity conditions are included, which have been sent to the anonymous ftp site zeta.lerc.nasa.gov. Char was produced and sent to NASA Lewis for use in future microgravity experiments.

April
The experimental apparatus has been disassembled and cleaned to insure accurate data collection. Further tests at flow rates of 0.1 mm/sec ignition and 0.7 mm/sec burn have been made to outline ignition curves. The latest version of the MSC Post Processor has been tested and found to work without incident.

May
Several more tests at flow rates of 0.1 mm/sec ignition and 0.7 mm/sec burn have been made. These tests have been made varying the ignitor power and the time the ignitor is on in order to better develop the ignition curve.

June
More char has been made and sent to NASA Lewis. Several more tests at various flow rates have been made to fill in the ignition curve.

July
Further test conditions on the ignition curve are being plotted. Points which seem to lie in outer lying areas are being checked for errors by rerunning at the given conditions. The curves seem to show a threshold value for causing melting of the char near the heater of approximately 55 watts. The radiation from the thermocouples in close proximity to the heater is vital in this threshold value.

August
Test cases have been run for various power levels and times for air flows of 0.1 mm/s ignition and 0.7 mm/s during smoldering. No unusual trends were noted during the test procedures for the tested conditions. It is noted that for input heat fluxes less than about 0.2 Watts/cm², of the sample will not maintain a self propagating condition for the given conditions.

A model that matches a requisite flux required for ignition of the smolder wave to the provided heat flux by the Nichrome wire heater. The model predicts test conditions that will produce a self propagating smolder over a range of input heat fluxes on the order of 0.1 to 1.0 watts/cm².
II. MSAD Program Tasks — Flight Research

September
Further experiments to determine ignition criteria for opposed-downward smoldering. The latest parameters to be varied are the flow velocity. Present tests are being conducted with flow velocities of 0.7 mm/s during both ignition and burning.

Estimates were made of the CO and CO\textsuperscript{2} production for the STS-69 experiments for post combustion gas analysis and sent to NASA Lewis.

Task 2: Forward Smoldering

October
Experiments with 2mm/s and 4mm/s flow velocity have been run. Cursory inspection indicates a strong dependence on the flow velocity, as temperature and exhaust gas concentration data are significantly different. Although detailed analysis has not yet been done, there is a trend of higher smolder velocities and temperatures with increasing flow velocity.

November
Experiments at 2mm/s to 10mm/s flow velocity are being run. Preliminary results indicate that smolder velocity is linear with air flow velocity. Also at high flow velocities, only one reaction front appears as opposed to the three (pyrolysis, smolder, and char oxidation) observed at low flow velocities.

December
The combustion chamber has been cleaned and rebuilt. We have made several runs with a 0.2mm/s ignition flow rate. Preliminary examination of the data shows little if any effect. Higher ignition flow rates will be studied in the future to determine the velocities at which the ignition flow rate becomes important.

January
Several more runs using 0.1mm/s ignition flow rate varying the power to the heater have been made.

February
It has been determined that the char left behind by the smolder reaction can be relit under similar conditions as the virgin foam. It appears that the pyrolyzate is providing most of the fuel for this second-burn reaction. Runs using only a nitrogen purge for the flow gas have been made also. The data from this will be used for determining the effect of the combustion reaction during ignition.

March
All of the data points gathered so far have been plotted on a semi-log chart of power vs. time. There is the possibility of a linear relationship among the data when viewed in such a fashion. Several more runs have been made along this boundary to improve the resolution of the chart and to make certain of the linearity.

April
More tests were run for determining the ignition curve.

May
Several more tests have been run using 0.1mm/sec ignition and 1mm/sec burn flow rates. These have been run in the low power, long time area of the ignition curve. A preliminary program for controlling the Thermo-Gravimetric Analyzer has also been completed.

June
Tests to determine the low power requirements for ignition have been completed. It has also been found that it is possible to cause a "melt" even at this low power. Several tests have been conducted in the high power region as well (80W-90W).
II. MSAD Program Tasks — Flight Research

July
Several more runs in the high power region have been completed. Much of the program to take data from the Thermo-Gravimetric Analyzer has been completed. Debugging is still in progress.

August
Several more runs have also been made in the high power region. Some strange behavior was noted and the combustion chamber has been rebuilt to see if this can be accounted for via experimental error. The foam no longer seems to melt at high power, only at intermediate power ranges.

September
Several more experiments have been made for the 0.2 mm/s case at low power settings. Experiments running a 0.5 mm/s ignition velocity have also been run. Preliminary results indicate that even at low power, the 0.2 mm/s gives the same power/time curve as the 0.1 mm/s case.

Task 3: 2-D Smoldering And The Transition To Flaming
January
We have made significant progress in finding a method to image the inside of the foam sample during burning. Ultrasound is presently very promising. We have acquired 25kHz ultrasound transducers and discovered that this frequency penetrates our foam (at our present experimental sizes) with reasonable attenuation—sending at 8V rms and receiving at 2mV rms for a 13cm sample. There is very good transmission through the char, distinguishing itself from unburnt foam. We will investigate higher frequencies to determine the conditions that provide the best combination of foam transmission and spatial resolution.

February
40kHz ultrasound transducers have been tested. They do not penetrate the foam as well as the 25kHz ultrasound transducers. Consequently, we will most likely restrict our imaging technique to frequencies between 25kHz and 40kHz.

March
The homepage for the University of California, Berkeley Microgravity Combustion Lab has been setup and can be found at http://www.me.berkeley.edu/mcl. Gases for the gas chromatograph have been ordered. 40kHz ultrasound transducers with higher sensitivity have been sent from Japan (from Prof. Miyasaka) and await testing—if these transducers perform well, then we will employ them in our ultrasound imaging apparatus, otherwise, we will go ahead with the 25kHz transducers. We are working in close collaboration with Professor Miyasaka (Fukui University, Japan) on the signal amplification and reconstruction aspect of the ultrasound imaging technique, as an e-mail connection has recently been established.

April
The high sensitivity 40kHz ultrasound transducers have been received from Professor Miyasaka and have been tested. They performed exceptionally and will be used in a prototype testing of the imaging system next month. The signal responses are as follows: sending at 12Vrms through a 13cm piece of foam, we can detect as signal of 3mVrms. The detection for the char is on the order of 1Vrms and for the Fiberfrax insulation (at 1/2 inch) is 35mVrms. Professor Miyasaka will bring more of these speakers next month, when we work on the imaging technique. Final manuscript for the ISTP-8 paper has been sent for publication.

May
Professor Miyasaka visited for a week and helped work on the GC sampling technique, TGA, and ultrasound imaging technique. The gases for the GC have arrived. The TGA has been repaired and hardwired to a PC for data acquisition and awaits calibration.
June
The GC has been setup (i.e. piping to gases, dedicated power line, computer interface). A capillary column to measure basic HC's and O$_2$, CO, and CO$_2$ has been ordered. We are still trying to develop a linear positioning system for the ultrasound transducers, but are experiencing difficulty in designing one at low cost (under $2000). The new pentium 100 computer has been setup.

July
The GC has been tested and works satisfactorily. Liquid CO$_2$ has been ordered for the cryogenic oven. Professor Miyasaka has arrived from Japan to help work on the linear position system and data acquisition for the ultrasound imaging system.

August
A PorAPlot Q capillary column for hydrocarbons, CO$_2$, O$_2$, and CO has been installed in the gas chromatograph. The TCD has been connected to the FID in series. A linear positioning system for the ultrasound transducers has been obtained (UniSlide, Velmax Inc.). An array of speakers and receivers has been setup. Professor Miyasaka designed and constructed an eight channel multiplexer and rms converter for the receiver signals, a power amplifier for the speaker signals, and some preamplifiers for receiver signals. Additional preamps have been made along with all connections and ancillary components, so that a working prototype of the imaging system is ready to be tested.

September
The ultrasound imaging system has been set up and attached to the linear positioner. Scanning movement, multiplexing, and data acquisition are done by computer. Software has been written for preliminary testing. Scans of the foam/char are being made.

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BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals


II. MSAD Program Tasks — Flight Research

Discipline: Combustion Science

Ignition and the Subsequent Transition to Flame Spread in Microgravity

Principal Investigator: Dr. Takashi Kashiwagi
National Institute of Standards and Technology

Co-Investigators:
Baum, Dr. H. National Institute of Standards and Technology (NIST)
MacGratton, Dr. K. National Institute of Standards and Technology (NIST)

Task Objective:
The objective is to conduct radiative ignition followed by transition to flame spread over various, different combustible solid surfaces in microgravity in order to understand the transition mechanisms from the ignition to subsequent flame spread, and to determine the effects of preheating, oxygen concentration, external flow velocity, geometrical configuration, and sample materials on the transition and flame spread characteristics.

These results are needed for comparison with the numerically calculated data taking advantage of microgravity to determine their accuracy and to examine the validity of the chemical and physical processes used in the calculations.

Task Description:
This study consists of a theoretical part and an experimental part. The latter has two different flights; one is a planned Glovebox experiment called RITSI (USMP-3, STS-75) and the other is a future flight experiment being called TIGER-3D which is currently in the definition stage. A numerical model which can calculate time-dependent phenomena of ignition and subsequent flame spread over a thin cellulosic paper has been developed in three different configurations — axisymmetric, two and three-dimensional Cartesian. Ignition can be initiated locally by an external thermal radiation or a heated wire.

The unique feature of this study is that localized ignition is initiated in the middle part of the sample instead of at the end of a sample. Therefore, there could be two possible flame fronts; one travels toward upstream and the other toward downstream under an external wind. A slow external wind up to 5 cm/s is studied in the calculation. It is planned that this model will be extended to apply to a thermally thick material, a plastic material, and smoldering. The results can be directly compared with the experimental results which will be obtained in the future RITSI and TIGER-3D flight experiments.

Task Significance:
For the first time, it is possible to study the transition from a radiative ignition to flame spread in the absence of overwhelming buoyant convection. The elimination of natural convective flow in the microgravity experiment simplifies both the formulation and subsequent computation of these time-dependent, three-dimensional problems that include other complexities such as finite rate chemical kinetics in both the gas and solid.

Progress During FY 1995:
The flight hardware for the RITSI experiment is nearly complete for a planned Feb. 1996 flight. In addition, the SRD for TIGER-3D was detailed further, and a DC-9 experiment apparatus is being fabricated for low gravity testing of TIGER-3D's laser ignition in Jan. 1996. An SCR for TIGER-3D is planned after the low gravity tests are completed.

Fifteen microgravity experiments were conducted using the RITSI engineering hardware in the Japan Microgravity Center (JAMIC) 10 second drop tower in March. Experimental parameters were atmospheric oxygen concentration (air, 35, and 50%), external slow wind velocity (0, 2, and 5 cm/s), experimental configuration (2D and 3D), and ignition mode (an electrically heated wire as a pilot and a tungsten lamp without any pilot). Whatman ashless filter papers (44) were used as a test sample. Ignition and subsequent flame spread were observed for all tests.
Since theoretically calculated results indicate that transition from ignition to flame spread is marginal in air, four tests in air were intended to test feasibility for the RITSI Glovebox experiment. Although flame spread rate could not be accurately measured during the short time after ignition in air, it appears that transition to flame spread was achieved after a sample was ignited using a heated wire and also the lamp with a heated wire above the irradiated area. In 35 and 50% oxygen concentration, the transition was achieved for all cases even some of them were ignited by the lamp only. Detailed data analysis of front view video pictures, 35 mm photographs of flames, and thermocouple data is in progress. Our calculated results show faster flame spread along the open edge of a paper sheet than along the center part of the paper. This trend was examined by igniting circularly a center part of a narrow paper by the radiation from the lamp in 50% oxygen with 2 cm/s slow wind. Shortly after ignition, the flame front reaches to the open edges of the narrow paper and flame spread very quickly along the open edges. Flame spread rate along the open edges was roughly three times faster than that along the center part of the paper.

The calculations show that the most interesting phenomena for ignition and flame spread under an external flow occurs when the external flow velocity is very small. In this case, the governing equations have an elliptic nature (i.e., information can travel upstream and downstream). Because of this, there is some concern on effects of the side walls and ceiling on the transition for the RITSI experiment. Since the experiment will be conducted in the glovebox, the size of the flow chamber is relatively small and the side walls and ceiling and the floor are relatively close to the sample. The 3D code has been modified to conduct several calculations varying the distance between the walls and the sample and also between the ceiling and the sample surface. All combustion experiments in microgravity must start from some kind of ignition but often the initial effects of ignition on intended combustion are ignored or neglected. Therefore, an analytical model to study the effects of sudden, localized well-defined heat release on the slow flow field has been constructed. The results indicate that the heat release rate creates a large modification to the flow field, whose magnitude is proportional to heat release rate.

STUDENTS FUNDED UNDER RESEARCH:

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals

Proceedings

Presentations
II. MSAD Program Tasks — Flight Research

Discipline: Combustion Science

Studies of Premixed Laminar and Turbulent Flames at Microgravity

Principal Investigator: Prof. Paul D. Ronney
University of Southern California

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
The objective of this work is to study the effects of gravity-induced buoyancy on the combustion limits of premixed gas flames. The following sub-tasks have been pursued during FY95:

1. Flame structure and stability at microgravity.
2. Evaluation of alternative ignition sources for premixed gases.
3. Experimental simulation of combustion processes using autocatalytic chemical reactions.

Task Description:
The three primary tasks related to this study are as follows:

For task 1, we are studying flames in a variety of gas mixtures having low Lewis number (Le) in drop tower and aircraft tests, particularly those exhibiting stationary spherical flame structures (flame balls). These tests are in support of the Structure of Flame Balls At Low Lewis-number (SOFBALL) flight experiment to be performed using the Combustion Module-1 (CM-1) hardware on Space Shuttle Mission MSL-1 in April 1997. Numerical modeling of flame balls is being performed to refine the flight test matrix and provide a theoretical comparison to the experimental results. Additionally, high-Le flames propagating in tubes are being studied to examine the different instabilities found under these conditions.

For task 2, we are measuring the minimum ignition energies of combustible gases using laser ignition sources and comparing these results to spark ignition results.

For task 3, we have introduced the use of aqueous autocatalytic chemical fronts for simulating premixed gas flame fronts. The aqueous fronts propagate at steady rates but are unaffected by heat losses, have practically no change in transport properties across the front and exhibit very small thermal expansion. Three flows have been employed: Taylor-Couette and Capillary-Wave flows to produce nearly isotropic turbulence, and Hele-Shaw flow to study buoyancy-driven front propagation in a flow described by linear equations.

Task Significance:
It is anticipated that the results of these studies will lead to an improved understanding of the fire hazards that may exist in orbiting spacecraft and of ways to minimize these hazards. Furthermore, such studies may lead to an improved understanding of the mechanism of combustion limits in Earth-based devices, which in turn could lead to the development of cleaner and more efficient engines through the use of lean premixed combustors.

Progress During FY 1995:
1. Flame structure and stability at microgravity

Eight weeks of tests have been performed on NASA's KC-135 research aircraft in support of the SOFBALL flight experiment using a flight-like combustion chamber, ignition system, and instrumentation. The issues studied include: accuracy of the gas mixing system, sensitivity and optimal gain settings for the intensified video cameras.
II. MSAD Program Tasks — Flight Research

Discipline: Combustion Science

and radiometer system, false coloring schemes to aid the flight crew in setting video intensifier levels, and final refinement of the space flight test matrix. While these tests are primarily for engineering development, some new science results have been obtained, notably that the radiometer signal levels are consistent with those predicted by theory.

A one-dimensional, unsteady flame code employing detailed chemical and transport sub-models has been installed and run on HP workstations. These predictions are generally in qualitative agreement with experiments and analytical theories. Additionally, these computations have determined the stability limits of flame balls in regimes inaccessible to analytical theories.

Theory predicts at high Lewis number, pulsating and traveling-wave instabilities should occur. In conjunction with Dr. Howard Pearlman of NASA-Lewis, experiments have been performed that show, in addition to these modes, spiral-wave flame fronts, multiple, and other instabilities found in other nonlinear chemical and biological systems. None of these modes had been conclusively observed experimentally in previous works, probably because the recent experiments employed more advanced diagnostics and mixtures with higher Le than any previous work of its type.

2. Evaluation of alternative ignition sources for premixed gases

Several flight PI's have considered the use of some alternative to electric sparks for flame ignition because of the potential difficulties with high voltages and electromagnetic interference. The PI had evaluated the use of heated wire sources and found them to be inadequate for some cases, particularly very lean or dilute mixtures, because of limitations on the amount of energy that could be deposited in the gas in a short period of time without melting the wire. To test a different alternative ignition source, we have measured the minimum ignition energies (MIE) of methane-air and methane-oxygen-argon mixtures by laser sources in collaboration with The Aerospace Corporation (El Segundo, CA). The most notable trends observed are summarized as follows: 1) the laser ignition results lie at higher energy than the electric discharge results, however, the difference decreases toward the flammability limits; 2) the experimental MIEs are higher than the predictions of detailed computations by Sloane and Ronney; and 3) the MIE for picosecond-duration pulses lies at higher energy than for nanosecond pulses, however, the difference decreases toward the lean and rich flammability limits. These results may be a consequence of the size of the energy deposition region with some possible influence of gas dynamic shock losses.

3. Experimental simulation of combustion processes using autocatalytic chemical reactions

Comparison of the results of experiments on front propagation rates in TC and CW flows to theory suggested that Yakhot's Renormalization Group (RNG) theory provides the best description of turbulent flame propagation for large Damkohler number (Da) (ratio of mean chemical reaction rate to mean turbulent strain rate). At lower Da, Damkohler's original (1940) hypothesis fits these data well. The fractal dimensions of these fronts compare favorably with Kerstein's heuristic model. Recently we have studied propagating fronts under buoyancy-dominated conditions in a Hele-Shaw cell, i.e., between two closely-separated vertical walls. Initial results indicate that fingering of the rising, propagating front can occur even in this very simple system. The reasons for this are not yet well understood, since the classical effects of Lewis number, surface tension, viscosity differences between reactants and products, etc., do not seem to apply to this system.

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**TASK INITIATION:** 3/91  **EXPIRATION:** 12/98

**PROJECT IDENTIFICATION:** 962-22-05-06  **NASA CONTRACT NO.:** NAG3-1523  **RESPONSIBLE CENTER:** LeRC

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Printed: 28 FEB 1996  II-57
II. MSAD Program Tasks — Flight Research

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

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Presentations

**II. MSAD Program Tasks — Flight Research**

**Discipline: Combustion Science**

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**Ignition and Flame Spread of Liquid Fuel Pools**

**Principal Investigator:** Dr. Howard D. Ross  
**NASA Lewis Research Center (LeRC)**

**Co-Investigators:**  
Sirignano, Prof. W.A.  
University of California, Irvine  
Miller, Dr. F.J.  
Case Western Reserve University

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**Task Objective:**

For flame spread over liquid fuel pools, the existing literature suggests three gravitational influences: (a) liquid-phase buoyant convection, delaying ignition and assisting flame spread; (b) hydrostatic pressure variation, due to variation in the liquid pool height caused by thermocapillary-induced convection; and (c) gas-phase buoyant convection in the opposite direction to the liquid-phase motion. No current model accounts for all three influences. In fact, prior to this work, there was no ability to determine whether ignition delay times and flame spread rates would be greater or lesser in low gravity.

Of special interest to this work is the determination of whether, and under what conditions, pulsating spread can and will occur in microgravity in the absence of buoyant flows in both phases. The objective of this study is to determine the influence of buoyancy on flame spread over liquid fuel pools.

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**Task Description:**

The approach we have taken to resolving the importance of buoyancy for these flames is (a) normal gravity experiments with advanced diagnostics, (b) microgravity experiments, and (c) numerical modeling at arbitrary gravitational levels.

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**Task Significance:**

The modelling and experimental data provide a more complete understanding of flame spread, an area of textbook interest, and add to our knowledge about on-orbit and Earth-bound fire behavior and fire hazards.

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**Progress During FY 1995:**

**Experiment:** The experiment was flown on a Terrier-Black Brant sounding rocket; this was a major milestone since it represented the first μg combustion experiment to be performed in a sounding rocket and included more advanced diagnostics than any previous microgravity combustion experiment (on any spaceflight carrier). A 30 cm long x 2 cm wide x 2.5 cm deep fuel tray was located inside a 10 cm x 10 cm cross-sectional area flow duct to provide a low-speed forced air flow over the fuel tray. The top and sides of the flow duct were also fitted with windows to allow recording of the propagating flame by eight camera systems. Two top cameras and two side cameras each recorded half of the flame spread. Two side-viewing Particle Image Velocimetry (PIV) systems recorded liquid-fuel flow patterns as the flame crossed the midsection of the tray. An Infra-Red (IR) camera recorded the liquid surface temperature field ahead of the flame crossing this same region. And finally, A Rainbow Schlieren Deflectometer (RSD) system recorded liquid-phase temperature gradients.

The sounding rocket flight provided just over six minutes of μg time to perform the experiment. The butanol fuel circulated internally for 60 seconds before filling the tray to ensure the PIV particles were well distributed in the fuel. The tray was then filled with fuel in low gravity; only a very few small bubbles located near the bottom of the fuel tray were observed and they did not apparently affect subsequent observations. This was an important finding of the experiment, since fuel management in μg was a key technological issue. Once the tray was filled with fuel, a bulk air flow was started and a waiting period began to allow any pool surface deformations to damp out and any internal fluid flows from the filling process to cease. When the downlinked PIV showed no residual fluid

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motion, a command was telemetried for the primary igniter to be energized. At the end of the test, all experiment systems were de-energized and a valve was opened to the vacuum of space to evacuate the payload for safe handling upon retrieval. The evacuated payload returned to Earth via parachute and was recovered without damage an hour after the flight.

**Preliminary Experimental Results**

**First Flight Test:** In general the intensity of the μg flame was much less than the 1 g flame; the μg flame monotonically decreased in intensity as it propagated; the flame stood off the surface more in μg, as well. The top camera view showed that the 1 g flame front was much less curved than the μg flame front, due most likely to the slower rate of propagation (discussed next) and increased sidewall heat loss in the latter case.

Ignition and spread are completed in 6-7 second in 1g. As expected, the 1g flame exhibits rapid, pulsating spread alternating in 1-2 second intervals between the slow and fast propagation phases of the pulsating cycle; the crawling velocity was 1-2 cm/second and the jump velocity was 8 cm/sec. Ignition and spread took more than twice as long in μg. The character of the spread was also distinctly different than in 1g. In the first half of the tray, the flame moved slowly, without pulsation, and reached a near-steady spread velocity of about 1.5 cm/second; this value is an order of magnitude less than that which occurs on Earth in the uniform spread regime, and thus is more similar to the crawling velocity described above. Just before the halfway position, the flame jumped forward at about 6 cm/sec. It then paused at about the 20 cm position, which coincides with a thermocouple rake. After this, it rapidly and erratically spread. A number of hypotheses have been conceived as to why the flame spread behavior changed and while these are all vigorously being pursued, none is fully verified; some of these are: air flow irregularities; thermocouple rake interferences; mass flow reductions in the air stream due to the combustion's heat release and product formation; and ignition transients.

A review of the PIV video indicates slow particle motion begins shortly after the igniter is energized in both 1g and μg. The flow accelerates sharply as the flame and its accompanying vortex approach. The center of the vortex preceding the flame is deeper in μg. The 1g RSD image shows a series of vortices, close to the pool surface, which formed during times when the flame slowly propagated. The μg structure extends much deeper into the fuel depth and shows a much longer preheat distance. This clearly indicates that buoyancy serves to stratify the warmer liquid near the surface in 1g; absent this force, as in μg the warm liquid is carried into the fuel depth by the thermocapillary-driven motion of the liquid fuel.

The 1g IR image shows a unique structure we have called twinning. As the 1g flame began the crawling portion of the pulsating cycle, the preheat region extended, as expected, a greater distance ahead of the flame. It then developed a very symmetric vortical structure toward the sidewalls of the fuel tray. These vortices rolled up while a central portion of preheated liquid continued to proceed upstream of the flame. When the surface temperature exceeded the flash point, the flame then jumped forward and obscured the twin vortices. The cycle then repeated itself with the reformation of the twin vortices. The μg flames showed an entirely different structure; recall, in this case, the flame was spreading steadily and much more slowly. As also seen by the RSD, the IR revealed the preheat distance ahead of the flame was much greater in μg than in 1g. While the flame itself showed excellent side-to-side symmetry, no twin structures were observed in the IR image. Instead, asymmetric vortical motion was clearly apparent in μg.

**Second Flight Test:**

In the second microgravity tests, however, the flame spread very slowly and uniformly across the entire length of the pool. Unlike 1g, the microgravity flame was completely blue (soot-free), without any noticeable plume or wavering. The diagnostic instruments showed a large liquid-phase vortical flow extending deep into the pool ahead of the flame, very different than the stratified liquid flow which occurs in 1g. These observations showed conclusively that flame spread can persist in microgravity and that it is substantially different than at Earth's gravity. The acquired data will allow detailed verification of the numerical models in areas such as liquid-phase velocity and temperatures, as well as the flame spread rate.

**Modelling:** The 2-D, transient, state-of-the-art numerical model, which incorporates finite-rate chemical kinetics, variable properties, and a partially adaptive grid scheme for a planar configuration, also reached a major milestone. Numerical convergence was obtained, enabling the prediction of flame spread behavior in microgravity for both
quiescent conditions and, for the first time, opposed air-flow conditions. A series of parametric runs determined that the results are sensitive to the choice of chemical kinetic coefficients for the second-order reaction rate expression. For example, a 20% decrease in the value selected for the activation energy, causes the character of flame spread to change from pulsating to uniform spread (for the same conditions as was flown in the sounding rocket experiment). The code was used to simulate previous drop tower and the aforementioned sounding rocket experiment. Similar to drop tower experiments and previous numerical calculations, the flame is predicted to extinguish in quiescent air in zero gravity. The sounding rocket experiment behavior was predicted qualitatively. There was a long ignition transient period during which the flame spread slowly. During this period liquid-phase flow develops far ahead of the spreading flame. After this initial period, the ensuing flame spread consists of a series of slow-moving and raid-moving progressions across the liquid surface. In some cases, the flame moves backward before accelerating forward. The pulsation frequency increases with increasing values of the pre-exponential constant or decreasing activation energy. In general, the model predicts that a combination of forced, opposed air flow and thermocapillary-driven concurrent flow can cause a gas-phase recirculation cell to form in front of the leading edge of the flame in microgravity. As was previously shown for normal gravity flame spread without forced flow, this recirculation cell entrains fuel vapor and leads to a prediction of pulsating flame spread. In μg, the mean flame spread rate and pulsation frequency increase with opposed flow velocity. This provides some guidance for the selection of conditions for the next sounding rocket test.

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**TASK INITIATION:** 2/91  
**EXPIRATION:** 2/97

**PROJECT IDENTIFICATION:** 962-22-05-07  
**NASA CONTRACT NO.:** NCC3-1212  
**RESPONSIBLE CENTER:** LeRC

**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Journals**

**Presentations**


Printed: 27 FEB 1996
II. MSAD Program Tasks — Flight Research

Combustion of Solid Fuel in Very Low Speed Oxygen Streams

Principal Investigator: Prof. James S. Tien
Case Western Reserve University

Co-Investigators:
Sacksteder, K.R.
Ferkul, P.V.

NASA Lewis Research Center (LeRC)
Analex Corporation

Task Objective:
The objectives are to:

1. Enhance the understanding of flame spreading above solid-fuel surfaces through a systematic investigation in the low-speed, forced and buoyant flow regimes.

2. Determine the mechanisms that induce flammability limits of flames over solid surfaces in low-speed flows.

3. Resolve the structure of flames spreading in low-speed flows through a characterization of the velocity, temperature and selected-species fields in their vicinity.

Task Description:
This task consists of the development and flight of a space-based experiment, the continued pursuit of ground-based results in drop towers and aircraft experiments, and the continued development of a comprehensive predictive model of concurrent-flow flame spreading.

The flight experiment is to be accomplished in three phases. An experiment to be conducted in a Glovebox Facility is to provide insight into global flame characteristics of thin and thick fuels. In the second phase, thin fuels are to be burned as they are dispensed from a continuous supply in such a way that the flame is fixed in space relative to the test apparatus. In this way, detailed diagnostic measurements are greatly simplified and transient flame behavior can be observed as the test conditions are slowly varied. In the third phase thick fuels will be burned to address the effect of transverse heat conduction into the fuel, yet with flames spreading slowly enough that the same diagnostic measurements can be made.

The ground-based experimental program consists of observations of flame spreading in partial gravity environments (first attempted as part of this program), the ignition behavior of flames over thin and thick solid fuels in microgravity forced flows, and the implementation of temperature, velocity and species concentration measurement systems.

The model development consists of integrating a fully elliptical fluid mechanics formulation with surface and gas phase radiative interactions in a transient model that attempts to predict the behavior of the selected experimental test fuels in concurrent-flow burning.

Task Significance:
In normal gravity, buoyant air motion in flames is at least 20 to 30 cm/sec. Flame spreading mechanisms that are present only in lower flow velocities, such as spacecraft air-ventilation currents, cannot, therefore, be studied in normal gravity. This research seeks to contribute to fundamental combustion science, and to improve the basis for engineering improved spacecraft fire safety.
Progress During FY 1995:

1) A device for evaluating the proposed technology for the flight experiment was completed and flight tested aboard the NASA parabolic aircraft. This device, named the Solid Fuel Delivery System or SFDS, consists of a low-speed wind tunnel enclosed around a spooled strip of thin solid fuel. The fuel is fed into the flow duct under the control of an automated system that detects the leading edge of the fuel as it burns away such that the burnout front of the fuel is kept at a fixed location in the duct. The flame surrounding the fuel is also held stationary in the duct, facilitating the probing of the flame with various candidate diagnostic systems.

In flight tests aboard the KC-135 and the DC-9, the SFDS demonstrated the feasibility of the continuous fuel supply concept in low-speed purely forced concurrent flows. The initial concept for sample ignition provided reliable ignitions, but introduced complexity in the fuel exit plane. The ignition system has been replaced with a retractable, reusable ignitor. This ignition has been successfully tested aboard the aircraft. At year's end the difficulties of spooling unburnt or partially burnt fuel has been resolved in normal gravity tests, and is awaiting demonstration tests in planned aircraft tests.

Mr. Richard Pettegrew, a student of Professor James S. T'ien of the Case Western Reserve University, defended his Master's Thesis entitled, "An Experimental Study of Ignition Effects and Flame Growth Over a Thin Solid Fuel in Low-Speed Concurrent Flow Using Drop Tower Facilities." This work clarifies the extent of concurrent flow flame spreading data that can be obtained in drop tower facilities beyond the earlier work of Grayson and Tien. In each case over a range of flow velocities from 1-10 cm/sec, flames were observed to be unsteady during the 5.2 seconds of test time. However, images of growing flames and pyrolysis regions and fuel and flame temperature data were obtained for the first time in these tests for preliminary evaluations of the (steady) theory.

2) The Forced Flow Flamespreading Test (FFFT) glovebox experiment was expanded for an additional flight opportunity aboard the Russian MIR Space Station as part of the Priroda Module. This additional flight opportunity was used to plan for additional flight data for thickness effects on concurrent flow flame spreading and for some retesting of the fuels that were flown in the USML-1 Wire Insulation Flammability Experiment. During this year, the MIR version of the experiment was proposed, awarded, built and delivered to Russia. The original version of the FFFT experiment was manifested to fly aboard the Space Shuttle during the USMP-3 mission on STS-75. These tests will focus on the spreading of flames in different speed concurrent flows using flat samples, and over cylindrical, heated samples. At year's end, this experimental hardware was being completed in preparation for a November shipment for flight integration.

3) Dr. Ching-Biau Jiang, a student of Professor James S. T'ien of the Case Western Reserve University, defended his PhD dissertation entitled "A Model of Flame Spread Over a Thin Solid in Concurrent Flow With Flame Radiation." This work consists primarily of the addition of gas-phase radiation to the concurrent flow flame spreading model of Ferkul and T'ien. The gas-phase radiation reduces the size of the calculated flames, more in line with experimental results and identifies a shift in the reduced gravity flammability boundary due to reduced chemical reaction rates. At high speeds the model identifies the importance of gas phase radiation as a heat feedback mechanism that increases flame spread rates but does not change the blowoff velocity.

Since Dr. Jiang's dissertation was completed, his model has been extended to consider purely buoyant flows, providing predictions of quenching in reduced, but non-zero, gravity levels, which can only be concluded in the presence of radiative losses from the flame. Another enhancement of the numerical model was also added to account for the possible effects of the finite flow duct that will be required in the proposed flight experiment. By reconfiguring the model boundary conditions to simulate the duct walls, the computed flames are forced closer to the fuel surface by the boundary layer growth, increasing heat transfer to the surface and increasing flame spread rates and fuel burning rates. These results will be important to the development of the science requirements for the flight experiment.
II. MSAD Program Tasks — Flight Research  Discipline: Combustion Science

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BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals


Proceedings


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II. MSAD Program Tasks — Flight Research

Droplet Combustion Experiment

Principal Investigator: Prof. Forman A. Williams
University of California, San Diego

Co-Investigators:
Dryer, Prof. F.L.
Princeton University

Task Objective:
The objective of this collaboration is to provide scientific support for fundamental, single component, isolated (free and tethered) droplet burning experiments to be performed on board space platforms. The specific objectives of these studies are to provide fundamental results which impact fire safety issues in low gravity environments and to advance the scientific understanding of droplet burning/flame/extinction dynamics over characteristic times which can not be studied in ground based configurations. Engineering and design support functions contribute to: developing and refining methods to perform isolated droplet combustion experiments in general; determining and testing methods to acquire appropriate experimental observations; developing and evaluating methods for presenting and analyzing the collected data. Experimental support functions include: theoretical analyses to define appropriate experimental conditions and to analyze the experimental results obtained; bench scale and drop tower experiments to test and verify methodologies to be applied in the space-based experiments.

Task Description:
The objectives are pursued experimentally in ground-based bench facilities at the respective institutions and NASA Lewis, as well as in the 2.2 Second and 5 Second Drop Towers located at NASA-Lewis. Studies include isolated, filament-suspended and free droplet studies in bench scale facilities (200 to 1000 micro-meter diameter), 2.2 Second Drop Tower experiments in tethered and free droplet modes (1000 to 1500 micro-meter diameter), and 5 Second Drop Tower experiments in tethered and free droplet modes. Theoretical analyses based on asymptotic methods are pursued at the University of California San Diego, while uni-dimensional (sphero-symmetric) and two-dimensional axi-symmetric numerical computations are pursued at Princeton. The fuels of interest to this work include: normal-heptane, normal decane, methanol, and methanol-water mixtures.

Experiments are performed at varying ambient pressures, oxygen contents, and with varying diluent species to develop an experimental data base over parameter ranges which can be studied in the available test times in each facility. Flight experiments are required because of the very long test times and low gravity conditions required for significant portions of the work. Data typically obtained include back-lighted images of the droplet, and direct light images of the flame structure as functions of the experimental test time. Data are acquired film as well as through video imaging (inclusive of intensified imaging methods to obtain direct light intensity distributions over specific wavelengths) as a means of producing quantitative comparisons of flame image with model predictions. Image data are processed using high precision methods to obtain droplet diameter history, flame structure history, and extinction diameter. These parameters are then directly compared with theoretical predictions. In ground-based experiments, supplemental experimental measurements such as integrated droplet chemical composition history, and direct light imaging measurements at several wavelength ranges can be obtained for comparison with model predictions. Data obtained in ground-based experiments complement that which can be obtained in flight experiments in that the characteristic times of the former extend from (with some overlap) to times considerably shorter and with droplet diameters much smaller than can be studied in the latter.

Task Significance:
The overall purpose of the research is to achieve fundamental advances in the science of droplet combustion, which is central to a number of energy-producing devices and systems. In particular, unsteady liquid and gas phenomenon, and extinction chemistry of normal alkanes and simple alcohols are investigated.

Printed: 27 FEB 1996
Progress During FY 1995:

Studies at University of California-San Diego
Analytical modeling on the structure and extinction of quasi-steady, spherically symmetric diffusion flames around methanol droplet continued during this reporting period. Analyses corresponding to two, three, and four-step reduced mechanisms applied to the study of n-heptane droplet burning have been completed previously, and similar studies are now complete for methanol. For methanol droplets, the equilibrium of water-gas shift reaction is a very good assumption so that extinction diameters obtained from the two and three-step analysis are almost the same. The asymptotic analysis corresponding to the four-step mechanism, obtained by relaxing the H atom steady-state assumption, gives extinction diameters 50% smaller than those of the two and three-step analyses. Initial comparisons between the initial asymptotic results and the numerical calculations performed at Princeton showed significant disagreement with the asymptotics predicting extinction diameters roughly one order magnitude smaller. While the early work did not account for the possible absorption and dissolution of water into the droplet as a function of burning time, a more recent effort now treats this condition fully. When this effect is included, the calculated results agree well with those found for the fully transient numerical analyses performed at Princeton (see below) for one atmosphere pressure.

Studies at Princeton University
In addition to supporting the engineering development of the flight experiment for DCE, and experimental studies in the 2.2 Second Drop Tower (see below), efforts have further advanced the computational tools and sub-models for numerically predicting the combustion of methanol and normal heptane droplets. Progress in each of these areas is summarized below.

Methanol and N-Heptane Droplet Combustion Modeling
A report describing in detail the numerical modeling which was performed to develop the test envelopes for the DCE second flight, methanol test matrix entitled, "Theoretical Basis for Estimating Test Times and Conditions for Drop Tower and Space-Based Droplet Burning Experiments with Methanol and N-Heptane" was issued as Princeton University MAE Report No. 1999.

Sphero-Symmetric Numerical Modeling of the Combustion of Methanol/Water Mixture Droplets
The one-dimensional numerical model developed earlier was updated to more fully consider the effects of pressure-dependent chemical kinetics and internal liquid phase transport effects. These modifications have led a refined understanding of previously published data and consideration of the combustion of bi-component droplets. The effect of liquid phase transport on the combustion of methanol/water droplets has been more fully explored. In the presence of only diffusive transport, little water should absorb into an initially pure methanol droplet during combustion. Experimental evidence in the form of measured extinction diameters and deviation from 42-law burning, both from ground-based laboratory experiments at Princeton, and from 2.2 Second Drop Tower data, suggest the presence of significant absorption/dissolution and integrated water accumulation over the droplet burning time. The presence of internal liquid transport due to fluid motions is apparently responsible for this result. Further study of the possible sources which produce such motions are under investigation. Extensive numerical calculations for pure methanol and methanol/water mixture droplets of 1-5 mm diameter in various N2/O2 and He/O2 oxidizing environments compare favorably with new experiments performed both by Princeton as well as in collaboration with NASA personnel in 2.2 Second Drop Tower (see below) when the effects of internal liquid motion are accounted for. Further comparisons will be made to the results of the FSDC glove box experiments (USML-2 and MSL-1). FSDC experiments not only include studies on pure methanol droplets, but on methanol droplets which initially contain water as well as ethanol and ethanol/water droplets.

Numerical Modeling of the Combustion of Pure Normal Alkane Droplets and Mixtures and Chemical Mechanism Development
A one-dimensional, transient numerical model has been used to simulate the combustion of bi-component liquid droplets of methanol/water and normal heptane/hexadecane. The results will be compared to those obtained in the FSDC glove box experiments (USML-2). The methanol/water calculations show that even gross droplet burning parameters can be affected by liquid transport phenomena. Initial calculations for n-heptane/hexadecane droplets were
performed using semi-empirical chemistry, as well as the detailed chemical kinetic mechanism proposed earlier by Warnatz (for pre-mixed flame propagation). More recently, and in collaboration with work underway for the Department of Energy, chemical kinetic experiments on n-heptane oxidation have been performed, and a new skeletal mechanism has been developed for high temperature n-heptane oxidation. Detailed droplet combustion calculations underway presently, and derivation of a reduced mechanism for this skeletal kinetics, along with comparative asymptotic droplet combustion calculations currently underway at UC-San Diego will be compared in the future.

Finally, a particular difficulty in comparing experimental data with theory has typically been how to quantitatively relate the light emission imaging of flames to the model predictions. Princeton has pursued appropriate modifications of the n-heptane model chemistry so as to predict chemiluminescent emissions observed from the flame structure surrounding burning-heptane droplets. Work in progress shows considerable promise for defining the emission intensity distribution as a function of burning time, thus giving quantitative comparisons not only as to the flame positions, but gross flame structural features.

Development of 2-D Droplet Combustion Model

Early drop tower experiments performed under this program showed compelling evidence that under slow gas/drop relative motions, gross droplet burning parameters might be significantly modified from those for spherically-symmetric burning conditions. Princeton has pursued development of a two dimensional, axi-symmetric, time dependent droplet combustion/vaporization model using spectral element techniques. In collaboration with members of the computational fluid dynamics group at Princeton (Dr. S. Orszag), the developed model is now being applied with simple-step chemistry to study the combustion of particles under at relative particle/gas Reynolds numbers from 1 to 10. While this initial testing is proceeding with simple chemistry, the formulation of the approach is directed toward including much more detailed descriptions of chemical and transport processes.

Experiments

An extensive range of droplet combustion experiments were performed using the 2.2 Second Drop Tower at NASA Lewis. Pure methanol and methanol/water mixture droplets were tested in O2/N2 and O2/He environments. Pure methanol burning rates have now been measured in 18%-35% O2/N2 and 30%-70% O2/He. These tests as well as those carried out for n-heptane burning in O2/He environments at the NASA Lewis 5-second ZGF were instrumental in developing an ultraviolet OH-emission imaging technique. The data analysis technique which, along with detailed numerical modeling, were used to determine the instantaneous flame position are described in an upcoming paper. Additional tests are planned, including collaborative testing with NASA-Lewis personnel.

Program Reviews:

During this reporting period a successful RDR/PDR for the Droplet Combustion Experiment (DCE) was completed in collaboration with the NASA personnel.

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BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals

Proceedings
Presentations


II. MSAD Program Tasks — Flight Research

Surface Controlled Phenomena

Principal Investigator: Prof. Robert E. Apfel
Jet Propulsion Laboratory (JPL)
Yale University

Co-Investigators:
Holt, R.G.
Jet Propulsion Laboratory (JPL)
Tian, Y.
Yale University

Task Objective:
The goals of this research are to investigate the rheological properties of surfactant-bearing liquid drops. By comparing experimental results in the ideal environment of the Spacelab to our theory for spherical equilibrium drops, we can validate the model. We can then synthesize a generic theory which can handle arbitrary acoustic fields and static deformations, in order to have a technique for studying static and dynamic surface properties for surfactant-bearing drops which can be successfully applied in 1-g experiments.

Task Description:
Single liquid drops are introduced into the center node of an acoustic standing wave in DPM's Near-Ambient Chamber. They are allowed to reach quiescent equilibrium. Then, shape oscillations about either a spherical or a spheroidal equilibrium shape are excited - either by a momentary increase and release of the z-axis acoustic pressure or by a periodic modulation of that pressure. The resulting oscillations are recorded on video tape and ciné film for later analysis.

Three sample materials will be investigated on USML-2: triply distilled water, water with small amounts of Triton X-100 or bovine serum albumin at five different concentrations. The additives provide contrasting time scales: Triton X-100 is a nonionic fast-sorbing surfactant while BSA is sorption-inhibited. A range of drop sizes from 4 to 12 cc will be investigated for each of concentration.

Task Significance:
The flight experimental data will be used to validate the theory which describes drop shape-oscillations as a function of various surface parameters. These parameters will be obtained in microgravity from drops oscillating about a spherical equilibrium shape as well as about an acoustically induced oblate shape. By comparing the differences in the natural frequency and damping constant for these oscillations, the theory and experimental techniques can be used to perform measurements of the surface properties on the ground. Theoretical development and ground-based experiments to support the microgravity work are also performed.

Progress During FY 1995:
Ground-based work, primarily in preparation for the USML-2 mission has focused on assuring ourselves that a) improved DPM will work up to expectations, b) the crew will be well trained for the mission, and c) the quality of the science and the ultimate science return will be maximized.

In preparation for USML-2, tests have been performed to determine an optimal tip configuration for drop deployment; the best of the ground based designs were tested in KC-135 flights as well. Tests have been performed on whether the flexible tubing used to transport fluids for forming drops can produce bubbles. Both levitated and suspended samples are being used to study spurious sample rotation of the type observed on USML-1; from our understanding techniques for compensation are being developed.

We have designed a computer simulation of drop dynamics that can be used in training the USML-2 crew. It has been delivered to the Payload Crew Training Complex at MSFC.
Ten papers covering the experimental, analytical, and computational studies into the rheology of surfaces covering the work done in this fiscal year will be submitted within the calendar year. Experimental measurements of surface tension from the static shape of a very small levitated drop compare well with theoretical predictions. Oscillating drop experiments analogous to those of USML-1 and USML-2 have been carried out on the ground, including measurement with the samples to be flown on USML-2. An automated system for reducing the video image of a drop to half a dozen parameters is now processing three frames a minute. The deformation on both static and oscillating drops due to the levitating and oscillation-driving acoustic field has been studied using a boundary-integral technique. The interaction of speakers in various configurations and the resulting torques have been studied with levitated and suspended samples.

Initial experiments at 1-g have been performed with arrays of charged drops in the presence of both acoustic and electrostatic fields. Drops are uniform and the drop patterns are regular. Studies have been performed on the evaporation of single drops and of multiple drops, as a precursor to studies of the evaporation or combustion of dense sprays.

**Students Funded Under Research:**

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**Task Initiation:** 2/90  **Expiration:** 6/96  **Project Identification:** 963-24-04-03  **Responsible Center:** JPL

**Bibliographic Citations for FY 1995:**

**Journals**


Tian, Y., Holt, R.G., and Apfel, R.E Investigations of liquid surface rheology of surfactant solutions by droplet shape oscillations. II. Experiments in 1g and 0g (USML-1) environments. J. Colloid Interface Sci., (submitted).

The Dynamics of Disorder-Order Transitions in Hard Sphere Colloidal Dispersions

Principal Investigator: Prof. Paul M. Chaikin
Princeton University

Co-Investigators:
Russell, Prof. W.B.
Princeton University

Task Objective:
To study the disorder-order transition in a hard sphere colloidal particle system, utilizing laser light scattering to detect (1) viscosity changes as the transition from fluid to crystal to glass occurs, (2) diffusion of particles, (3) nucleation and growth of the crystals and (4) rigidity after the transition is complete.

Task Description:
The approach has focused on simulating low-gravity by fluidizing a bed of specially prepared hard spheres (silica particles with short polymer coatings) with a counterflow of solvent. The counterflow allows an approximation of microgravity.

Task Significance:
The study of crystal formation in the microgravity environment can lead to a greater understanding of how gravity affects the formation of many kinds of materials. The focus of the experiment is to use a material which can exist in multiple phases depending on its concentration in a colloidal suspension. Laser light scattering images will provide structural information about the material as it goes through liquid, ordered crystal, and disordered glass phases.

Progress During FY 1995:
A novel colloidal fluidized bed was assembled to gain some insight into the behavior of hard spheres in the absence of gravity. Using this method it is possible to support particles by the viscous drag of upwardly flowing fluid. Since an isolated sphere settles at about 3 mm/day, this requires an extremely slow fluidized bed by any conventional standards. The resulting steady state then mimics the equilibrium conditions expected under microgravity if the Péclet number is sufficiently small, i.e. for very slow sedimentation relative to diffusion.

The fluidized bed at Princeton is coupled with a light scattering instrument capable of detecting Bragg diffraction from the crystalline phase and dynamic light scattering from either the disordered or ordered phases. The scattered light is detected at any desired angle by a photomultiplier tube (Hamamatsu H4730-01 PMT and Amplifier/Discriminator Assembly) through optics mounted on an arm that rotates with the goniometer. The detection optics consist of a lens that images the scattering volume to the photo cathode of the PMT and two apertures to limit the acceptance angle and define the scattering volume. The signal is collected by a digital correlator (ALV 5000) which constructs the temporal autocorrelation function and computes the static intensity of the sample. To investigate the structure factor at low angles, a CCD camera is used (GBC CCD-500E) to capture the intensity of the pattern on a screen.

The shear modulus and viscosity of the hard sphere dispersion are detected via Bragg Spectroscopy. A function generator drives a speaker attached to the rotational stage holding the fluidized bed, which gently rotates it at a prescribed frequency, creating an oscillatory disturbance within the dispersion. When the amplitude of the oscillation is small relative to the inverse of the scattering wave vector, the scattered intensity fluctuates at the same frequency, and the ensemble averaged temporal autocorrelation function of the scattered intensity from the center of the bed indicates the amplitude of the response. The rheological properties are then extracted by modelling the colloidal system as a linear viscoelastic solid.
The scattering intensity pattern at low angles from a crystalline dispersion in the fluidized bed forms five symmetric rings on each side. The Bragg angles indicate an fcc structure for a fluidized hard sphere crystal, since the ratios of the corresponding scattering wave vectors match all lines for the fcc crystal. However, the intensity of the second Bragg peak is much less than the first, suggesting that some crystal grains may have randomly stacked hexagonal structures which contribute to the intensity of all but the second peak. Thus the structure observed may be a mixture of face-centered-cubic and randomly stacked crystallites. The flight experiment should confirm if the structure is pure fcc or a mixture of fcc and randomly deposited hcp structures.

The samples needed for the flight experiment have been prepared by Princeton and are ready to be integrated into the flight instrument. The instrument itself has been designed and is in the advanced stages of assembly with a partially capable breadboard located at Princeton. The breadboard will be used to develop operational procedures for the flight experiment and to verify software functionality.

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BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals


II. MSAD Program Tasks — Flight Research

Discipline: Fluid Physics

Critical Dynamics of Fluids

Principal Investigator: Prof. Richard A. Ferrell
University of Maryland

Co-Investigators:
Moldover, Dr. M.
Wilkinson, Dr. R.A.
National Institute of Standards and Technology (NIST)
NASA Lewis Research Center (LeRC)

Task Objective:
This work has three objectives all relating to second-order phase transitions in fluids. Part one seeks to confirm the untested dynamic scaling of the asymptotic limiting acoustic attenuation in simple liquid-vapor systems. The second part seeks to confirm the fast dynamics of adiabatic heat transfer in compressible liquid-vapor critical systems using a near-term space experiment. The third objective is to expand the theory of the specific heat at the lambda transition of liquid $^4$He. The theory would allow for comparison to data from a currently planned space experiment, namely CHEX.

Task Description:
The work involved three critical fluid investigations: 1) ground-based definition of an acoustic attenuation in a pure liquid-vapor system experiment, 2) Principal Investigator activities for a fast adiabatic thermal/density relaxation space experiment which flew on IML-2 in 1994, and 3) theoretical work on the specific heat near the lambda transition of $^4$He. Item 1 involves repeating the Garland & Thoen experiment to establish techniques and demonstrate that adiabatic heat transfer losses are detectable and predictable. That knowledge will be useful in defining a space experiment that will not be hampered by the effect. It is the space experiment that would fulfill the first objective.

Item 2 employed ESA's Critical Point Facility (CPF) with visual and interferometric data. A heat pulsed wire and a 500 Volt charged wire were used to stimulate fast transients and electrostriction. This activity also allowed a refight of an IML-1 experiment to study slow density relaxation with better geometric and thermal boundary conditions.

Task Significance:
Significance is based on the fundamental character of this work, all of which is driven by experiments only possible in low-gravity. The $^4$He work is to bring current theory up to par with experiments in process. The liquid-vapor acoustics work is to probe the unverified dynamic scaling predictions of older theory, while properly accounting for adiabatic heat transfer loss mechanisms. The experiments on IML-2 also works toward the confirmation of the theory describing adiabatic heat transfer in highly compressible critical fluids.

Progress During FY 1995:
During the past twelve months, considerable effort has been devoted to the analysis and interpretation of the experiments Fast Adiabatic Equilibrium (AFEQ) and Temperature Equilibrium Bis (TEQB) that were flown aboard the space shuttle Columbia in July, 1994. This work culminated in an invited talk given by the P.I. at a Gordon Research Conference in July, 1995 and in a final report prepared by Dr. Greg Zimmerli to be presented by him in November, 1995 at the Investigators' Working Group in Frascati, Italy.

AFEQ: Dr. Min at NASA/Lewis has noted a complication in the analysis of the electrostriction portion of the AFEQ that is due to the non-linear response of the fluid to the applied electric field around the wire that passes through the sample cell. This is because the electrostrictive pressure, $P$, produced by the field is sufficient to change the density near the wire, $r$, significantly away from its critical value, $r_c$. By making use of the accepted scaling
A further impediment to a direct comparison of the theory of electrostriction with the experimentally observed interferograms is the fact that the optical system of the Critical Point Facility (CPF) does not image the sample cell onto the recording CCD camera. The "virtual" plane that the CPF does actually map onto the CCD plane is at a non-negligible distance, \( s \), away from the mid-plane of the sample cell. As a consequence, a light ray that has been influenced by the electrostrictive density inhomogeneities in the fluid, and thereby deflected by a small angle, \( q \), becomes laterally displaced by \( q \), when it impinges on the "virtual" plane. It is essential to allow for this displacement when interpreting the fringe pattern of the interferograms. A further complication is that the additional path length traversed by the ray, namely, \( q^2 /2 \), superposes on the ray a supplemental phase shift, \( p q^2 \), where \( l \) is the wavelength of the light in air. Thanks to the collaboration of visiting Professor Jayanta K. Bhattacharjee, it has been possible to analyze these refraction effects in considerable detail. A full report on the electrostrictive portion of AFEQ, that will expose and delineate all of the complications that are involved in analyzing the interferometric data, is in preparation for publication in a scientific journal.

TEQB: The primary goal in TEQB was to measure the rate of heat diffusion at a temperature closer to the critical point than is possible on Earth. The heat flow across the sample cell imposes an initial condition for the temperature relaxation that has an angular dependence that is described by a cosine (or sine) function. An expansion in normal modes is feasible. This has, in fact, been carried out, but it can be argued that the analysis of the relaxation in terms of the "tilt" function that corresponds to the presumed uniform temperature gradient of the initial condition promises greater accuracy. This is because, at every stage of the relaxation, the actual temperature distribution is more closely similar to the "tilt" function than it is to any of the Bessel's functions that describe the individual normal modes. Two alternate methods of implementing the "tilt" function approach have been followed at the University of Maryland and at NASA Lewis Research Center. The Maryland method, at any given stage of the relaxation, generates by means of Fourier analysis, an "orthogonal" fringe pattern. Multiplying this with the evolving pattern and summing numerically over all of the pixels in the pattern reveals the subsequent changes. The values inferred for the thermal diffusion coefficient, \( D \), at 100mk and 30mk agree with the values obtained by Jany and Straub from light scattering. The value for \( D \) at 10mk, which is closer to the critical point than was possible for Jany and Straub, agree with a dynamic scaling with a relatively small correction for the non-critical thermal conductivity background, as expected for this temperature. The Lewis method, on the other hand, generates explicitly a phase map which is then analyzed in terms of the "tilt" function. The Maryland and Lewis results agree above 10mk but exhibit a difference at 10mk. A further troubling discrepancy, also not understood, is the failure of the phase map to satisfy the isothermal boundary condition at the cell perimeter.

When the heat flow is turned off in TEQB, the initial condition can be expected to excite not only the "tilt" mentioned above, but also the cylindrically symmetric modes. Alternatively, one expects a flat relaxing temperature distribution with the spatial dependence consisting only of some rounding at the cell perimeter. This is a non-isobaric non-one-dimensional phenomenon for which the theory has not yet been worked out. It is necessary to include adiabatic temperature changes, as described by the now familiar Onuki-Ferrell-Hao differential-integro-equation. An expansion in eigenfunctions of the Laplace operator yields a solution in terms of a doubly infinite series. This work is still in progress.

Critical Sound Propagation: Early in FY95, measurements were made by Dr. R. Kusner of the acoustic spectrum of a critically filled SF\(_6\) resonator at low frequencies (200Hz \(< f < 3000\)Hz). The resonator was an annulus of height 3 mm and circumference 28 cm. The resonator was designed to oscillate at low frequencies in order to probe the effects of surface to volume ratio (or the thermal boundary layer) on the acoustic resonances of a critical fluid. From an analysis of these resonances, the critical exponent for the specific heat at constant volume was found to be 0.11, in agreement with present theory and recent direct measurements by Straub et al. Acoustic attenuation was significantly larger throughout the temperature range, which was attributed to mechanical losses in the resonator. In mid FY95, a phase lock technique was used to track a particular acoustic resonance as the temperature approached the critical point. An analysis of these data showed that corrections due to the boundary layer could account for deviations of the data from a simple power law relationship to within 100 mK of the critical point. Calculations by
Prof. George Tuthill, made in the fourth quarter of FY95, showed that corrections due to stratification of the critical fluid are significant within 100 mK of the critical temperature. Careful and thorough measurements of the acoustic spectrum of the annular resonator produced data that displayed the same behavior as the stratification calculations; however, the measured resonances were five percent higher than the calculated resonances within 10 mK of the critical temperature, whereas above 100 mK the measurements and calculations agreed to less than one percent. An investigation continues in an effort to understand the source of this discrepancy, which may be a failure of the equation of state or an incomplete treatment of the effects of stratification on thermal boundary layer losses.

Theoretical Studies: A further ramification of TEQB is the approach to equilibrium in the two-phase state of the fluid. We have limited our investigation of this problem to the layered one-dimensional geometry of the liquid $^3$He experiment of Zhang and Meyer. The more complicated 3-D configurations encountered in TEQB are much more difficult to analyze. By means of a completely analytical treatment, we have verified the Straub-Eicher solution for CO$_2$, a study that was prompted by the Zhang-Meyer experiment. We have noted that the predicted behavior of the vapor phases is in stark and qualitative disagreement with that reported by Zhang and Meyer. Straub and Eicher’s ascribing this to convection seems untenable. A more likely explanation, suggested by M. Moldover, is that there is an additional meniscus resulting from the wetting of the upper pair of the capacitor plates. A publication on this subject is in preparation.

In collaboration with Visiting Professor J. K. Bhattacharjee, Dr. Hao has been making a detailed comparison of his analytic $e$-expansion Ph.D. thesis critical viscosity exponent calculation with the numerical solution of Siggia et. al. A publication on this is in preparation.

In the low temperature realm, Prof. Bhattacharjee has developed a method for taking into account the effect of boundary conditions on order parameter fluctuations, with a view toward making a prediction for CHeX.

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**STUDENTS FUNDED UNDER RESEARCH:**

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**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Journals**


**Presentations**


Microscale Hydrodynamics Near Moving Contact Lines

Principal Investigator: Prof. Stephen Garoff
Carnegie Mellon University

Co-Investigators:
Weislogel, M.
NASA Lewis Research Center (LeRC)

Task Objective:
The objective of this study is to characterize the relationship between macroscopically observed dynamic contact angles and the microscopic fluid physics occurring near the contact line.

Task Description:
This task consists primarily of low-gravity experimental studies on moving contact lines. The experiments will simultaneously measure flow fields and the fluid/fluid interface very near the moving contact line using video microscopy and particle image velocimetry. The experiments will be conducted in low gravity since it is in low gravity that interfacial phenomena dictate the fluid/fluid interface shape and location.

Task Significance:
Our ground based research on moving contact lines focused on five areas: (1) confirming the validity and delineating the limitations of present asymptotic hydrodynamic models of spreading; (2) measuring the velocity field near moving contact lines; (3) examining the changes in inner scale physics for a systematically varying set of fluids and solid surfaces; (4) studying thin film formation for advancing contact lines; and (5) probing receding contact lines.

Progress During FY 1995:
We have completed our studies confirming the validity of present asymptotic hydrodynamic models for spreading. We measured the dynamic interface shape over distances on the order of the capillary length. Far from the contact line, the interface shape becomes static-like; although within the accuracy of our experiments, the interface shape at capillary numbers (Ca) as low as 0.01 is measurably different from the static shape even at distances of 80% of a capillary length from the contact line. We found that the parameter w0 determined by fitting the dynamic data near the contact line to the composite model is same as the contact angle for the static-like interface shape far from the contact line. This result confirms a fundamental hypothesis of the model. We discovered a region of the interface at moderate distances from the contact line (on the order of 1/3 of the capillary length for Ca=0.001) which is neither static-like nor described by the asymptotic model. Within this region, viscous and gravitational forces compete to determine the interface shape. This competition is not described by the present model. The residual dynamic bending of the interface even at distances on the order of a capillary length and relatively low Ca raises doubts about the accuracy of measurements of dynamic interfaces made by other techniques and reported in the literature. This work is first reported in a manuscript accepted for publication in Journal of Colloid and Interface Science.

We also completed the studies begun last year of interface shapes at higher Ca. We have made the first measurements of dynamic interface shapes in depression. We find that the present models are adequate for Ca<0.1. Above this Ca, the model predicts too large a curvature at small distances from the contact line. The region where the model is inadequate expands as Ca increases. The breakdown is either due to an insufficient number of terms in the Ca expansion describing the intermediate region or to the inner scale hydrodynamics affecting the interface shape at unexpectedly large distances. The model of the intermediate region can be extended to higher Ca. It then provides an additional parameter describing the inner scale physics. However, our measurements show that this expansion cannot be used in terrestrial measurements because of contamination from geometry dependent terms which cannot be expressed analytically. This result proves that microgravity measurements, which increase the outer length scale, will provide a unique opportunity to measure an additional parameter with which to characterize inner scale physics. This work is first reported in a manuscript accepted for publication in Physics of Fluids.
We have completed our first study of the velocity field near the moving contact line using particle image velocimetry. The polymer fluid is seeded with small bubbles 1-10 mm in diameter. Particle trajectories and velocity fields are measured and compared to the fields predicted by the modulated wedge approximation, an approximation central to the model of the intermediate region which we use to fit the interface shape. We have carefully examined our data and proved that the bubbles are tracking the fluid flow field even near the contact line where the streamlines exhibit a sharp bend. At \( \text{Ca} < 0.15 \), the model correctly predicts the flow field for distances 20-500 mm from the contact line. At higher \( \text{Ca} \), the flow field is not correctly described by the modulated wedge approximation due to the higher curvatures exhibited by the interface at larger distances from the contact line. The velocity field measured in such experiments provide necessary input to numerical modeling of systems with moving contact lines. A manuscript describing this study is presently in preparation.

Our materials studies show that the inner scale hydrodynamics of polymer melts is sensitive to local polymer/surface interactions. We have probed the effects of: polymer end termination, polymer molecular weight, ultrathin water layers on the solid surface, and interfacial energy of the solid surface. For all materials systems we probed, the parameters describing the inner scale hydrodynamics must be dependent on the velocity of the moving contact line. The spreading of polydimethylsiloxanes (PDMS) across a 7740 Pyrex surface is strongly affected by the end termination chemistry of the polymer: hydroxyl terminated PDMS exhibiting a strong velocity dependence and methyl terminated PDMS exhibiting a weak dependence. The parameter describing dynamic wetting increases with the molecular weight of the polymer. Ultrathin (~30 Å), mobile layers of water condensed from a humid atmosphere onto the pyrex surface enhance slip of the polymer relative to the solid surface. By spreading the same polymer across derivatized surfaces, we have found that the dependence of the parameter describing dynamic wetting on solid surface energy is complex. None of these conclusions can be drawn from traditional methods of measuring dynamic wetting. Our method of measuring the viscous bending of the interface near the contact line is essential.

Thin precursing films exist in front of static menisci and have been proposed as inner scale mechanisms for moving contact lines. We have explicitly measured the extent of films near moving contact lines using condensation figures and videomicroscopy. Our results show that for speeds as slow as 3 mm/sec (\( \text{Ca} = 1.5 \times 10^{-4} \)), films of 10 poise PDMS on pyrex extend less than 2 mm, the resolution limit of our present experiments. Thus, thin film mechanisms are not likely to be the appropriate inner scale mechanism for our material system.

We have begun studies of receding contact lines. For 10 poise PDMS on a surface with a grafted, fluorinated surfactant, the interface shape is compatible with the asymptotic hydrodynamic model for \( \text{Ca} < 2.5 \times 10^{-4} \). For \( 2.5 \times 10^{-4} < \text{Ca} < 5 \times 10^{-4} \), the interface shape is not compatible with the model. However, condensation figures and videomicroscopy show no PDMS is left behind on the surface! We have shown that a monolayer of PDMS would be detectable. For \( \text{Ca} > 5 \times 10^{-4} \), a thick film, visible to the naked eye, is pulled from the retreating meniscus.

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**Presentations**


II. MSAD Program Tasks — Flight Research

Discipline: Fluid Physics

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II. MSAD Program Tasks — Flight Research

Geophysical Fluid Flow Cell

Principal Investigator: Dr. John E. Hart
University of Colorado, Boulder

Co-Investigators:

Leslie, F. NASA Marshall Space Flight Center (MSFC)
Miller, T. NASA Marshall Space Flight Center (MSFC)
Toomre, J. University of Colorado, Boulder
Ohlsen, D. University of Colorado, Boulder

Task Objective:
The Geophysical Fluid Flow Cell experiment (GFFC) takes advantage of the unique environment of the microgravity laboratory, which permits forces that would otherwise be swamped by normal terrestrial gravity to become dominant. The GFFC uses electrostatic forces to warp gravity into a radial vector field, centrally directed towards the center of the cell. This allows us to perform visualizations of thermal convection in a spherical shell of liquid subject to imposed differential heating and basic rotation, where the active buoyancy forces are radially directed, as in planetary atmospheres and stars. The objective of the experiments is to categorize the types of convective patterns that can occur in highly nonlinear and turbulent flows with varying amounts of basic rotation and thermal stress.

Task Description:
1. A significant modification to the original GFFC experiment that flew previously on Spacelab-III is the addition of real-time video visualizations of the fluid turbulence that can be downlinked from the shuttle to scientists on the ground. This permits interactive experiments that can identify and focus on important flow regimes. Design, construction and verification of space qualified hardware to effect this video downlink is a major hardware task.

2. In preparation for flight, theoretical, computational, and terrestrial laboratory simulations of rotating convection are required in order to suggest particular experiments that can critically advance our basic understanding of such phenomena, as well as make contributions to the science of geophysical and astrophysical fluid dynamics. Theoretical and computational models typically involve assumptions that will be checked in the Spacelab flights, and terrestrial experiments are planar in geometry, allowing comparison of fundamental differences between "flat" and "spherical" convection.

Task Significance:
The spherical configuration and radial gravity of the Geophysical Fluid Flow Cell is significant because large-scale motions of the atmospheres of planets and stars are constrained by the inherent spherical geometry of these bodies. Furthermore, the flows on them are constrained by rotation, under the action of the same Coriolis forces that shape Earth's weather, and by buoyancy forces which result in hot fluid rising in a radial direction. It is impossible to study such motions in the terrestrial laboratory because gravity is uniformly directed. The GFFC experiments will provide basic laboratory data that can be applied to problems of cloud patterns on the giant planets, differential rotation on the Sun, and motions in Earth's core, mantle and atmosphere.

Progress During FY 1995:
1. The GFFC instrument and video acquisition module were integrated and tested at MSFC. They were subsequently shipped to the Kennedy Space Center for integration into the USML-2 Spacelab payload. Functional tests there were successfully completed.

2. Computational simulations of low voltage GFFC runs were completed using both the Tim Miller and the Gary Glatzmaier codes. These indicated several interesting and surprising flow regimes that will be investigated during
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II. MSAD Program Tasks — Flight Research

USML-2. No pulsating modes were found. However, at low rotation rate, multiple states of convection were seen in the models at modest (5KV) voltages. Such multiple states are important for understanding the predictability and long-time "climates" of slowly rotating spherical shells (like the Earth's mantle).

3. Detailed thermal and velocity measurements in rotating convection were completed in the terrestrial laboratory. These illustrate the surprising presence of a non-adiabatic interior, as compared with non-rotating convection.

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BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals


II. MSAD Program Tasks — Flight Research

**Interfacial Phenomena in Multilayered Fluid Systems**

**Principal Investigator:** Prof. Jean N. Koster  
**University of Colorado, Boulder**

**Co-Investigators:**  
Biringen, Prof. S.  
University of Colorado, Boulder

**Task Objective:**
The main objective of the research is to design a three-liquid-layer flight experiment that will study the interaction of two interfacial tension forces of different magnitude, and their effects on thermocapillary fluid flow induced in adjacent liquid layers. The thermocapillary flow results from temperature gradients parallel to the fluid interfaces. In addition, the mechanical coupling between the immiscible layers and the suppression of convective flow will be investigated. The conditions for the existence for oscillatory flow, and the effects of g-jitter may also be studied.

**Task Description:**
The general approach is to conduct ground-based normal-gravity testing and develop theoretical models of the combined buoyant and thermocapillary convection phenomena. The instrumentation and diagnostics are centered around the physics of interest; namely, flow fields, temperature fields, and interfacial shapes. The theories and numerical models developed and verified with the one-g data were used to predict the anticipated results of the flight experiment for the IML-2 mission which flew July 1994.

**Task Significance:**
The results are expected to significantly advance our knowledge in the area of surface-tension-driven convection in multilayered fluid systems. The scientific results will find applications related to encapsulated float zone processing. Float zone processes are techniques in which space processing of crystals can be done while minimizing the number of imperfections.

**Progress During FY 1995:**
As was previously reported, the IML-2 flight experiment was not successful. Consequently, the analysis of flight data which would have normally occurred during FY 1995 has not taken place. The experiment was considered for a re-flight on BDPU for the LMS mission in June 1996, however, it failed to receive approval from a re-flight review in December 1994. ESA, however, has decided to re-fly the previously flown test container on the LMS mission under the auspices of a European investigator, J. C. Legros of Belgium. Koster has consulted with Legros on the nature of this LMS experiment.

The grant activities, therefore, have continued making progress in the ground-based areas only. The multifaceted ground-based program of Prof. Koster continued to study the many aspects of multilayered flows. The specific areas of study included: natural convection in multiple layers; stability in multiple layers; natural convection in two-phase miscibility gap fluids; free convection of a vertical plate; combined and Rayleigh Benard convection; thermocapillary waves in thin layers; natural convection with density inversion. Much of this work is being done by European students who are supported by their home institutions.

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**Task Initiation:** 11/89  
**Expiration:** 12/95  
**Project Identification:** 963-24-00-05  
**NASA Contract No.:** NAG3-1094  
**Responsible Center:** LeRC
BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals


Theses


II. MSAD Program Tasks — Flight Research

Discipline: Fluid Physics

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Extensional Rheology of Non-Newtonian Materials

Principal Investigator: Prof. Gareth H. McKinley
Harvard University

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
The objective of this work is to determine the extensional viscosity in uniaxial stretching flow for dilute polymer solutions. To generate a simple homogeneous shear free flow in the material while measuring the stress response of the material during this constant strain rate deformation.

Task Description:
This effort will require an initial ground-based experimental design, and ultimate payload implementation, of a novel experimental apparatus to measure accurately the rheological response of non-Newtonian fluids under shear-free conditions that are characteristic of those experienced in the containerless processing of materials.

The proposed instrument generates a homogeneous uniaxial elongation through an exponential stretching of the test sample, and the spatial uniformity of the deformation rate experienced by the fluid is verified by digital particle image velocimetry. Direct measurements of the tensile force exerted by the material then allow calculation of the extensional viscosity of the fluid.

The design of the apparatus utilizes several of the existing or planned fluid diagnostic modules being considered by NASA, and in addition, provides a completely new fluid-science flight capability, which can be used repeatedly to support multi-user rheological measurements for a wide range of non-Newtonian fluids.

Task Significance:
The extensional viscosity is a fundamental physical property of all non-Newtonian materials, and cannot be determined from simple viscometric shear flow experiments. Constitutive equations for viscoelastic fluids such as dilute polymer solutions predict large changes in the extensional viscosity as the elongation rate is increased; however, the validity of these theories cannot be confirmed due to the lack of experimental data obtained in extensional flows. To date, quantitative rheological measurements in shear-free flows have only been possible for highly elastic or "stiff" materials such as polymer melts which can easily be elongated without sagging under a gravitational body force. By performing similar experiments in an extended microgravity environment it will be possible for the first time to obtain accurate measurements of the extensional viscosity for more 'mobile' fluids such as polymer solutions, suspensions and liquid crystalline materials. This rheological data will allow designers of both space- and ground-based material processes to use improved constitutive models in numerical simulations of complex two- and three-dimensional fluid flows.

In a simple stretching flow, fluids such as water or syrup exhibit a resistance to stretching that is exactly three times the value of their Newtonian viscosity. This resistance is usually termed the "extensional viscosity of the material." However, for fluids containing long macromolecules (e.g., synthetic polymers, liquid crystals, or DNA) it is predicted that the extensional viscosity can be anywhere from 100 to 10000 times greater than the viscosity of the fluid. In the near future, NASA plans to develop "containerless processing" operations under microgravity and a detailed understanding of extensional properties in fluids will be absolutely critical -- since, in the absence of container walls, the only way to mix, pour, and shape fluids will be through pure stretching motions. Knowledge of such nonlinear material functions is fundamental to the development and verification of relatively cheap ground-based computational modeling techniques which can be used effectively in the "a priori" design of advanced microgravity material processes thereby alleviating the need for costly in-flight pilot experiments. Finally, in addition to their significance in many industrial processes such as fiber-spinning and film coating operations,
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Discipline: Fluid Physics

stretching flows of non-Newtonian fluids are of fundamental scientific importance to a number of complex fluid dynamics phenomena, including the stability and breakup of jets, enhanced oil recovery and turbulent drag reduction for advanced aircraft, boats, and submarines.

Progress During FY 1995:

The birefringence system has been built and assembled with acceptance testing in progress. Optical measurements of the fluid filament radius and seeded fluid particle velocities have been successfully demonstrated. DPIV software was developed at Harvard University and used for the velocity and velocity gradient computations. A laboratory electronic force balance has been identified and tested. A linear drive unit was designed, built, and used to conduct numerous axial stretching tests of a Boger fluid column. The extensional viscosity, and trouton ratio was computed from the resulting force and radius measurements.

Future plans include further development and construction of the following:

1. The drive mechanism breadboard testing - Inertial motion of neutral density fluid: viscous diffusion of Boger fluid
2. Reducing diameter device (RDD) breadboard testing
3. Load cell and microbalance testing (.01 to 300 grams range required)
4. Sridhar Apparatus testing - using RDD with electronic balance
5. Adiabatic heating - Theoretical estimation
6. Fluid choice - rheological analysis of varying concentrations of polyiobutylene (monodisperse)
7. Lubrication - Analysis of slip on wheels in plateau tank
8. Surface tension - Determine correlation of dynamic surface tension to extensional rheology experiment

STUDENTS FUNDED UNDER RESEARCH:

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McKinley, G.H., "Steady and transient motion of a sphere sedimenting through shear-thinning and constant viscosity elastic fluids." A presentation given to the British Society of Rheology on April 20, 1995.

Pool Boiling Experiment

Principal Investigator: Prof. Herman Merte, Jr. University of Michigan

Co-Investigators: No Co-I's Assigned to this Task

Task Objective:
The program described here seeks to improve the understanding of the fundamental mechanisms that constitute nucleate pool boiling. The vehicle for accomplishing this is an investigation, including experiments to be conducted in microgravity and coupled with appropriate analyses, of the heat transfer and vapor bubble dynamics associated with nucleation, bubble growth or collapse, and subsequent motion. Certain effects that can be neglected at normal Earth gravity, such as surface tension and vapor momentum, can become quite significant in microgravity. Momentum imparted to the liquid by the vapor bubble during growth tends to draw the vapor bubble away from the surface, depending on the rate of growth, which in turn is governed by the temperature distribution in the liquid. Thermophoretic forces, arising from the variation of the liquid-vapor surface tension with temperature, on the other hand, tend to move the vapor bubble toward the region of higher temperature. The bubble motion will be governed by which of these two effects prevail.

The elements of nucleate boiling, for which research conducted under microgravity would advance the basic understanding, are:

1. Nucleation or onset of boiling. Indications are that both heater surface temperature and temperature distribution in the liquid are necessary to describe nucleation.
2. The dynamic growth of a vapor bubble in the vicinity of the heater surface. This includes the shape as well as motion of the liquid-vapor interface as growth is taking place. These are influenced by the liquid temperature distribution at the initiation of growth.
3. The subsequent behavior of the vapor bubble. This includes the motion, whether departure takes place or not, and the associated heat transfer.

Task Description:
In the proposed experiment, a pool of liquid—initially at a precisely defined pressure and temperature—will be subjected to a step-imposed heat flux from a semitransparent thin-film heater forming part of one wall of the container, such that boiling is initiated and maintained for a defined period of time at a constant pressure level. Transient measurements of the heater surface and fluids temperatures near the surface will be made, noting especially the conditions of the boiling process in two simultaneous views, from beneath the heating surface and from the side. The conduct of the experiment and the data acquisition will be completely automated and self-contained.

Task Significance:
The outcome of the experiment is expected to include the following:

1. Observation of the liquid-vapor behavior, including bubble growth and motion as functions of heat flux, initial subcooling and time, and correlation with observed heater surface temperature variation.
2. Use of initial liquid temperature distribution at nucleation to compute vapor bubble growth rate for comparison with observation.
3. Measurement of delay time to nucleation for correlation with nucleation theory.

Anyone who has ever boiled water on a stove is familiar with nucleate pool boiling. Even though it is an everyday event, scientists do not understand precisely how it works, because Earth's gravity influences how bubbles form and grow in boiling liquids.
NASA is interested in results from this experiment, because boiling liquids generates bubbles which are very efficient at transferring large amounts of heat. Finding new ways to dissipate heat from the space shuttle or future manned space platforms will be vital to the success of long term missions.

There are potential benefits closer to home as well, including more effective air conditioning and refrigeration systems and improvements in power plants that could reduce the cost of generating electricity.

Progress During FY 1995:
The prototype hardware for the Pool Boiling Experiment was flown aboard the SL-J mission on September 12–20, 1992. Performance of the hardware was “near perfect.” The data clearly reveal that pool boiling in reduced gravity (10^{-3}g) is a transient process and not a steady periodic one. At the higher-heat flux tests (8 W/cm²), the temperature, as well as the vapor content continued to increase. Tests conducted at the lower-heat flux levels resulted in a rapid spreading of the vapor across the heater as compared to the high-heat flux levels. In low gravity, the vapor bubbles adhered to the heater surface and were 1 cm to 5 cm in diameter. In normal gravity, the vapor bubbles lift off the heater surface due to buoyancy and are approximately 1.5 mm in diameter.

The flight hardware was flown on STS-57 mission in June 1993. Eight of nine test points were successful. The Pool Boiling Experiment was flown again on the STS-60 mission, in February 1994. All nine test points were successful.


The results from these three flights can be summarized as follows:

In microgravity the absence of buoyancy causes large bubbles to form and often remain on the heater surface as surface tension plays a dominant role. Some dryout and rewetting was observed. In normal gravity (1g) convection causes the bubbles to depart from the heater surface while they are quite small.

Correlation of dry out area to mean, h_o and boiling, h_w (wetted) heat transfer coefficients and mean heater surface temperature, T_w, for microgravity pool boiling tests is the first of its kind.

Professor Merte successfully passed a Microgravity Hardware Reflight Reviews held on June 24, 1994 and his proposed experiment, "Study of Pool Boiling in Microgravity-Rewetting Following Dryout," was approved by NASA. Two flights are planned using the same pool boiling hardware with a few modest changes. There will be higher subcoolings (20, 30 and 40°F) in the first reflight and lower heat fluxes (0.5/and 2 w/cm²) in the second. The first mission is to be launched on STS-72 in January 1996.

### BIBLIOGRAPHIC CITATIONS FOR FY 1995:

**Journals**


**Presentations**
II. MSAD Program Tasks — Flight Research

Discipline: Fluid Physics

Surface Tension-Driven Convection Experiment (STDCE-1, STDCE-2)

Principal Investigator: Prof. Simon Ostrach  Case Western Reserve University

Co-Investigators:

Kamotani, Prof. Y.  Case Western Reserve University

Task Objective:

The objective of this research is to further the understanding of the physical mechanisms associated with non-oscillatory (STDCE-1) and oscillatory (STDCE-2) thermocapillary flow by (a) developing an accurate description of the physical mechanisms, (b) developing an accurate numerical model, and (c) obtaining ground-based and flight experimental data to verify the physical mechanisms and the numerical model. The thermocapillary flows result from the fluid motions generated by the surface-tractive force that is caused by surface-tension variation due to the temperature gradient along the free surface.

Task Description:

STDCE-1: The basis of Surface-Tension-Driven Convection Experiment 1 (STDCE-1) flight experiment is a copper test cell 10 cm in diameter and 5 cm deep, filled with silicone oil, able to provide both flat and curved free surfaces in a microgravity environment. The outer wall of the test cell is water cooled. The silicone oil can be centrally heated either externally by a carbon dioxide laser (constant heat flux, CF) or internally by an immersion heater (constant temperature, CT). The cross section is illuminated by a 1-mm-thick sheet of light, which scatters from small aluminum oxide particles mixed into the oil, allowing observation and measurement, using a particle-tracking technique, of the axisymmetric flow velocity. An infrared imager is used to measure surface temperature, and thermistors are used to measure fluid and wall temperature. The velocity and temperature measurements are compared with the numerical predictions.

STDCE-2: The center of Surface-Tension-Driven Convection Experiment 2 (STDCE-2) is an interchangeable module containing a test cell and fluid reservoir. Six modules containing copper test cells of 1.2, 2.0 and 3.0 cm diameter, each with the depth equal to the radius, will be filled with 2 centistoke silicone oil, to provide both flat and curved free surfaces in a microgravity environment. In three of the modules, one of each size, the fluid will be heated by a carbon dioxide laser, imposing a Gaussian heat flux on the free surface, and in the remaining three the fluid will be heated internally by an axially located heater which is ten percent of the chamber diameter. The outer walls of the test chambers will be cooled. This modular approach was taken to accommodate the large test matrix.

During the experiment, the surface temperature—which is the driving force in the flow—is measured non-intrusively by an infrared imager. The free-surface deformation, felt to play a critical role in the oscillation phenomenon, is measured quantitatively using a Ronchi deflectometer. The flow field is observed by illuminating the entire test chamber volume with laser light which is scattered from 20 micron aluminum oxide particles mixed in the fluid, allowing for three-dimensional qualitative visualization. Thermistors are used to measure bulk fluid, wall and heater temperatures.

At the start of each test the heater power will be slowly increased until the flow transitions from steady and axisymmetric to periodic and three dimensional. This will be performed for 43 combinations of test chamber size, heating mode and free surface shape. The temperature difference at the transition point will be used to calculate non-dimensional parameters which are used to characterize the onset of oscillations. The flow field, surface and bulk temperature distributions, and the free surface deformations will be correlated to support verification of the proposed physical mechanism for the oscillatory phenomenon.
II. MSAD Program Tasks — Flight Research

Discipline: Fluid Physics

Task Significance:
The Surface Tension Driven Convection Experiment is designed to study the nature and extent of steady and oscillatory thermocapillary flows using state of the art diagnostics to measure and characterize these flows over a wide range of parameters. Valuable data, which can only be gained from low gravity-based experimentation, will be obtained resulting in an understanding of the fundamental physical mechanisms and improved implementation of related industrial processes such as life support systems, containerless processing of materials, crystal growth, propellant storage management, and bio-fluids engineering both in space and on earth.

Progress During FY 1995:

STDCE-1

STDCE-1 was flown on USML-1 in June, 1992. 38 tests were completed returning over 12 1/2 hours of data. To date approximately 75% of the data has been analyzed and compared to the numerical model. The comparisons show good agreement. This represents the relevant data from the experiment. The analysis of the data has been broken into sections and numerous presentations of those sections of the reduced flight data have been made. These include flow data from the 1hr CT and CF tests, temperature data from the 1hr tests, flow data from the shorter CT flat surface tests, and flow data from the shorter CF curved surface tests. All of the reduced data has been compiled by a graduate student for the final contract report. In addition, no flow oscillations were observed in any of the tests corroborating the Principal Investigator's theory that the Marangoni number alone is not sufficient to indicate the onset of oscillatory flow.

STDCE-2

Flight Experiment:

STDCE-2 is scheduled to fly on USML-2 in September of 1995. In preparation, the PI and Co-I participated in 2 USML-2 Investigator Working Group meetings at the Marshall Space Flight Center in addition to numerous meetings and reviews at the Lewis Research Center. The PI/STDCE operations team have participated in pre mission simulations and are currently awaiting the launch of USML-2.

Ground-based work:

1. Analysis

A scaling analysis of axisymmetric thermocapillary flows was conducted. Various important velocity and length scales were determined. The scaling laws were shown to agree well with the results of a numerical analysis. Based on the scaling analysis a surface deformation parameter (S-parameter) was derived for each CT and CF heating mode. Both the Marangoni number and the S-parameter must be larger than certain values in order to obtain oscillatory thermocapillary flow. The work has been written up as a Ph.D. thesis.

2. Experimental Work

The deformation of the free surface has been measured in oscillatory thermocapillary flow. The free surface motion was measured by observing the surface in one radial cross-section through a micro-video system. An organized free surface motion was found. The frequency of the free surface oscillations was the same as that of the temperature oscillations detected by a thermocouple probe in the fluid. Both oscillation patterns were closely related.

An optical system is being set up to measure the free surface motion over the entire free surface instead of only sections. This optical measurement will provide us with information regarding a wave-like motion of the free surface. The information is important in establishing a coupling among the surface motion and the rotating temperature oscillation pattern.

An experiment on oscillatory thermocapillary flow in cylindrical containers is being performed. The objective is to confirm the S-parameters obtained by analysis. In order to do so the effect of buoyancy on oscillatory thermocapillary flow must be understood. The buoyancy effect is being investigated numerically and experimentally.
II. MSAD Program Tasks — Flight Research

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Project Identification: 963-25-0D-09  NASA Contract No.: NAG3-1568  Responsible Center: LeRC

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

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Presentations


II. MSAD Program Tasks — Flight Research

Modeling and New Experiment Definition for the VIBES

Principal Investigator: Prof. Robert L. Sani

University of Colorado, Boulder

Co-Investigators:

Azuma, Dr. H.
Doi, Dr. T.
Kamei, Dr. S.
Ohnish, Dr. M.
Kida, Dr. T.
Yamamoto, Dr. K.

Japanese National Aerospace Laboratory (NAL)
NSDA — Japanese Space Agency
Mitsubishi Research Institute
Japanese National Aerospace Laboratory (NAL)
Japanese National Aerospace Laboratory (NAL)
Japanese National Aerospace Laboratory (NAL)

Task Objective:

The Vibration Isolation Box Experiment System (VIBES) is an IML-2 flight experiment designed by the Japanese National Aerospace Laboratory. Its primary goal is to evaluate the performance of a vibration isolation device in conjunction with typical fluids experiments. The IML-2 flight experiment contains two experimental units: the Convection Diffusion Unit (CDU) and the Thermal-Driven Flow Unit. The purposes of the CDU experiment (the one of interest herein) are to observe natural convection and diffusive transport in a micro-g environment and to observe the effect of g-jitter with and without the vibration isolation due to the vibration isolation box. The objective of this project is to provide numerical modeling for the CDU experiment for aiding in design refinements and evaluation of terrestrial benchmark experiments as well as post-flight evaluation of the experimental data.

Task Description:

The numerical modeling will utilize a Galerkin finite element algorithm for the linear momentum, energy and species balance equations using the Boussinesq approximation. This project will make comparisons of two codes (PI's research code and a commercial code, FIDAP) in a transient, 3-D calculation to determine their efficiency and accuracy. Timing comparisons will also be made between FIDAP and the research code. The numerical experiments will include example cases with and without the test cell being subjected to g variation; both single and multiple frequency variations will be considered. The numerical experiment will also consist of simulating the g-environment (to be provided by the Japanese research team) both inside and outside the isolation box. Comparison of these results should allow a quantitative assessment of the isolation capability of the apparatus.

Task Significance:

The microgravity environment available for space experiments is not quiescent but is subjected to significant background vibrations generated by aerodynamic and machinery vibrations, crew motion, etc. Such g-jitter can be relatively random in orientation and can attain significant magnitudes. There is a growing list of observations and data analyses that demonstrate the existence of significant g-jitter episodes and their potential for having very deleterious effects on many proposed flight experiments. A potential solution to this problem in the micro-g environment is the use of vibration isolation for the experiments which require it. The assessment of such an apparatus is one of the main thrusts of the research proposed in this project.

Progress During FY 1995:

1. Continued development, testing and benchmarking of the semi-consistent mass finite element projection algorithm for 2-D and 3-D transient Boussinesq flow.
   a. The modification of the algorithm to include the option of a skew-symmetric form of the advection operator and concomitantly the implementation and tuning of a bi-conjugate gradient stabilized routine has continued.
   b. The implementation of a variable time step capability has recently been finished and currently various error indicators are being investigated.
II. MSAD Program Tasks — Flight Research

Discipline: Fluid Physics

c. The inclusion of the capability to model additional fields, for example, concentration, is being addressed. Its eventual complementation will be an essential tool in the definition of potential new flight experiments behavior of coupled fluids in a g-jitter environment.

2. Time-varying gravity Boussinesq flow simulations.
   a. Simulations of ground based experiments done by Japanese research team have been compared with experimental observations.
   b. The data analysis of the gravitational data from the VIBES experiment was received from NASA LeRC and simulations of the VIBES experiment utilizing a more realistic gravitational field temporal signature, obtained from the data analysis, has been initiated.
II. MSAD Program Tasks — Flight Research

Discipline: Fluid Physics

Studies in Electrohydrodynamics

Principal Investigator: Dr. Dudley A. Saville
Princeton University

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
This research is designed to strengthen and test the experimental foundations of the theory of electrohydrodynamics. Electrohydrodynamic forces can be used to manipulate fluids, especially fluid interfaces. As such, they offer a means of controlling fluid motion on very small length scales.

Task Description:
Theoretical studies will center on adapting the existing leaky dielectric theory for the stability of a fluid cylinder to account for pinning the contact line at the upper and lower boundaries in a liquid bridge configuration. Experimental studies will be carried out to (1) evaluate the influence of ionic surfactants on conductivity so as to enable us to control the time scale of the electrohydrodynamic fluid motion and (2) test the existing electrohydrodynamic theory for the stability of a cylinder subject to an axial field with isopycnic systems.

Task Significance:
Despite substantial efforts over the past two decades, the foundation of electrohydrodynamics is weak. Relatively few experiments have been done to test the leaky dielectric theory, the most promising model of behavior, and much of the work has been of limited scope because of the need to use isopycnic systems to avoid sedimentation and hydrostatic pressure effects. This restricts the range of fluid properties that can be studied and as a result there are many gaps in our knowledge. In addition to its scientific importance, there are a wide range of applications where electrohydrodynamic phenomena play important roles.

Progress During FY 1995:
In December 1994, a “Science Concepts Review” was completed and we began preparations for a flight experiment. The experiment will be part of the LMS mission scheduled for June 1996. The experiment itself will test the leaky, dielectric model of electrohydrodynamics by studying the behavior of liquid bridges in the presence of strong electric fields. Our experiment will be conducted in the BDPU facility which is part of the European Space Agency’s activities. Since December we have designed and built apparatus to test various concepts during parabolic flights on a KC-135. Two flights were completed and another is scheduled. Flight apparatus has also been designed and tested in a “breadboard” configuration. This whole activity is, in some sense, a “test case” to see if laboratory science on board the Space Shuttle can be carried out within a short time period. In our case, only eighteen months will elapse from the Science Concepts Review to completion of the microgravity experiment.

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BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Presentations

Printed: 28 FEB 1996
II. MSAD Program Tasks — Flight Research

Discipline: Fluid Physics

Mechanics of Granular Materials

Principal Investigator: Dr. Stein Sture
University of Colorado, Boulder

Co-Investigators:
Costes, N. NASA Marshall Space Flight Center (MSFC)

Task Objective:
The objective of this research is to gain a quantitative understanding of the mechanical behavior of cohesionless granular materials under very low effective confining pressures.

Task Description:
Ground-based displacement-controlled triaxial experiments are conducted on a cohesionless granular material at the lowest effective confining pressures possible, that do not result in material instability, to assess constitutive properties, stability phenomena, and control parameters that will be applied to in-space experiments on 75 mm (diameter) and 150 mm (length) right cylindrical specimens. The ground-based tests on similar-sized specimens are conducted in the range 3.5-69 kPa, while the microgravity tests will be conducted at effective confinement levels in the range 0.05-1.30 kPa.

The displacement-controlled mode of loading confined specimens was chosen mainly to maintain overall specimen-apparatus stability while strain-softening resulting from continuous or discontinuous bifurcation and discontinuous deformation fields are allowed to take place. Optical and other noncontacting displacement-sensing techniques are used to measure specimen response during experimentation. Prescribed displacements are transmitted in terms of loading, unloading, and reloading histories, while volume change is measured in "drained" tests, and pore fluid pressure is measured in "undrained" isochoric tests. Confinement pressure is transmitted to the granular material assembly through a thin flexible latex membrane surrounding the specimen. A subangular and uniform Ottawa quartz sand constitutes the specimen.

Specimens tested both in space and on ground will be subjected to nondestructive and destructive (thin-slicing) testing to assess degrees of material uniformity and isotropy before and after experimentation. It appears that instability phenomenon associated with specimens of certain configurations result in curved internal surfaces of localized deformation and high rates of dilatancy, whose structure depends on bifurcation mode.

Task Significance:
Specifically, the purpose is to study the influence of particle interlocking and other fabric properties on the strength criterion near the effective stress space origin, i.e., can it be represented by a straight-line envelope passing through the origin or does it have a curved shape with shear (cohesion or interlocking) or tensile strength intercepts. The experiment will determine whether cohesionless granular materials under very low effective confining pressures/effective stresses tend to dilate or contract regardless of their initial state of compaction, and whether their mechanical behavior under relatively large displacement or quasi-static cyclic loading is according to conventional constitutive theory. In addition, bifurcation and material instability phenomena resulting in formation of shear bands, before and after peak strengths have been reached, will be studied. Based on terrestrial experiments and theory, it has been found that critical hardening, strain-softening behavior, and shear band orientation are dependent on confining stress.
II. MSAD Program Tasks — Flight Research  

Discipline: Fluid Physics

Progress During FY 1995:
The progress in the MGM project during FY95 includes the following:

1. Completion and testing of the MGM apparatus;
2. Improved analysis of tested specimens;
3. Training of payload/mission specialists;
4. Improved constitutive models;
5. Improved analysis of specimen bifurcation instability conditions; and,
6. Improved optical-imaging and measurement systems.

STUDENTS FUNDED UNDER RESEARCH:

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TASK INITIATION: 12/92  EXPIRATION: 9/97

PROJECT IDENTIFICATION: 963-55-05

NASA CONTRACT NO.: NAS8-38779

RESPONSIBLE CENTER: MSFC

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals


Proceedings


Presentations

II. MSAD Program Tasks — Flight Research

Discipline: Fluid Physics

Thermocapillary Migration and Interactions of Bubbles and Drops

Principal Investigator: Prof. R. S. Subramanian
Clarkson University

Co-Investigators:
Balasubramanian, Dr. R.
Wozniak, Dr. G.

NASA Lewis Research Center (LeRC)
Tu Bergakademie Freiberg (Germany)

Task Objective:
The objectives of the research are to experimentally measure, both in one-g and low-g, the thermocapillary migration velocities and the shapes of single and interacting gas bubbles, and liquid drops in a continuous phase under the action of an applied temperature gradient. Comparisons between the observed velocities and shapes with those that are predicted from theory will be made. The low-g results will bear data that will be free from the effects of buoyant convection in the matrix liquid as well as buoyancy effects on the bubbles/drops.

Task Description:
The general approach has been to conduct ground-based normal-gravity testing and to develop theoretical models of the thermocapillary migration phenomena. The instrumentation and diagnostics are centered on the physics of interest, namely, flow fields, temperature fields, and bubble/droplet velocities and shapes. The theories and numerical models developed and verified with the one-g data will be used to design and predict the results of the flight experiments. Assessment of flight IML-2 data was done in FY95. In addition, LMS preparatory activities continued during FY95.

Task Significance:
The results from these bubble migration experiments are not only expected to advance our knowledge in the area of surface tension driven motion, but are, in addition, relevant to several applications with respect to space processing of materials. Some examples of the latter include solidification, glass processing, and composite preparation. The physics studied in the experiments offer a method by which undesirable void formation in metals and composites can be avoided.

Progress During FY 1995:
Efforts in FY 1995 concentrated on analysis of the flight data from their successful IML-2 flight experiment. Video and film images have been analyzed for bubble and droplet speeds. Results show good agreement with theoretical predictions in the case of bubbles. Analyses are continuing for the drop cases for isolated objects. Results show good qualitative agreement and some quantitative discrepancies with theoretical predictions where available. Results for an interesting pair of drops show that a small leading drop, which appears unaffected in its motion, can significantly reduce the motion of a trailing drop in its vicinity that is almost twice as large.

The investigators were made aware of plans to re-fly BDPU in the LMS mission. They successfully presented their re-flight objectives and a preliminary test matrix to a science review panel in December 1994. The emphasis for the LMS mission will be studying bubble/bubble and drop/drop interactions.

Recommendations to the European Space Agency (ESA) had been made for the implementation of a shearing interferometric system in the BDPU. A dual purpose composite prism with two angles (to accommodate the differing requirements of the Subramanian and Straub experiments) was recommended. ESA has accommodated this request. The selection of tracer particles to be used in the test cells is nearly complete. These particles will be mixed with the host fluid to enable the visualization of the flow fields around the bubble. Normal activities have progressed in support of the LMS mission. This includes feedback to ESA regarding the definition of functional objectives for the LMS experiments; updating application software; and attending IWG meetings.

Printed: 28 FEB 1996
II. MSAD Program Tasks — Flight Research

Discipline: Fluid Physics

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BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals

Presentations


Drop Dynamics Investigation

Principal Investigator: Prof. Taylor G. Wang

Co-Investigators:

Lee, C.P.  
Anilkumar, A.V.  
Hmelo, A.B.  
Hussein, H.J.

Task Objective:

The objective of this program is to understand the behavior of free liquid drops, primarily by studying them in a microgravity environment. The Drop Physics Module (DPM) operated in the Space Shuttle provides an opportunity to address outstanding fluid-dynamics issues of rotating and oscillating simple and compound drops. To maximize the return from this short on-orbit opportunity, ground-based experiments will be performed to verify concepts and experimental techniques, and modeling will be done to select the parameters for the DPM experiments.

This investigation will use a triple-axis acoustic positioning chamber to study the static shape and the dynamics of simple and compound drops. Equilibrium shapes and the stability of rotating and nonrotating drops, their associated internal flow patterns, and the centering force associated with shape oscillations of compound drops—will be the principal scientific areas of interest.

Task Description:

A variety of experiments will be performed in space. Compound drops and liquid shells will be formed to study their oscillation modes and the effectiveness of those modes in centering the core. The interaction between the acoustic field and the drops will be studied: the drops' static shape, the stability of distorted shapes, and the generation of any flows in the drops. The dynamics and stability of rotating drops near the point of fission will be explored.

Task Significance:

This investigation uses the low gravity provided by the Space Transportation System, the working laboratory of Spacelab, and the Drop Physics Module hardware to study large drops. Studying drops of size 1-3 cm in diameter shows dynamic phenomena to time scales, which can be observed by the experiment operator (video) as well can be captured on high-speed film.

Progress During FY 1995:

Due to the late arrival of the DPM video data, the reduction and analysis of most of the USML-1 data was performed in FY 94; three papers have been prepared using this data and have been submitted for publication. Support to the DPM Project in the area of understanding tumble rotation progressed from flow visualization experiments at Vanderbilt to preparations for torque measurements on the flight system at KSC.

One of the primary objectives of USML-1 was to perform careful experiments on rotating drops. The DDM results from Spacelab-3 deviated from analytical and numerical predictions. The USML-1 results showed that flattening due to acoustic forces would cause experimental measurements to deviate from theoretical predictions that assume a spherical geometry; when the drops are spherical, there is no discrepancy. The results have appeared in the Journal of Fluid Mechanics (Oct. 94).

Another set of USML-1 experiments studied the tendency of compound drops to become concentric under the influence of capillary oscillations. A compound drop is composed of two relatively immiscible fluids one totally
II. MSAD Program Tasks — Flight Research  

Discipline: Fluid Physics

 contained within the second fluid drop. Studies of both bubbles and liquid-liquid compound drops showed that the core moved to the center after several periods of oscillation and remained concentric. These results which can not be explained by existing inviscid models were published in the Journal of Colloid and Interface Science.

A third paper has been generated from USML-1 sequences of drop oscillation and natural decay. The experimental results were obtained using rotating and non-rotating drops with small but finite viscosities. The latter data agree with current theory while the flattening inherent in a rotating drop caused deviation from the simple models. This work has been accepted for publication in the Journal of Fluid Mechanics.

A major reason that the science return from USML-1 was less than expected was the appearance of an uncontrollable rotation. In support of the DPM Project a team at Vanderbilt has been studying how the air moves inside the DPM chamber under various combinations of acoustic signals. Flows were observed to be due to both to the quartz wind from the individuals drivers as well as to the interaction of DPM's unique signals. The strength of the former is larger and correlates with the weak DPM driver on USML-1. Characterization measurements of the torque on a suspended ball in DPM-like chambers at Vanderbilt, the trainer at Marshall SFC, and the flight system at KSC are being carried out.

**STUDENTS FUNDED UNDER RESEARCH:**

| BS Students: | 0 | BS Degrees: | 0 |
| MS Students: | 3 | MS Degrees: | 2 |
| PhD Students: | 1 | PhD Degrees: | 0 |

**TASK INITIATION:** 8/92  **EXPIRATION:** 7/96  

**PROJECT IDENTIFICATION:** 963-24-04-01  

**RESPONSIBLE CENTER:** JPL

**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Journals**


Physics of Colloid in Space

Principal Investigator: Dr. David A. Weitz

Co-Investigators:
Pusey, Prof. P.N.

Task Objective:
This experiment entails the study of the physics of colloidal particles in microgravity. It consists of two distinct parts. The first deals with ordered structures while the second deals with highly disordered structures. The study of ordered structures entails the growth of colloidal superlattices formed with mixtures of different-sized particles. The goal is to develop useful periodic structures using colloidal particles as precursors, through "colloid engineering." The study of the highly disordered structures entails the formation of fractal colloidal aggregates of much greater extent than has ever been done, and the formation of very weak structures that would collapse under their own weight in normal gravity.

Task Description:
The work within this effort is ground-based research to study the formation of novel materials from colloidal dispersions, and to study the physical properties of such materials. As part of the effort, space experiments to be carried out in a space shuttle middeck carrier, will be defined. These experiments will utilize the laser light scattering apparatus currently being developed at NASA LeRC.

The focuses of the ground-based experiments are the study of colloidal superlattices formed from mixtures of different-sized colloidal particles, the in-depth study of the formation of fractal colloidal aggregates. While considerable knowledge exists about the formation or growth of fractal colloidal aggregates, much less is known about the unique properties of these objects and the consequences of their scale invariance. A major reason for this is the relatively small scale over which the aggregates exhibit scale invariant behavior. By growing structures that are scale invariant over a much greater range of length scales, the properties of these objects can be studied much more directly. This will provide the first detailed information about the consequences of scale invariant structure on the properties of these materials.

Task Significance:
Very little is currently known about the structures of binary colloidal crystals, and these experiments will initially be directed at determining the phase diagrams of the superlattices for mixtures of different sizes of particles. In addition, virtually nothing is currently known about the kinetics of the formation of these superlattices, and about their dynamics once they are formed. This will also be studied by these experiments. This will represent the first in-depth study of the growth and properties of colloidal superlattices.

Progress During FY 1995:
This project successfully completed the Science Concept Review (SCR). The final draft of the Science Requirements Document (SRD) was completed. It was proposed that PCS be supported by two Mir Space Station Glovebox missions. The first mission, the experiments on superlattice crystal formation in bidisperse colloid mixtures and gelation of colloid plus polymer mixtures, is tentatively scheduled for the August 1996 Mir flight. These experiments require PMMA colloidal particles. Initial difficulties with their synthesis have now mostly been overcome, although some problems remain in the preparation of highly monodisperse small particles. Enough monodisperse material was prepared to supply training and flight samples for the first proposed mission. A ground-based experiment was begun to see if superlattice structures form in zero-gravity as simulated by slow tumbling. A well developed theoretical understanding was attained for the behavior of density-matched polystyrene (PS) gels, as it has been observed in all experiments made to date. It is now possible to measure the elasticity of gels by an entirely optical technique. Development continued of the PCS in-software correlator, necessary for
determining the behavior of PS gels in a regime currently inaccessible to measurements. More understanding was reached on the phase diagram and gelation kinetics of colloid plus polymer mixtures. With this system it will be possible to determine the consequence for gels of a flexible bond between particles, rather than the rigid bond of PS gels, but since the colloid plus polymer mixture system cannot be density-matched, a zero-gravity environment is essential for experiments. Training and flight samples were prepared for the first Glovebox flight. Emulsion droplets are likely to provide one of the components of future heterogeneous systems. Therefore, much work was done in order to understand the light-scattering, rheological, and crystal forming behavior of oil droplet emulsions. New techniques were developed for the emulsification of liquid crystals. These techniques may eventually be applied in the assembly of novel order materials in zero gravity.

**STUDENTS FUNDED UNDER RESEARCH:**

| Students |  
| BS Students: | 0  
| MS Students: | 0  
| PhD Students: | 2  

**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Journals**


**Presentations**


Weitz, David A., "Elasticity of emulsions, or how solid sludge slips." International Meeting on Complex Fluids and Monte Carlo Methods, July 24-August 5, 1995.
II. MSAD Program Tasks — Flight Research

Discipline: Low Temperature Microgravity Physics

Critical Dynamics in Microgravity

Principal Investigator: Dr. Robert V. Duncan
Sandia National Labs, and Univ. of New Mexico

Co-Investigators:
Chui, T. C.P.
Israelsson, Dr. U.E.
Jet Propulsion Laboratory (JPL)
Jet Propulsion Laboratory (JPL)

Task Objective:
The objectives of the Critical Dynamics in Microgravity Experiment are as follows:
1. Measure the dependence of the normal state thermal conductivity ($\lambda$) on the heat flux $Q$, and on the proximity of the cell endplate:
   - Nonlinear transport very close to $T_c$;
   - $Q$ sets distance from criticality, as predicted by the Dynamic Renormalization Group Theory (DRGT);
   - Explore boundary suppression of fluctuations near criticality;
   - $0.3 \text{ nW/cm}^2 < Q < 10 \mu\text{W/cm}^2$ with no convection in a 2 cm cell.

2. Measure the temperature profile very near, and through, the He I-He II interface:
   - Measure $\Delta T_x(Q)$ and explore "supercooled" He I region as a function of small temperature and pressure changes
   - Determine the boundary conditions on $j$, and possibly $\varphi$;
   - Determine if the interfacial width ($w$) scales as predicted by DRGT and Dynamic Scaling: $w = w_{J}/\sqrt{Q}$;
   - Measure the superfluid $\Delta T$, hence He II vorticity.

3. Search for hysteresis in the superfluid transition under heat flux:
   - Up/down reproducibility;
   - Latent heat.

Task Description:
A high-resolution, all-aluminum thermal conductivity cell containing a sample of ultra-high purity helium will be employed for these measurements. The high resolution thermometers (HRT) developed for and used successfully on the Lambda Point Experiment will also be used on DYNAMX to measure temperature gradients when a small heat current is applied.

Task Significance:
The improvements to the HRT's developed for the CHEX mission will also be applied to the DYNAMX HRT's. The all-aluminum construction will reduce the mass of the sample cell to reduce the degradation of the measurement resolution caused by ionizing radiation in orbit.

Progress During FY 1995:
Critical Dynamics in Microgravity (DYNAMX) has reached a singular time in its scientific progression at the end of FY 1995. The scientific concept for the DYNAMX experiment in microgravity is now mature, and the Prototype Cryoprobe for DYNAMX is now complete and fully operational at the University of New Mexico. The Prototype Cryoprobe is the first in three phases of the flight instrument development. The next phase, namely the Engineering Model Cryoprobe, shall be constructed in FY 1996, and the final Flight Hardware shall be built beginning in FY 1997. The basic scientific premise motivating the Shuttle flight of DYNAMX has been clearly...
established through the combined effort of the Science Team at Sandia, JPL, and UNM. Only a few aspects of the detailed science requirements for DYNAMX have yet to be firmly established, such as the need for vibration isolation in the microgravity environment. As detailed below, these few remaining issues shall be clarified during FY 1996, permitting the final Scientific Requirements Document to be completed by the end of FY 1996. Both the Preliminary Implementation Plan review and the Scientific Concept Review shall be conducted within the next month. Successful passage of both reviews shall permit DYNAMX to enter "phase B" and to progress towards the requirements Definition Review (RDR) to be held at the end of FY 1996.

The construction of the ground-based prototype of the DYNAMX instrument has been completed at the University of New Mexico (UNM). It has been used to measure the thermal profile in the liquid helium sample as the superfluid - normal fluid (HeI - HeII) interface moves past a sidewall thermometry platform located 0.25 mm from the warm end of the cylindrical thermal conductivity cell. These thermal profiles have been measured at heat flux (Q) values ranging from 20nW/cm² to 240nW/cm² with high resolution thermometry (HRT) capable of sub-nanokelvin resolution in a one-hertz bandwidth. With the cell entirely within its superfluid phase, the HRT located on the 0.25 mm thermometry stage tracked the temperature of the HRT located on the 14 mm stage to within 0.3%, demonstrating the isothermal nature of the superfluid over this entire range of heat flux Q. These experimental results detected no thermal gradient within the superfluid over this range in Q, consistent with previous experimental work. Once the HeI-HeII interface was located at about 1 to 2 mm above the bottom of the cell, the 0.25 mm stage HRT tracked the normal fluid temperature profile as the superfluid temperature read on the 14 mm stage was increased by a servo at the rate of 2.5nK/s. The resulting normal fluids gradients, all outside of the nonlinear region, agreed well with predictions based on other Earth-based thermal conductivity data taken further from the transition, validating our experimental ability to obtain adequate thermal control to make these highly sensitive measurements. The region of greatest interest, namely the region where the interface position was within about 0.2 mm of the 0.25 mm stage, was heavily rounded by cell design effects. Although some information may be obtained within this region through advanced data analysis techniques, modifications to our original cell design should permit us to much more clearly detect and study the non-linear transport region. These cell design improvements are discussed below.

Our original cell design consisted of ultra-pure (99.999% pure) aluminum sidewall thermometry stages welded at a constant height from bottom plate of a cell all around the circumference of the cylindrical cell. The end plates were made of the 99.999% pure aluminum, while the sidewalls were made of the AL5456 alloy, which was thought to have an extremely low thermal conductivity (<10 mW/cmK) in stark contrast to the extremely high conductivity of the ultra-pure aluminum (>20 W/cmK). Unfortunately, the thermal conductivity of this sidewall alloy was measured to be 20 mW/cmK, which was unacceptably high for making measurements in the non-linear region. On close examination of the electron beam welds of the high conductivity aluminum sidewall stages to the AL5456 sidewalls it became apparent that some small separation occurred. Foiling four thermal cycles the end-cap welds, which were hermetic against superfluid leaks initially, developed microcracks which ruined the cell. These complications have led us to develop a new cell design and construction technique which shall be evaluated experimentally in the engineering model cell during FY 1996.

Our new cell design shall consist of alumina-impregnated composite sidewalls with either ultra-high purity copper or aluminum endplates and sidewall stages. This cylindrical sidewall made of the alumina composite has a measured thermal conductivity of 0.1 mW/cmK at 2K, which is less than the background thermal conductivity of normal fluid well away from the superfluid transition. Hence, the sidewall heat flux will not create a substantial radial heat flux near the interface, permitting the sharp onset of thermal resistance at the interface to be clearly observed. Adhesives have been used to seal the cell against superfluid heat leaks successfully, and to provide good anchors of the sidewall stages to the sidewall. Comprehensive two-dimensional thermal models have verified that these alumina-composite sidewalls will round the transition by no more than 1.5 nK on Earth, corresponding to a special resolution of about 8μm at the thermometry stages. A flight-like cell shall be built at JPL and integrated into the prototype cryoprobe at UNM for science verification of these anticipated thermal properties by the end of March, 1996. Once complete, this flight-like cell shall be integrated into the "critical thermal path" and again tested at UNM. The "critical thermal path" shall consist of the cell, bubble pressure regulator, cryogenic valve, burst rings, and the HRTs. The "critical thermal path" shall then be integrated into the Engineering Model cryoprobe, which will complete the second generation of the DYNAMX hardware built.
The measurement scenario for DYNAMX while on Earth-orbit has been defined. The DYNAMX cell shall be cooled through a weak thermal conductance of about 7,000 K/W by a platform, called "stage 4," which will be located above the isothermal shield which surrounds the critical thermal path. By setting the stage 4 temperature with a precision of 10 nK to a temperature of a few mK below the cell temperature, a precisely known amount of power will be removed from the cell. The time constant for the thermal relaxation between the cell and stage 4 shall be about 20,000 seconds, while a typical measurement shall require only 300 seconds to complete. Hence, the power out of the cold end of the cell (Q) shall be approximately constant and very well known throughout the measurements. A heat current of 1.02Q shall be applied to the opposite, hot end. Hence, a total of 0.02Q rate of heating shall be available to build up the enthalpy in the normal fluid region once the normal fluid interface forms out of the superfluid phase in the cell. Even if the heat flux does not stabilize the superfluid - normal fluid interface (as predicted by A. Onuki), this thermal design shall ensure that the rate of the interface advance will be well controlled. Through one-dimensional simulations we have determined that at 5mm from the hot end of the cell the rate of interface advance shall be optimal for capturing the nonlinear thermal profile near the interface in quasi-static conditions. We shall use heat flux values ranging from 5nW/cm² to 70 nW/cm², resulting in interface advance rates which will increase with decreasing Q and which are typically a few microns per second. This range of Q was chosen to observe the nonlinear thermal conductivity region near the superfluid transition over the range where such measurements would be difficult and/or impossible on Earth.

In addition to the nonlinear thermal conductivity measurements, the DYNAMX experiment shall measure other nonlinear effects near the superfluid transition which can not be observed on Earth. The temperature drift rate in the superfluid phase, while a known rate of heat is being extracted from the cell, will result in an accurate measurement of the superfluid heat capacity while the superfluid transports a heat flux Q. This heat capacity is expected to become strongly dependent on Q very close to the superfluid transition. Once in the interfacial region, the captured thermal profile shall display how the interfacial width varies with Q. This anticipated scaling of the interfacial width has been predicted to be Q⁻⁴, where x has been calculated at approximately 0.5 by two different theories. Finally, qualitative questions concerning the superfluid transition under a heat flux which have not been observed on Earth, such as the possible existence of hysteresis, and heat-flux stabilization of the interface, shall be examined in the microgravity conditions on-orbit.

Some open scientific questions remain unsettled at this time. Measurements at UCSB have indicated that an unexplained thermal resistance appears in the superfluid phase over a temperature range which scales as Q⁻⁴⁵, while the nonlinear thermal conductivity region is anticipated to scale as Q⁻⁷⁵. Hence this unknown dissipative phase should not create a major contribution to the dynamics of the dissipative phase at very small Q, as has been observed experimentally down to Q=0.45μW/cm². While measurements at UNM have shown no detectable thermal resistance in the superfluid phase, the nature of the onset of thermal resistance once the superfluid phase breaks down. These pre-transition dissipative effects possible result from vorticity in the superfluid, and further ground-based investigations should be conducted to better understand the effects of superfluid vorticity on the superfluid transition. Richard Packard, Talso Chui, and David Goodstein have all been considering such effects near the superfluid transition.

The cryogenic design of DYNAMX has been published at the Space Cryogenics Workshop sponsored by the Goddard Space Flight Center during FY 1995. These proceedings shall be published in Cryogenics. Dynamics has participated in a number of educational outreach activities, including the development of a videotape teacher's guide which introduces superconductivity and superfluidity to High School students. Other educational outreach activities have included our involvement in a science fair in Pasadena, and a number of talks on DYNAMX to different organizations by various members of the DYNAMX team.

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Printed: 27 FEB 1996
BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Proceedings

Presentations


Satellite Test of the Equivalence Principle (STEP)

Principal Investigator: Prof. C. F. Everitt

Co-Investigators:
Worden, P.

Task Objective:
The objective of the Satellite Test of the Equivalence Principle (STEP) experiment is to investigate the foundation of gravitational theory, the equivalence of inertial and gravitational mass.

Task Description:
The mission, now called Quick STEP, is a NASA led experiment with possible collaboration with the French space agency. The Quick STEP mission is a descoped M2 mission with the essential Equivalence Principle and geodesy science intact. The overall cost has been reduced from approximately $600 m to $150 m.

Task Significance:
The STEP experiment may be thought of as a modern version of the experiment attributed to Galileo of dropping two weights from the Leaning Tower of Pisa. Any difference in the ratio of gravitational to inertial mass causes a corresponding difference in the rate of fall. The detection of a difference would substantially alter present theories of relativity and gravitation.

Progress During FY 1995:
During FY 1994-95 we completed two major projects:
1) completion of new office space in "mag gen" building (June 1995)
2) completion of "mechatronics" laboratory in "mag gen" building (September 1995)
We have occupied the newly completed space. About half of the potentially available laboratory space remains to be developed, but may be completed in FY 1995.

Technical progress in FY95:
(1) We made a number of minor improvements to the Flux Microscope. These are essentially to make it easier to use. We are still waiting for usable NbEuSe samples to be delivered by JPL.
(2) We made some major improvements to the SQUID Position Sensor Experiment. These include
   a. A new stepper motor and controller
   b. A much improved translation stage
   c. A new sample holder
   d. Much improved data acquisition software
   e. A new capacitance displacement sensor
   f. More accurate inductance calculations
   g. Good temperature control
   This apparatus is now adequate for our immediate sensor R&D effort.
3) We made significant improvements to the Exposure System which will expose niobium circuits on cylinders. This system was tested to be reproducible to better than 1 micron and has a resolution of about 10 microns. We also developed special software for the bitmaps that drive this system since commercial software was not up to the job.
4) The Electrostatic Positioning system, which will be used in the tipper table and other systems, has gone through two prototypes. The latest improved the resolution from 8 bits to 12 bits and the performance from 90 to 110+ Db.
(5) We acquired a number of major items of equipment for the Mechatronics Laboratory, including an RF etcher and...
clean room equipment. The Mechatronics laboratory has been moved from the Varian Physics building to its permanent quarters in the STEP building. It is being reassembled and significantly upgraded.

(6) All major components of the tipper table were acquired, and assembly and preliminary tests of subsystems have begun. We made some tests of the seismic background in the STEP building (a limiting factor for this facility) and found that it is about 10 times quieter than in Varian.

(7) We built a vacuum probe for helium storage dewars, and constructed a simple magnetometer for flux motion measurements. We used a Quantum Design DC SQUID. Without coupling the SQUID, we are as sensitive as listed in the SQUID specification sheet (actually, we are a little better). With the pick-up coil connected to the SQUID, we lose a factor of 10 in sensitivity. We are currently trying to improve this. However, the probe sensitivity is more than adequate to do the things we want to do at this point in time. The probe is so useful that we are building a second one to study superconducting-superconducting junctions and heat switches.

**STUDENTS FUNDED UNDER RESEARCH:**

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**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**NASA Tech Briefs**


**Presentations**


Critical Fluid Light Scattering Experiment - ZENO

Principal Investigator: Prof. Robert W. Gammon

Co-Investigators:
Shaumeyer, Dr. J.N.

Task Objective:
The objective is to measure the decay rates of critical density fluctuations in a simple fluid (xenon) very near its liquid-vapor critical point using laser light scattering and photon correlation spectroscopy. Such experiments are severely limited on earth by the presence of gravity which causes large density gradients in the sample. The goal is to measure fluctuation decay rates with 1% precision two decades closer to the critical point than is possible on earth, with a temperature resolution of +/- 3 microKelvin. This will require loading the sample to 0.1% of the critical density and taking data as close as 100 microKelvin to the critical temperature \( T_c = 289.72 \text{ K} \). The minimum mission time of 100 hours will allow a complete range of temperature points to be covered, limited by the thermal response of the thermostat and correlation averaging times. Other technical problems have been addressed such as multiple scattering and the effect of wetting layers.

Task Description:
We have demonstrated the ability to avoid multiple scattering by using a thin sample (100 microns), a fast optical thermostat with microKelvin temperature control and measurement, and accurate sample loading. Further important engineering tasks of mounting the experiment to maintain alignment during flight have been confirmed.

The experiment entails measurement of the scattering intensity fluctuation decay rate at two angles, 12 and 168 degrees, for each temperature and simultaneously recording the scattering intensities and sample turbidity (from the transmission). The analyzed intensity and turbidity data gives the correlation length at each temperature and locates the critical temperature.

The fluctuation decay rate data set from these measurements will provide a severe test of the generalized hydrodynamics theories of transport coefficients in the critical region. When compared to equivalent data from binary liquid critical mixtures they will test the universality of critical dynamics.

Task Significance:
Such experiments are severely limited on Earth because gravity causes a large density gradient in the fluid due to the divergence of the fluid compressibility as the critical temperature is approached. The data from this experiment will provide a test of critical phenomena theories in a temperature realm that has not been adequately tested to date, due to the limitations imposed by gravity. The data tests the current theory of crossover from asymptotic behavior near \( T_c \) to pure background behavior far from \( T_c \). Such a crossover theory is useful for predicting thermophysical properties in supercritical fluid solvents.

Progress During FY 1995:
After a science panel review, approval was given to prepare the Zeno instrument for a second flight as part of the USMP-3 payload to fly in February, 1996. The major theme of the second flight would be to avoid the density perturbations caused by moving the temperature too quickly. This would be accomplished by using a calculated set of maximum ramp rates which get slower and slower as \( T_c \) is approached. \( T_c \) itself would be determined with increasing resolution from the data taken in a single sequence moving to the critical point.

The key personnel at Ball Aerospace were retained and testing of the flight hardware began in November 1994. We were not able to improve on the slight amount of container heater perturbation of the sample temperature and so decided to fly with no change in hardware. We had thought that a simple radiation baffle could improve
performance. We also did extensive further testing of the laser/sample cell alignment and finally concluded that the fill was correct and stable and the focused beam was at the correct location. Thus our confidence in the inherent stability of the Zeno apparatus has increased.

After the final vibration testing, the inside of the Optical Module was cleaned and sealed up. The instrument began P+ characterization on April 13, 1994 and the testing continued to June 26, 1995. In this time we learned that our previous scanning technique for locating $T_c$ was flawed, and though precise, it gives a value for $T_c$ up to 1 milli-Kelvin below the actual temperature. The reasons are that the preparation of the sample for the scanning cooled the sample too quickly leaving the density at the laser beam position too low. Secondly, the scan itself was too fast causing further errors. A rich array of behavior was seen as various patterns of ramp rates were chosen to move to density testing temperature and to the data temperatures. These were eventually understood and separated into contributions from dimensional creep of the cell, real density changes at the beam location, and sample turbidity. A baseline set of procedures was written as code and tested for an earth baseline data set.

In July 1995 the final testing of the instrument was done off-line at KSC in three weeks. A final baseline was run. Using all the known parameters and a refined calculation of the rounding caused by gravity induced density stratification, we were finally not able to use the set of correlogram data to refine the value of $T_c$. We believe that the procedure will work in low gravity but we have to wait for the flight to demonstrate it. We used as a backup a very slow scan through $T_c$ from 300 micro-Kelvin. A consistent and reliable value for $T_c$ was found but it did not have the ~10 micro-Kelvin precision that we require.

We are continuing to study the behavior of the xenon sample in our back-up flight cell mounted in the Engineering Development Model located at the University of Maryland. We expect to improve our understanding of the complex interference effects seen in the flight cell from data taken with the back-up cell which has solid sapphire windows.

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**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Journals**


Presentations
Confined Helium Experiment (CHeX)

Principal Investigator: Prof. John A. Lipa
Stanford University

Co-Investigators:
Chui, T.C.P.
Israelsson, U.E.
Gasparini, F.M.
Jet Propulsion Laboratory (JPL)
Jet Propulsion Laboratory (JPL)
State University of New York, Buffalo

Task Objective:
Of significant current interest in the field of condensed matter is the study of crossover behavior as a bulk system if confined more and more tightly in one or more dimensions. Crossover occurs when the effect of the boundaries is significant but not dominant. An ideal way to explore this effect is to perform measurements on films of ever decreasing thickness until the lower dimensional behavior is dominant. Unfortunately, in most cases it is not possible to vary the film thickness without totally changing the sample, making it difficult to keep track of intrinsic changes in the parameters. Also, in real systems other effects often dominate the behavior of matter at very small length scales. Near the lambda transition of helium the correlation length diverges, magnifying the effects of the confinement while simultaneously decreasing the importance of extraneous effects. This situation gives us a unique tool to look at a diverse set of conditions in a controlled way, opening a new window on the general question of finite size phenomena in condensed matter systems. For example, at 0.1°C below the transition, the correlation length is on the order of a few Angstroms, whereas 10°9 degree below the transition the correlation length is about 0.1 mm. This means that, if we can access the inner region very close to the transition, we will have for the first time a finite size system with a truly macroscopic length scale, allowing exceptional control of the effects of boundaries. This situation appears to be absolutely unique in condensed matter systems. The Confined Helium Experiment should lead to dramatically improved tests of the theory of finite size effects.

Task Description:
We plan to measure the heat capacity of helium confined between closely spaced parallel plates and compare the results with the bulk heat capacity data obtained on the Lambda Point Experiment (LPE). The relationship between the two data sets is predicted by theories of confinement. Most of the LPE flight hardware will be reused to perform the required measurements.

Task Significance:
The Confined Helium Experiment should provide a much improved test of the theory of confinement and may provide a firm basis for its extension to other properties of confined materials.

Progress During FY 1995:
In the past year we have been able to transition from instrument development to systems level performance testing with integrated instrument and flight electronics.

A significant effort was undertaken at Northeastern University to develop a suitable confinement system. This resulted in a stack of 408 silicon wafers with 57 micron average gap, suitable for use in a flight calorimeter. A new caging mechanism was built and the calorimeter successfully assembled. A 77 K shake test was performed and no damage was detected. This cleared the way for assembly of the complete flight instrument and the commencement of low temperature testing. In parallel with this effort the flight high resolution thermometers were built, based on the prototype design. Detailed simulations were performed on the cosmic ray noise problem and the results were encouraging. It appears that with an on Hz sampling rate most of the degradation seen on LPE can be avoided. Recent noise measurements of the flight thermometers have confirmed the good results seen with the prototype devices.
The flight electronics was upgraded slightly to meet the CHEX needs and take advantage of the experience from LPE. The sampling rate of the science-critical A/D converters was increased to 100 Hz from 1 Hz, and the main heater drive circuits were modified to operate at higher power levels and shorter pulse times. This latter change was made to minimize the effects of stray heat input on the net energy increments made to the sample.

The flight code development work encountered significant difficulty implementing the 1553 interface used to communicate between the instrument and facility computers. Vendor support was almost non-existent, and new device drivers had to be written virtually from scratch. This delayed our flight code development significantly and impacted testing. However we now have a minimal operating environment functioning and are testing science code. The software development environment is substantially improved over LPE and bug fixes are quick, primarily limited by the time required to understand the cause of the problem.

Improvements to the flight cryostat were accomplished at JPL and tested to verify the required performance. Modifications to the facility electronics to provide the 1553 communications interface to the instrument electronics and improve functionality for ground testing have been completed and are under test. This effort was also impacted by the difficulties with the 1553 interface, discussed above. Early mission integration activities have been accomplished with MSFC, including establishing the Mission Verification Plan and a baseline Instrument Interface Agreement and completion of Phase 2 Ground and Flight Safety Reviews. Preparations are now under way for instrument and cryostat integration and the following round of performance and environmental testing.

**Students Funded Under Research:**

- BS Students: 0  
- MS Students: 0  
- PhD Students: 2

**Task Initiation:** 12/92  
**Expiration:** 12/97  
**Project Identification:** 963-24-04-02  
**Responsible Center:** JPL

**Bibliographic Citations for FY 1995:**

**Journals**


**Proceedings**


II. MSAD Program Tasks — Flight Research

In Situ Monitoring of Crystal Growth Using MEPHISTO

Principal Investigator: Dr. Reza Abbashian
University of Florida

Co-Investigators:
Coriell, Dr. S.R.
National Institute of Standards and Technology (NIST)
Favier, Dr. J.J.
CENG (France)

Task Objective:
The objective is to determine the morphological stability of solid/liquid interfaces and resulting macro and micro segregation patterns, and to determine the attachment kinetics at the freezing interface deduced via measurements of the growth-rate/interface-supercooling relationship.

Task Description:
To investigate the solidification behavior and stability of solid/liquid interfaces during the growth of pure Bi (a facet forming material), and Bi alloyed with small amounts of Sn, in 1-g and μg. The experiments were designed to make use of the second flight of MEPHISTO on USMP-2 (3/94).

The experiments make use of the Seebeck technique to measure the interface temperature in-situ and non-invasively during crystal growth in both the ground-based and flight experiments. Both 1-g and μg experiments make use of the measured resistance change across the sample to determine interfacial velocity and Peltier pulsing for demarcation of the interface shape.

Task Significance:
The experiments were performed to gain a detailed understanding of the role of gravity driven convection during the solidification of faceted materials. Two fundamental and interrelated aspects of the liquid to solid transformation have been investigated: (a) Morphological stability of the solid/liquid (s/l) interfaces and the resulting macro- and micro-segregation patterns and (b) atomic attachment kinetics at the freezing interface, deduced via measurement of the growth rate-interface supercooling relationship(s).

Initial ground-based experiments were carried out using high purity Bi and dilute Bi-Sn alloys. Bi-Sn alloys were chosen to complement the experiments conducted by CENG/CNES on the first flight of MEPHISTO (MEPHISTO-I): on this flight, dilute, non-faceted Sn-Bi alloys were used, while this research program (the second flight of MEPHISTO, or MEPHISTO-2) used strongly faceted Bi-0.1 at% Sn alloys. In this manner, the results of the two flights are being used to compare and contrast various fundamental aspects of solidification without and with a strong influence of atomic attachment kinetics, respectively, in the presence (ground-based studies) and near-absence (μ-g studies) of gravity induced convection. It is expected that this comprehensive investigation approach will significantly further our understanding of key crystal growth parameters.

We expect to use these data to test and improve current solidification theories. In particular, the interplay between morphological stability and interface kinetics is not well understood. The microgravity experiments will yield an integrated database involving interface velocity/interface shape/interface supercooling. Such data are important from both practical and theoretical standpoints. For example, a knowledge of the transition from a faceted to a rough interface (from the Seebeck data) and the interface shape (from solute-dump-demarcated interfaces) under identical growth conditions has important applications in practical crystal growth situations: the information can be used to understand the correlation between defect generation and solute banding. In addition, because the information has been obtained in diffusion dominated conditions without the overriding effects of gravity-induced thermo-solutal convection, meaningful tests (and appropriate refinements) of the current crystal growth theories can be made.
II. MSAD Program Tasks — Flight Research

Progress During FY 1995:

A comprehensive directional solidification experiment was recently carried out successfully on the USMP-2 mission (STS-62) utilizing the MEPHISTO directional solidification facility. The 14 day shuttle flight was launched on Friday, March 4, 1994. A total of 45 cm of dilute Bi-Sn alloys were solidified directionally in microgravity. Prior to the final directional solidification, extensive measurements were performed on the samples, consisting of Seebeck measurements to measure the solid/liquid (s/l) interface temperature, resistance measurements to track the position of the s/l interface and thermal gradient measurements in the solid and liquid during freezing and melting. The final solidification also included a procedure for marking the shape of the s/l interface via mechanical perturbations, as well as rapid quenching of a 2 cm section of one of the samples.

A three-pronged strategy for achieving the stated goals was used: (a) Development of experimental apparatus for ground based kinetics and morphological stability studies at University of Florida (UF), (b) Scientific and technical collaboration with the MEPHISTO teams at CENG and CNES and (c) Analytical modeling of morphological stability and interface kinetics in collaboration with Sam Correll (NIST). Particular reference is also made to an extensive collaboration between the various scientific and technical personnel from NASA-Lewis for developing a comprehensive flight program.

The research team at UF developed the facilities necessary for ground based experiments to ensure maximum conformity with the MEPHISTO space hardware. In addition, four "campaigns" were conducted prior to the USMP-2 mission (three on the MEPHISTO engineering model at CENG and one on the MEPHISTO flight model at CNES). Each campaign required three samples approximately 1 meter in length, which were prepared at UF according to MEPHISTO specifications. The campaigns not only proved the integrity of the samples produced, but also provided valuable ground based data, which is currently being compared with the flight experiments. Concurrently, NIST has carried out analytical modeling of the morphological stability of faceted solid/liquid (s/l) interfaces for the alloy system under investigation.

These flight experiments used a novel technique (termed the Seebeck technique) to measure the interface supercooling directly, non-invasively and in-situ (i.e. in real time during growth). The interface velocity was measured by monitoring the resistance change across the sample, while the interface shape was delineated by subjecting the sample to electrical current pulses (for ground based studies) and mechanical perturbations (for μ-g studies) to cause a momentary demarcation of the interfaces.

We will be able to obtain values of key parameters, such as liquid diffusivities, via this investigation. In addition, the novel and non-intrusive technique used to measure the interface temperature can potentially be used for monitoring and controlling the space-based single crystal growth of technologically important semiconductors.

We are currently analyzing the approximately 6 gigabytes of USMP-2 MEPHISTO data. During the mission, extensive use was made of the telemetry commanding capability to modify and refine experimental procedures for better scientific yield. Preliminary analysis of the data acquired during the first 28 hours of mission shows excellent correlation of the Seebeck signal with melting/freezing as well as solute build-up/decay. Numerical calculations are being carried out concurrently to correlate the Seebeck signal with thermal/solutal decay and hence to back calculate an accurate value of the diffusion coefficient of tin in liquid bismuth.

Metallurgical analyses of the samples have shown that the interfacial kinetics play a key role in controlling the morphological stability of faceted alloys. We believe this to be the first unambiguous demonstration of such an effect during directional solidification under diffusion-dominated conditions. In addition, analysis of the differential Seebeck thermoelectric signals for very slow growth velocities was carried out. The analysis indicates that it is possible to calculate the kinetic undercooling from the Seebeck signal decay at the end of solidification. Such information is not accessible from ground-based studies due to effects associated with convective destabilization.

Recent results of the analysis work on the USMP-2 flight samples are contained in the papers listed in the bibliography section of this report. With the collaboration of the French, anomalies experienced during the flight have been analyzed and plans to present their reoccurrence made.
II. MSAD Program Tasks — Flight Research

Discipline: Materials Science

Students Funded Under Research:  
BS Students: 1  
MS Students: 4  
PhD Students: 0  

Task Initiation: 1/90  Expiration: 9/95  
Project Identification: 963-25-05-04  
NASA Contract No.: NAG3-1096  
Responsible Center: LeRC

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Presentations


II. MSAD Program Tasks — Flight Research

Coupled Growth in Hypermonotectics

Principal Investigator: Dr. J. B. Andrews
University of Alabama, Birmingham

Co-Investigators:
Cortell, S.
National Institute of Standards and Technology (NIST)

Task Objective:
The objective of this investigation is to gain an improved understanding of solidification processes in immiscible alloy systems. A portion of this study involves the development of experimental techniques which will permit steady-state coupled growth of hypermonotectic composition samples to produce aligned microstructures. A parallel effort is underway to develop a model for the coupled growth process in monotectic systems. This analysis starts with the basic equations for diffusion controlled growth and avoids many of the simplifying assumptions often utilized in similar analyses. Once results are obtained from experimentation, they will be compared to predictions from the model and utilized to improve the model. In order to permit steady-state coupled growth in hypermonotectic composition samples, experimentation must be carried out under low-gravity conditions.

Task Description:
This project includes the major research tasks of experimentation, including ampoule development and testing, and theoretical modeling. Ampoule development first involves the selection of an appropriate ampoule material based on wetting characteristics and resistance to attack by the molten aluminum-indium alloy. This first step is then followed by design of an appropriate ampoule assembly to control thermal end effects and to eliminate free surfaces during processing. The modeling segment involves stability calculations to determine the gravity level limits and thermal gradient requirements for proper processing followed by modeling of the solidification process. In addition, the project is concerned with the evaluation of current flight hardware for use in experimentation and with input into the development of new hardware. Experimentation will require directional solidification of immiscible aluminum-indium alloys under low-gravity conditions in order to avoid convective instability and promote steady-state coupled growth.

Task Significance:
The significance of this project lies primarily in the scientific gains to be made in truly understanding the coupled growth process in immiscible alloys. Many alloys in immiscible systems have great promise for potential applications in areas which include superconductors, magnetic materials, catalysts, and electrical contacts. However, there are many details of the solidification process that are poorly understood for these alloys because these details are masked by gravity-driven phenomena. This project is aimed at using the unique environment available in space to improve this understanding in order to make possible the production of new materials using specialized processing techniques.

Progress During FY 1995:
A tremendous amount of progress has been made in both the experimental and theoretical segments of this project. In the experimental area, aluminum nitride (AlN) has been selected as the ampoule material of choice due to its favorable wetting behavior and inertness when utilized with hypermonotectic Al-In alloys. An ampoule assembly has been designed and tested which utilizes a spring and piston arrangement to accommodate thermal contraction and solidification shrinkage of the sample during directional solidification. This design is being utilized to minimize the likelihood of void formation during processing which would provide free surfaces on the melt and the possibility of surface tension driven convection. Thermal contraction of the Al-In alloy requires a spring that can accommodate over 2 cm of travel while maintaining a consistent spring constant at temperatures up to 1260 °C. This requirement has been satisfied by using a specialized high performance carbon spring produced from a polymer precursor. The ampoule design also includes a three piece sample assembly consisting of a main sample sandwiched between two...
smaller "dummy samples." This arrangement is utilized to minimize thermal end effects in the main sample and provide a more uniform solidification rate during processing.

A vacuum ampoule loading and sealing technique has been developed which permits loading the ampoule at absolute pressure levels approaching 1x10^-7 Torr. The aluminum nitride ampoule is then sealed by melting a sealant material using a focused radiant heating facility while the ampoule assembly is under vacuum. Vacuum sealing minimizes residual gas entrapment in the ampoule which could lead to void formation. Prototype testing of the ampoule assembly has been carried out using the Advanced Gradient Heating Facility (AGHF) at the French Space Agency (CNES) and at using a breadboard model of the High Gradient Furnace with Quench (HGFQ) at the Marshall Space Flight Center (MSFC).

Major advances have also been made in the modeling area. Calculations have been made to emulate the flow patterns and velocities anticipated at different gravity levels while processing. These two- and three-dimensional analyses have permitted determination of the sensitivity of the experiment to residual acceleration levels and orientations with respect to the ampoule axis.

The solidification model, which starts with the basic equations for diffusion controlled growth, has been further refined. This model has already shown that some of the assumptions which were previously thought to be valid placed major restrictions on the phase equilibria conditions. The model has now been expanded to permit determination of the influence of a high diffusivity in one of the product phases during coupled growth. This addition is significant because one of the product phases is a liquid. The results indicate that diffusion in one of the phases has a surprisingly small impact on the resulting microstructure. Work is continuing in this area.

**Students Funded Under Research:**

| BS Students: | 0 | BS Degrees: | 0 | Project Identification: 963-25-08-09 |
| MS Students: | 2 | MS Degrees: | 1 | NASA Contract No.: NAG8-39717 |
| PhD Students: | 2 | PhD Degrees: | 0 | Responsible Center: MSFC |

**Bibliographic Citations for FY 1995:**

**Journals**


**Proceedings**


II. MSAD Program Tasks — Flight Research

Presentations


II. MSAD Program Tasks — Flight Research

Discipline: Materials Science

**Effects on Nucleation by Containerless Processing**

**Principal Investigator:** Prof. Robert J. Bayuzick  
Vanderbilt University

**Co-Investigators:**
Hofmeister, W.  
Vanderbilt University  
Robinson, M.  
NASA Marshall Space Flight Center (MSFC)

**Task Objective:**
The primary scientific objective is to further the understanding of nucleation of solids from their melts. A secondary objective is to determine if ground-based methods, such as drop tube processing, electromagnetic levitation, and electrostatic levitation, are equally useful for containerlessly processing bulk samples of pure metals as compared to electromagnetic heating and positioning in low Earth orbit. Within this secondary objective is a focus on identifying and quantifying any possible technique specific factors that influence nucleation behavior.

**Task Description:**
The presently existing containerless ground-based methods are being used to study nucleation of solid from the liquid. This includes drop tube processing, electromagnetic levitation, and electrostatic levitation. Since nucleation is a statistical process, approximately 100 undercooling measurements are desired for each type of sample. This number of measurements facilitates the interpretation of results through the application of statistical techniques. Much care is taken in the measurement of temperature due to the sensitivity of the approach to the precision of the measurements. Increasing and investigating the absolute precision is an important part of the experiments. The precision affects the width of the distribution of undercoolings, which consequently determines the activation energy for the phenomena. A large half-width of a distribution yields low values for the pre-exponential and exponential factors in the nucleation equation, thereby indicating heterogeneous nucleation by mechanisms other than contact with a container. Comparisons of the data from the ground-based and flight techniques give clues as to the nature of the nucleation of the solid from the liquid. Different processing methods have different environments and other factors that may affect the amount of undercooling in bulk samples.

**Task Significance:**
Solidification processing is one of the most prominent methods for the production of materials and most of these processes begin with a nucleation step. The regime of nucleation known as deep undercooling, where liquids are cooled considerably below their equilibrium freezing temperatures prior to the formation of solids, has become particularly distinctive. With deep undercooling, a unique condition for microstructural development and control exists and, therefore, a unique condition for improving and controlling the properties of materials exists. Hence, an understanding of the rudiments of nucleation in the unique regime is most important.

**Progress During FY 1995:**
Each major objective of this research has been addressed in order to complete the theoretical and statistical foundation of the project. The first objective is to quantitatively describe the observed nucleation behavior in terms of Classical Nucleation Theory. The second is to determine the processing factors which promote higher undercoolings and thus a decrease in the heterogeneous nucleation behavior. The third is to interpret the results from statistical analyses in order to determine if there is a specific heterogeneous nucleation mechanism responsible for the observed behavior.

For the first objective, a complete error analysis has been accomplished on the statistical analysis method which leads to calculated values for the pre-exponential and exponential factors of the classical nucleation rate equation.
The error analysis was performed with Monte Carlo simulations for a predescribed set of parameters. This set of parameters has been optimized by the use of a factorial design in analysis of variance. The results of the error analysis indicate that there is a strong correlation between the accuracy of the calculated values and temperature error. The magnitudes of the calculated values decrease as temperature error increases.

Another part of the error analysis concerns the actual temperature data used to determine the undercooling temperatures. All of this data, including the data from the flight experiment aboard TEMPUS and the ground-based data from the electromagnetic levitator and electrostatic levitator, has been analyzed. The results indicate that there is a one standard deviation temperature measurement error of ±1.4 K using the TEMPUS hardware and an error of ±5 K using ground-based hardware. Further error analysis concerning the dependence of undercooling measurements on individual sample and on processing cycle number are being continued.

For the second objective, statistical comparisons based on analysis of variance techniques have been employed in order to quantitatively determine the effects of several processing parameters and to give the comparisons a sound statistical foundation. The existing data sets have also been interweaved into a factorial design framework that will allow the determination of the factors that influence nucleation behavior. Some of these factors may be intrinsic to Classical Nucleation Theory, whereas others may not be explicitly contained in the nucleation rate equation. In the investigation of processing parameters, different containerless processing methods have been used. Different processing methods have different environments and/or adjustable parameters which may promote higher undercoolings or a greater success rate of achieving higher undercoolings. The specific parameters that will be investigated by the factorial design are sample size, sample purity, cooling rate, overheat, initial vacuum level, and overall processing method. Interactions between processing parameters as evidenced by the results of the factorial design analysis may be indicative of specific heterogeneous nucleation mechanisms such as surface nucleation, if these interactions are coupled with low calculated values for the pre-exponential and exponential factors. Further investigation is being done in order to interpret the results of possible results from the factorial design. The end result of the factorial design analysis will identify those factors which show the promise of promoting higher undercoolings in ground-based processing methods.

**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

Presentations

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**STUDENTS FUNDED UNDER RESEARCH:**

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**TASK INITIATION:** 4/90  **EXPIRATION:** 6/95

**PROJECT IDENTIFICATION:** 963-35-10

**NASA CONTRACT NO.:** NAG8-978

**RESPONSIBLE CENTER:** MSFC

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Printed: 28 FEB 1996
II. MSAD Program Tasks — Flight Research

Discipline: Materials Science

Alloy Undercooling Experiments in Microgravity Environment

Principal Investigator: Prof. Merton C. Flemings  Massachusetts Institute of Technology (MIT)

Co-Investigators:
Matson, D.M.  Massachusetts Institute of Technology (MIT)

Task Objective:
The objectives of this task are to perform solidification experiments on undercooling binary alloys, to compare results of ground-based and microgravity experiments, and to examine effects of microgravity on solidification behavior and microstructure characteristics.

Task Description:
We will collect thermal history, nucleation and growth history, and resulting solidification microstructures through experiments applying direct, high speed, high resolution pyrometric and cinematographic measurements during melting, undercooling and recalescence of nickel-tin binary alloys of different compositions, both on the ground and in microgravity.

Task Significance:
With experiments carried out in microgravity, it is expected to have improved specimen shape and stability and reduced convection during cooling, resulting in the possibility of higher undercooling, less microstructure alteration, reduced coarsening, and improved specimen observation in order to gain a complete understanding of the solidification kinetics of undercooled melts, including: primary dendrite tip velocities; rapid thickening of primary and secondary arms during recalescence; ripening, remelting, and solute redistribution; dendrite fragmentation and grain refinement; primary phase solidification and ripening; and eutectic solidification with concurrent primary phase ripening.

Progress During FY 1995:
Work during the current grant period resulted in the successful completion of post-flight analyses. Metallographic analyses of the flight samples were conducted and post-processing condition was documented. Analysis results of alloy interdendritic spacing was compared to ground-based experiments using samples processed under similar conditions on earth. These results show a slight increase in dendrite arm spacing for the IML-2 samples but the deviation was less than expected due to the lower undercooling achieved. Analysis of the video record showed that samples were fully molten and surface oscillations were readily apparent, especially under higher superheat conditions. Documentation of significant ground-based and flight results has been completed.

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TASK INITIATION: 4/90  EXPIRATION: 6/95

PROJECT IDENTIFICATION: 963-35-10
NASA CONTRACT NO.: NAG8-971
RESPONSIBLE CENTER: MSFC

Printed: 28 FEB 1996
II. MSAD Program Tasks — Flight Research

Discipline: Materials Science

**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Journals**

**Proceedings**

II. MSAD Program Tasks — Flight Research

Compound Semiconductor Growth in Low-g Environment

Principal Investigator: Dr. Archibald L. Fripp

NASA Langley Research Center (LaRC)

Co-Investigators:

Crouch, R.K.
Debnam, W.J.
Clark, I.O.

NASA Headquarters, Code UG (MSAD)
NASA Langley Research Center (LaRC)
NASA Langley Research Center (LaRC)

Task Objective:
The objective of the Langley flight program is to determine the effects of gravity driven convection on the growth and crystal properties of the compound semiconductor alloy, lead tin telluride which is miscible over the entire compositional range. The electronic properties of this material are dependent on the ratio of the two, pseudobinary, components and consequently, the uniformity of an array of devices is dependent on good compositional control. Lead tin telluride is amenable to study for it is easily compounded; it has a relatively low vapor pressure; and there is existing, though limited, literature on its growth and properties.

Task Description:
This material was chosen for microgravity research for a number of reasons. Lead tin telluride is not only a useful semiconductor material which has been used for construction of infrared detectors and tunable diode lasers. It also has a phase diagram similar to other compound semiconductors of interest such as mercury cadmium telluride and mercury zinc telluride.

Lead tin telluride is also interesting from a purely scientific point of view in that it is both solutally and thermally unstable. Both the temperature gradients and the compositional changes in the liquid near the melt/solid interface produce density gradients which, in turn, produce driving forces for convection when coupled with gravity.

Task Significance:
Earth based Bridgman growth of lead tin telluride has only produced inhomogeneous crystals that are a result of strong convective forces in the liquid during growth. The temperature gradients are required for growth and the solutal changes at the interface are a fundamental property of the material system. However, for convection to occur these gradients must be coupled to a gravitational field. Growth in low Earth orbit offers an unique and fascinating opportunity to study the effect of convection on this class of materials. The resultant gravitational force is not zero in low Earth orbit hence convection is not completely eliminated but the fluid velocity, due to convection, will be greatly reduced.

Two flights are planned in the Advanced Automated Directional Solidification Furnace (AADSF). The primary objective of both flights is to study the effect of gravity reduction, hence convection reduction, on the growth of lead tin telluride. In one experiment the growth rate of the crystal will be changed in steps to test the effect of varying the relative speed of the interface movement and the fluid velocity. In the other experiment the Space Shuttle will be rotated to vary the relative orientation of the gravity vector and the crystal growth axis. Both sets of experiments are expected to affect the compositional homogeneity of the crystal.

Progress During FY 1995:
We are ready for flight on USMP-3 presently schedules for launch on February 22, 1996. Three cell ampoules have been constructed, tested both thermally and machenically, and a flight item delivered to Cape Kennedy and installed in the AADSF on the Shuttle.
II. MSAD Program Tasks — Flight Research

**Discipline:** Materials Science

**STUDENTS FUNDED UNDER RESEARCH:**

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**TASK INITIATION:** 10/78  **EXPIRATION:** 9/98

**PROJECT IDENTIFICATION:** 963-35-00-01

**RESPONSIBLE CENTER:** LaRC

**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Journals**


Zhao, A.X., Motes, C., and Narayanan, R. Rayleigh convection in a closed cylinder- experiments and a three dimensional model with temperature dependent viscosity effects. Physics of Fluids, 7, 1576-1582 (1995).

**Presentations**

Melt Stabilization of PbSnTe in a Magnetic Field

Principal Investigator: Dr. Archibald L. Fripp

Co-Investigators:
- Debnam, W.J.  NASA Langley Research Center (LaRC)
- Szofran, F.R.  NASA Marshall Space Flight Center (MSFC)
- Chait, A.  NASA Lewis Research Center (LeRC)

Task Objective:
The objective of this research is to further elucidate the gravity driven physical phenomena on the growth of the alloy compound semiconductor, PbSnTe. This work, coupled with the past microgravity experiment with the MEA and the existing flight program to grow PbSnTe in the AADS, will form the most comprehensive set of space processing experiments performed to date.

Task Description:
The effect of the gravitational body force on the convective properties of the alloy compound semiconductor, PbSnTe, with that body force modified by both reduced gravity and by magnetohydrodynamic (mhd) damping is the subject under investigation. PbSnTe is an ideal material for this study in that it was the material of both a past flight experiment and a planned 1996 AADS experiment. Both of these experiments are without magnetic fields. Subsequent experiments, both Earth based and in Space, using mhd damping will form a complete set of experiments that will further elucidate the gravity dependent physical phenomena on the growth of this class of materials.

The application of a magnetic field to PbSnTe growth will dampen convective flow. The anticipated results are that even in the MSFC superconductor magnetic furnace the growth will not become diffusion controlled but that the combination of magnetic field and low gravity environment will produce diffusion controlled growth.

Task Significance:
Numerical modeling is an integral part of this endeavor. Computer simulation can aid in the design of the space experiment by its predictive capacity to optimize conditions for the growth. The key purposes of this portion of the study will be to optimize the growth for both the Earth and the space experiments and to obtain an estimate of the required magnetic field strength for low gravity growth.

This proposed work will complete the set. It will compare the effects of convection, as modified by a magnetic field, on the growth of this material both on Earth and in the Microgravity environment found in low Earth orbit.

Progress During FY1995:
The progress within this first year of the research on the magnetic stabilization of PbSnTe consists primarily of preparations for future quantifiable, modelable experiments.

Numerical modeling is an essential part of this program. The primary thrust for modeling is within the Materials Division at the Lewis Research Center. Modeling, both two and three dimensional, will evaluate both the sensitivity of the thermophysical parameters to determine if better measurements, in addition to electrical resistivity, are needed and attempt to determine the combination of low gravity and magnetic field strength required to attain diffusion controlled growth in PbSnTe. Preliminary modeling is complete. Diffusion controlled growth is predicted, with modest magnetic fields, in microgravity but heavy mixing is predicted on Earth even with a five Tesla field.

The ampoule configuration has been designed for ground based tests in the five Tesla magnetically stabilized furnace at the Marshall Space Flight Center. Seven crystals have been grown to date, six with full magnetic field and one
with no field. The primary variable was ampoule pull rate. All crystals showed complete compositional mixing as was predicted by the numerical modeling.

Additional modeling is underway to evaluate the ampoule diameter as it affects both viscous damping and extent of flux line intersection. The effect of thermal gradient, both magnitude and shape, will also be evaluated.

**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

*Journals*

*NASA Tech Brief*
Gravitational Role in Liquid-Phase Sintering

Principal Investigator: Prof. Randall M. German
Pennsylvania State University

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
The purpose of this research is to establish the gravitational role in liquid phase sintering with respect to both the macro-scale distortion and phase separation, and the microstructural evolution over time at elevated temperatures.

Task Description:
The investigation has graduated to flight status, with 21 samples (seven compositions of tungsten-nickel-iron) subjected to liquid phase sintering treatments on STS-65 during July 1994. The experiments involved isothermal hold times of 1, 15, and 120 minutes at 1500°C in the Japanese-developed Large Isothermal Furnace on the IML-2 mission. Identical ground-based experiments have been conducted to provide the baseline for contrast with the microgravity samples, and post-flight analysis will focus on quantitative assessment of distortion and microstructural evolution.

Task Significance:
The microgravity experiments will establish a modeling basis for the gravitational role in a viscous flow distortion of sintered components. It will further assess the agglomeration of solid grains and possible coalescence contributions to grain growth for upgrading of current theories. These results will lead to the development of manufacturing techniques which will permit formulation of new unique alloys.

Progress During FY 1995:
"Sintering" means welding or fusing of metal or ceramic powders by heating them without melting. Frequently, it is aided by applying pressure in a special high-temperature press to squeeze the particles together. This experiment explores a different mechanism, by adding a portion of a powder that melts at a lower temperature and surrounds the powders that remain solid. This liquid then lets particles and materials move more easily, allowing the powders to move rapidly form a solid compact. Problems such as separation of the solid and liquid due to gravity (manifested by settling of the solid particles) still remain. Access to the low gravity environment of Earth orbit provides a unique opportunity to study liquid-phase sintering without separation, settling, or other gravity-induced complications. The results will increase our understanding of the process, and the new knowledge will help introduce new industrial application of liquid-phase sintering, such as automotive components, resulting in new and improved products.

In this investigation, mixed powders of tungsten, nickel, and iron are initially cold compacted under pressure in the shape desired for the final product. The compacts are then heated to just below the nickel-iron alloy melting temperature to provide handling strength, a process called "presintering." In the experiment, they are heated above 1465°C to form a liquid-solid mixture. The tungsten, with its very high melting point (3370°C), remains a solid, while the nickel and iron, with much lower melting points, become liquid. The liquid permits more rapid transport of material for faster sintering than would be possible if all the material were solid. After sintering, the microstructure of the samples (i.e., the structure when viewed under very high magnification), consists of connected tungsten grains surrounded by the solidified liquid.

This experiment flew as part of the second International Microgravity Laboratory (IML-2) mission in July 1994 aboard the Space Shuttle Columbia. The experiment was conducted in an apparatus called the Large Isothermal Furnace (LIF), which could operate at the high temperatures required. The LIF was developed by

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Ishikawajima-Harima Heavy Industries Co., Ltd., for the National Space Development Agency of Japan (NASDA). This project was supported by the NASA Headquarters Microgravity Science and Applications Division, and was managed by the NASA Lewis Research Center.

The test specimens for the LIF consisted of three different cartridges, each containing seven samples 10 mm in diameter by 10 mm high. One cartridge was tested at each of the three critical sintering periods identified in earlier ground-based experiments: 1, 15, and 120 minutes. In one minute, liquid penetrates along existing solid-solid boundaries. Fifteen minutes is the time needed for full densification. The 120-minute time is needed to observe grain rotation and coalescence events.

In summary, a nominal temperature of 1506°C was achieved for each of the desired test times. Free-drift of Columbia during the appropriate times was confirmed. All of the functional objectives were achieved. Analysis will begin upon return of the samples to the laboratories of the Pennsylvania State University.

October 1995 update:

Upon return of the samples to the laboratories of the Pennsylvania State University in November of 1994, each sample was photographically recorded, its physical data was collected, and they were readied for metallurgical examination using traditional sectioning, mounting, and polishing techniques.

The microgravity environment present during the sintering of these samples has had a drastic impact on their microstructural development. The frequency and magnitude of the microstructural anomalies is larger in samples containing lower volume fractions of tungsten. Features are present which have never been seen before, despite the fact that tungsten-nickel-iron heavy alloys are well characterized metallurgically. New scientific explanations are needed to describe the formation of the aforementioned anomalies; however, specific observations can be made about the microstructures observed. First, gas-filled pores appear to be a stable, discrete phase in microgravity liquid-phase sintered materials. These pores undergo coalescence as they become very large compared to the scale of the microstructure.

The lack of gravity also produced very unusual pore morphologies. Pore shape is probably influenced by attachment to tungsten grains. In the next several months, work will begin on reconstructing the three-dimensional microstructure of these samples.

At the edge of the sample where there was no constraint exerted by the crucible, liquid protrusions were observed on the sample sintered for 120 minutes. In terrestrial conditions, these protrusions would not exist, rather there would be a meniscus between grains produced by the capillary force of the wetting liquid. The lack of periodic structure in these structures creates additional questions. Had the structure been periodic, it could have been described as a columnar solidification front created by constitutional undercooling.

Clearly, there are some very unusual features in these microstructures. It will be some time before all of these features are adequately described by fundamentals of powder metallurgy and solidification.

The data obtained from this shuttle flight opens the doors to many unanswered questions. The next immediate step is to dissect the samples layer by layer using a micromilling instrument, and then to electronically reconstruct the microstructure using image analysis equipment. This should provide an absolute picture of the microgravity microstructures. Additionally, it may be possible to fly additional samples on an upcoming shuttle flight (e.g., MSL-1 on STS-83) that will provide information on the effect of surface energy, solid content, etc., on microstructural development in a microgravity environment.

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BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals


Proceedings


Printed: 28 FEB 1996
II. MSAD Program Tasks — Flight Research


Presentations
Iacocca, R.G. and German, R.M. "Liquid phase sintering in microgravity." 22nd Seminar & Exhibit of the Pittsburgh Section of APMI International on November 8, 1994.

II. MSAD Program Tasks — Flight Research

Discipline: Materials Science

Isothermal Dendritic Growth Experiment

Principal Investigator: Prof. Martin E. Glicksman
Rensselaer Polytechnic Institute

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
Successfully analyze the resulting space flight and terrestrial data from the Isothermal Dendritic Growth Experiment (IDGE) on its first Space Shuttle mission (STS-62), as part of the Second United States Microgravity Payload (USMP-2). Determine dendritic tip velocity and tip radius as functions of supercooling. Use velocity and radius data to evaluate the validity of theoretical models that purport to predict velocity and radius. These models are used to improve industrial metal production on Earth. Publish reports and give papers to inform the scientific community of our findings.

Acquire and analyze terrestrial data gathered by the IDGE apparatus at three different supercoolings, in order to characterize the three-dimensional shape of dendritic tips. This data will ultimately be compared with the resulting space flight data from the second mission. This is the primary scientific objective of the second flight of IDGE aboard USMP-3 on STS-75, currently scheduled for February 1996.

Purify and characterize Pivalic Acid (PVA), the sample material to be used for the third flight of IDGE aboard USMP-4.

Task Description:
The Isothermal Dendritic Growth Experiment is a fundamental Materials Science Experiment performed on the Space Shuttle. The specific topic is dendritic solidification which is relevant to virtually all industrial manufacturing of metals and alloys on Earth.

IDGE is an experiment performed in the cargo bay of the Space Shuttle using an apparatus designed, built, and tested at the NASA Lewis Research Center in Cleveland, Ohio, USA. Over 400 photographs of dendrites that solidified in space along with over 800 photographs of dendrites solidified on Earth will be produced on each flight. Each photograph will be accompanied by 8 or more scientifically important measurements of time, temperature, and local acceleration. While in space, the apparatus will be operated by IDGE personnel located in the Payload Operations Control Center (POCC) at the George C. Marshall Space Flight Center (MSFC) in Huntsville, Alabama, the Tele-operations Support Center at the Lewis Research Center, and at Rensselaer Polytechnic Institute (RPI).

Personnel at RPI will analyze terrestrial data produced by the IDGE flight apparatus prior to its turn over to KSC personnel on September 1, 1995. During the mission, LeRC and RPI personnel will work together to analyze the IDGE CCD camera dendrite images which are transmitted down to Earth in near real time, in order to replan the remaining portion of the mission.

After the flight, RPI personnel will analyze the resulting 35 mm photographs and other data to determine the dendrite tip velocity, as well as characterize the three-dimensional size and shape of the dendrite tip. This data will be used to assess the validity of various solidification theories which are currently being used to try to improve industrial metal production on Earth.

RPI personnel will publish reports and give papers at relevant conferences to disseminate the information to the scientific and industrial community.

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II. MSAD Program Tasks — Flight Research

Task Significance:

IDGE will study dendrites. Dendrites are tiny crystalline structures that form inside molten metals and metal alloys when they solidify. Nearly all industrially important metals solidify dendritically from the molten state. The dendrites formed inside metals during manufacturing result in microscopic zones of strength, weakness, ductility, and brittleness. Consequently, understanding precisely how dendrites form can lead to improvement of alloy strength and ductility by eliminating weak and brittle areas in the metal. Metal products can be improved, while lowering production costs.

During the past 50 years, numerous scientists have attempted to develop practical theoretical models to predict important dendritic growth parameters. However, years of experimentation on Earth has not produced a dataset capable of testing the models to find out which one, if any, is correct. This is due to the influence of gravity. On Earth, dendritic solidification can be strongly affected by gravitationally driven convective currents in the molten metal. These currents exist during virtually all metal solidification processes. However, their effect on dendritic solidification cannot be accurately be modeled without knowledge of dendritic solidification in the absence of such currents.

The microgravity environment of space effectively eliminates convection in the IDGE experiment. Extensive non-advocate peer review indicated that a comparison between space experiment data and Earth experiment data is the only practical way to separate the effects of convection from the underlying mechanism of dendritic solidification.

Ultimately, IDGE may result in the improved industrial manufacturing of steel, aluminum, super alloys, and other metals that are used on Earth every day. Moreover, the data returned from space will remain relevant indefinitely. IDGE data can be used to test current theories, as well as theories that will be developed perhaps far into the future. In fact, the Schriffer Committee report (1987) declared that the IDGE was one of NASA's four "world-class" microgravity science experiments due to the quality, scope, and long term relevance of its science.

Progress During FY 1995:

First Flight of IDGE

The Isothermal Dendritic Growth Experiment (IDGE) is a tele-operated microgravity science experiment payload that flew on the space shuttle Columbia in March of 1994. The first mission was an unqualified success. Analysis of the flight data revealed that current solidification theories need significant modification. Moreover, convection, both on Earth and in space, may have greater effect than previously believed. These findings are unexpected and immediately important to the science of solidification, and longer term, to industrial alloy production. Personnel at RPI have concluded all the analysis of the data from the first mission.

Second Flight of IDGE

RPI purified sample material and filled a growth chamber required for the second flight of IDGE. RPI analyzed terrestrial data produced by the IDGE flight apparatus prior to its turnover to KSC personnel on September 1, 1995.

Third Flight of IDGE

RPI personnel have continued to improve their techniques required to purify pivalic acid (PVA), the sample material to be used in the third flight. RPI now has enough PVA to fill several growth chambers.

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BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals


Proceedings


Presentations
II. MSAD Program Tasks — Flight Research

Discipline: Materials Science


II. MSAD Program Tasks — Flight Research

Thermophysical Properties of Metallic Glasses and Undercooled Alloys

Principal Investigator: Dr. William L. Johnson
California Institute of Technology

Co-Investigators:
Lee, D.
California Institute of Technology

Task Objective:
The objective is to study thermophysical properties of undercooled alloy melts and how they relate to glass formation. Toward this end, we have developed non-contact calorimetric methods to investigate the specific heat and thermal conductivity of these melts, both in the liquid and undercooled region. These quantities are essential for the development of newer, more advanced processing technologies for both existing and future materials.

Task Description:
Non-contact AC calorimetry was successfully demonstrated on the IML-2 flight in July, 1994. We obtained information on the specific heat and thermal conductivity of liquid and undercooled Zr_{55}Ni_{54} and Ni_{50}V_{15} melts using TEMPUS. This data is currently being analyzed to calculate entropy and free energy functions for these melts. We will compare these quantities to their values for the corresponding equilibrium and metastable crystals to compare the relative stability of the phases. Also, we will determine the Kauzman isentropic temperature of the alloys and compare it to the observed glass transition temperature.

In addition, the ground-based total radiance bolometer is currently being integrated onto a UHV levitation chamber for total hemispherical emissivity measurements. Measurement of temperature-dependent total hemispherical emissivity functions will allow us to unwind specific heat from undercooling data in an unambiguous manner.

Task Significance:
The non-contact AC calorimetry experiment is significant for many reasons. First, the thermodynamic properties of these advanced materials are a prerequisite to the development of processing technologies for them. Without knowledge of heat capacities and thermal conductivities, it is not possible to define, for example, how much power is needed to melt and cast the materials. In addition, the specific materials chosen for our experiment are the parent compounds for a new class of bulk metallic glasses that have recently been discovered by our group here at Caltech. By studying the properties of these parent compounds, we hope to better understand the bulk metallic glasses and how they form. These materials will revolutionize metallic processing technologies with their novel, superior properties. These materials can be engineered to be more ductile, slipperier, harder, lighter and more corrosion resistant than the typical materials used today. It is essential that the processing technologies for these materials be developed as quickly as possible and that, therefore, the thermophysical properties be measured.

Progress During FY 1995:
Flight data from IML-2 has been analyzed and the results have been both submitted for publication and presented at meetings - including an invited talk at the 1994 Gordon Conference on Microgravity Research and an invited talk and paper at the 4th Asian Thermophysical Properties Conference in Tokyo, Japan.

The technique is constantly being refined. Better filtering and analysis routines are being written and the phase difference between the RF coil current and temperature response is being analyzed for possible additional information on the thermal conductivity of the samples. Also, an impedance change technique for determining the electrical conductivity of the liquid sample is being developed.

Samples for reflight are being chosen and tested for vapor-pressure, melting point, undercoolability, etc. The ground-based support program for reflight from DLR has begun, as has TEMPUS refurbishment. Samples are also being prepared for the January 1996 KC-135 flight of TEMPUS.
In the ground-based program the total emissivity measurements are being refined. Low-noise pyrometers have been built and are in the process of being calibrated for accurate temperature measurement of RF-levitated samples. In an effort jointly undertaken with the Szekely group at MIT and Mike Robinson's group at MSFC, dynamic liquid drop viscosities of good glass-forming alloys are being measured in the drop tube at Marshall.

**Students Funded Under Research:**

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**Bibliographic Citations for FY 1995:**

**Journals**


**Proceedings**


**Presentations**

Orbital Processing of High Quality Cadmium Telluride

Principal Investigator: Dr. David J. Larson, Jr. State University of New York, Stony Brook

Co-Investigators:
- Dudley, M.
- Moosbrugger, J.
- Carlson, F.
- Alexander, J.
- Gillies, D.
- DiMarzio, D.

Task Objective:
The objective of this research is to investigate the influences of gravitationally-dependent phenomena (hyostatic and buoyant) on the growth and quality of doped and alloyed Cadmium-Zinc-Telluride (CdZnTe) crystals grown by the modified seeded Bridgman-Stockbarger technique. It is hypothesized that the damping of the gravitationally-dependent buoyancy convection will substantially enhance chemical homogeneity and the near-elimination of hydrostatic pressure will enable the significant reduction in defect density.

Task Description:
The primary objective of this program is to grow CdZnTe crystals in orbit (μ-g) for comparison with the best crystals grown terrestrially (1-g). Our comparative baseline includes crystals grown within the program and at the best facilities commercially. Our hypothesis suggested that crystals grown in μ-g could be grown under diffusion controlled growth conditions, due to the damping of gravitationally-dependent convective (buoyant) flows. Further, in the near-absence of hydrostatic pressure in μ-g, crystals could be grown virtually without melt/crystal/ampoulewall contact. It was hypothesized that this would significantly reduce the thermo-mechanical stresses experienced by the growing and cooling crystal and improve the defect density (twins and dislocations) of the resulting CdZnTe crystals.

As a critical part of this task, 2 crystals were grown in μ-g using the seeded Bridgman-Stockbarger growth technique on USML-1/STS-50 in 1992. and 2 additional crystals were processed on USML-2/STS-73 in October/November 1996. The seeded Bridgman-Stockbarger crystal growth technique, is accomplished by establishing isothermal hot zone and cold zone temperatures that bracket the solidification temperature (1095°C) with a uniform thermal gradient in between. After sample insertion into the Crystal Growth Furnace (CGF) the furnace's hot and cold zones are ramped to temperature, establishing a thermal gradient of 35°C/cm and melting the bulk of the sample. The furnace is then equilibrated and then is moved back towards the seed crystal, causing the bulk melt to come in contact with the seed crystal, thus seeding the melt. The seed crystal prescribes the growth orientation of the crystal grown. Having seeded the melt, the furnace is again equilibrated, the furnace translation is reversed, and the sample is solidified directionally by passing the thermal gradient down the length of the sample at a uniform velocity (1.6cm/h).

Task Significance:
The family of II-VI compound semiconductors, of which CdZnTe is a commercially significant member, is used in the fabrication of medium and long wavelength IR sensors and beta and gamma ray nuclear detectors. Orbital processing offers a unique opportunity to advance toward the goal of greatly increased structural perfection within bulk crystals of increased chemical homogeneity, by growing, characterizing, and testing paradigm material. Further, seeding technologies are being developed which will be useful to industry, and insight will be generated with respect to the generation of twins, which are pervasive terrestrially.
Progress During FY 1995:

Three (3) of the above crystals, two (2) on USML-1 and one (1) on USML-2, were grown successfully using a standard Bridgman-Stockbarger seeded growth configuration. The fourth sample was successfully processed on USML-2 in an identical manner thermally, except that the ampoule was designed to minimize wall contact between the growing and solidifying crystal and the growth ampoule. This maximized the volume of material which solidified without wall contact, regions which had been shown to minimize thermo-mechanical stresses experienced by the crystal during growth and post-solidification cooling. This experiment could only be attempted in μ-g where there is a near-absence of hydrostatic pressure on the bulk liquid.

It was found that the chemical homogeneity of the crystals grown in μ-g was outstanding longitudinally and radially. This suggested that gravitationally-dependent thermo-solutal convection had been damped and diffusion-controlled growth had been achieved, as anticipated. This result also suggests that in this alloy system surface driven convection is not a significant detriment to the attainment of diffusion-controlled growth conditions in microgravity.

The experiments also demonstrated that in the near-absence of hydrostatic pressure the non-wetting liquid separated from the ampoule walls as proposed, depending on influences including: volumetric fill-factor, level of constraint, residual g-vector, ampoule geometry and growth conditions. Regions solidified without wall contact were found to virtually eliminate twinning, suggesting that these pervasive defects terrestrially are surface nucleated. Further, these regions without wall contact showed dramatic reductions in (111)(110) dislocation density, from 800,000 (1-g) to 800 (μ-g) epd. This resulted from thermo-mechanical stress reduction within the flight samples during growth and post-solidification cooling. Regions of partial wall contact showed defect gradients, with high densities on the wall side and low densities on the free surface side. These results are consistent with our high-fidelity thermal and thermo-mechanical stress models.

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Task Initiation: 8/90 Expiration: 12/96
Project Identification: 963-21-08-02
NASA Contract No.: NAS8-39721
Responsible Center: MSFC

Printed: 28 FEB 1996
II-138
Crystal Growth of II-VI Semiconducting Alloys by Directional Solidification

Principal Investigator: Dr. Sandor L. Lehoczky
NASA Marshall Space Flight Center (MSFC)

Co-Investigators:
Szofran, F.
Su, C.-H.
Scripa, R.
Sha, Y.
NASA Marshall Space Flight Center (MSFC)
University of Alabama, Birmingham (UAB)
Universities Space Research Association (USRA)

Task Objective:
The objective of this research is to investigate the effects of reduced gravity on the crystal growth of mercury zinc telluride (HgZnTe) and mercury zinc selenide (HgZnSe) alloys with respect to their compositional, metallurgical, and optical properties.

Task Description:
The investigation includes both Bridgman-Stockbarger and solvent growth methods, as well as growth in a magnetic field. The alloys are prepared by reacting pure, elemental constituents in evacuated, sealed, fused-silica ampoules. The crystals are grown in a multizone furnace. The hot zone is heated above the liquidus temperature of the given alloy and the cold zone is maintained at lower temperatures to provide temperature gradient sufficient to prevent constitutional supercooling. Crystal growth is accomplished by slowly moving the ampoule from the hot zone to the cold zone of the furnace. The flight portion of the investigation is being performed by using the Crystal Growth Furnace (CGF). Preparation of the samples is being done in the Space Sciences Laboratory of Marshall Space Flight Center. Characterization and analysis of the samples after processing is being done primarily in the same laboratory with substantial microstructural analysis being done at the University of Alabama at Birmingham. Device fabrication and characterization will be done primarily at the Rockwell International Science Center.

Task Significance:
The anticipated results of this study will have both scientific and technological significance. The advancement in science will result from the increased understanding of the role of gravity on the fluid dynamic and compositional redistribution phenomena during the crystal growth of solid-solution semiconducting alloys having large separation between the liquidus and solidus of the constitutional phase diagrams, and from the more accurate values of material properties that can be measured using the high-quality, bulk crystals grown in space. Any advance in quality of these electronic materials has a great technological impact because of the application to infrared detectors for NASA and DOD requirements.

Progress During FY 1995:
A new seeded method has been developed for the growth of HgZnTe crystal ingots from pseudobinary melt by the Bridgman-Stockbarger type directional solidification. A vapor transport method developed by us was used to grow 2 cm ZnTe seed crystals in the fused silicon ampoules. Then a stack of precast pseudobinary alloys of varying compositions were loaded in the remaining ampoules.

The alloy compositions and their distribution were chosen to correspond to the expected melt composition variation along the growth axis for steady-state diffusion-controlled growth conditions. A series of Hg$_{0.48}$Zn$_{0.32}$Te and Hg$_{0.48}$Zn$_{0.12}$Te crystals were grown using the CGF Ground Control Experiment Laboratory (GCEL) furnace, as well as MSFC heat-pipe furnaces. Several crystals were also grown under the influence of a 5T axial magnetic field. Detailed compositional and microstructural characterization of the samples indicated that the alloy stacks could be successfully back-melted within 0.5 mm of the seed interface to assure nearly steady-state growth conditions. The
applied magnetic fields had a significant influence on radial alloy segregation and interface constitutional supercooling breakdown underlying the importance of gravity-induced fluid-flow effects.

Two Hg_{60.36}Zn_{12}Te seeded ampoules were prepared for processing in the CGF during the Second United States Microgravity Laboratory (USML-2) mission. The results from similar experiments that were inadvertently terminated during the previous USML-1 mission strongly indicated that residual accelerations to the growth axis are detrimental for achieving the primary experiment objectives. A Shuttle flight attitude that minimizes such accelerations was requested for the USML-2 mission. Just prior to launch the attitude was disallowed because of programmatic constraints and a decision was made not to perform the flight portion of the experiment under unfavorable growth conditions.

**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Journals**


**Proceedings**


II. MSAD Program Tasks — Flight Research  

Discipline: Materials Science


Presentations


II. MSAD Program Tasks — Flight Research

**Growth of Solid Solution Single Crystals**

| Principal Investigator: Dr. Sándor L. Lehoczky | NASA Marshall Space Flight Center (MSFC) |
| Co-Investigators:                          | NASA Marshall Space Flight Center (MSFC) |
| Szofran, F.                                | NASA Marshall Space Flight Center (MSFC) |
| Gillies, D.                                 | NASA Marshall Space Flight Center (MSFC) |

**Task Objective:**

The major objective of this research is to establish the limitations imposed by gravity during growth on the quality of bulk solid solution crystals having large separation between their liquidus and solidus temperatures. The important goal is to explore the possible advantages of growth in the absence of gravity.

**Task Description:**

The alloy system being investigated is Hg_{1-x}Cd_xTe with x-values appropriate for infrared detector applications in the 8 to 14 cm region. Both melt and Te-solvent growth methods as well as growth in magnetic fields are being considered. The study consists of flight experimentation and ground-based experimental and theoretical work needed to establish material properties and optimum experimental parameters for the on-going flight experiment and to assist material evaluation. Hg_{1-x}Cd_xTe is representative of several alloys which have electrical and optical properties that can be compositionally tuned to meet a wide range of technological applications in the areas of sensors and lasers with applications to optical computing and communications as well as the national defense. The investigation includes both Bridgman-Stockbarger and solvent growth methods, as well as growth in a magnetic field. The alloys are prepared by reacting pure, elemental constituents in evacuated, sealed, fused-silica ampoules. The crystals are grown in a multizone furnace. The hot zone is heated above the liquidus temperature of the given alloy and the cold zone is maintained at lower temperatures to provide temperature gradient sufficient to prevent constitutional supercooling. Crystal growth is accomplished by slowly moving the ampoule from the hot zone to the cold zone of the furnace.

The majority of the ground-based studies are being performed in the Space Sciences Laboratory of the George C. Marshall Space Flight Center. The flight portion of the investigation is being conducted using the Advanced Automatic Directional Solidification Furnace developed by the Marshall Space Flight Center and manifested for flights on the United States Microgravity Payload series of missions. The first flight of the instrument took place in March 1994.

**Task Significance:**

The anticipated results of this study will have both scientific and technological significance. The advancement in science will result from the increased understanding of the role of gravity on the fluid dynamic and compositional redistribution phenomena during the crystal growth of solid-solution semiconducting alloys having large separation between the liquidus and solidus of the constitutional phase diagrams, and from the more accurate values of materials properties that can be measured using the high-quality, bulk crystals grown in space. Any advance in quality of these electronic materials has a great technological impact because of the application to infrared detectors for NASA and DoD requirements.

**Progress During FY 1995:**

An approximately 16 cm long Hg_{1-x}Cd_xTe alloy crystal was successfully grown over a period of eleven days during the STS-62 Second United States Microgravity Payload (USMP-2) mission. Detailed microstructural and
compositional analysis has been performed for the crystal. A rate change inserted into the growth timeline sequence produced in the crystal an effective time marker for correlating orbital and residual accelerations to various crystal features and alloy compositional changes. This allowed a detailed evaluation of the effects of the magnitude and direction of residual acceleration on crystal homogeneity and perfection to be made for the first time. The rate change did not cause the propagation of new crystals. Circumferential variation of composition and topographic features around the boule indicated that residual acceleration vectors were present and having a considerable effect on the growth process. Preliminary x-ray topographs of the portion grown in the most favorable attitude (-XLV, -ZVV) indicate that this region is of significantly higher quality than usually grown on the ground. The OORE acceleration measurement instrument proved to be an accurate predictor of the direction of residual acceleration vectors, as evidenced by the surface features of the boule. The measured transient surface compositions indicate a lower effective mass transport coefficient than those observed in ground-based studies. Certain attitude maneuvers of the orbiter can dramatically affect the growth stability. This is illustrated by the roll-around in tail-down attitude which reversed the direction of the residual acceleration perpendicular to the interface and caused thick compositional striations at 11 cm along the crystal. Further microstructural, optical, and electrical characterizations of the crystal promise to provide a wealth of additional information on the growth in low earth orbit of solid solution alloy crystals having a large separation between their liquidus and solidus. A series of Hg0.5Cd0.5Te crystals were also grown under the influence of axial magnetic fields up to 5T. The application of the magnetic fields greatly reduced the radial compositional variations in the crystals, underlying the importance of gravitationally-induced fluid flows. The observed radial segregation agreed well with theoretically predicted results.

**Student Funded Under Research:**

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**Task Initiation:** 10/84  **Expiration:** 10/95

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

**Journals**


Mazuruk, K., Su, C.-H., Lehoczky, S.L., and Rosenberger, F. Novel oscillating cup viscometer - application to molten HgTe and Hg0.5Cd0.5Te. J. Appl. Phys., 77, 5098-5102 (1995).


**Proceedings**


II. MSAD Program Tasks — Flight Research

Diffusion Processes in Molten Semiconductors

Principal Investigator: Prof. David H. Matthisen
Case Western Reserve University

Co-Investigators:
Arnold, Dr. W.
Case Western Reserve University
Chait, Dr. A.
NASA Lewis Research Center (LeRC)
Dunbar, Dr. B.
NASA Johnson Space Center (JSC)
Stroud, Prof. D.
Ohio State University

Task Objective:
To provide purely diffusive experimental measurements of the isothermal diffusion coefficients of Ga, Sn and Sb in molten germanium with sufficient accuracy and precision to:

a) Differentiate between model predictions of the temperature dependence.
b) Determine the effect of dopant size and type.
c) Determine if a "wall effect" is present.
d) Provide input to continuum and atomistic model development.

To provide purely diffusive experimental measurements of the thermomigration diffusion coefficients of Ga, Sn and Sb in molten germanium with sufficient accuracy and precision to:

a) Determine the effect of dopant size and type.
b) Determine if a "wall effect" is present.
c) Provide input to continuum and atomistic model development.

to develop a 3-dimensional, fully time dependent continuum numerical model of the germanium diffusion column, shear cell, cartridge and furnace for both earth-based and space-based experiments which accurately predicts the measured concentration profile as a function of distance in the diffusion column.

To develop atomistic models which accurately predict:

a) The purely diffusive isothermal diffusion coefficient of a dopant in a molten semiconductor,
b) The temperature dependency of dopants in molten semiconductors, and which:
c) Attempts to explain the "wall effect."
d) Develops new empirical potentials useful for predicting other diffusion and transport properties for other molten semiconductor systems.

Task Description:
This program of study is directed at the fundamental and applied issues pertaining to diffusion of species in the liquid state as driven by concentration gradients (Fickian diffusion) and thermal gradients (Soret diffusion). The fundamental material systems of interest for near term study are the dilute binary systems of gallium (Ga), tin (Sn) and antimony (Sb) in germanium (Ge). Systems of commercial interest for future study include the dilute binary systems of dopants in gallium arsenide (GaAs). This research program consists of three major components: an experimental measurement portion, a continuum numerical simulation portion and an atomistic numerical simulation portion.

The experimental measurement portion is designed to provide definitive measurements of the purely diffusive component of mass transfer in molten semiconductor systems. A shear cell technique will be used to directly measure the diffusion coefficients in semiconductor melts. For the Fickian diffusion case, isothermal measurements will be used to determine the diffusion coefficients. An experimental matrix will be used to determine the dependence of the diffusion coefficients on temperature, dopant type and column diameter. For the Soret diffusion case, measurements will be made in a thermal gradient.
II. MSAD Program Tasks — Flight Research

Discipline: Materials Science

Task Significance:

The fundamental mechanisms of mass diffusion in the liquid state are still unclear to the degree necessary for the prediction of diffusion of one species into another or even within itself. This observation is especially true with respect to the dependency of diffusion mechanisms on temperature and on concentration levels, as well as on the dopant type. Present estimates of diffusivity in molten semiconductors can typically provide an order of magnitude estimate only, without any information on their dependency on concentration levels and types, and on temperature and temperature gradients.

The availability of these data is of paramount importance for practical reasons as well. The relevancy of numerical modeling for the analysis and design of ground based and space experiments is directly dependent upon the accuracy of the fundamental material properties used in these simulations. These data are also important for the correct characterization and interpretation of experimental results from ground based and space experiments.

The subject of how a mass of one species diffuses through a matrix of another is, at the same time, both a very old and very new research area. That this area can encompass the small, i.e., movement of electrons in a plasma, to the very large, i.e., the depletion of the global ozone layer, merely serves to emphasize the fundamental aspects of this subject. Most manufacturing technologies at some stage, rely on diffusion processes in the solid, liquid or gas.

The need for precise measurements of the diffusion coefficients in molten semiconductors has been repeatedly pointed out. These data are required both to interpret the experimental results from previous space-based (and Earth-based) experiments and also to optimize newly envisioned experiments. Difficulties in experimental techniques and theoretical interpretations are cited for the lack of these data. This is a comprehensive program which addresses both of these issues.

Progress During FY 1995:

- Quantified the level of mixing between adjacent shear cell segments due to the initial fluid-fluid shear.
- Numerically evaluated shearing rate.
- Numerically quantified the effect of convection on the measured diffusion coefficient.
- Established machining tolerances for shear cells.
- Numerically evaluated Soret diffusion.
- Numerically evaluated the use of an applied magnetic field to suppress convection.
- Quantified error bars to establish confidence level in the measured diffusion coefficient.

We are currently running diffusion experiments with Ga-doped Ge and Sb-doped Ge. We are evaluating the experimental data.

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Presentations

Printed: 28 FEB 1996
GaAs Crystal Growth Experiment

Principal Investigator: Prof. David H. Matthiesen
Case Western Reserve University

Co-Investigators:
Kafalas, Dr. J.A.
Ditchek, Dr. B.M.
GTE Laboratories, Inc. (retired)
GTE Laboratories, Inc.

Task Objective:
The objective is to determine the magnitude of effects of buoyancy-driven convection on the crystal growth of bulk gallium arsenide (GaAs).

Task Description:
Selenium-doped (~10⁻¹⁷) GaAs crystals are grown in controlled environments at selected environments affecting fluid flow as follows: (a) low-gravity (minimal convection), and (b) normal gravity in three separate orientations (vector stabilizing the temperature gradients, vector destabilizing the thermal gradient, and vector transverse to the thermal gradient), and a magnetically damped flow (the three normal-gravity orientations with either axial or radial magnetic field). The distribution of dopant is measured and compared to numerical predictions. Selected electrical and chemical properties are measured and correlated with the dopant distribution. Both macro- and micro-segregation are determined.

Task Significance:
Gallium arsenide (GaAs), an electronic material, has two principle advantages over silicon for producing solid state electronic "chip:" its ability to emit light, useful for making lasers, and its inherently high speed, useful in high-speed computers, communication satellites, etc. To fully exploit these characteristics, the material must be of the highest quality and be uniformly doped with traces of impurities. Typically, such uniformity is determined by convection in the molten material from which GaAs crystals are grown.

This materials processing experiment, part of a larger effort to better understand and control the crystal growth process, was undertaken to investigate the effects of buoyancy-driven convection on crystal growth.

Progress During FY 1995:
During late 1994 and 1995, an advanced Ph.D. candidate (Mr. Eugene Garza) was provided access to the flight samples in order to complete the secondary ion mass spectroscopy (SIMS) measurements of selenium distribution. He implemented improved techniques for these quantitative measurements on advanced mass spectrometers at the University of Illinois and at the Wright Patterson Materials Laboratory. The measurements were completed and the results summarized as part of Garza's thesis (thesis submitted in FY95 and degree to be granted in FY96). These results and correlated analyses of thermal and fluid transport (due to A. Chait at LeRC) will be summarized in the project final report and presented as part of a final coordinated characterization meeting at LeRC in Spring, 1996 with the Air Force Materials personnel and participants in the characterization program from LeRC.

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TASK INITIATION: 9/85  EXPIRATION: 1/96
PROJECT IDENTIFICATION: 963-21-05-02
NASA CONTRACT NO.: NAG3-1478
RESPONSIBLE CENTER: LeRC

Printed: 28 FEB 1996
II. MSAD Program Tasks — Flight Research

Discipline: Materials Science

The Study of Dopant Segregation Behavior During the Growth of GaAs in Microgravity

Principal Investigator: Prof. David H. Matthiesen
Case Western Reserve University

Co-Investigators:
Kafalas, J.
Carlsan, Dr. D.
Kaforey, Dr. M.

Viable Systems, Inc.
M/A Com Associates
Case Western Reserve University

Task Objective:
To design, fabricate, process, and analyze selenium-doped gallium arsenide crystals grown in the Crystal Growth Furnace (CGF) on the ground and during the Second United States Microgravity Laboratory mission (USML-2).

Task Description:
Selenium-doped gallium arsenide crystals will be grown in the CGF during ground-based experimentation and on board USML-2. Characterization of these crystals will include optical (infrared transmission, Fourier Transform Infrared Spectroscopy (FTIR), and Quantitative Infrared Imaging (QIR)), electrical (Hall effect, Capacitance-Voltage (C-V), spreading resistance, and Deep Level Transient Spectroscopy (DLTS)), and chemical (Secondary Ion Mass Spectroscopy (SIMS)) analysis. Experimental results will be compared with theoretical predictions.

Task Significance:
The Current Pulse Interface Demarcation (CPID) system on the CGF will be used to demark the melt-solid interface. These demarcation lines will show the interface shape and location at specific times during solidification. This should lead to an improved understanding of the axial and radial segregation contributions to the concentration distribution.

Progress During FY 1995:
During this task, the design of the sample/ampoule configuration was finalized. Samples were prepared and processed in the CGF on the ground. Characterization of these samples was started. Flight experimental timelines were designed based on analysis of the ground-based experiments. In addition, flight samples were prepared for processing in the CGF on the Second United States Microgravity Laboratory (USML-2).

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Task Initiation: 5/92
Expiration: 10/96

Project Identification: 963-35-05
NASA Contract No.: NAS8-39722
Responsible Center: MSFC

Printed: 28 FEB 1996
II. MSAD Program Tasks — Flight Research

Temperature Dependence of Diffusivities in Liquid Metals

Principal Investigator: Prof. Franz E. Rosenberger
University of Alabama, Huntsville

Co-Investigators:
Banish, R. M.
University of Alabama, Huntsville

Task Objective:
This research aims at advancing the understanding of diffusion mechanisms in liquid metals and alloys through accurate diffusivity measurements over a wide range of temperatures, including the proximity to the materials' melting points.

Task Description:
Toward the task objective we are pursuing the following tasks:

- Development of an efficient technique for dynamic in situ measurements of diffusivities in melts as a function of temperature;
- Development of a flight-certified hardware package to automatically perform such diffusivity measurements under reduced gravity conditions and on Earth;
- Investigation of the significance of the "wall effect" in diffusion capillaries;
- Exploration of convective contamination of the diffusivity measurements on Earth through numerical modeling;
- Exploration of the possibility to simulate low gravity diffusion conditions in conducting liquids on Earth through the application of magnetic fields; and,
- Measurements of diffusivities of selected materials that will be chosen according to class-like molecular interaction behavior in the liquid.

Diffusivities will be determined from temporal records of evolving concentration profiles through multi-detector measurements of radioactive tracer emission. An initially solid, cylindrical sample contains a radioactive isotope at one end. After melting, radiation escaping through small bores in an isothermal liner/radiation shield is monitored via a chain of detectors. Data evaluation is facilitated by a novel algorithm, which is not limited to the simple initial conditions traditionally used in diffusivity studies. The algorithm permits data deduction from any sequence of concentration distributions. Hence, diffusivity data can be gathered over a range of temperatures in a single experiment. Utilizing the different radiation absorption behavior of different photon energies, we will investigate the significance of the "wall" effect. This effect is currently believed to contaminate diffusion studies in narrow capillaries used to suppress convection at normal gravity.

Task Significance:
The diffusion of species in melts and its temperature dependence is important for the product quality in numerous metallurgical and semiconductor manufacturing processes. Hence, a detailed understanding of diffusion in liquid metals and alloys is essential for an efficient improvement of numerous technological processes. Neither accurate measurements nor reliable theories of diffusion in liquids are available at this point. The uncertainties in the experimental values are due to difficult-to-control transport contributions from convection. The complex structure if liquids has resulted in an abundance of theoretical models of liquid diffusion. This investigation will provide accurate data for selected materials over wide temperature ranges that can be used for both process development and verification of theoretical models.

Progress During FY 1995:
- Development and construction of a low temperature (200° C) version of the our liquid metal diffusion experiment in response to an immediate flight opportunity utilizing the Microgravity Isolation Mount (MIM) aboard Mir.
II. MSAD Program Tasks — Flight Research

Discipline: Materials Science

Measurements to be conducted under defined reduced accelerations will provide data for comparison with our numerical modeling efforts and guidance for the later high temperature experiments.

- Development of solid state diffusion bonding between radioactive and nonradioactive sample sections, and of a spring-loading the whole diffusion sample. These measures will mitigate the formation of voids and bubbles during the liquid diffusion experiment.
- Redesign of the radiation shield/isothermal liner to increase the signal-to-noise ratio and, thus, to decrease the specific activity required for the experiments. As a consequence both the flight experiment timeline and radiation safety issues became less critical.
- Verification of various experiment design assumptions through tracer diffusion experiments in both solid and liquid samples.
- Numerical modeling of the time-dependent and three-dimensional diffusive-convective transport in typical experiment geometries with realistic boundary conditions. In particular, the results show that the magnetic field strengths required to assure dominantly diffusive transport at unit gravity are so high that the diffusivity itself would be modified.

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Printed: 28 FEB 1996
Particle Engulfment and Pushing by Solidifying Interfaces

Principal Investigator: Dr. Doru M. Stefanescu
University of Alabama, Tuscaloosa

Co-Investigators:
Curren, P.
NASA Marshall Space Flight Center (MSFC)

Task Objective:
The primary objective of this task is to further develop the existing understanding on pushing and engulfment of particles by planar liquid/solid interfaces during solidification of metallic alloys.

The approach towards achieving this objective is by developing an analytical and then a numerical model describing this phenomenon, with a parallel experimental validation effort. The numerical model is required in order to include the effect of convection in the liquid on interface morphology and the particle/interface interaction. The interaction between an insoluble ceramic particle and a liquid-solid interface during solidification is significantly influenced by gravitational acceleration. Since the interaction between an insoluble ceramic particle and a liquid-solid interface during solidification is significantly influenced by gravitational acceleration, microgravity experiments will be performed to validate these models and to further contribute to the experimental database for metals/ceramic particulate mixtures.

Task Description:
To acquire further insight in the physics of particle behavior, directional solidification experiments with transparent organic materials doped with polystyrene particles were scheduled. It was planned to use results from these experiments to validate the analytical model developed so far.

Ground-based experiments under controlled solidification conditions with Al-SiC systems to document pushing/engulfment phenomena in metallic systems were also scheduled. In addition several tasks required in the flight requirements definition effort were to be performed.

Task Significance:
It is of fundamental and practical importance to understand and control particle behavior at the solid-liquid interface during solidification. Particle behavior determines the uniformity of their distribution in the matrix. Uniformity of particle distribution is of great significance as it dictates the mechanical and physical properties of the composites.

Introduction of insoluble ceramic particles in a metal matrix primarily involves three stages: transfer of particles from gas to liquid, interaction of particles in the liquid state and finally transfer of particles from liquid to solid. It is the last stage that is the most important and yet the least understood since it is the outcome of the interaction of numerous solidification variables. The anticipated results from this research program will provide a much better understanding of this stage of metal matrix composite processing and therein lies its significance.

Progress During FY 1995:
During FY95 ground experiments were conducted to formulate the particle pushing experiments to be done on the Shuttle flight. It was found that SiC particles used were non-spherical and also, if their size was small, they tended to agglomerate. Therefore, for further experiments spherical zirconia particles of larger diameters (500 μm dia.) were chosen.

In order to calibrate the actual melt interface velocities with respect to the furnace translation velocities two approaches were used. In one case, the distance between the initial melt interface and the quench interface was divided by the time of translation to obtain the actual average interface velocities. In the other case, Al-Ni eutectic
matrix samples were run at different velocities and the interlamellar spacings obtained were calibrated against the furnace translation velocities.

The samples have been examined under x-ray microscopy before running to locate the position of the zirconia particles. This is a new technique developed at the Marshall Space Flight Center. Similarly, the samples were again analyzed under the x-ray microscopy after running at different translation velocities. The new location of particles provided information whether the particles were pushed or engulfed at those translation rates. This demonstrated the utility of this new non-destructive testing method of characterizing the location of the particles accurately. Concurrent work went on during this period to investigate the critical velocities of particle pushing in the pure Al-zirconia and Al-Ni-zirconia samples. The results so far had clearly demonstrated the effect of convection on the particle pushing phenomena.

Thermal tests were performed on the samples incorporated in the cartridge in the AGHF furnace in France. The results of the tests have shown that the method of testing samples with respect to their design and translation behavior is satisfactory and that the samples can be successfully run in the similar furnace aboard the Shuttle.

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BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals
Crystal Growth of ZnSe and Related Ternary Compound Semiconductors by Physical Vapor Transport

Principal Investigator: Dr. Ching-Hua Su

Co-Investigators:

Brebrick, R.  Marquette University
Volz, M.  NASA Marshall Space Flight Center (MSFC)
Sha, Y.  Universities Space Research Association (USRA)
Noever, D.  NASA Marshall Space Flight Center (MSFC)
Sanghanitra, S.  Santa Barbara Research Center
Johnson, S.  Santa Barbara Research Center

Task Objective:
The investigation consists of extensive ground-based experimental and theoretical research efforts and concurrent flight experimentation. The objectives of the ground-based studies are to obtain the experimental data and conduct the analyses required to define the optimum growth parameters for the flight experiments, perfect various characterization techniques to establish the standard procedure for material characterization and quantitatively establish the characteristics of the crystals grown on Earth as a basis for subsequent comparative evaluations of the crystals grown in a low-gravity environment, and develop theoretical and analytical methods required for such evaluations.

Task Description:
The crystal growth experiment will use a novel vapor transport three-thermal-zone heater translating method. The Crystal Growth Furnace (CGF) or Advanced Automated Directional Solidification Furnace (AADSF) will be ideal for this experiment because they provide two high-temperature end zones and a booster heater at the center of the furnace with translation capability. Using this technique, large single crystals of CdS, CdTe, PbSe, and ZnTe have been grown successfully in this laboratory.

Task Significance:
The materials to be investigated are ZnSe and related ternary semiconducting alloys, e.g., ZnS, Se, ZnSe, Te, and Zn, Cd, Se. These materials are useful for opto-electronic applications such as high efficient light emitting diodes and low power threshold and high temperature lasers in the blue-green region of the visible spectrum. The recent demonstration of its optical bistable properties also makes ZnSe a possible candidate material for digital optical computers. Compositional non-uniformity, microstructural crystal defects (e.g., dislocations, small-angle grain boundaries, and second phase precipitates), and deviation from stoichiometry can seriously limit state-of-the-art device performance and future device applications. The reduction of gravity-driven convective fluid flows in a low-gravity environment is expected to be advantageous in minimizing these compositional variations and structural defects.

Progress During FY 1995:
1. Mass flux of ZnSeTe and ZnSeS was measured and the results were compared to theoretical calculations.
2. Horizontal and vertical growth of ZnSe, ZnSeTe, and ZnSeS were performed.
3. The grown crystals were assessed by characterization methods established earlier.
II. MSAD Program Tasks — Flight Research

Discipline: Materials Science

STUDENTS FUNDED UNDER RESEARCH:  TASK INITIATION:  2/93  EXPIRATION:  2/96
PROJECT IDENTIFICATION:  963-35-23
NASA CONTRACT NO.:  NAS8-39718
RESPONSIBLE CENTER:  MSFC

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals


Presentations


Measurement of Viscosity and Surface Tension of Undercooled Melts

Principal Investigator: Dr. Julian Szekely  
Massachusetts Institute of Technology (MIT)

Co-Investigators:
Hyers, R.  
Massachusetts Institute of Technology (MIT)

Task Objective:
The objective of this investigation is to utilize the electromagnetic levitation unit, TEMPUS, on IML-2 to measure the viscosity and surface tension of undercooled metallic melts. To date, little study has been made of the thermophysical properties of undercooled melts, and a controversy exists over whether the temperature dependence of the viscosity obeys an Arrhenium-type or a power-law relationship.

Task Description:
In this investigation, a "squeezing" force will be applied to a suitably-positioned sample to induce oscillations. The rate of decay of the amplitude of these oscillations will be observed in order to measure the viscosity at a number of temperatures in the undercooled regime, while the frequencies of the oscillation modes will be used to deduce the surface tension at these temperatures.

Our effort consists largely of a comprehensive program of mathematical modeling designed to give a detailed understanding of what can be expected from the flight experiment. To date, the main thrust of the modeling work has been to develop the methodology and to perform calculations predicting the behavior of levitation-melted/electromagnetically-positioned metallic droplets under both Earthbound and microgravity conditions.

Task Significance:
The main purpose of the work was to be able to predict the electromagnetic forces and heating rates, electromagnetically-driven velocity fields within the sample, the transient behavior of the system, and the deformation of the sample. The accuracy of the computational models has been checked by comparison with available analytical results and the results of ground-based experiments.

Progress During FY 1995:
Further analysis of the IML-2 mission data was carried out. These data were collected during the flight in July 1994. Surface tension data were presented for Au, AuCu, and ZrNi. This data provides experimental support for theories about ground-based electromagnetic levitation that cannot be obtained except in microgravity.

Viscosity data was not as expected. New theories were developed and tested against numerical models in an attempt to determine the cause of the enhanced viscosity observed. The leading theory is that the enhanced viscosity was caused by turbulent or transitional flows in the liquid sample. This investigation has led to planned experiments, which will be carried out under support related to the MSL-1 mission (April 1997) to define the conditions, in terms of process variables, under which the flow in the molten sample ceases to be simple laminar flow and becomes turbulent or transitional.

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Printed: 28 FEB 1996
BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals

Proceedings

II. MSAD Program Tasks — Flight Research

Discipline: Materials Science

Test of Magnetic Damping of Convective Flows in Microgravity

Principal Investigator: Dr. Frank R. Szofran

Co-Investigators:
- Cobb, S.D.
- Robinson, M.B.
- Volz, M.P.
- Motakef, S.

NASA Marshall Space Flight Center (MSFC)
Computer Assisted Process Engineering (CAPE)

Task Objective:
The objectives of this study are: to test experimentally the validity of the modeling predictions applicable to the magnetic damping of convective flows in conductive melts as this applies to the directional solidification of semiconductor and metallic materials in the reduced gravity levels available in low Earth orbit; and to assess the effectiveness of magnetic fields in reducing the fluid flows occurring in these materials during space processing that result from density gradients (driven by the residual steady-state acceleration or g-jitter) or surface tension gradients (Marangoni flow). To achieve these fundamental objectives, the following specific objectives will be pursued:

- To determine the relative effectiveness of an axial magnetic field in suppressing convective flows in 1g driven by gravity, vibration, or surface tension gradients;
- To test the validity of magnetohydrodynamic modeling predictions in characterizing the effectiveness of an axial magnetic field for suppressing convective flows in 1-g.

Task Description:
To achieve the objectives of this investigation, we will carry out a comprehensive ground-based program using a carefully chosen set of materials. Some of these materials have been intensely studied in environments that have not simultaneously included both low gravity and an applied magnetic field. These include a dilute alloy (Ga-doped Ge) in which solutal effects will be negligible and three solid solutions (Ge-Si, InSb-GaSb, and Cu-Ni) with liquid density ratios of 2.18, 1.07, and 1.012, respectively. Thus, during Bridgman-Stockbarger solidification with the solid on the bottom, Ge-Si has a strongly stabilizing solutal density variation, InGaSb is very mildly stabilizing with previous results showing substantial mixing, and Cu-Ni is even less stabilizing. All four systems will be processed by the Bridgman method using two diameters. In addition, the Ga-doped Ge and Ge-Si systems will be float-zoned to study the effects of magnetic suppression of Marangoni convection.

Task Significance:
During directional solidification of semiconductors, generation of destabilizing temperature gradients in the melt is unavoidable, resulting in buoyancy-induced convective mixing of the liquid phase. On Earth this convective mixing is generally very intensive and interferes with segregation of melt constituents at the growth front leading to less than optimum quality crystals. Crystal growth in space provides the opportunity to reduce the convective intensity and, for some classes of systems and charge sizes, achieve mass transfer diffusion-controlled growth. Magnetic damping of convection in electrically-conductive melts, however, can be used to provide a higher degree of control on convection in the melt. Thus our understanding of convective influences can be further advanced, and our ability to interpret space experimental results may be significantly improved.

Progress During FY 1995:
During the past year, experimental work has included Bridgman and floating-zone growth experiments and numerical modeling of the Bridgman experiments has continued. The floating-zone experiments were done with silicon.
samples and were carried out in collaboration with the Kristallographisches Institut (KI) of the University of Freiburg, Germany under a collaborative agreement between KI and NASA.

Significant accomplishments of the project include:

- Spreading resistance measurements confirmed earlier four-point probe results that diffusion controlled growth has been obtained in gallium-doped germanium.
- Experimental results confirmed the accuracy of the numerical model for predicting the magnetic field needed to obtain diffusion controlled growth in gallium-doped germanium.
- Problems of irregular growth rate and bubble formation on the outside surfaces of the germanium samples were eliminated by using boron nitride sample containers.
- A vacuum or inert gas growth cartridge that does not use fused silica was designed and built to permit current pulse demarcation experiments in germanium and germanium-silicon alloy samples.
- Silicon samples, including some which were coated to eliminate Marangoni convection and some which were pill doped, were grown by float zoning in fields up to 4 Tesla.
- At 3 Tesla, the float-zoned samples followed a diffusion-controlled profile but analysis of the pill-doped samples showed that there was still more convection than in reduced gravity with zero field.

**STUDENTS FUNDED UNDER RESEARCH:**

**TASK INITIATION:** 10/92  **EXPIRATION:** 10/95  
**PROJECT IDENTIFICATION:** 963-35-24  
**RESPONSIBLE CENTER:** MSFC

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**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Journals**

**Presentations**


II. MSAD Program Tasks — Flight Research

Coarsening in Solid-Liquid Mixtures

Principal Investigator: Prof. Peter W. Voorhees
Northwestern University

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
The objective of this project is to plan and perform a microgravity experiment on Ostwald ripening in solid-liquid mixtures. This experiment will serve two primary purposes.

1. It will allow experiments to be performed which can be directly compared to heretofore untested theories for coarsening in systems with low volume fractions of solid.

2. It will eliminate conclusively convection of the liquid matrix and small-scale particle motion within the skeletal structure as possible sources for the disagreement observed between theory and experiment in the high volume fraction experiments.

Task Description:
Previous NASA sponsored work clearly showed that solid-liquid mixtures consisting of Sn-rich particles in a Pb-Sn eutectic liquid are ideal, and perhaps unique, systems in which to explore the dynamics of the Ostwald ripening process. The high coarsening rate in these systems permit accurate kinetic data to be obtained, and the thermophysical parameters necessary to make a comparison between theory and experiment are known. However, in a terrestrial environment experiments can be performed only at the relatively high volume fractions of solid where the presence of a solid skeletal structure prevents large-scale particle sedimentation. In these high volume fraction experiments, a comparison between theory and experiment shows that the solid-liquid mixtures are coarsening faster than predicted by an approximate theory for purely diffusional controlled Ostwald ripening. Thus, the we are in the process of formulating a microgravity experiment. This experiment will serve two primary purposes: it will allow experiments to be performed which can be directly compared to heretofore untested theories for coarsening in systems with low volume fractions of solid, and it will eliminate conclusively convection of the liquid matrix and small-scale particle motion within the skeletal structure as possible sources for the disagreement observed between theory and experiment in the high volume fraction experiments.

Task Significance:
The spaceflight experiment will produce data which, for the first time, can be compared directly to theory with no adjustable parameters. This data will address the long standing controversy over the dependence of the coarsening rate of a two-phase system of the volume fraction on coarsening phase.

The results from the spaceflight experiment will further our understanding of the processes responsible for microstructural development in a broad range of commercially important materials ranging from turbine blade alloys to liquid phase sintered materials.

Progress During FY 1995:
We have finalized the science requirements for the Coarsening in Solid-Liquid Mixtures spaceflight experiment. We have also interacted extensively with NASA engineers in the design of a furnace for the spaceflight experiment. Our specimen preparation procedures have been refined and improved. We can now produce spatially uniform mixtures of Sn particles immersed in a Sn-Pb eutectic liquid at a variety of volume fractions of solid. The spatial uniformity of these mixtures was confirmed, even in low volume fraction mixtures, through short-time coarsening experiments. Our image analysis programs and system have been improved in preparation for the analysis of the spaceflight specimens.

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We have developed an automated serial sectioning machine. It consists of a micromiller and metallurgical microscope. The images from the microscope are digitized using a frame grabber. We can control the thickness between each serial section to two microns. The entire process is quite efficient; we can acquire approximately eight sections per hour. These digitized images are then used to reconstruct the three-dimensional image of the microstructure using a computer. This three-dimensional image can then be manipulated to analyze the morphology of the solid-liquid mixture. Preliminary measurements show clearly the interconnected nature of the solid-liquid mixture.

Terrestrial-based experiments using Pb-Sn solid-liquid mixtures have shown that grain boundaries occasionally form between particles. In an effort to quantify the nature of these grain boundaries, and hence the morphology of these solid-liquid mixtures, for comparison with the results of the spaceflight experiment, we are in the process of measuring the orientations of grain boundaries using a scanning electron microscope. This microscope will allow us to determine the orientation of many grain boundaries and thus we will be able to examine the temporal evolution of the distribution of grain boundary orientations as well as the dependence of the distribution on the volume fraction of solid. We have acquired high quality Kikuchi patterns from the tetragonal Sn particles and are in the process of examining the distribution of grain boundary orientations.

Finally, we have examined the effects of ternary alloy additions on the kinetics of Ostwald ripening. The objective of this work was to determine how the addition of a third alloying element, which could be employed to match the density of the solid and liquid phases, would change the coarsening kinetics of the two-phase mixture. The analysis is valid for a general, nonideal, nondilute solution, but is limited to extremely small volume fractions of coarsening phase and neglects off-diagonal terms in the diffusion matrix. The Gibbs-Thompson equation in a ternary system undergoing coarsening reveals that the concentrations at the particle-matrix interface are dependent on the far-field supersaturations as well as the particle radius. An asymptotic analysis shows that the exponents of the temporal power laws for the average particle radius, number of particles per unit volume and matrix supersaturations are the same as that found in the binary limit, however, the amplitudes of the power laws are modified. We find that the trajectory of the matrix supersaturation must lie along a tie line, but the trajectory of the average particle composition does not. Thus, the density difference between the two phases will be a function of time and matching the density of the two phases by the addition of a third element is nearly impossible during Ostwald ripening.

**Students Funded Under Research:**

**Task Initiation:** 1/92  **Expiration:** 10/95

**Project Identification:** 963-25-05-10  
**NASA Contract No.:** NAG3-1417  
**Responsible Center:** LeRC

**Bibliographic Citations for FY 1995:**

**Journals**


Vapor Growth of Alloy-Type Semiconductor Crystals

Principal Investigator: Dr. Heribert Wiedemeier
Rensselaer Polytechnic Institute

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
The objectives of this research are: (1) the establishment of experimental trends for the relation between convective flow, mass flux, and crystal morphology; and, (2) the identification of microgravity effects and crystal properties for the ternary semiconductor mercury cadmium telluride (HgCdTe).

For this purpose, thin epitaxial layers of Hg$_{1-x}$Cd$_x$Te will be grown on (100)CdTe substrates during the USML-2 mission to observe the effects of microgravity on the morphology of the substrate-layer interface and of the epitaxial layer.

Task Description:
This experiment requires the hot zone to be 625°C and the cold zone to be 455°C. The total duration of the experiment is 16 hours. The ampoule assembly is designed to be 160 mm in length, 18 mm outer diameter, and about 31 grams total weight. A cadmium telluride single crystal and a sapphire disc are used for the epitaxial crystal growth as substrate and substrate support, respectively. Four time intervals are required for crystal growth, namely, heat-up, annealing, growth, and cool-down periods.

Identical experiments, except for the level of gravity, are performed on ground and in space to provide a direct comparison of results.

Task Significance:
The lateral and axial compositional homogeneity (distribution) of the major and dopant components is expected to be more uniform for the space-grown epitaxial layers. The density of dislocations, of strain-induced defects, and possibly the number of inclusions are expected to be considerably reduced relative to ground-control specimens.

In addition, observations of the effects of reduced gravity on the formation of defects at the growth interface and on the propagation of these "birth defects" into the layer are of basic scientific and technological significance.

Progress During FY 1995:
Ground-based test experiments were continued in the PI's laboratory to confirm the transient times for island and layer growth of Hg$_{1-x}$Cd$_x$Te on (100)CdTe substrates under vertical, stabilizing conditions. The combined results of these and of corresponding GCEL tests were used to establish the final time line for the USML-2 experiments.

In addition to the flight, flight back-up, and flight spare sample-ampoule assemblies, six assemblies were prepared and delivered to MSFC for vibration and qualification testing.

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Wiedemeier, H., Ge, Y.R., Hutchins, M.A., and Sha, Y.G. Growth of Hg$_{x}$Cd$_{1-x}$Te epitaxial layers on (100)CdTe by chemical vapor transport under normal and reduced gravity conditions. J. Crystal Growth, 146, 610 (1995).
The Use of Bioactive Glass Particles as Microcarriers in Microgravity Environment

Principal Investigator: Prof. Portonovo S. Ayyaswamy
University of Pennsylvania

Co-Investigators:
Ducheyne, P.
University of Pennsylvania

Task Objective:
Our ultimate objective is to find optimal operating conditions which would produce the best three-dimensional tissue culture by using bioactive glass materials as microcarriers in a microgravity environment. This requires the determination of desirable characteristics of the bioactive beads, chemical make-up and control of the fluid medium in the microgravity environment (for example, a rotating-wall vessel), and actual operating conditions (for example, the rotational speed of the vessel, etc.). This undertaking involves both numerical and experimental aspects.

During the first year of the four year grant, preliminary studies of the fluid motion and forces on the bioactive glass particles caused by rotation of the vessel will be carried out. Actual extent of sedimentation of the particles as a result of vessel operation and the diffusive mass transport of different chemical species in the fluid medium will be numerically simulated. The most beneficial chemical composition of the fluid and the properties of this optimum fluid will also be experimentally investigated at the University of Pennsylvania laboratory.

Task Description:
During the first year of the four-year grant, the numerical aspects of the investigation will comprise of the following: a) Development of a numerical scheme to incorporate the effects of rotation. b) Appropriate modifications of governing equations and boundary conditions. c) Checking the convergence by analyzing situations of no rotation. d) Continuation of unit gravity study to develop confidence in the numerical procedure by comparison of results obtained with those available in published literature.

The experimental aspects to be studies are: a) Preparation of bioactive glass samples. b) Preparation of tissue culture. c) Familiarization with rotating vessels. d) Investigation of various rotating vessel parameters.

Task Significance:
It is anticipated that the numerical and experimental studies will ultimately lead to an improved understanding of the use of bioactive use particles in conjunction with the rotating-wall vessel. The use and production of bioactive glass particles and the use of the rotating-wall vessel for tissue culturing in microgravity conditions will in turn lead to an improved three-dimensional tissue culture technique.

Progress During FY 1995:
There is not much progress to report at this time as the project funding began in September 1995. However, the governing equations, the appropriate boundary conditions, and initial conditions for rotating fluid flow/mass transfer problems have been developed. These are being scaled to determine the importance of the various terms. Various designs of rotating-wall vessels are being considered for use in experiments.

Students Funded Under Research: BS Students: 0, MS Students: 0, PhD Students: 2

Task Initiation: 7/95 Expiration: 7/99
Project Identification: 962-23-01-23
NASA Contract No.: NAG9-817
Responsible Center: JSC
Evaluation of Ovarian Tumor Cell Growth and Gene Expression

Principal Investigator: Jeanne L. Becker, Ph.D.  University of South Florida

Co-Investigators:
Spaulding, G.F.  NASA Johnson Space Center (JSC)
Widen, R.H.  University of South Florida (Tampa Gen. Hospital)

Task Objective:
To examine growth and gene expression of LN1 ovarian tumor cells cultured in the NASA Rotating-Wall Vessel (RWV).

Task Description:
To develop an in vitro model for the growth of ovarian tumor cells under conditions which simulate the growth of these tumors in vivo, e.g., the development of complex multicellular aggregates. To apply the knowledge gained from this model towards the development of models to study other hormonally responsive gynecologic tumors.

Task Significance:
Reliable in vitro models to study human gynecologic cancer have been limited by the ability to maintain certain vital tumor cell characteristics during extended growth in tissue culture. Using the NASA RWV, we have established such a model for ovarian cancer using the LN1 human ovarian tumor cell line. During three-dimensional growth in the RWV, these tumor cells express characteristics of the original patient tumor specimens from which LN1 was derived, including the generation of multiple types of cells of varying lineages (epithelial and mesenchymal), as evidenced by immunohistochemical analysis. Moreover, we have observed changing patterns of gene expression during extended culture of LN1 cells in the RWV, as determined by reverse transcription polymerase chain reaction amplification. Because ovarian cancer is a hormonally responsive cancer, studies have been initiated to examine the effects of hormonal modulation on the growth of LN1. Furthermore, the RWV model has applied to the growth of endometrial tumor cells, another hormone dependent cancer which is exceedingly difficult to maintain for an extended time period in vitro.

Progress During FY 1995:
The sensitivity of LN1 to estrogen was examined as a function of two versus three-dimensional growth. Grown as both a monolayer and as three-dimensional aggregates in the RWV, LN1 exhibits expression of mRNA for estrogen receptor. However, monoclonal antibody studies showed that LN1 cells fail to stain positively for estrogen receptor protein. These results suggest the possibility of a post-transcriptional defect or, alternatively, that the estrogen receptor protein expressed by these cells is altered in some way such that it is not recognized by the monoclonal antibodies used for detection.

In order to determine whether LN1 cells were responsive to estrogen, cells were grown as monolayers or in the RWV in the presence of physiologic (1 nM) versus pharmacologic (1 uM) concentrations or 17-beta estradiol. Exposure to 1 nM 17-beta estradiol yielded no alterations in cell growth under either growth configuration. Exposure to 1 uM steroid induced a slight reduction in the rate of cell growth of LN1 grown as a monolayer. In contrast, cell growth was entirely inhibited when LN1 was cultured in the RWV, in the presence of 1 uM 17-beta estradiol, with cell death occurring in cultures grown 7 days with this concentration of steroid. Furthermore, LN1 cells grown in the RWV for up to 5 days with the estradiol could not be rescued from subsequent death by replenishing cultures with fresh media lacking the steroid. The mechanisms of these effects occurring as a function of two-dimensional versus three-dimensional growth are presently being examined, since non-receptor mediated effects of estrogen have been documented in the literature.
Studies have been initiated to establish a model of endometrial carcinoma growth in the RWV. For these experiments, the growth of the Ishikawa human endometrial carcinoma cell line has been examined. Because endometrial carcinoma is generally estrogen and progesterone receptor positive, Ishikawa cells represent a good model for this tumor since these cells exhibit both estrogen and progesterone receptor expression. Cultured as a monolayer or in the RWV, Ishikawa cells were appropriately growth stimulated in the presence of either 1 nM or 1 uM 17-beta estradiol, as has been well documented in the literature.

Additional work using this model has been aimed at investigating potential mechanisms for the development of chemotherapeutic resistance. For these studies, we have examined regulation of the expression of the multiple drug resistant gene (MDR1). This gene has particular relevance in endometrial carcinoma, since progesterone is known to upregulate MDR1 expression in normal secretory and gestational endometrium. The MDR1 gene product, p-glycoprotein, is a transmembrane pump which acts to remove toxic agents, including certain classes of chemotherapeutic drugs. P-glycoprotein (Pgp) is believed to function as a detoxifying protein in preventing the build up of toxic material in the endometrium during the time of embryo implantation and subsequent pregnancy.

When cultured as monolayer or in the RWV, Ishikawa cells show expression of mRNA and protein for MDR1. Interestingly, however, 17-beta estradiol inhibited the expression of MDR1 (both mRNA and protein) in cells cultured as a monolayer. This effort did not occur in cells cultured as three-dimensional aggregates in the RWV. Ishikawa cells cultured in the RWV in the presence of either 1 nM or 1 uM of the steroid both exhibited MDR1 mRNA and Pgp. Recent studies have demonstrated that the acquisition of drug resistance in tumor cells is facilitated by growth as three-dimensional spheroids. We, therefore, postulate that MDR1 expression can be regulated as a function of three-dimensional growth in the RWV, and additional work is aimed at investigating potential mechanisms involved in this effect.

**Students Funded Under Research:**

| BS Students: | 0 |
| MS Students: | 0 |
| PhD Students: | 1 |

**Task Initiation:** 11/92  **Expiration:** 11/95

**Project Identification:** 962-23-01-15  **NASA Contract No.:** NAG-648  **Responsible Center:** JSC

**Bibliographic Citations for FY 1995:**

**Journals**


**Presentations**


Expansion and Differentiation of Cells in Three Dimensional Matrices Mimicking Physiological Environments

Principal Investigator: Prof. Rajendra S. Bhatnagar
University of California, San Francisco

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
Our overall objective is to develop 3-D culture systems for mammalian cell cultures based on materials that mimic type I collagen, a major component of the extracellular matrix (ECM). We have identified a potent cell binding domain of type I collagen. Synthetic compounds based on this domain, as well as several conformational analogue peptides, exhibit many of the cell directed activities of collagen including the ability to attach cells and to promote their migration into several 3-D matrices. In this environment, cells exhibit remarkable differentiated behavior.

Objectives for FY 95:

1) Preparation of Culture Matrices: Design and synthesize 3-D matrices containing analogues of a cell binding domain of collagen.

2) Suitability for 3-D Cell Culture: Demonstrate attachment, migration and proliferation in these matrices, of (i) primary cultures of neonatal human dermal fibroblasts, (ii) an established line of human osteoblasts, (iii) an established line of human mammary epithelial cells, and (iv) a line of rodent liver cells.

Task Description:
We expect to perform the following tasks in FY 95:

1) Preparation and Characterization of Peptide Containing Matrices: Collagen-mimetic Peptides will be grafted on the following matrices, (i) Agarose hydrogel, (ii) Methyl cellulose hydrogel, (iii) Hydroxypropyl methylcellulose hydrogel, (iv) Polyglycolide (PGA) fibers, braided yarns, (v) Polylactide (PLA) fibric, sponges, sheets, and (vi) PGA-PLA composites fabric, sponges, sheets.

2) Synthesis (i) Development and optimization of procedures for grafting peptide to polymers, (ii) Determine the conditions for loading peptides at different levels.

3) Characterization: (i) Peptide content, (ii) Determine N- or C-terminal attachment of peptides.

4) Biological Activity: (i) Preliminary assays of cell attachment, (ii) Cell infiltration and formation of 3-D colonies.

5) We have selected cells which are known to exhibit differentiated behavior in 3-D matrices. The following cell lines will be used in the first year of the project: (i) CRL 1502 Fetal human dermal fibroblast, (ii) MG-63 Human osteosarcoma, (iii) HBL 100 Breast epithelium, and (iv) Hep G2 Human liver carcinoma.

Task Significance:
The proposed research will help in designing more efficient bioreactors by attaching cells in 3-D matrices via receptors for ECM. Since a cell in its native physiological environment is surrounded by the ECM and by other cells, receives informational messages from both the ECM and from the surrounding cells through specific receptors, and since collagen is presumed to be the primary transducing agent for mechanical signals transmitted through specific receptors, the synthetic environment proposed by us will promote the expansion of cell
populations in a differentiated state. Such matrices can be engineered to allow for a facile exchange of nutrients and wastes with a surrounding medium. Cultures in such matrices will, (i) facilitate expansion of differentiated cell populations, (ii) allow closer examination of the role of geometry and surroundings (ECM, cells) on differentiation, (iii) allow the determination of the role of mechanical signals in differentiation through the manipulation of applied force, and thus (IV) make possible the optimization of culture conditions for bioreactors.

Progress During FY 1995:

Peptide and polymer composite syntheses have been initiated and studies are in progress to characterize these composites both in terms of their physico-chemical properties and their ability to sustain cell binding and growth in 3-D geometry.
Quantitative, Statistical Methods for Pre-Flight Optimization, and Post-Flight Evaluation of Macromolecular Crystal Growth

Principal Investigator: Prof. Charles W. Carter
University of North Carolina, Chapel Hill

Co-Investigators:
Ducriux, A.
Ries-Kautt, M.
CNRS-Gif Laboratoire de Biologie Structure
CNRS-Gif Laboratoire de Biologie Structure

Task Objective:
To generalize the application of response surface methodology to a broad range of crystallization studies. To develop and provide tutorial workshops to distribute skills in designing response surface experiments and analyzing the resulting data for stationary points.

Task Description:
This approach involves making and testing models of crystal growth which involve quadratic polynomials, which are the simplest mathematical functions that can pass through a maximum or minimum. Because these are slowly varying mathematical functions, they are most appropriate for modeling a local neighborhood of a maximum or minimum value for the response a system makes to changes in the independent parameters. The polynomial form of the functions facilitates estimation of a small number of coefficients using a minimum number of experiments. It is also a simple matter to determine the coordinates of the optimum point by partial differentiation and solution of the resulting simultaneous equations. The model and its statistical descriptors become an valuable set of scientific documentation. Moreover, working at stationary points also enhances the reproducibility of experiments, because of the property that the partial derivatives vanish at stationary points of the quadratic function.

Task Significance:
A generally applicable protocol for determining that the conditions for crystal growth correspond to an optimum in the response surface will extend the value of flight-based microgravity experiments, by helping to assure that experiments are minimally vulnerable to experimental errors and hence as reproducible as possible. By eliminating experimental fluctuations as an important source of variation, this in turn will enhance the signal-to-noise ratio of all microgravity experiments done under optimal conditions. Ultimately, the same experimental strategy can be extended to the study of crystal diffraction quality. This work may therefore contribute to improvements in the detail with which protein structures can be analyzed, as well as the quantitative attribution of such improvements to microgravity.

Progress During FY 1995:
We have arranged for Robert Cudney of Hampton Research, Inc., to provide five proteins of his choosing, for which we will determine analytical response surfaces, to generalize the approach to as broad a selection of proteins as possible. I have hired a research specialist experienced in protein crystal growth, who will join the lab December 1, 1995 to supervise the project. Once she has learned to carry out response surface experiments, we will schedule and advertise the first of several workshops to teach the techniques to protein crystallographers involved in NASA microgravity research. As part of the theoretical development of the method, I formally derived a local approximation to supersaturation, applicable to any situation where estimates exist for the limiting solubility in the absence of crystallizing agent and the exponential decay constant for the dependence of solubility on the crystallizing agent concentration. This approximation facilitates a direct coordinate transformation for the solubility phase diagram, making it possible to represent the nucleation zone as a rectangle and to place experiments on a rectangular grid. Though apparently abstract, this result has important practical advantages. It will help to assure that sampling for response surface experiments can be done in an efficient manner as possible with minimal loss of data through inadvertent placement of experiments that do not crystallize and hence do not contribute to the analysis.
II. MSAD Program Tasks — Ground-based

Discipline: Biotechnology

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Printed: 28 FEB 1996
Crystallographic Studies of Proteins Part II

II. MSAD Program Tasks — Ground-based

**Principal Investigator:** Dr. Daniel C. Carter
NASA Marshall Space Flight Center (MSFC)

**Co-Investigators:**
Ho, J. (X. He)
NASA Marshall Space Flight Center (MSFC)

**Task Objective:**
This research involves the atomic structure determination of several protein structures. Key areas of study involve serum albumin structure and chemistry and HIV antibody complexes and structure. Aspects of this research generate flight experiment problems and contribute to facilities for the evaluation of flight experiment activities.

**Task Description:**
The overall goal of this research is to utilize ground-based and microgravity-grown protein crystals to improve our understanding in two important areas of structural molecular biology. The first involves the determination of the definitive structure of serum albumin together with the chemical basis for the molecules’ tremendous ability to bind and transport an immense variety of ligands throughout the circulatory system. The second area which is also broad in scope involves the structure determination of a series of human monoclonal antibodies expressed against the AIDS virus together with their respective antigen complexes. Both of these project areas are in an advanced stage where improvements in crystal quality will have significant impact on our understanding of the underlying chemistry.

Screens for optimum crystallization conditions or to determine crystallization conditions for new proteins will be conducted by the hanging-drop vapor-diffusion method. A Micromedics robotic crystal growth system is available to aid systematic surveys of pH, precipitant type, precipitant concentration, and protein concentration. The monoclonal antibodies expressed against the AIDS virus will be supplied by collaborator Professor Forian Rücker of The Institute of Applied Microbiology in Vienna, Austria. Cleavage of the antibody with papain or pepsin to produce the Fab fragments and subsequent purification will continue to be conducted. Antigenic peptides will be provided by Dr. Rücker and/or as a gift from IAF Biochemicals of Canada. X-ray diffraction data will be collected from both ground-based and flight crystals using a multi-wire area detector (Nicolet) and an imaging plate system (R-Axis) mounted on a Rigaku RU200 rotating anode generator. In favorable cases where the logistics can be arranged, diffraction data will be collected at synchrotron sources.

**Task Significance:**
High quality, single crystals are of tremendous value for a variety of industrial and research applications. Crystals of sufficient size and quality also provide invaluable avenues to understanding the detailed atomic structure and function of biological macromolecules and other substances. Efforts to produce higher quality protein crystals for application in x-ray crystallography have spawned numerous experimental approaches which range from the application of automated screening to the growth of protein crystals in microgravity.

**Progress During FY 1995:**
1. Complete details of the atomic structure of Canine serum albumin have been determined to high resolution. The structure has been refined to 2.0 angstroms including the water structure. This structure has revealed the atomic details of long-chain fatty acid transport via serum albumin which has resolved many important questions in the literature. Two manuscripts describing these results are in preparation.

2. The structure of several important ligand complexes with albumin have been determined and manuscripts describing these results are in preparation.
II. MSAD Program Tasks — Ground-based

Discipline: Biotechnology

3. The refinement of Cytochrome c5 was finished. The HEW lysozyme microgravity and ground-based refinements were finished and details were submitted for publication.

4. The work describing the atomic structure of glutathione S-transferase was published.

5. Crystallization conditions for a variety of new serum albumin crystal forms suitable for the atomic structure determination were published.

6. We have completed the second year on two government/industry agreements and both are in the process of being extended.

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Microgravity Simulated Prostate Cell Culture

Task Objective:
Prostate cancer is the most common malignancy found in the United States. In 1994, it is estimated that 200,000 new cases of prostate cancer will be diagnosed which accounts for over 32% of all cancer cases detected and the second leading cause of death in American men. As an endocrine organ, the prostate gland is highly responsive to the steroid androgen, which induces growth and differentiation in vitro. Consequently, endocrine therapy remains the leading therapeutic modality for the treatment of local and metastatic disease. Unfortunately, an escape of the blockade by androgen antagonists presents a serious threat to the success of endocrine therapy. Despite much effort in the past years to develop model systems to study the effects of androgen on prostatic epithelial cell growth and differentiation in vitro, there is little evidence that prostate epithelial cells respond directly to the mitogenic signal of androgen. One of the key reasons that these attempts have failed is the conventional prostate cell culture system will not retain an essential feature of the prostate gland, i.e., a 3-dimensional organization composed of interactive prostate stromal and epithelial cells.

Task Description:
The central theme of this proposal is to establish a 3-dimensional cell-cell interaction model in vitro to examine the influence of androgen, known and novel growth factors, extracellular matrices, and differentiating agents on the growth and differentiation of human prostate cancer. To accomplish this goal, we propose to pursue the following three objectives:

First, we propose to construct 3-dimensional prostate organoids in vitro under microgravity-based conditions. We will compare the growth and differentiative potentials of the prostate organoids formed through cell-cell interactions between prostate stromal cells obtained from normal, benign, or cancerous prostate and epithelial cells derived from normal prostate or the LNCaP cell line (which is derived from a human prostate cancer lymph node deposit). These comparisons will help us to understand the possible regulatory roles of prostate stromal cells on prostate epithelial growth and differentiation.

Second, we will test the biologic behaviors and the functionalities of the reconstituted prostate organoids. The growth and differentiative responses of the reconstituted prostate organoids to androgen (dihydrotestosterone, DHT), growth factors (transforming growth factor α, TGF α; transforming growth factor β keratinocyte growth factor, KFG; hepatocyte growth factor/scatter factor, HGF/SF; and basic fibroblast growth factor, bFGF), extracellular matrices (plasma membrane preparation from human prostate stromal cells, collagen IV, laminin, fibronectin, heparan sulfate proteoglycans, and Matrigel (R), and differentiating agents (phenylbutyrate, retinoid fenretinide) will be evaluated by histomorphologic, immunohistochemical, biochemical, and molecular methods. This microgravity-simulated model system may be developed and perfected for future use as a standard for drug screening programs and could serve as a valuable tool to complement existing animal model systems.

Finally, because of the possible parallelism of prostate growth and differentiation between the microgravity-simulated conditions and in vivo conditions, we propose to use this system to produce large quantities of potentially biologically relevant molecules that can be used to stimulate human prostate epithelial cell growth in vitro using conventional culture conditions on plastic dishes. It is hoped that the enriched source of these biologically relevant factors will assist our future attempts to purify, characterize, and close factors that may be useful in evaluating the possible roles of these factors in the clinic progression of human prostate cancer.
Task Significance:
The specific significance of this proposal involves the delineation of central questions in prostate cancer research. These include:

1. Although the prostate gland is a highly androgen-responsive organ, why do prostate cells, when cultured in vitro, generally not show growth responses to androgens? Despite the coculture of prostate epithelial cells with prostate fibroblasts, androgen only induces marginal growth responses in the cultured prostate cells in vitro (Chang and Chung, 1989).

2. What are the physiological mediators for androgen-induced prostate growth responses?

3. Can the growth mediators alone or differentiating agents promote prostate cancer differentiation under chemically defined cell culture conditions? With respect to question 1, there has been considerable speculation on the interrelationship of structure and function of the prostate gland. Reciprocal interaction between prostate stroma and epithelium, under proper configurations (e.g., 3-dimensional structure), may determine the differentiative function of prostatic sinus epithelium. Tissue recombinants composed of fetal UGM and UGE (urogenital sinus epithelium) have been demonstrated to undergo normal prostatic morphogenesis and cytodifferentiation when promoted by androgen (Cunha and Chung, 1981; Chung and Cunha, 1983) and selected growth factors (Sugimura et al., 1994) both in vivo and in vitro. These results illustrate the importance of proper organization and orientation of prostate stroma and epithelium; a 3-dimensional organization could be prerequisite for the androgen-induced growth and differentiation observed in the normal prostate gland.

Progress During FY 1995:
Funding for this research became available at the very end of FY 95. We have recently submitted a paper to In Vitro.

STUDENTS FUNDED UNDER RESEARCH: TASK INITIATION: 9/95 EXPIRATION: 9/99
PROJECT IDENTIFICATION: 962-23-01-19
NASA CONTRACT NO.: NAG9-823
RESPONSIBLE CENTER: JSC

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Task Description:
The following is a brief description of the work to be done in the first year to accomplish the two previously mentioned tasks.

1. The bench-top design will include delivery of spectral wavelengths in the 2.0 to 2.5 μm region which is a minimum for the water absorption signal. Both transmission and reflection signals will be investigated. A series of increasingly biologically complex solutions will be investigated, from glucose in water to the cell culture medium, to identify the minimum number of wavelengths that can be used without loss in production of glucose concentration. Temperature and pH effects on the spectrum will be investigated and, if needed, compensated for with the algorithms described under specific aim #2.

2. Algorithms will be developed to determine the optimal wave numbers that are most useful for the determination of glucose within the various cell culture media used. Filtering algorithms will be developed for pre-processing the raw spectra before application of the multivariate techniques. The optimal number of factors and model will be developed using a partial least squares multivariate technique.

Task Significance:
In this proposal, a plan is forwarded to accelerate the development of a three-dimensional tissue culturing system by building on previous data toward the design of an on-line, non-invasive, NIR optic sensor for use with rotating-wall bioreactors. The primary focus is to develop the technology to continuously and non-invasively sense glucose in the cell culture media to allow for long-term, on-line monitoring without potential contamination of the medium from invasive approaches.

The culturing of a variety of tissues under clinostatic suspension in rotating vessels has been enhanced by NASA's biotechnology program. Increased cell viability, adhesion, tissue formation and differentiation have been demonstrated. However, to increase the production of cell cultures, the design of bioreactors must include computer control of cell culture processes. In order to do this, on-line, preferably non-invasive, sensors must be implemented to monitor various parameters of the cell culture and feedback that information for regulation of the environment. One vital parameter of interest is the concentration of glucose in the cell culture media. The basis for cell culture media is an isotonic saline solution of nutrients and cofactors. In a typical medium, glucose is the major
carbohydrate energy source. Cell proliferation causes rapid consumption of glucose which in turn causes growth limitations of the cells. Thus, the relative concentration change of glucose is a determining factor for the rate of cell culture growth. By permitting closed-loop control of cell culture glucose, the growth rate will be optimized. Current techniques used to monitor the value of this parameter within cell culture media are invasive and cannot be used in a closed-loop system since they are not on-line measurements. In this research, a near-infrared spectroscopy system is proposed to perform the required glucose measurements on-line and non-invasively, without potential contamination of the cell culture media.

Progress During FY 1995:

With the receipt of the grant in September, 1995, we set about acquiring equipment and setting up a laboratory. Algorithms are being developed in order to proceed with the required research.

STUDENTS FUNDED UNDER RESEARCH:

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II. MSAD Program Tasks — Ground-based

Discipline: Biotechnology

A Comprehensive Investigation of Macromolecular Transport During Protein Crystallization

Principal Investigator: Dr. Lawrence J. DeLucas
University of Alabama, Birmingham

Co-Investigators:
Chait, A. NASA Lewis Research Center
Meyer, W. NASA Lewis Research Center

Task Objective:
The objective of this research is to systematically study the differences between protein crystallization transport mechanisms on Earth and in space. The protein, lysozyme, will be used for these studies since there exists a definitive and extensive growth mechanism database for this protein.

Task Description:
Ground-based studies with lysozyme will include dynamic light scattering on protein in solution and in gels and measurement of particle and concentration fields around the growing crystal. This data will be compared with continuum modeling of transport phenomena which will be followed by the design of new growth cells and optimization of parameters for flight experiments. The eventual flight experiment will measure the particle and concentration fields around a growing crystal and compare results with ground-based gel and solution experiments and modeling prediction.

Task Significance:
The proposed research represents a new and innovative approach that combines advanced in situ diagnostic techniques with detailed continuum computational models to describe transport in solution. The combination of diagnostics and modeling will allow critical examination of hypotheses emerging in the literature.

Progress During FY 1995:
No progress to date since the money for this grant was received very late in the fiscal year.

STUDENTS FUNDED UNDER RESEARCH: TASK INITIATION: 9/95 EXPIRATION: 8/99
PROJECT IDENTIFICATION: 962-23-08-35
NASA CONTRACT NO.: NAG8-1162
RESPONSIBLE CENTER: MSFC

Printed: 28 FEB 1996
Development of Robotic Techniques for Microgravity Protein Crystal Growth

Principal Investigator: Dr. Lawrence J. DeLucas University of Alabama, Birmingham

Co-Investigators:
Torre, Dr. L. Boeing Aerospace
Tillotson, Dr. B. Boeing Aerospace

Task Objective:
The objectives of this proposal are to increase the yield of large, high quality crystals from macromolecular crystal growth experiments and to enhance crystallographer's ability to evaluate crystal suitability for x-ray analysis.

Task Description:
The approach is multidisciplinary, combining engineering, robotics, optics, and crystallography. Machine vision techniques will be applied to monitor growth rate and solute concentration at the surface of growing crystals. Advanced automation will use this information to dynamically adjust conditions in the growth cell, maintaining optimal conditions throughout the crystal growth. Non-invasive methods for manipulating crystals in low gravity will be investigated.

Task Significance:
The proposed research in improving the yield of high quality crystals is important because of the increasing role of macromolecular crystallography in medicine and biology. Large, high quality crystals are critical to solving the structure of biologically important macromolecules, but many macromolecules are difficult to crystallize. Methods for improving the yield of large, high quality crystals are therefore fundamentally important to crystallographers. Because hard-to-crystallize molecules are often selected for microgravity crystallization experiments (where it is hoped that reduced gravity will improve crystalline quality), and because flight opportunities are infrequent and have very limited capacity, it is especially important to maximize the yield of usable crystals from microgravity experiments.

Progress During FY 1995:
No progress since money was received very late in the fiscal year.

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Printed: 28 FEB 1996
II. MSAD Program Tasks — Ground-based

Macromolecular Crystallization: Physical Principles, Passive Devices, and Optimal Protocols

Principal Investigator: Dr. George T. DeTitta Hauptman-Woodward Medical Research Institute

Co-Investigators:
Luft, J.R. Hauptman-Woodward Medical Research Institute

Task Objective:
The objective of our work is to design, test, and evaluate devices and protocols for growing single crystals of macromolecules suitable for x-ray diffraction experimentation. In the process we propose to identify those devices and protocols that will work optimally on earth, and to identify those that may benefit from a microgravity environment. Our objective is to quantitatively assess those parameters that have a profound effect on crystal quality.

Task Description:
The work centers on a fundamental understanding of the role of kinetics in determining the ultimate quality of macromolecular crystals grown in the 1-g laboratory. Our preliminary work was to propose and fabricate a number of prototype devices that can work to control the kinetics of equilibration in vapor diffusion crystallization experiments. Our present work is to understand the basic processes that underlie the vapor diffusion process, and to confirm the assertion that the rate-limiting step in vapor diffusion is transit of water across the vapor space.

Task Significance:
Macromolecular crystallography has become the foundation stone of much of modern biology. Protein engineering, molecular genetics, and rational drug design depend on an accurate description of the structure of a macromolecule (typically a protein) at the atomic level. Such a view is afforded by few disciplines, and the principal one is crystallography. A necessary first step in structure determination is the growth of a single crystal suitable for diffraction studies. Our work is aimed at a greater understanding of the processes of crystal growth, and at the design and testing of devices and protocols to grow better macromolecular crystals. Modern approaches to drug design rely on an accurate understanding of the target molecules of drugs and therefore our work has the potential to materially affect the general public who will benefit from new drugs that result from these approaches.

Progress During FY 1995:
The project was funded on July 27, 1995. Since that time we have completed a number of basic experiments designed to answer the question concerning the rate-limiting step in vapor diffusion. Our work on the relationship between the kinetics of equilibration of hanging drops on the distance from the drop to the surface of the reservoir shows that the further the drop is from the reservoir the slower is the equilibration. In the last few weeks we have concluded studies on the evaporation of equivolumetric droplet configurations, in which droplet configurations of one 24 µL droplet, two 12 µL droplets, three 8 µL droplets, ..., eight 3 µL droplets were allowed to equilibrate with uniform reservoirs. Six sets of experiments were conducted, differing in the distance from droplet to reservoir distance from set to set. Results indicate that for short drop-reservoir separations the kinetics of equilibration is linearly, and strongly, dependent on the surface area of the droplet, while at long separations there is a weak dependence on surface area. This indicates that the principal assumption of vapor diffusion theory is correct. In addition, three manuscripts of work preliminary to our project appeared in print. Finally, we are in the final stages of preparation of a manuscript describing the results of the kinetics versus drop-reservoir separation work (that manuscript will be submitted to Acta Crystallographica D as well).
II. MSAD Program Tasks — Ground-based

STUDENTS FUNDED UNDER RESEARCH:  

TASK INITIATION: 7/95  EXPIRATION: 7/99
PROJECT IDENTIFICATION: 962-23-08-41
NASA CONTRACT NO.:  NAG8-1152
RESPONSIBLE CENTER:  MSFC

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals


The Effect of Microgravity on the Human Skin Equivalent

Principal Investigator: Dr. S. D. Dimitrijevich                Univ. of N. Texas Health Science Ctr, Fort Worth

Co-Investigators:
Mills, MPh., J.G.                                      University of North Texas

Task Objective:
The objective of this project in the first instance is to effect morphogenesis of the Human Skin Equivalent under the conditions of simulated microgravity. We will then study the effects of fluctuation in several physiological parameters (e.g., glucose, calcium levels), encountered in intact organisms under zero gravity, on the development and functions using our in vitro model of the human skin.

Task Description:
We have developed living in vitro models of several human tissues (e.g., skin, cornea, conjunctiva), based on a non-contracting three-dimensional collagenous matrix populated with appropriate fibroblasts. These models feature epithelia and endothelia which structurally and functionally resemble ex vivo human tissue. These features provide an excellent opportunity for studies of heterologous cell-cell, and cell-matrix interactions during tissue development or wound healing. Equally important are studies of the responses of these models to changes in the microenvironment during development, wound healing or in quiescence. We will, therefore, examine all states involved in the development of the skin equivalent such as fibroblast behavior and functions, attachment of keratinocytes and melanocytes and epidermopoesis under the conditions of simulated microgravity. Using the RWV cell culture systems and the Human Skin Equivalent, we will also study the effects, on tissue, of changes in glucose and calcium hemoestasis which have been observed transiently in intact organisms (man) under zero gravity. Finally, we propose to determine if the simulated microgravity is a stress factor to which cellular tissue components respond by expression of stress proteins.

Task Significance:
Simulation of the microgravity environment using RWV has been found to provide unique conditions which allow growth of difficult cell type/lines, and of organoid cell aggregates. We expect to demonstrate that the simulated microgravity conditions will generate human tissue equivalents of superior mechanical and biological integrity, more appropriate for tissue replacement therapy. It is further hoped that a great deal may be learned about tissue tolerance to the transient physiological abnormalities experienced under zero gravity, and how this might impact on our understanding of processes such as tissue repair and design of new approaches to wound healing.

Progress During FY 1995:
The majority of studies utilizing reduced gravity microenvironment have been concerned with cultures of difficult-to-grow mammalian cells, or transformed cells and their involvement in the process of tumorigenesis. It has been shown that the benefits of reduced gravity, generated by various configurations of the Rotating Wall Vessel Cell Culture Systems, led to the production of "tissue granules" arising from aggregation of cells and subsequent differentiation.

Over the past few years, we have focused our attention on cell-cell and cell-matrix interactions involving normal human cells. The in vitro models which we have developed to facilitate our studies are three-dimensional cultures consisting of the collagen type I matrix populated with the appropriate fibroblasts. On the surface of these non-contracted Connective Tissue Equivalents are plated epithelial cells, which are allowed to proliferate and form confluent monolayers. Following exposure to the air-liquid interface, the monolayer differentiates to a fully stratified epithelium. Utilizing dermal fibroblasts and epidermal keratinocytes in this fashion, we have assembled the Human Skin Equivalent (HSE), and applying this concept to ocular tissue led to the development of Human Corneal and Conjunctival Equivalent.
In order to study the effect of the reduced gravity on the heterologous interactions using our tissue models, we have had to implement certain modifications of the RWV Cell Culture System. We have modified the 50 ml RWV cell culture system by introducing a cylindrical insert which divides the vessel into four compartments. This arrangement allows an HSE to be set up in each of the segments, so that simultaneous execution of four identical repeats of any experimental conditions are possible. In addition to helping with quantitation of the results, this approach also makes the experimental process more efficient. Alternatively, the Skin Equivalent has also been set up as a disc (5mm thick and 2.25" in diameter) attached to the end wall of the vessel nearest to the drive shaft. By using a suitable "cross shaped spacer" this tissue can also be subdivided into four identical compartments. We are advised that neither modification interferes with the principles which govern generation of the microgravitational environment.

Dermal Equivalents (DE) are made by inoculating collagen type I solution (5mg/ml) with $2.5 \times 10^5$ normal human dermal fibroblasts/ml, and "casting" 3.0 ml of this mixture in each compartment of cylindrical insert. The DE are then allowed to polymerize and form translucent gel segments. The RWV is then charged with medium, the system assembled and set to rotate under incubator conditions. After incubation for several days, visualization by neutral red uptake method shows a random distribution of viable fibroblasts throughout the matrix. There appears to be no extensive cell division, nor matrix contraction under the experimental conditions used. These findings parallel our experience with these three-dimensional cultures when normal conditions are used. When the keratinocytes are introduced into the vessel, they attach to the surface of the DE, and form a confluent monolayer. Some stratification takes place under "submerged" conditions but differentiation at the air-liquid interface has not yet been examined.

To date, we have established that the Human Skin Equivalent can be assembled in the 50 ml RWV Cell Culture System and have implemented some modification which are more appropriate for our experimental protocols. Studies are in the progress on quantitation of the findings briefly described above, keratinocyte attachment and incorporation of normal human melanocytes into the SE.

**Students Funded Under Research:**

| BS Students | 0 |
| MS Students | 0 |
| PhD Students | 1 |

**Task Initiation:** 9/95  **Expiration:** 8/99

**Project Identification:** 962-23-01-37  **NASA Contract No.:** NAG9-813  **Responsible Center:** JSC

**Bibliographic Citations for FY 1995:**

Presentations

II. MSAD Program Tasks — Ground-based

Discipline: Biotechnology

Use of Microgravity-Based Bioreactors to Study Intercellular Communication in Airway Cells

Principal Investigator: Dr. Ellen R. Dirksen
University of California, Los Angeles

Co-Investigators:

Woodruff, Dr. M.
UCLA School of Medicine

Biotano, Dr. S.
UCLA School of Medicine

Task Objective:

1. To develop protocols for optimal culturing of airway epithelial tissue in low shear stress simulated microgravity, rotating-wall zero-headspace vessels (RWV) using microbeads.

2. To measure the ability of airway tissues cultured in simulated microgravity to communicate intercellularly.

3. To measure and characterize mechanically-sensitive ion channels in the plasma membranes of isolated cells grown in low shear stress microgravity simulating RWV.

Task Description:

Recent experiments using microgravity-based systems to culture cells have demonstrated the potential for obtaining the viable tissues that can be used for biochemical and molecular biological analysis. Cells that do not grow well in cultures at unit gravity (e.g., cells obtained from salivary gland) have been induced to form three-dimensional assemblies of cells, with differentiated characteristics that mimic the in situ tissue when grown in simulated microgravity. In contrast, some other cell types (e.g., lymphocytes) do not grow or proliferate well in microgravity. Gravity has been shown to affect the stimulus-response characteristics of cells and this may underlie the differences observed in microgravity. We have been studying a cell calcium signaling mechanisms in our laboratory that is expressed in a wide range of tissues and that may be used to help evaluate normal cell and tissue functional integrity.

Our model system is based on primary tissue cultures of airway epithelia and the stimulus-response parameter that we have characterized is a mechanosensitive change in intracellular calcium concentration in an individual cell that is communicated over a large area of the cultured epithelial tissue by gap junctional mediated diffusion. We have developed precise and accurate techniques for observing the cellular calcium changes in an array of cells in real-time using a computer-based video imaging system. High resolution quantitative measures and qualitative analyses have allowed us to implicate particular components of the signal transduction pathway, including mechanosensitivity release of effector molecules (e.g., inositol 1,4,5-trisphosphate and calcium ion), and mechanosensitive membrane ion channels (calcium-conductance channels). We have recently discovered that interference with the signaling has a long-term effect on cell migratory behavior and are exploring the possibility that the mechanosensitivity relies on the function of the cytoskeleton.

The experiments that we are proposing will assay the effect of earth-based, simulated microgravity on the intercellular communication of the mechanically-induced calcium signal and on the expression and control of mechanosensitive cation specific and calcium channels that are related to the calcium signaling. Cells will be grown in a Rotating-Wall Vessel and then characterized for 1) whole tissue growth characteristics such as aggregation, morphological differentiation, viability and tissue growth, 2) second messenger signaling such as mechanosensitivity, magnitude of intercellular calcium increases and intercellularly communicated calcium responses, and 3) channel activity including single ion channel currents and whole-cell mechanosensitive ion channel currents. We will use as controls cells cultured in parallel at unit gravity in organ and tissue cultures.

This type of cell communication may be fundamental over a broad range of tissue types, including all tissues that express gap junctional communication and it is important that it be fully understood. These experiments will use...
the microgravity environment to help us dissect the molecular mechanisms involved in the pathway in order to advance new ways to manipulate the culturing of cells in microgravity.

Task Significance:
Mechanically induced intercellular communication is a complex tissue function involving the expression of mechanoreceptors, mechanically sensitive ion channels, IP$_3$-dependent CO$_2$ mechanisms, and the expression and assembly of gap junctions between cells. This type of communication is essential for coordinated cellular activity and proper tissue response. It is presently unknown whether tissues produced under microgravity conditions have intercellular communication. In contrast, with cells that are grown under conditions that reduce mechanical shear stress, where the effect of gravity is randomized, might demonstrate a potentiation of intercellular communication.

The epithelial cells, as they are presently cultured, have several limitations. The morphology of cells in monolayer cultures, unlike those in the intact epithelium, take on an amorphous, flattened shape and the percentage of ciliated cells is markedly reduced as cells migrate away from the explant. Attached to the collagen substrate is minimized after 2 to 3 weeks in culture. Because the cells do not stay attached, they do not survive for an extended time in culture. We are through limited in our ability to manipulate the cultures--to observe the long-term effects of agents that are added extracellularly and intracellularly. This is a serious limitation since we would like to study biomolecules that control differentiation and maintenance of the cells and relate this effect to the expression and elaboration of gap junctional communication. Microgravity has been shown to extend the lifetime of cultures of salivary glands and may also produce this beneficial effect in airway epithelia.

Microgravity may influence the expression and maintenance of the IP$_3$ signaling pathway and hence, intercellular communication. Many of the deleterious effects of microgravity, such as reduced exocrine and endocrine secretion, reduced biosynthetic activity, and reduced cell proliferation in the immune system may involve an altered IP$_3$ signal as well as an altered PKC pathway.

It has been suggested that microgravity-based culture systems may have great potential for medical science in the area of tissue engineering. The success of this hypothesis is dependent on developing fully functional tissues under microgravity conditions. Our proposed research will elucidate cell-cell interaction mechanisms that are sensitive to gravitational forces. These results may lead to information for improving tissue growth so that the full potential of biological systems under microgravity conditions can be realized.

Progress During FY 1995:
The research project has commenced with the purchase of a bioreactor. Techniques are being developed to facilitate attachment of airway cells to microbeads for use in microgravity experiments. Previous findings indicate that the cells prefer collagen-coated beads for attachment. Therefore, successful attachment of the cells to the microbeads is a major step in the experiments being developed to manipulate the culturing of cells in microgravity.

**STUDENTS FUNDED UNDER RESEARCH:**

| TASK INITIATION: | 8/95 |
| PROJECT IDENTIFICATION: | NASA CONTRACT NO.: |
| NASA CONTRACT NO.: | NAG9-814 |
| RESPONSIBLE CENTER: | JSC |
Microgravity Thresholds for Anti-Cancer Drug Production on Conifer Cells

Principle Investigator: Dr. Don J. Durzan
University of California, Davis

Co-Investigators:
Falk, R.
University of California, Davis

Task Objective:
Objectives are to develop a model system for anticancer drug production under microgravity using highly adaptable conifer cell lines, and more specifically, to evaluate the effects of microgravity on the cellular production of free and covalently bound taxanes.

Task Description:
1. The postulated enhanced production of free and bound taxanes is explored in suspensions of haploid and diploid cells using bioreactors that can be designed to establish better process controls with plant cells.

2. Free taxanes are recovered by solvent extraction and the residual bound taxanes are removed from cellular structures by enzymatic cleavage. Taxane precursors and substrates are evaluated for their possible use as synthons for the enhanced production of paclitaxel (Taxol™).

3. Microgravity is examined for the enhanced production of taxanes on catalytic surfaces by gold-immunological localization at the subcellular level using scanning electron microscopy.

4. Cell viability is monitored cytochemically for apoptosis (unscheduled and programmed cell death). Protein crystals in organelles are evaluated as an additional marker for effects of microgravity.

Task Significance:
1. A model for enhanced paclitaxel and taxane production by haploid-derived cell lines provides a novel experimental basis and a possible alternative to alleviate the anti-cancer drug supply problem. This opportunity is indicated by the current unavailability of a total chemical synthesis of paclitaxel. The numerous and complex steps make total chemical synthesis uncommercial. Moreover, dependency on natural or cultivated sources of Taxus brevifolia are unrealistic because this tree is endangered, undomesticated, highly variable in taxane content, and slow-growing. Other faster-growing Taxus sp. make taxol but usually at lower levels. However, other species may produce more baccatins and substrates that can be used as synthons to shorten the number of steps in a chemical synthesis. Process control strategies with the appropriate bioreactor designs are aimed at enabling a better evaluation of how gravity alters cell cultures as drug factories.

2. Texane production is enhanced over current extractive methods by enzymatic recovery of covalently bound taxanes. The yet unidentified new taxanes offer opportunities to investigate their anti-cancer properties as more water-soluble analogues of paclitaxel. Cell lines can be evaluated for their ability to biochemically transform synthons via the isoprenoid pathway for enhanced drug production.

3. Taxus cells under 1xg synthesize catalytic surfaces that covalently bind taxanes. An assay is needed to show how distributions of taxanes on these surfaces are affected by microgravity. Since some cell lines are initially haploid, mutations can be directly and more easily screened than in diploid cells. In the long run, mutant lines will help to clarify the steps in the overall biosynthetic process and could serve as more effective cell factories for taxane production. Recovery of proteins from catalytic surfaces would enable a more specific examination of how specific proteins are used in the biosynthetic process.
4. The effects of gravity and microgravity on the viability of cells in terms of enhancing scheduled and unscheduled cell death and terminal differentiation are needed. Endonuclease assays and evaluation of protein crystal growth in subcellular organelles provide internal markers as to physiological states and viability of cells in the bioreactor. This information is needed to understand opportunities with taxane-producing cell lines in bioreactors designed for continuous and batch culture.

Progress During FY 1995:

Three genotypes of *Taxus brevifolia* cells on semisolid plates were subjected to 2xg on a large centrifuge for 2 weeks. Delays in the manufacture of bioreactors led to preliminary work that showed that other conifers gave antibody reactivity to taxanes. Suspensions of these cells were also subjected to 2xg using 1 liter rotating nipped flasks on a clinostat in 50 gallon buckets of the centrifuge.

Results indicated that growth was not inhibited by 2xg. Cells from both studies were harvested for taxane content and microscopic examination. A shortage of parts for the scanning electron microscope has temporarily delayed cell surface examinations. Compatibility of cell suspensions for the Synthecon rotary cell culture system will start now that the bioreactors have arrived. We have considered future modifications to the bioreactors that would make them more amenable for use with a light source without the heat input.

**STUDENTS FUNDED UNDER RESEARCH:**

**TASK INITIATION:** 8/95  **EXPIRATION:** 8/99

**PROJECT IDENTIFICATION:** 962-23-01-28

**NASA CONTRACT NO.:** NAG-9-825

**RESPONSIBLE CENTER:** JSC
Laser Scattering Tomography for the Study of Defects in Protein Crystals

Principal Investigator: Prof. Robert S. Feigelson

Stanford University

Co-Investigators:

DeLucas, L.
DeMatti, R.C

University of Alabama, Birmingham
Stanford University

Task Objective:

The principal objective of this research program is to apply the laser scattering tomography technique (LST) to the study of defects in protein crystals and how these defects relate to growth conditions and x-ray diffraction performance.

Task Description:

This research effort is designed to provide an in-depth understanding of the nature of the defects present in protein crystals using the fast and non-destructive LST technique. A major component of this program will be a comparison between microgravity-grown and terrestrial-grown crystals to help establish the link between microgravity conditions and the improved crystal quality of space-grown crystals. The program will begin with the design and construction of the LST apparatus. The system will be tested on Lysozyme and used to study at least four other protein systems representative of crystals which have been or are being grown in µg. Crystals grown in the laboratory will be evaluated along with space-grown crystals. After the LST crystal characterization studies, these same crystals will be sent to the University of Alabama for x-ray performance evaluation.

Task Significance:

This investigation will clarify the relationship between growth conditions and defects in protein crystals. It will lead to both a better understanding of the mechanisms involved in protein crystal growth and an ability to make a rational choice of growth parameters, thereby permitting an improvement in crystal size and diffraction resolution (quality). The LST technique will also provide an important means of pre-screening protein crystals prior to diffraction experiments with a significant savings in manpower and beam time.

Progress During FY 1995:

Work on this project started in September 1995 when funding became available. During this short reporting period, we have begun the LST equipment design and selection phase. Various equipment suppliers have been contacted and specifications developed for the laser and other parts of the optical system.

STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 7/95 EXPIRATION: 6/99
PROJECT IDENTIFICATION: 962-23-08-33
RESPONSIBLE CENTER: MSFC
Role of Fluid Shear on 3-D Bone Tissue Culture

Principal Investigator: Prof. John A. Frangos
University of California, San Diego

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
The ultimate goal of this research is the optimization of growth and differentiation of three-dimensional bone cell cultures in vitro. The research has medical implications in arresting bone loss due to microgravity, bedrest, and disuse; and in developing better implant devices and grafting materials. Our objective is to study the role of fluid shear stress on three-dimensional growth and differentiation of osteoblasts and manipulate fluid shear stress throughout the culture period to maximize viable and differentiated tissue. Specifically, we will:

1. Determine the role of different levels of fluid shear stress on osteoblast growth differentiation when grown on porous collagen beads in a Slow Turning Lateral Vessel (STLV). We will:
   a. Study different levels of shear stress experienced by the cells in the bioreactor by using collagen beads that are either unweighted (minimal shear), weighted with small amounts of titanium particles (moderate shear), or weighted heavily with titanium particles (high shear) and compare them to similar beads in control situations (static in the incubator or immobilized in a trickle bed reactor system).
   b. Evaluate the growth characteristics of the osteoblasts by determining growth rate and glucose consumption.
   c. Perform cell cycle analysis by propidium iodine staining and flow cytometry on cells subjected to no, low, and high fluid shear stress. For completeness, we will also perform this analysis on monolayer cells exposed to well defined levels of shear in our two-dimensional laminar flow system.
   d. Assess differentiation by measuring alkaline phosphatase activity on the cell membrane, hydroxyapatite deposition onto the beads, and osteocalcin release into the media.
   e. Assess cell organization and growth patterns on the porous beads by SEM analysis of bead samples.
   f. Evaluate media PGE$_2$ levels for comparison to previous work on fluid shear stress on osteoblasts grown as monolayers on glass slides. This will serve as a comparison of two- and three-dimensional growth under flow and shear conditions.

2. Extend our knowledge of the role of fluid shear stress on cell differentiation to optimize the reactor conditions. In this part of the study, we will progress from constant shear level during the culturing period to variable shear levels throughout the culturing period in order to optimize growth and differentiation. These studies will include the following:
   a. Determine if altering (increasing) the shear stress within a culturing period can promote differentiation.
   b. Determine if reducing shear stress after the onset of differentiation results in the cells reverting to a proliferative phase.
   c. Evaluate whether intermittent periods of increased shear stress are capable of providing the stimulus required for initiation.
Task Description:

Specific Aim 1: We propose to differentiate between the effects of no, low, and high fluid shear stress on osteoblasts in three-dimensional culture.

Specific Aim 2: We propose to optimize the reactor growth condition of osteoblasts on matrix beads by manipulating the hydrodynamics of the system. Altering the fluid hydrodynamics within a single culturing period will reveal the importance of fluid shear stress in bone tissue growth and differentiation. Changes in fluid shear stress within a culturing period may be achieved by altering the media density. The major force creating fluid shear stress on the beads is the force of gravity resulting in bead settling. The settling velocity is dependent on the density difference between bead and fluid. The greater the difference, the greater the settling velocity, and thus the greater the fluid shear stress on the beads and cells. Thus, we propose that manipulation of media density, achieved with the normal medium changes, can alter the fluid shear in the system and promote osteoblast differentiation. Ficoll 400 (Pharmacia) may be used to achieve altered densities. Ficoll 400 is a neutral, highly branched, hydrophilic polymer of sucrose. It dissolves readily in aqueous solutions up to a concentration of 50% by weight and a specific gravity of 1.2 without exceeding normal osmolality. It is also cell culture tested by Sigma, but will be tested extensively on our osteoblast system before use. By utilizing media density to manipulate shear stress within the reactor, we will be able to alter the shear stress within a culturing period by approximately 3.5 dyne/cm² (i.e., from 0.5 to 4 or from 2 to 6 dyne/cm²). We will study the following situations.

Task Significance:

In our lab we have previously cultured primary rat osteoblasts and studied their response to fluid shear stress when grown as monolayers on glass slides (Reich et al., 1990; Reich and Frangos, 1991; 1993; Appendix) and when grown on macro-porous collagen beads in a fluidized bed bioreactor (VERAX System One) (Hilsley and Frangos, 1994; Appendix). The flow-induced shear stresses on the cells on the beads, assuming no bead tumbling and applying Stokes Law, were estimated to be on the order of dyne/cm². The osteoblasts were evaluated for markers of differentiation, including alkaline phosphatase activity and hydroxyapatite formation. A control experiment was conducted in which osteoblasts were grown on the same macro-porous collagen beads under static conditions. Static beads had a better seeding efficiency, but the final cell density was similar to that of beads exposed to flow. The alkaline phosphatase (AP) levels per cell dropped earlier and more dramatically for the cells subjected to flow in the bioreactor than for the static cells. Studies by Owen et al., (1990) have shown that alkaline phosphatase mRNA levels peak just prior to the onset of mineralization. We observed a similar peak in alkaline phosphatase activity near the time that mineralization began. In comparable monolayer shear experiments, we also observed a drop in AP MRAN levels, followed by decreased AP activity (unpublished data). Hydroxyapatite greatly increased during the three-dimensional flow experiment. Yet in comparable static control studies, no measurable hydroxyapatite was detected in this time frame. This indicates that when coupled with three-dimensional growth, fluid flow promotes mineralization by osteoblasts in vitro. This type of cell culturing system shows great promise for growing a large number of osteoblasts.

The above described three-dimensional fluidized bed osteoblast culturing system produced differentiated cells. However, this system would be greatly improved if larger numbers of cells could be cultured prior to the onset of mineralization, and if a mixed cell population of differentiated and proliferating osteoblasts could be maintained throughout the culturing period. Both static and flow beads resulted in limited cell density due most probably to diffusion limitations in control beads and mechanical disruption of surface cells on reactor beads resulting from bead-bead and bead-reactor collisions. The STLV offers an opportunity to increase the cell density prior to eliciting differentiation with increased shear stress.

The STLV imposes shear based on settling velocities, a principle also utilized by the fluidized bed reactor. However, the STLV configuration controls bead to wall bumping and turbulence better due to elimination of a need to recirculate media through a peristaltic pump. These are the main impediments to growth and differentiation (Cherry and Papoutsakis, 1988) within the fluidized bed system.
II. MSAD Program Tasks — Ground-based

By studying the optimal culturing conditions of osteoblasts and bone tissue, we can gain an understanding of the requirements for healthy bone formation and of the missing elements of microgravity which result in reduced bone mass and possibly also in bone diseases such as osteoporosis. The results of this study will also aid in developing techniques for propagating autologous grafts, and maximizing bone healing.

Progress During FY 1995:

During the last five months, we have re-established our protocol for culturing primary osteoblasts from rats. These cells are now characterized and we have begun flow studies. We are establishing ties with the Institute of Aging at UCSD to acquire human bone tissue as well. We have also established the culture of osteoblast-like cells UMR-106. During this period, we have acquired an STLV unit (from Dr. B. Palsson who is instructing us on its use). We are now in the process of characterizing the hydrodynamic environment in the bioreactor with the use of shear-sensitive bioluminescent dinoflagellate marine organisms. This latter technique is one we are pioneering here at UCSD for the flow characterization of shear stresses by visualizing bioluminescence emitted by *pyrocystis lumula* species of marine plankton.

In related research, we have developed an animal model of altered flow through bone. Since our ultimate objective is to determine the role of flow perfusion of bone in osteoporosis of disuse and microgravity-induced osteoporosis, this rat model will provide an appropriate complement to our in vitro models. Preliminary results suggest that increased perfusion of rat tibia by femoral vein ligation or venous tourniquet stimulates increased bone mass.

<table>
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STUDENT IDENTIFICATION: 962-23-01-22
NASA CONTRACT NO.: NAG9-837
RESPONSIBLE CENTER: JSC
Microgravity Studies of Cell-Polymer Cartilage Implants

Principal Investigator: Lisa E. Freed, M.D., Ph.D. Massachusetts Institute of Technology (MIT)

Co-Investigators:
Langer, R. Massachusetts Institute of Technology (MIT)
Goodwin, T.J. NASA Johnson Space Center (JSC)
Vunjak-Novakovic, G. Massachusetts Institute of Technology (MIT)

Task Objective:
The long-term objectives of our current studies are: (1) to assess the effects of in vitro culture conditions, microgravity in particular, on cartilage tissue morphogenesis, (2) to correlate the characteristics of engineered cartilage with bioreactor fluid dynamics in the form of physically and biologically sound mathematical models, and (3) to elucidate the mechanisms underlying the effects of microgravity on the structure and function of the engineered cartilage. Related practical objectives are to optimize bioreactor design and develop operating strategies for cultivating tissues under conditions of simulated and actual microgravity.

Task Description:
A simulated microgravity environment can be used to engineer cell-polymer tissue constructs, the size and shape of which are determined by an FDA approved, biocompatible, biodegradable scaffold with a defined three dimensional (3D) shape and structure. In particular, fibrous polyglycolic acid (PGA) can be seeded with isolated cells (chondrocytes) and cultivated in rotating bioreactors to make cartilaginous tissue constructs for in vitro studies of tissue morphogenesis and/or in vivo implantation.

Task Significance:
Engineered cartilage (i.e., tissue constructs grown in vitro using isolated cells and biomaterial scaffolds) can be used in vivo, to create subcutaneous neocartilage (in nude mice) and for joint resurfacing (in rabbits), and thus represents a biologically based therapy for repairing cartilage damaged by congenital defects, arthritis, or trauma. We have shown that the structure of engineered cartilage depends on hydrodynamic forces during in vitro cultivation; this is similar to the known effects of environmental forces on tissue morphogenesis in vivo. For example, structural organization of bone and cartilage depends on the mechanical distribution of compressive and tensile stresses. Ground-based research utilizing rotating vessels (simulated microgravity) is expected to enhance our understanding the principles governing tissue morphogenesis. Space studies (actual microgravity) can further extend the operating limits of these vessels. The same approaches and methodologies can also be applied to other cell-polymer model systems, in order to engineer other clinically useful tissues.

Progress During FY 1995:
Specific Aim (1):
Establish methods to culture chondrocytes on 3D synthetic, biodegradable polymer scaffolds in a rotating vessel to regenerate cartilaginous tissue.

Constructs based on bovine calf chondrocytes and PGA scaffolds were seeded in spinner flasks and transferred into rotating vessels (n=12 per 110 cc STLV). Within 1 month of cultivation, cartilaginous tissues were produced that were 7-8 mm diameter x 3-4 mm thick and resembled explants of natural bovine articul cartilage with respect to morphological appearance, biochemical composition and biomechanical properties. Morphologically, constructs consisted of uniformly distributed lacunae containing round cells within a compact cartilaginous extracellular matrix and a thin surface layer of flat cells. Biochemically, 2-3 week constructs contained 18% more water, 19% less glycosaminoglycan, GAG, and 55% less collagen than natural cartilage. The biomechanical properties of tissue engineered cartilage improved continuously over the course of in vitro cultivation as assessed in creep-recovery.
II. MSAD Program Tasks — Ground-based

Discipline: Biotechnology

studies: 2-3 week constructs were seven-fold more stiff than the initial PGA scaffold and half as stiff as natural cartilage.

Specific Aim (2):
A perfused rotating vessel was designed that consisted of the annular space \(120 \text{ cm}^3\) between two concentric cylinders with diameters of 5.75 and 2cm. The vessel containing cartilage discs (n=12) was simultaneously perfused with tracers (aluminum powder and blue dextran dye) and rotated such that the discs were maintained in a state of continuous free-fall. Convective mixing was generated by the settling discs, which oscillated, tumbled, formed wakes and shed vortices. Fluid-dynamic parameters including construct settling velocity (2-3 cm/s), settling Reynolds number (114-207), average hydrodynamic stress (1.5 dyn/cm²), non-dimensional dispersion \(0.918\), Peclet number (3.8), and equivalent number of perfectly mixed vessels in series (1.1) demonstrated excellent mixing in conjunction with relatively low shear in rotating vessels under the operating conditions selected for the cultivation of tissue constructs.

Specific Aim (3):
Correlate the characteristics of engineered tissues with specific fluid dynamic parameters in order to optimize bioreactor operating conditions.

Mixing affected the structure and function of tissue engineered cartilage as follows. Constructs cultured statically were thin and irregularly shaped with rough surfaces while those grown under mixed conditions were thick and regularly shaped with smooth surfaces. Biochemically, constructs grown in mixed flasks contained up to 50% more cells, 50% more GAG, and 125% more collagen than constructs grown in static flasks. A 300mm thick capsule consisting of multiple layers of flat cells and collagen formed at the surface of constructs grown in flasks. Constructs grown in rotating vessels were more elastic and less dissipative than those grown in mixed spinner flasks, in which the surface capsule increased the time required for constructs to recover from an applied compressive load.

Collaborations: A space study within the Shuttle/Mir Program is currently scheduled to begin in August 1996. In brief, bovine calf chondrocytes will be seeded on polymer scaffolds at M.I.T. and transferred into the Engineering Development Unit - Mir (EDU-M) prior to launch. Cell-polymer constructs will be cultivated in space for five months and then returned to M.I.T. for structural and functional analyses. Parallel ground studies will be done in order to study the effects of actual microgravity on cartilage tissue.

**Students Funded Under Research:**

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**Task Initiation:** 3/94 **Expiration:** 3/95

**Project Identification:** 962-23-01-11

**NASA Contract No.:** NAG-9-655

**Responsible Center:** JSC

**Bibliographic Citations for FY 1995:**

**Journals**


II. MSAD Program Tasks — Ground-based  
Discipline: Biotechnology

Books


Presentations

II. MSAD Program Tasks — Ground-based

Microgravity Tissue Engineering

Principal Investigator: Lisa E. Freed, M.D., Ph.D. Massachusetts Institute of Technology (MIT)

Co-Investigators:
- Vunjak-Novakovic, Dr. G. Massachusetts Institute of Technology (MIT)
- Cohen, Dr. R. Massachusetts Institute of Technology (MIT)
- Gooch, Dr. K. Massachusetts Institute of Technology (MIT)
- Schmidt, Dr. C. Massachusetts Institute of Technology (MIT)
- Nerem, Dr. R. Georgia Technical University
- Stamenovic, Dr. D. Boston University

Task Objective:

Engineered tissues can be grown in vitro using isolated cells, biodegradable three-dimensional (3-D) polymer scaffolds, and tissue culture bioreactors. Our working hypothesis is that NASA rotating vessels offer unique fluid dynamic and mass transfer conditions that selectively stimulate cells to maintain their differentiated phenotype and form functional tissue constructs. Our scientific and practical objectives are as follows:

1) Characterize mixing and mass transfer in bioreactors designed for the cultivation of 3-D cell-polymer tissue constructs. Establish flow conditions that promote tissue regeneration. Quantitate hydrodynamic forces acting on the constructs over the course of in vitro cultivation.

2) Extend the chondrocyte-polymer-bioreactor system to a variety of cell types including cardiac myocytes, osteoblasts, neuronal and endothelial cells, and tumor cells.

3) Develop structure-function relationships for the engineered tissues described in objective 2 in the form of mathematical models based on experimentally measured tissue properties (histological, physiological, biochemical, and biomechanical).

4) Optimize a bioreactor design for microgravity tissue engineering. Design and characterize bioreactor prototypes to be incorporated into flight hardware for space studies of tissue morphogenesis.

Our long-term goal is to improve our basic understanding of tissue morphogenesis. The use of NASA bioreactors in conjunction with cells and 3-D polymer scaffolds is expected to form a basis for microgravity tissue engineering for a variety of clinical applications.

Task Description:

Tissue morphogenesis will be studied using isolated cells, synthetic biodegradable polymer scaffolds, and tissue culture bioreactors (including rotating vessels) in complementary ground and space studies. The project will be realized through the joint effort of three research teams located at the Massachusetts Institute of Technology, Georgia Institute of Technology and Boston University, in cooperation with the NASA Johnson Space Center.

Aim 1: The effects of flow, mixing and mass transfer on tissue growth will be assessed using a variety of tissue culture bioreactors, including rotating vessels. Baseline tissue culture parameters will be those established for tissue engineered cartilage in rotating vessels (i.e., 110 cm³ STLVs containing n-8-12 chondrocyte polymer constructs 5-10mm diameter x 3-5mm thick and rotated at a speed at which the constructs are maintained in a continual state of free fall). Bioreactor operating conditions will be defined that provide for the changing construct requirements over the course of in vitro cultivation.
II. MSAD Program Tasks — Ground-based

Discipline: Biotechnology

Aim 2: The model system will be extended to a variety of cell types in addition to chondrocytes, including cardiac myocytes (to form contractile tissue constructs up to 1 cm in diameter x 0.2 cm thick for physiological or pharmacological studies), osteoblasts (to form bone implants for orthopedic surgery), neuronal and endothelial cells (to study innervation and vascularization of tissue engineered constructs), and tumor cells (for recombinant protein production). For each cell type, appropriate in vitro culture conditions will be established with respect to mass transfer requirements for nutrient supply and waste removal and acceptable levels of shear stress.

Aim 3: Construct structure will be correlated with function over the course of in vitro tissue morphogenesis. For example, tissue engineered cartilage will be compared with natural cartilage and the initial polymer scaffold with respect to histological appearance (distribution of structural elements), biochemical composition (amounts of water, cells, collagen and glycosaminoglycan) and biomechanical properties (apparent stiffness and permanent deformation).

Aim 4: Bioreactor design and operating conditions for further ground studies and the Shuttle/Mir biotechnology experiment scheduled for August 1996 will be optimized based on the results of research described in Aims 1-3. Several bioreactor designs will be tested to select the appropriate vessel configuration and operating conditions for tissue morphogenesis in actual microgravity. In order to provide the required levels of shear stress and mass transfer in the absence of gravity, other mechanisms to generate fluid motion at the construct surface will be explored. Parallel ground and flight studies will be done in order to compare the effects of simulated and actual microgravity on cartilage tissue.

Task Significance:
The proposed studies are motivated by the need for basic research in the area of microgravity tissue engineering. Tissue morphogenesis is known to depend on exogenous forces, presumably due to hydrodynamic effects on cell function and mass transfer. Requirements for the in vitro cultivation of engineered tissues include: (1) 3-D scaffolds for cell attachment and tissue regeneration, (b) an adequate supply of nutrients and gases, and (c) a hydrodynamic environment that is permissive for tissue morphogenesis or even designed to promote selected cell functions.

Our current studies indicate strong correlations between cartilage tissue morphogenesis and hydrodynamic forces in the in vitro tissue culture environment. Simulated microgravity conditions in rotating vessels permitted chondrocytes to maintain their differentiated phenotype and form biomechanically functional cell-polymer constructs, while pilot space flight studies (STS-62, 2/94) showed chondrocyte growth and metabolism in actual microgravity. These studies demonstrated the feasibility and advantages of using cell-polymer cartilage constructs cultured in rotating vessels as a model system for microgravity tissue engineering.

In addition, polymer scaffolds and rotating vessels will be used in conjunction with a variety of other cell types to cultivate cardiac tissue constructs for in vitro physiological or pharmacological studies, engineered bone for orthopedic surgery, nerve and blood vessel constructs for implant innervation and vascularization studies, and tumor cell aggregates for recombinant protein production. In all cases, complementary ground-based and flight studies will permit us to correlate cultivation conditions, including the effect of gravity, with tissue morphogenesis. Microgravity tissue engineering holds promise for advancing our basic understanding of cell growth and function, and developing new tissues to study and/or treat human diseases.

Progress During FY 1995:

Specific Aim (1): Characterize mixing and mass transfer in tissue culture bioreactors. Establish flow conditions that promote tissue regeneration. At MIT, long-term (up to 10 weeks) cultivations of cell-polymer cartilage constructs (5-10 mm diameter, 3-4 mm thick) were carried out under three different sets of mixing conditions: static, rotating vessels (STLV), and turbulent spinner flasks. Assessment of construct morphology, biochemical composition, metabolic activity (nutrient consumption and byproduct synthesis), and biomechanical properties are underway. In separate studies, spinner flasks were seeded with various numbers of chondrocytes per polymer scaffold and the kinetics, yield and uniformity of cell attachment are being evaluated. At Georgia Tech, a flow chamber for cultivating chondrocyte monolayers and tissue constructs under
defined fluid dynamic conditions is being fabricated and assays to assess cell morphology and gene expression are being developed.

Specific Aim (2): Extend the cell-polymer-bioreactor system to a variety of cell types (cardiac myocytes, osteoblasts, neuronal, endothelial and tumor cells). Various cell types are being studied with respect to acceptable levels of mass transfer and shear during in vitro cultivation. At MIT, cardiac tissue constructs based on chick embryonic cardiac myocytes and polymer scaffolds were cultivated for up to one month in rotating vessels (HARV) and spinner flasks and evaluated functionally (microelectrode recordings of spontaneous and paced signals) and histologically (light and electron microscopy); biochemical assays (e.g., myosin content) are under development. Methods were established to culture rat calvarial osteoblasts on two types of polymer scaffolds (polyglycolic acid with and without polyactic acid coating) in rotating vessels and spinner flasks. Pilot innervation studies were done in which chick embryo sympathetic neurons and heart cells were co-cultured on polymer scaffolds. At Georgia Tech, mouse pituitary tumor cells (AT-T-20 cells) are being assessed with respect to cell aggregation and recombinant protein production.

Specific Aim (3): Develop structure-function relationships for engineered tissues in the form of models based on experimentally measured tissue properties. At MIT, correlations are being made between the morphology, composition and biomechanical properties of cartilage constructs cultured for different time intervals (up to one month) and natural cartilage exposed to various culture conditions (rotating vessels, spinner flasks, static). At BU, correlations are being made between confined and unconfined compression-recovery data obtained from tissue constructs and natural cartilage. This information is needed to establish methods (experimental, theoretical) to assess construct biomechanical properties.

Specific Aim (4): Optimize bioreactor design for microgravity tissue engineering. Cell-polymer cartilage constructs were cultured for one month in rotating vessels (110 mL STLSv) under defined conditions (50% refeeding every other day, continuous gas exchange through a 0.0036m^2 membrane). Medium levels of pH, dissolved gases and glucose were monitored and constructs were evaluated histologically and biochemically. The following preliminary set-ranges were established for baseline metabolic parameters:

\[
7.2 < \text{pH} < 7.45, \quad 40 < \text{pO}_2 < 150 \text{ mm Hg}, \quad 35 < \text{pCO}_2 < 80 \text{ mm Hg}, \quad \text{and} \quad 1.5 < \text{glucose level} < 4.5 \text{ g/L}.
\]

Cartilage tissue constructs from MIT were cultured for up to one month in a prototype rotating perfused vessel (125mL EDU-M) at JSC with daily infusions of 60 mL (50%) and twice-daily perfusions (through a 0.4 m^2 membrane) and the parameters could be maintained within the above set-ranges. A separate set of tissue constructs have been cultivated at MIT for a flight unit (EDU-I) at JSC for a long-term (five month) ground-based study in preparation for the Shuttle-Mir study scheduled to launch in August 1996. In addition, a perfused system is being fabricated to assess the effects of nutrient and oxygen levels on cellular metabolic activities at Georgia Tech and a rotating perfused vessel is being fabricated for further cultivations of cell-polymer tissue constructs at MIT.

**STUDENTS FUNDED UNDER RESEARCH:**

- BS Students: 0
- MS Students: 0
- PhD Students: 9

**TASK INITIATION:** 9/95  **EXPIRATION:** 8/99

**PROJECT IDENTIFICATION:** 962-23-01-11
**NASA CONTRACT NO.:** NAG8-836
**RESPONSIBLE CENTER:** JSC

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Printed: 28 FEB 1996

II-194
II. MSAD Program Tasks — Ground-based

Protein and DNA Crystal Lattice Engineering

Principal Investigator: Dr. D. T. Gallagher

Center for Advanced Research in Biotechnology

Co-Investigators:

Gilliland, G.L.

Center for Advanced Research in Biotechnology (CARB)

Task Objective:

1) To enhance scientific understanding of the molecular interactions that control crystal growth.

2) To produce crystal contact mutants of the enzyme subtilisin and compare their crystal growth to that of wild-type.

3) To produce branched-DNA oligomers and study their structure using crystallography and other methods.

Task Description:

Standard molecular biology techniques will be used to make site-specific mutants of the enzyme subtilisin BPN\(_\text{e}\), which will then be crystallized using acetone. DNA that has been designed to contain stable branched structures will be synthesized and characterized by electrophoresis, UV spectroscopy, and other methods. Crystallization of the DNA will be attempted.

Task Significance:

Current advances in medical and other biotechnologies depend on knowing the precise structures of molecules as determined through crystallography. To do this you need crystals, but how protein and DNA crystals grow is still largely a mystery. These studies are aimed at the fundamental mechanisms of crystal growth. The balance of forces that operate in one well-characterized protein crystal system (primitive monoclinic subtilisin) will be measured by modifying key residues in the crystal contacts. In addition, the structure of branched DNA, which is important in many biological processes as well as potential synthetic biomaterials, will be investigated.

Progress During FY 1995:

Funding for this project began in September 1995. Thus there was only 1 month of progress in FY95. During this time starting materials were ordered and experimental designs were double-checked. Specifically, nucleotides were ordered for DNA synthesis and the design of the first two subtilisin mutants (Y91F and D259N) has been established.

STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 9/95  EXPIRATION: 8/99

PROJECT IDENTIFICATION: 962-23-08-37

RESPONSIBLE CENTER: MSFC

PRINTED: 28 FEB 1996
II. MSAD Program Tasks — Ground-based

Discipline: Biotechnology

Microgravity-Based Three-Dimensional Transgenic Cell Models

Principal Investigator: Dr. Steve R. Gonda

NASA Johnson Space Center (JSC)

Co-Investigators:

Yang, Dr. T.C.
Wu, Dr. H.
Richmond, Dr. R.C.
Short, Dr. J.

NASA Johnson Space Center (JSC)
KRUG Life Sciences
Dartmouth College
Stratagene

Task Objective:
The FY'95 objective is to develop and optimize microgravity-based three-dimensional cell culture models that have been genetically engineered for genomic containment of multiple copies of a defined target gene for mutational analysis.

Task Description:
Using the NASA bioreactor, we will examine and evaluate the growth characteristics of transgenic cells. We will focus on (i) the attachment phase, (ii) growth and proliferation phase, and (iii) the three-dimensional formation phase. Mono- and co-cultures of transgenic fibroblasts and epithelial cells will be studied.

Task Significance:
Advances on two fronts of biotechnology are merged to provide new and fundamental information on the roles of multicellular organization in genetic alterations caused by exposure to environmental conditions. Advances in genetic engineering of cells and advances in the development of three-dimensional models in the NASA bioreactor that are representative of multicellular tissue will be utilized to develop three-dimensional models for risk assessment of environmental insults.

Progress During FY 1995:
Initial studies are underway to establish the optimal culture of selected genetically engineered mammalian cells that contain multiple copies of a defined target gene. High Aspect Ratio Vessels (HARV) have been selected to culture 21 fibroblasts (Stratagene's Big Blue™ rat cell line) into three-dimensional models. Culture of rat 21 fibroblasts as three-dimensional aggregates was achieved using microcarrier beads as the attachment matrix.

STUDENTS FUNDED UNDER RESEARCH: TASK INITIATION: 9/95 EXPIRATION: 8/99
PROJECT IDENTIFICATION: 962-23-01-30
RESPONSIBLE CENTER: JSC
II. MSAD Program Tasks — Ground-based

Discipline: Biotechnology

Lymphocyte Invasion Into Tumor Models Emulated Under Microgravity Conditions In Vitro

Principal Investigator: Thomas J. Goodwin, M.S. NASA Johnson Space Center (JSC)

Co-Investigators:
- Pellis, Dr. N.R. NASA Johnson Space Center (JSC)
- Wolf, Dr. D.A. NASA Johnson Space Center (JSC)
- Becker, Dr. J. University of South Florida

Task Objective:
This application tests the hypothesis that microgravity provides a unique environment to create three-dimensional tumor models for the investigation of solid tumor invasion by human lymphocytes in vitro. We predict that this research will demonstrate that human lymphocytes oriented proximally to solid tumor models will be induced to invade and kill modeled tumor tissue. Two solid tumor models will be adopted from the NASA Rotating-Wall Vessel (RWV) bioreactor to propagate materials currently investigated by the applicant laboratory. Colon and breast cancer tissues co-cultivated with mesenchymal components yield tissue masses consistent with the investigation of the lymphatic invasion of human solid tumors. Tissue masses will be co-cultivated in stationary phase and RWVs with human peripheral blood lymphocytes (PBL) and with IL-2 activated PBL known as lymphokine activated killer (LAK) cells. Invasiveness will be analyzed quantitatively for adherence to lymphocyte populations to target tumors masses, the extent of infiltration, and the effect on tumor viability.

Previous investigations have shown that lymphocyte movement in simulated and true microgravity is substantially impaired when measured in type I collagen. The tumor models will be analyzed under the same conditions to determine if lymphocyte movement in native tissue rather than gelled collagen is profoundly affected by alterations in gravity load and vector.

The investigation will use the RWV technology in two applications, 1) production of tissue organoids and 2) analysis of the effect of gravity on lymphocyte movement. Completion of this proposed investigation will provide the experimental basis for establishment of three-dimensional models to investigate therapeutic strategies irrespective of whether immunological, chemical, or radiological. Furthermore, it will facilitate identification of the utility of microgravity in two areas of cell biology, tissue production and cellular movement.

Finally, in the later stages of experimentation, solid tumors created in simulated microgravity environment will be transitioned into true microgravity in a Space Shuttle experiment where they will be inoculated with LAK cells and the progress of these lymphocytes, activated and non-activated, will be measured via histological, immunohistochemical and biochemical techniques.

Task Description:
Hypothesis:

Microgravity provides a unique environment to create three-dimensional tumor models for the investigation of the solid tumor invasion by human lymphocytes in vitro.

Specific Aim 1: Develop the models of choice: a) colon tumor model and b) breast tumor model.

Specific Aim 2: Quantitate the degree of invasion into the tumor models by resting and activated normal PBL: a) Analyze adherence of lymphocytes to tumor models, and b) Immunohistochemical evaluation of invasion by location and distance traveled.
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Discipline: Biotechnology

Specific Aim 3: Determine if lymphocyte invasion into tissue involves a normal locomotory response.: a) Test invasion in the RWV known to inhibit PBL locomotion into solid matrix., b) Determine if lymphocyte activation in situ facilitates invasion in unit and microgravity, and c) Determine the relationship of cytokines produced by tumors to invasion.

Specific Aim 4: Determine the effect of tumor invasion by lymphocytes on cell metabolism and death: a) Determine changes in cellular metabolic activity as a result of LAK invasion in tumors, b) Analyze cell death in LAK infiltrated tumors, and c) Distinguish if cell death is necrotic or apoptotic.

Specific Aim 5: Determine the effect of true microgravity on invasive process by LAK cells.

Task Significance:

It is of paramount importance to understand the response of the immune system to tumors. In many cancers, the host immune system is triggered, deployed systematically, and even has its cells accumulate at the site of the tumor. Despite what appears to be an overwhelming hemostatic response to destroy the tumor, the immune system seems to lose effectiveness at the tumor site. Therefore, it is critical that we model the tumor site to address strategies that will augment host immunity in cancer. Colorectal and breast carcinoma are the second and third largest cancer killers in the United States with 57,000 deaths occurring from colon and 46,000 deaths occurring from breast carcinoma in 1993. An estimated 153,000 new cases of colorectal carcinoma will occur along with an estimated 183,000 new cases of breast carcinoma. These carcinomas kill through either local or distant spread when the tumors metastasize. Survival of patients afflicted with colonic and breast carcinoma has not improved dramatically in recent decades, and the mechanisms for control of the metastatic process are poorly understood. Over the last four years, the NASA Johnson space Center has developed in vitro tumor models using RWVs in several cell systems. Colon, breast, ovarian, and prostate cancer are but a few. Construction of these three-dimensional, in vitro tumor models has revealed striking similarities to tissues grown in vivo. In addition to the solid tumor models that have been created for study of the respective diseases, normal cellular models have also been created to study cell/cell interactions and the development of interstitial and cellular matrices which form in vivo. Recently, space flight experiments conducted in actual microgravity aboard the Space Shuttle have revealed characteristics of locomotion through solid matrix by normal human PBL. The mechanisms involved in the locomotion of PBL initiated in microgravity is currently under study; however, investigations that are on-going have elucidated methodologies to elicit locomotion from PBL under normally non-locomotory conditions which rule out the necessity for chemical induced. These methodologies may be employed to arrest solid tumors prior to metastasis by activation of lymphocytes at the tumor site. Three-dimensional tumor models developed in microgravity allow the study of this scenario.

Progress During FY 1995:

Substantial progress has been made establishing that randomized gravity (simulated microgravity, [SM])provides a unique environment of low shear and turbulence to create three-dimensional (3D) tumor tissue models. Solid tumor models of colon (LS174T) and breast (MCF-7) cancers were established in rotating-wall vessel (RWV) culture to study the induction of lymphokine-activated killer (LAK) cells that invade and kill solid tumors. LS174T and MCF-7 were grown in RWV and stationary culture for 21 days on cultispher-GL microcarriers (Hyclone Labs) and GTSF-2 medium (Goodwin, et al., 1993). Cells were seeded at 2.0 X 10^5 cells/ml with 1 mg/ml microcarriers. Stationary LS174T and MCF-7 cultures achieved average densities of 9.6 X 10^3 and 1.07 X 10^4, while RWV cultures achieved 1.92 X 10^4 and 8.3 X 10^6 cells/ml. Guacose utilization rates at 21 days of culture for LS174T ranged from 4.2 and 1.27 mg/dl/hr in stationary phase to 6.4 and 2.98 mg/dl/hr in RWV culture, respectively. LS174T RWV cultures were positive for known differentiation markers such as sucrase-isomaltase, villin, desmin and expressed an increase in normal colon antigen (NCA). Similarly, MCF-7 RWV cultures displayed Estrogen receptor (ER) Keratin 8,18,19 and polymorphic epithelial mucin -positive cells. LS174T and MCF-7 models both expressed apical polarized, microvillus surfaces. Thus the development of differentiated 3D tissue assemblies in SM of colon and breast provide the plausible models to investigate the process of LAK cell adherence and invasion into solid tumor in vitro.
II. MSAD Program Tasks — Ground-based

STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 8/95  EXPIRATION: 8/99
PROJECT IDENTIFICATION: 962-23-01-32
RESPONSIBLE CENTER: JSC

Printed: 28 FEB 1996
Differentiation of Cultured Normal Human Renal Epithelial Cells in Microgravity

Principal Investigator: Dr. Timothy G. Hammond
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Co-Investigators:
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Goodwin, T.J.
Albrecht, R.M.
Univ. of Wisconsin - Milwaukee Clinical Science Center
NASA Johnson Space Center (JSC)
University of Wisconsin, Madison

Task Objective:
Renal tubular injury is a common, lethal problem costing many billions of federal health care dollars annually. Although the molecules postulated to mediate many forms of this injury have been identified in vivo (The "Heymann antigen" megalin (gp330) and an intermicrivial glycoprotein (gp280)), the field is impeded by the lack of culture conditions in which kidney epithelial cells contain to express these highly differentiated nephrotoxin receptors; recent evidence suggests that gp330 is the polybasic aminoglycoside receptor. The purpose of this proposal is to explore microgravity as an innovative modality of cell culture which will lead to organ-specific differentiation of normal human renal epithelial cells expressing gp280 and gp330 in vitro.

Normal human renal cells have been difficult to obtain, cumbersome to purify, and impossible to maintain in differentiated form in culture. Our hypothesis is that simulated microgravity culture will provide a source of differentiated normal human epithelial cells expressing gp330 and gp280 in culture.

Task Description:
The specific objectives of this proposal are:

1. To compare enhancement of adhesion, proliferation, and organ specific differentiation renal epithelial cells in simulated microgravity (NASA rotating-wall perfused vessels) to conventional cell culture techniques (2-D monolayer, suspension culture, beads, polarization on Milipore inserts).

2. To test whether organ specific co-culture with cortical microvascular endothelial cells enhances adhesion, proliferation, and organ specific differentiation of renal epithelial cells in simulated microgravity (NASA rotating-wall perfused vessels) and conventional cell culture techniques.

Indices of organ specific epithelial cell differentiation include expression of the postulated injury mediators gp280 and gp330, as well as some structural and functional measures of properties specific to polarized cellular domains.

Task Significance:
Endocytosis by epithelial cells of the renal proximal tubule, serves to reabsorb and degrade the proteins that have escaped the glomerular filter, as demonstrated for a number of proteins. This specialized activity, "degradative endocytosis", is associated with a high turnover of plasma membrane which entails very efficient recycling carried out by specialized structures, the dense apical tubules. Understanding of this process is key to defining mechanisms of toxicity of common nephrotoxins from the polybasic aminoglycoside antibiotics, through myeloma light chains. A cell culture model expressing the receptors for these and other toxins, will greatly enhance studies of renal toxicity of pharmaceuticals.
II. MSAD Program Tasks — Ground-based

Discipline: Biotechnology

Progress During FY 1995:
We have collected substantial preliminary data pending funding of our application.

We have grown normal human proximal tubular cells in culture, both highly purified by differential trypsinization, and in co-culture with the physiological cellular mixture of the normal renal cortex. Culture of the renal proximal tubular cells in Slow-Turning Lateral Vessels compared to non-adherent bags, stirred fermentors, or conventional 2-dimensional culture is associated with a significant increase of polarized features. Polarity is characterized by increased number of microvilli, and endosomal differentiation including the formation of dense apical tubules. These effects are much more marked in the cells co-cultured in the presence of the cortical cellular mixture, than in pure proximal tubular cultures. There is expression of gp330 in the polarized renal proximal tubular cells in culture.

STUDENTS FUNDED UNDER RESEARCH:

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TASK INITIATION: 9/95  EXPIRATION: 8/99

PROJECT IDENTIFICATION: 962-23-01-29

NASA CONTRACT NO.: NAG9-811

RESPONSIBLE CENTER: JSC

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Presentations


II. MSAD Program Tasks — Ground-based

Excitable Cells and Growth Factors under Microgravity Conditions

Principal Investigator: Dr. Charles R. Hartzell

Co-Investigators:

Schroedl, N.
Gonda, S.

Task Objective:

Peptide growth factors are intimately involved in the regulation of normal muscle growth and differentiation. Invaluable ground work has been laid by investigators using numerous muscle cell lines to elucidate the contributions made by these growth factors in myogenesis, and significant advances in understanding these mechanistic pathways have been achieved. We underscore, however, the importance of confirming these results in primary muscle cultures.

Task Description:

Using the NASA bioreactor, we will examine the effects of a three-dimensional architecture on the growth and differentiation of neonatal rat heart cells and young adult muscle satellite cells. The bioreactor allows muscle cells to orient and grow within constraints normally determined by the basal lamina in vivo, yet permits experimental parameters to be cleanly delineated. Once muscle cultures are established, the role of neuromuscular junction formation on myogenesis will be explored by co-culture of heart or skeletal muscle cells with cholinergic neurons. Alterations in the differentiation program induced by fibroblast growth factor, insulin-like growth factor-I and transforming growth factor-b will be evaluated.

Task Significance:

The two-dimensional, unit-gravity constraints of conventional cell culture do not optimally model the three-dimensional cytoarchitectural design of the in vivo system. Limitations inherent in standard cell culture systems encourage us to continue the development of an innovative in vitro model system that is not limited by gravity-induced constraints and that promotes the formation of three-dimensional, in vivo-like tissue that is critical to understanding myogenic regulation.

Progress During FY 1995:

1. Evidence for multiple satellite cell populations

We have initiated studies to determine if different populations of satellite cells provide nuclei to growing and regenerating muscle fibers. Thirty-one percent of cells isolated from bupivacaine-induced regenerating muscle and 66% of cells from controls were identified by desmin staining as satellite cells. Within this muscle population, 28% of the colonies contained 8-44 large cells only whereas the remainder of the colonies were composed of 60-226 mostly small and some large cells. Regeneration did not effect either the percentage of muscle colony type isolated or the ability to proliferate in vitro. Satellite cells from regenerating and control muscles formed myotubes and expressed myosin heavy chain at similar levels. Treatment of regenerating cultures with dexamethasone resulted in a 16% increase in the number of desmin-positive colonies. These results suggest that two distinct populations of satellite cells can be isolated from muscle and a third population can be induced by glucocorticoids to express desmin and possibly to participate in myogenesis.

2. Characterization of the muscle cell line RMo

To eliminate some of the intrinsic heterogeneity associated with primary rat satellite cell cultures, we were interested in obtaining a cell line that was derived from the rat and was responsive to growth factors, formed...
contractile myotubes and expressed myosin isoforms and myogenic transcription factors like adult satellite cells. The better known L6 line did not meet these standards, however, the rat myoblasts omega or RMo line (Merrill, 1989) was intriguing to us because its growth and differentiation properties in culture was like satellite cells. Since little information concerning the phenotype of RMo cells was available, we characterized the expression of myogenic factors (MyoD, myogenin, MRF4 and myf5), intermediate filament proteins (desmin and vimentin), myosin heavy chain isoforms (embryonic, neonatal, and adult fast IIB) and the α and β subunits of the acetylcholine receptor of RMo cultures. Data from these experiments suggest that RMo cells express a mature myogenic program in vitro that is significantly more like that in rat satellite cells than the program expressed by the L6 line.

3. Growth factor regulation of muscle development in three-dimensional (3D) tissue culture

What affect growth factors have on the myogenic program when cell-cell or cell-substratum interactions are not confined to two dimensions (2D) is unknown. We investigated the response of RMo myoblasts to basic fibroblast growth factor (bFGF) and insulin-like growth factor-I (IGF-I) in 3D tissue culture. Myoblasts were grown on microcarrier beads suspended in the High Aspect Ratio Vessel (HARV). Proliferation of RMo myoblasts in the HARV was responsive to different serum concentrations, and cells underwent limited proliferation for 72h in a chemically defined medium (DMC). Addition of bFGF and IGF-I separately to defined medium increased the proliferation of myoblasts 1.7-fold and 1.3-fold, respectively. When bFGF and IGF-I were added together, proliferation of myoblasts increased 4.4-fold, suggesting the two factors acted synergistically to enhance proliferation in 3D culture. Importantly, this enhanced proliferation of RMo cells by bFGF and IGF-I, either alone or in combination, was not observed in flat (2D) culture.

4. Development of three-dimensional muscle grafts in vitro for transplantation

Although growth of muscle cells on microcarrier beads in the HARV resulted in 3D interactions of cells, the arrangement of muscle fibers on microcarriers was random. For this reason, we chose to develop a system that promotes 3D interactions of cells but also facilitates the parallel arrangement of myotubes in vitro with the idea of grafting this tissue into a muscle bed in vivo. Using a modified culture system developed by Stohman (1990), we were able to grow macroscopic (1.5cm x 1mm) pieces of skeletal muscle that actively contracted. Cross sections stained with trichrome demonstrated that the interior of the muscle was composed of a mixture of muscle and connective tissue and a well defined layer of connective tissue surrounded the piece, appropriate in size and composition, for grafting back into the adult rat. To follow the fate of grafted muscle in vivo, we have developed several RMo myoblast clones that have been transfected with a replication-defective retrovirus containing the β-galactosidase gene. Hybrid muscle fibers composed of primary rat satellite cells and transfected RMo myoblasts are identified using simple histochemistry and should provide us with a marker system to follow the fate of grafted muscle in vivo. This important model system can now be used to monitor changes in biochemical, genetic or gravity parameters either in vitro or in vivo.

5. Biological Characterization of Cardiac Cell Cultures in 3D

Increasing evidence suggests that cells cultured in microgravity exhibit profound alterations in both detectable phenotypes and cellular responses; however, the mechanism(s) by which these alterations occur remains unclear. At unit gravity, neonatal rat cardiac cells beat spontaneously and rhythmically after 16 to 20 hours in culture; this coordinated contractile activity is dependent on specific cellular interactions as well as on the organization of the cytoskeleton and organelles, precise Ca²⁺ handling, and efficient energy metabolism. Since alterations in the structures and activities of the myocardial cells represent adaptations to specific culture conditions, we have characterized some of the effects of microgravity on the morphology and contractile functions of neonatal rat heart cells in the HARV bioreactor.

Cardiac cells form distinctive three-dimensional structures along the surfaces of the support matrices and among groups of unsupported cells. These structures are contractile; however, the contractile activity (i.e., beat frequency) of HARV-cultured cardiac cells is invariably reduced compared to parallel control cultures carried out on traditional tissue culture flats. The degree of beat frequency reduction varies, but HARV cultures typically beat at less than
half the rate of controls and this beating is often non-rhythmic. The explanation for this decrease in contractility may have to do with the method of observation: HARV-cultured cells must be sampled through the access ports, then placed into a tissue culture dish prior to viewing on a microscope; control cultures are viewed directly but can be similarly affected by moderate levels of shear (e.g., feeding). Alternatively, differences in contractility may be due to the consistent exposure of the cells to the low level of shear inherent in HARV culturing, they may be due to differences in gas tension (O2/CO2) between the HARVs and traditional culture, or they may be due to differences in either paracrine stimulation or cellular signal transduction.

6. Ultrastructural Examination of Cardiac Cell Cultures

The organelles of cardiac cells become organized into a distinct pattern when cultured using traditional culture methods: nuclei tend to localize toward the basolateral surface (toward the culture dish), and the majority of sarcomeres occur between the nucleus and the apical cell surface. We have investigated the distribution of organelles in HARV-based cultures to determine whether their distribution is directed by the extra-cellular matrix or by the direction of the gravity vector. Our observations suggest that the distribution of organelles in the HARV-cultured cells more closely resembles that seen in vivo (nuclei are more centrally located). We are currently confirming our preliminary observations of HARV-cultured cardiac cells, but we can tentatively conclude that the distribution of organelles in traditional cardiac cultures results from the gravitational vector and not from an extra-cellular matrix signal. We are continuing and extending our ultra-structural observations in order to specifically identify the inter-cellular junctions formed in HARV cultures.

7. Effect of Growth Factors on Cardiac Cell Myosin Expression

Neonatal rat cardiac cells are sensitive to changes in the hormonal status of the animal. In vitro, this sensitivity is maintained; specifically, the levels of a and b myosin change with exposure to triiodothyronine (T3, causes a switch from b to a myosin) or basic fibroblast growth factor (bFGF, mimics hypertrophy and induces b myosin). We have begun characterizations of the dose responsiveness of cardiac cells in HARV and traditional (flat) culture methods. The effects of T3 on contractility have been established: prolonged exposure of cells to T3 results in an elevation in beat frequency over control. These results are consistent with a myosin isoform switch from b myosin to a myosin. Differential electrophoretic migration assays have been used to determine protein levels of a and b myosin in flat cultures using doses of T3 ranging from 0 to 3 mM. Our results support the published descriptions of myosin regulation in cardiac cells and coincide with the physiological effects on contractile frequency noted above. In addition, we are developing a PCR-based method for the quantitation of relative messenger RNA levels.

8. Culture of cholinergic neurons

The cholinergic neuronal cell line, NG08-15 was obtained from Dr. Marshall Nirenberg at NIH. We found that the cells would proliferate well in either the medium specified by Dr. Nirenberg or in the serum-supplemented, DMEM-based medium normally used for the culture of satellite cells. Although dibutyryl cAMP was considered by Nirenberg, et al., to be essential for the induction of differentiation in the NG108-15 cells, we observed significant neurite outgrowth in two different serum-free media developed in our laboratory. Thus, we were able to successfully coculture the neuronal cells with cardiac cells (in SFHM medium) and satellite cells (in DMC supplemented with hemin). We are currently sub-cloning NG08-15 cells to generate a more homogenous population for subsequent assays, and we are characterizing NG108-15 differentiation in culture by using a combination of two molecular markers: choline acetyl transferase and monosialyl-ganglioside (GM2).

9. Coculture of Cardiac and NG108-15 Cells

The coculture of neonatal rat cardiac cells with the neuroblastaoma/glioma fusion cell line NG108-15 leads to differentiation of NG108-15 cells and cholinergic innervation of the cardiac cells. Standard cardiac cell cultures maintain a spontaneous beat frequency about 5-6 times faster than co-cultured cardiac cells. Treatment of co-cultures with atropine (2 mg/ml) results in an increase in cardiac cell beat frequency by approximately 1.8 fold; the subsequent addition of the adrenergic agonist isoproterenal (16 mg/ml) further stimulates cardiac cell contractility by increasing beat frequency another 1.7 fold. Interestingly, a atropine had no effect and isoproterenal had little effect...
on cardiac cells cultured without NG108-15 cells; both control and co-cultured cells exhibited negative chronotropic responses to the adrenergic antagonist (10 mg/ml) propranolol. These observations will be extended to HARV cultures in which both NG108-15 cells and cardiac cells have been cultured.

10. Co-culture of neuronal cells with satellite cells

We hypothesized that cholinergic neurons would affect the maturation and neuromuscular junction formation of primary muscle cells in culture. Cells were grown in the customary serum-supplemented medium and at day 6 in culture NG108-15 neurons were added in defined medium containing 5 mM hemin. Neurons had no distinguishable effect on differentiation as indicated by myosin content. Conversely, acetylcholinesterase activity was increased 3-fold in the presence of neurons and clustering of acetylcholine receptors was significantly greater, suggesting enhanced formation of neuromuscular junctions.

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals


Presentations


Determining the Conditions Necessary for the Development of Functional Replacement Cartilage Using a Microgravity Reactor

Principal Investigator: Prof. Carole A. Heath  
Iowa State University

Co-Investigators:  
No Co-I's Assigned to this Task

Task Objective:  
The objectives of the project are to determine i) the level, frequency, and time course of applied compressive and/or shear stresses which result in regenerated tissue that is biologically relevant and functional over the long term, and ii) how chondrocyte growth and cartilage regeneration are affected by microgravity in vitro.

Task Description:  
Mechanical forces have been shown to affect the structure of developing cartilage in vitro. With the goal of achieving regenerated tissue that is biologically relevant and useful, we will determine the effects of varying the frequency and time course of applied stress(es), and the effects of microgravity on the development process. Isolated chondrocytes, the cells responsible for producing the components of cartilage, will be cultured in a three-dimensional mesh of resorbable polymer in a modified microgravity reactor which can be operated over the range from very low stress to high shear and/or high compression. By mimicking key aspects of the in vivo environment, i.e., structure and stress, we will determine the conditions necessary for regenerating functional cartilage in vitro from cultured chondrocytes as well as the effects of microgravity on the process.

Task Significance:  
The knowledge gained from this project will bring us closer to a treatment for osteoarthritis and other diseases and injuries of the articulating joints and to an understanding of the effects of microgravity, as experienced in space flight, on cartilage development. The generation of an in vitro engineered sheet of replacement articular cartilage, if it can be successfully implanted in vivo, would precipitate a revolution in the management of severely impaired dysfunctional joints.

Progress During FY 1995:  
Funding for this research became available at the very end of FY 95. Progress to date has included investigations of cartilage development under intermittent compression at low levels of pressure; studies at higher pressure levels are underway.

STUDENTS FUNDED UNDER RESEARCH:  
TASK INITIATION: 9/95  
EXPIRATION: 8/99  
PROJECT IDENTIFICATION: 962-23-01-34  
NASA CONTRACT NO.: NAG9-827  
RESPONSIBLE CENTER: JSC
II. MSAD Program Tasks — Ground-based

The Effects of Microgravity on Viral Replication

Principal Investigator: John H. Hughes, Ph.D. Ohio State University

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
The overall goal of this project is to assess the effects of microgravity on various aspects of viral replication. The specific objectives are:

1) To determine viral yields for different viruses grown in different cells at microgravity.

2) To determine the effects of either acute or chronic microgravity on the replication of several different human viruses that are grown in different cells.

3) To determine if cells latently infected with viruses are activated to produce more virus when grown at microgravity than when the same cells are grown at 1g.

4) To determine if viruses that cannot be grown in conventional cell culture systems at 1g can be grown in cells at microgravity.

For comparative and control purposes, viral replication in cells grown at hypergravity and 1g (standard culture) will also be examined.

Task Description:
For this project, we will use a simulated microgravity environment to investigate viral replication. The simulated microgravity environment will be generated by one of the NASA-designed Rotating-Wall Vessels (RWVs). Some key issues to be addressed are whether microgravity plays a role in viral-cell interactions and whether microgravity can effect viral pathogenesis. To study any effects of microgravity on viral replication, both DNA and RNA viruses will be used. For the effects of microgravity on latent viruses, we will test lymphoblastoid cell lines infected with the Epstein-Barr Virus and mouse cell lines infected with retroviruses. Lastly, differentiated cells induced at microgravity will be studied to determine if they will be permissive for the replication of viruses that currently cannot be grown in vitro. Viral yields for viruses produced at microgravity and 1g will be determined by plaque assay. For viruses that only replicate in vivo, a quantitative polymerase chain reaction will be used to monitor viral replication.

Task Significance:
Information about the replication of infectious agents at microgravity is limited. Our studies are aimed at understanding and determining the effects of microgravity on infected cells. If microgravity can activate latent viral infections, then the activation of varicella-zoster virus and the induction of shingles during spaceflight could be a significant health problem. Our proposed studies should help determine if microgravity has any adverse effects on latent viral infections.

In the past, virological studies with infected cells have opened new avenues of research involved with cellular functions. Currently, certain viruses such as some hepatitis viruses and wart viruses, etc., will only replicate in vivo but not in vitro. Since these viruses replicate in differentiated cells in vivo, there are obviously some cellular factors that permit their replication or inhibit their replication. Differentiated cells induced at microgravity may provide the critical cell or host-factors needed for the replication of certain viruses. If this is the case, then microgravity conditions could provide a new biotechnology tool for developing control measures (new vaccines and antivirals) as well as allowing for the production of new diagnostic products.
II. MSAD Program Tasks — Ground-based  

Discipline: Biotechnology

Progress During FY 1995:

The project started in September, 1995. After receipt of grant funds, equipment was purchased and the set-up of the laboratory was begun. Interviews were conducted lab personnel brought on-board to conduct the studies. Certain experiments have been started; however, there is minimal results to report at this time.

STUDENTS FUNDED UNDER RESEARCH: TASK INITIATION: 9/95 EXPIRATION: 8/99
PROJECT IDENTIFICATION: 962-23-01-26
NASA CONTRACT NO.: NAG9-818
RESPONSIBLE CENTER: JSC
Sensitized Lymphocytes for Tumor Therapy Grown in Microgravity

Principal Investigator: Dr. Marylou Ingram
Huntington Medical Research Institutes

Co-Investigators:
Techy, G.  
Saroufem, R.  
Narayan, S.  
Yazan, O.  
Goodwin, T.  
HMRI

Task Objective:
The NASA High Aspect Ratio Vessel (HARV) bioreactor is used to generate spheroids of human tumor cells. These spheroids are then used to sensitize the patient's lymphocytes to the patient's own tumor. The population of tumor-sensitized lymphocytes (TSL) is then expanded in culture in the presence of interleukin-2 (IL-2) and used in immunotherapy of the patient.

Task Description:
This is a small scale, in depth pilot study in which nonspecifically stimulated autologous lymphocytes (ASL) and tumor-sensitized lymphocytes (TSL) are administered serially by intralesional injection as local immunotherapy of invasive carcinoma of the urinary bladder. This is a modification of cellular immunotherapy that we have applied with encouraging results in clinical trials to treat recurrent malignant gliomas (1). For the bladder cancer, the urological surgeon will inject cells intralesionally at intervals of approximately three weeks. Response to therapy will be determined by direct observation of the lesions, serial biopsies for histological and cytological studies, clinical evaluations and radiological exams. The ability to biopsy lesions at various times after injection of stimulated immune cells is an important advantage in understanding response to therapy and in refining the therapy. Serial evaluation of lesions by histology, measuring the expression of major oncogenes and tumor suppressor genes, assays of cytotoxic effectiveness of ASL and TSL to tumor obtained from urine specimens or bladder washings can provide additional information between biopsy specimens. The pilot study provides an excellent opportunity to evaluate the NASA HARV bioreactor as a method for culturing tumors as spheroids as contrasted with conventional monolayer cultures. Tumor spheroids in vitro have a number of characteristics present in vivo but absent from monolayer cell cultures. These characteristics, some of which will undoubtedly be important in the generation of TSL cells, include the presence of specific tumor markers, angiogenesis and growth factors, biological response modifiers, gap junctions and tumor suppressor gene expression. Changes in the level of expression and distribution of intermediate filaments and the extracellular matrix elements have also been observed.

Task Significance:
Cancer of the urinary bladder is of special interest in immunotherapy because it has already been shown that immunotherapy in the form of BCG treatment is efficacious. It is now widely used in clinical management of invasive bladder cancer. Thus, there is sound evidence that appropriate mobilization of the patient's own cellular immune mechanisms can have therapeutic efficacy. A number of investigators have recognized the possibility that TSL may offer significant advantages in adoptive immunotherapy and there has been a recent resurgence of interest in this approach (2). TSL can be expected to show increased tumor cell killing, as do tumor-infiltrating lymphocytes (TIL) and they also hold forth the promise of serving as probes for identifying immunogenic gene products in the tumor.

Many elderly patients with multiple tumors of the urinary bladder cannot tolerate extensive surgery or extensive chemotherapy and may either have failed to respond to BCG immunotherapy or are too unwell to tolerate side effects of that therapy. These patients present a frustrating therapeutic problem to the urologist. We believe that local
immunotherapy would be well tolerated by these patients and that it is a rational therapeutic strategy. A clinical trial of this therapy will also provide valuable information about how stimulated immune cells select and identify their target cells, migrate through tissue, whether or not they continue to proliferate after injection and other important considerations that will advance our understanding of cellular immunotherapy and aid in refining it for treatment of bladder cancer and ultimately other cancers.

Bladder cancer has many advantages as a model for refining methodology. It permits serial administration of immunologically stimulated cells into the bladder lesions and yields serial biopsy specimens in which the interactions of immune cells and their tumor targets can be studied directly.


Progress During FY 1995:

During FY 95 we have expanded the set of human tumor cell lines cultured successfully without carrier beads in the HARV bioreactor. All these cell lines are otherwise anchorage dependent. Cultures are initiated with a monodisperse suspension containing 1-2 million cells per ml of nutrient medium and are maintained for periods of a few days to approximately a month. The spheroids produced are fixed in Carnoy's/Bouin's fixative, then sectioned and stained. Some sections of each specimen are stained with hematoxylin and eosin and other sections from the same specimen are stained immunohistochemically to demonstrate various markers of interest.

When cultured in the bioreactor, each cell line produces spheroids that demonstrate cell line-specific microscopic architecture. Thus, gliomas tend to form small, compact spheroids that sometimes fuse to form larger bodies; the PC3 cell line that was developed from prostate carcinoma metastatic in bone forms discrete spheroids in which cells are very loosely associated. Another prostatic carcinoma cell line, LNCaP, typically forms elongated tubule-like or folded structures. The bladder carcinoma cell line, HBL2, recently developed here, shows a definite tendency towards epithelial differentiation. The tendency for the HBL2 cells to assemble in such a pattern suggests that various cell adhesion molecules play a role in determining spheroid morphology. Immunohistochemical staining to demonstrate fibronectin, collagen, vimentin, CD44 and other cell adhesion molecules can be expected to provide further insight into spheroid formation and differentiation. So far, we have found cytokeratin, CD44 and E cadherin to be definitely up-regulated in spheroids as contrasted with monolayer cells.

Our clinical collaborator, Dr. Michael Bishai, urological surgeon, has provided additional surgical specimens of bladder tumors and these have been invaluable. They have allowed us to refine tissue culture methods specifically for bladder cancer. Most of the patients from whom the specimens were obtained did not meet protocol requirements (patient selection criteria) for the immunotherapy trial and Dr. Bishai managed these patients clinically using other therapeutic modalities. The fifteenth patient did meet protocol requirements and became the first protocol patient.

The immunotherapy protocol for bladder cancer derives from previous experience in this institute with local cellular immunotherapy that was applied with encouraging results in treating more than 150 patients with recurrent malignant gliomas. The general plan of the study is to give a series of intrallesional injections of stimulated autologous lymphocytes directly into individual tumors in the urinary bladder. The first injections consist of lymphocytes stimulated with phytohemagglutinin (PHA) and then cultured in the presence of interleukin-2 (IL-2) in a manner analogous to our earlier clinical trials of implantation immunotherapy for recurrent malignant gliomas. These autologous, non-specifically stimulated lymphocytes (ASL) are intended to initiate an immune reaction against the tumor while other lymphocytes are becoming specifically sensitized in vitro to a biopsy specimen of the patient's tumor. The latter lymphocytes are tumor sensitized (TSL). Once they appear in the tissue culture, they are expanded by further culture in the presence of IL-2 and included in subsequent injections.

The treatment plan is to inject stimulated lymphocytes into the base of individual tumors approximately every three weeks for the first quarter then quarterly thereafter. The first tumor biopsy obtained is the source of tumor for preparation of tumor-sensitized lymphocytes in vitro. Since this step usually takes at least a month, the initial injections of stimulated lymphocytes consist entirely of non-specifically stimulated ASL which are generated by
exposing resting peripheral blood lymphocytes to phytohemagglutinin in vitro to transform resting lymphocytes into proliferating lymphoblasts. This is followed by culture in the presence of IL-2 typically for about two weeks, to augment their numbers. When TSL become available, they are included in the population of cells injected, the goal being to achieve a 1:1 mixture ASL and TSL. Each tumor will have 500 million to one billion stimulated lymphocytes, in a volume of 1-2 ml of physiological saline, injected into the base at each injection. Ten thousand units of Amgen recombinant human IL-2 (r-met Hu IL-2 (ala-125)) per ml of cell suspension is included in the injected volume. IL-2 is not administered systemically.

The first patient received three intraleisional injections of ASL at intervals of approximately three weeks. Injections were very well tolerated and had no significant adverse effects. The patient died in his sleep the day before a scheduled fourth injection of cells and before receiving any TSL. Cause of death was myocardial infarct unrelated to immunotherapy. An autopsy was performed and tissue was obtained from various areas of the bladder. These sections showed multifocal and diffuse, dense collections of lymphocytes in all areas of the bladder and no evidence of residual tumor. Further case studies in which the therapeutic course is completed and there is adequate follow-up will, of course, be required before we can draw any conclusions about the therapeutic efficacy of the procedure. It is, however, somewhat encouraging that whereas multiple new tumors had invariable appeared in previous serial examinations in this case, no new tumors were found after the second injection of lymphocytes.

From the bladder cancer specimens obtained so far, we have established one bladder cancer cell line and at least one additional specimen may also yield a permanent cell line. The new cell line, designated HBL2, has been cultured in both monolayer culture and in the NASA bioreactor culture vessel. HBL2 cells, as well as minced tumor from some of the other bladder cancers and glioma and prostate cancer cell lines, provide important experimental and reference specimens for immunohistochemical demonstration of "markers" of interest and for comparison of "marker" expression in cells grown in monolayer as contrasted with the same cell lines grown as spheroids or examined in tumor mince specimens. Although we cannot, of course, expect to establish a permanent cell line (i.e., at least 25 passages) from every tumor, primary cultures and tumor mince will be available for use in preparing TSL in all cases. Where possible, lymphocytes to be tumor-sensitized for bladder cancer immunotherapy will be exposed to tumor mince, monolayer cultures and tumor spheroids in parallel so that we may compare the relative immunological effectiveness of the three modes of tumor presentation. We also hope to establish, from at least one tumor, a line of fibroblasts and a line of endothelial cells as well as tumor cells and to use these autologous cells in a structured series of co-cultures to observe histogenesis and the effects of active histogenesis on the expression of antigenic gene products.
Three-Dimensional Tissue Interactions in Colorectal Cancer Metastasis

Principal Investigator: Dr. J. M. Jessup  
New England Deaconess Hospital

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
The objective of this project is to test the fidelity with which microgravity models three-dimensional tissues by assessing how well microgravity effects the molecular and biological function of MIP-101, a human colorectal carcinoma.

Task Description:
MIP-101 is a poorly differentiated adenocarcinoma of the colon that was derived from the ascites of a patient who had widespread metastases within the abdominal cavity. MIP-101 cells are grown in the rotating wall vessel (RWV) with or without normal host cells and assessed for the production of carcinoembryonic antigen (CEA - a 180 kDa glycoprotein that is produced by carcinomas and used clinically as a tumor marker) and for biological behavior including adhesion and metastasis. Cells grown in microcarrier bead cultures in the RWV under simulated microgravity are compared to similar MIP-101 cultures grown on microcarrier beads in stationary culture as well as in conventional monolayer cultures.

Task Significance:
One of the prime goals of the biotechnology program at NASA/Johnson Space Center is to determine whether cultivation of cells in microgravity produces three-dimensional cultures that mimic the morphology and function of tissues in living animals or humans. The MIP-101 cells are an excellent test of this because they are poorly metastatic in experimental models of metastasis in athymic nude mice and do not produce CEA in conventional monolayer culture systems. We have shown that CEA injected into mice enhances production of liver metastases by MIP-101 cells and that MIP-101 cells will metastasize when implanted into the abdominal cavity after producing CEA. MIP-101 cells placed in the subcutaneous tissue of the mouse do not metastasize and do not produce CEA. Furthermore, cells grown on plastic or conventional substrates such as Matrigel or laminin are neither metastatic nor induced to produce CEA.

The conventional interpretation of these results is that the microenvironment (the three-dimensional environment) of the abdominal cavity induces MIP-101 cells first to produce CEA and then to develop blood-borne metastases in the liver and lungs of nude mice. Early experiments in the RWV question this conventional interpretation of the effects of host microenvironment, because they demonstrated that CEA production may be induced in MIP-101 cells when they are grown in the RWV in the absence of any abdominal cavity stromal cells. This suggests that the MIP-101 cells may produce CEA when they are allowed to grow in three dimensions. Thus, the absence of CEA production in subcutaneous tumors is due to an inhibition of CEA production by the subcutaneous tissue rather than promotion of CEA production and metastatic potential by the abdominal cavity.

This system is an excellent model in which to test the fidelity of the RWV culture system because the first stage in metastasis by MIP-101 cells appears to be the induction of CEA production, followed by the acquisition of the ability to develop experimental metastases after injection into the spleen of nude mice. Assessment of fidelity of the RWV culture system is simplified by first testing for the production of CEA, tumor marker, then assessing the biological aspects of metastasis.
Progress During FY 1995:
The primary goal of the Biotechnology Program at NASA/Johnson Space Center is to determine whether cells cultivated in microgravity produce three-dimensional cultures that mimic the morphology and function of tissues in living humans or animals. The MIP-101 cancer cell line is an excellent test of this functionality because these poorly differentiated human colon carcinoma cells are not metastatic and do not produce either carcinoembryonic antigen (CEA) or non-specific crossreacting antigen (NCA, a member of CEA gene family) when grown in monolayer culture. However, when MIP-101 cells are implanted in the abdominal cavity of nude mice, they produce CEA and NCA as well as develop spontaneous, blood-borne metastases in liver and lung. We have found through this project that MIP-101 cells grown in the rotating-wall vessel (RWV) that simulated microgravity produce CEA and NCA and that this may have an effect on the potential to metastasize. Considerable effort has been expended during the past year to precisely define the amount of CEA and NCA that are increased in the three-dimensional culture provided by the RWV. On a protein level, western blots have demonstrated that CEA is increased 1.5-fold in cells grown in the RWV compared to the level expressed in MIP-101 cells cultured in monolayers. The amount of NCA is increased even more in MIP-101 cells since it is 5 to 6-fold greater in RWV cultures than in monolayer cultures. When compared at the gene transcript level by the semi-quantitative technique of competitive RT-PCR, no CEA mRNA is detectable in either type of culture while MIP-101 cells grown in the RWV produce 0.04 attomoles of mRNA per mcg of DNA compared to 0.001 attomoles for NCA mRNA in monolayer cultured cells. These results are important because they indicate that three-dimensional growth is an important stimulus for the production of molecules that are involved in the spreading, or metastasizing, of cancer.

Last year we observed that MIP-101 cells grown in three-dimensional cultures appear to produce more H+ and carbon dioxide than monolayer cultures. In follow-up, we find that it appears to be consistent in both RWV and nonstick petri three-dimensional dish cultures. It is not clear if this is a process that is associated with the lack of diffusion in cells grown in three-dimensions. We are currently continuing to analyze this and will assess the production of key enzymes in the glycolytic pathway in three-dimensional cultures to their expression in monolayer cultures.

We have observed that as cultures of MIP-101 cells mature in the RWV, the cells generally get bigger as they begin to slow their proliferative rate. We have begun to analyze the cell cycle by flow cytometry as well as to assess the production of key proteins that regulate the cell cycle. We have found that, in certain cultures, cells accumulate in G2+M, whereas our preconception would suggest that they accumulate in G1+S, which is the major branch point for differentiation. These results are not consistent and have to be repeated numerous times to be sure that this trend is real. However, MIP-101 cells cultured three-dimensionally in either a static non-stick Petri dish or in the RWV produce more cyclin D1 and cyclins A and B1 than do MIP-101 cells grown in monolayers. Work is in progress to define the significance of this and how it relates to the changes in metabolism and the expression of NCA and CEA.

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals
II. MSAD Program Tasks — Ground-based

Discipline: Biotechnology

Presentations


Fibril Formation by Alzheimer's Disease Amyloid in Microgravity

Principal Investigator: Prof. Daniel A. Kirschner
University of Massachusetts

Co-Investigators:
Inouye, Dr. H.
University of Massachusetts

Task Objective:
(1) To identify specialists whose experience is in micro-fabrication of synthetic polymers into fibers.

(2) To initiate experiments whose objective is to obtain oriented fibrils of the full-length (or nearly full-length) amyloid-beta peptides.

Task Description:
Our previous experiments mainly utilized peptide fragments corresponding to the putative extracellular domain of amyloid-beta, although some 40-mers were studied (primate, rodent, and Dutch-haemorrhagic analogues). The difficulty in undertaking fiber diffraction from 40-mer samples is that, unlike most of the shorter peptides, the assembled fibers do not easily orient in a magnetic field, which results in more overlap among the x-ray reflections, in turn making the diffraction patterns considerably more difficult to interpret. However, considering the preponderance of the 1-40 and 1-42 amyloid proteins in AD tissue, we are focusing our fibrillogenesis efforts on these full-length peptides. Obtaining well-oriented fibers and interpretable x-ray patterns will have important bearing on our subsequent molecular modeling.

Task Significance:
In Alzheimer's disease, a normally-occurring protein termed beta-amyloid accumulates between the nerve cells. The formation and accumulation of this material profoundly affects the functioning of the central nervous system, as amyloid can be neurotoxic. To characterize the mechanism by which the amyloid forms and exerts its effects, we have been studying fibrillogenesis using the tools of structural biology (x-ray fiber diffraction, electron microscopy). A detailed understanding of the three-dimensional organization of amyloid will complement the information currently being obtained from analytical protein chemistry and immunocytochemistry, and more recently from light scattering and magnetic resonance, and will provide valuable insights into the pathogenesis of pre-amyloid deposits, neuritic plaques, and cerebrovascular amyloid. By establishing the molecular organization of amyloid, we hope to provide a rationale basis for developing agents for diagnostic imaging of the deposits and therapeutics for preventing or arresting fibrillogenesis.

Progress During FY 1995:
During the nearly two months of FY95 that this grant was in effect, at a time when we were preoccupied with moving our lab from Neurology Research at Children's Hospital in Boston to the Department of Biological Sciences at UMass-Lowell, we were able to identify scientists at the new institution, as well as at UMass-Amherst and at the Army Materials Research Laboratory in Natick, Massachusetts, who have considerable experience in microfabrication of fibers from synthetic polymers. Contacts with these scientists have been initiated, and we hope to begin experiments to assemble oriented fibers of the full-length peptides within the next two months, as our new lab becomes functional.

Students Funded Under Research: Task Initiation: 8/95 Expiration: 8/98
Project Identification: 962-23-08-32
Responsible Center: MSFC

Printed: 28 FEB 1996
II. MSAD Program Tasks — Ground-based

Applications of Atomic Force Microscopy to Investigate Mechanisms of Protein Crystal Growth

Principal Investigator: Dr. John H. Konnert
Naval Research Laboratory

Co-Investigators:
Ward, K.
D'Antonio, P.

Task Objective:
The research objective is to use Atomic Force Microscopy (AFM) to study protein crystal growth mechanisms by extending the pioneering work of Durbin et al (1992) to include crystals other than lysozyme, and by applying image analysis methods not generally available to others to aid in the interpretation of crystal face images observed by this technique. Specific objectives include:

• To modify an AFM liquid sample cell for use as a crystal growth cell for protein crystals. This cell has a flow-through system which will allow protein supersaturation to be controlled by varying the temperature of the crystallization solution bathing the crystal being observed;

• To determine by direct observation of developing crystal faces the mechanism of crystal face growth for a variety of growth conditions including solution composition, degree of protein supersaturation, temperature, growth rate, and microgravity;

• To use crystal etch figures methods and direct observation of growing crystal faces observed by AFM to classify and determine the number of crystal imperfections which occur under a variety of crystal growth rates and conditions, including the examination of faces of single crystals prepared under microgravity conditions;

• To demonstrate that the observed diffraction quality of protein crystals prepared under a variety of conditions can be correlated with the number and type of crystal defects observed using AFM; and

• To determine whether any observed changes in the appearance of the surface of protein crystals can be correlated with the growth cessation phenomena of protein crystals.

Task Description:
Proteins will be prepared for examination by AFM using conventional vapor diffusion, hanging drop methods, and in the temperature-controlled crystallization cell described by Ward, Perozzo and Zuk (1992).

The goal will be to prepare crystals of a given protein using different growth rates by carefully controlling the degree of supersaturation and other growth parameters. Single crystals prepared for these studies will be characterized by x-ray diffraction analyses using standard data reduction and analysis techniques. The diffraction quality will be quantified using the relative Wilson plot analyses described by DeLucas et al (1991).

In addition to preparing crystals under unit gravity conditions, we also intend to submit these proteins for crystallization experiments under microgravity conditions. These experiments will be performed by Keith Ward either as part of the co-investigator protein crystallization program at the University of Alabama-Birmingham or as part of his own Flight Investigation Project which has been submitted in response to the recent NRA.

A number of proteins have been selected for this application of AFM to protein crystal growth studies. Each one is readily available, easily crystallized, and exhibits unique crystallization properties.
Task Significance:

This project will, for the first time, provide unique information about protein crystal growth processes by direct observation of crystal faces in their growth medium. Successful results will provide new evidence for the effect of crystal growth conditions, including microgravity, on the defect structures and diffraction quality of protein crystals. Results derived from this project will, therefore, be of direct significance to NASA-funded efforts aimed at preparing high-quality protein crystals for structural investigations by effectively utilizing the unique microgravity environment of space platforms.

Progress During FY 1995:

The data collection and processing techniques developed in FY94 have been applied to study the growth kinetics of protein crystals in solutions whose concentrations span those employed for the growth of protein crystals suitable for single crystal diffraction experiments. Growth kinetics of the (110) and (101) faces of lysozyme crystals have been observed within a temperature range of 10-30° C (employing a newly constructed stage for temperature control) and at concentrations up to 10 fold supersaturation. It has been observed for the first time that the rate for nucleation of new features is greater on the (101) faces than the (110) faces at low supersaturation. The relative rates of growth due to nucleation of new, ordered regions relative to growth building upon existing defects is crucial to determining the perfection of the final crystal. Two journal articles are being prepared.

Our observation of the preferred molecular arrangement on the (110) faces has aided Marc Pusey and co-workers at NASA/Marshall Space Flight Center in developing theoretical models for the growth of protein crystals. Subsequently their work has suggested features that we should look for on the (101) faces.

Studies with the green fluorescent protein have demonstrated that the techniques may be applied to other proteins.

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Presentations


Regulation of Skeletal Muscle Development and Differentiation In Vitro by Mechanical and Chemical Factors

Principal Investigator: Dr. William E. Kraus
Duke University Medical Center

Co-Investigators:
Truskey, G.A.
Duke University

Task Objective:
This is a biomedical engineering project dealing with the de novo growth of three-dimensional skeletal muscle tissue in a culture system. The thrust of the project is the bioengineering of skeletal muscle fibers in culture and involves: 1) construction of a device adequate for the study of mature muscle cells in culture and that mimics some of the mechanical responses experienced by skeletal muscle fibers during physical exercise; 2) comparison of biologic maturation of skeletal muscle fibers grown in this device with those grown in the NASA bioreactor; and 3) characterization of the physical and chemical parameters of the culture conditions (e.g., physical stress and strain, metabolite and gas diffusion) that contribute to fiber type alterations in mature muscle fibers.

Task Description:
I. Investigations of the cell culture conditions that support the development of intact muscle fibers in vitro: This phase includes construction of and comparison of several devices that will support the growth of mature skeletal muscle fiber bundles in vitro. Preparation for Phase I has required identification of a suitable flexible membrane upon which freshly derived mammalian (rabbit) myoblasts grow and proliferate, identification of a suitable mechanical attachment for the fibers, and identification of stretching parameters that promote muscle fiber alignment. The device has also been designed to support studies of various deforming frequencies on skeletal muscle phenotype. This phase also addresses considerations of the influences of a) extracellular matrix substrate, b) original muscle fiber type from which the cells were derived, c) co-culture of non-muscle cell types in the culture (e.g., fibroblasts, neurons), d) media conditions and e) uniform, gradual physical deformation (mechanical loading) on the ultimate phenotypic characteristics of the resulting fibers. Muscle fiber phenotype is to be determined using histological staining, immuno-histological staining with fiber type specific antibodies and by the ratio of gene expression for oxidative to glycolytic enzymes. Primary muscle cells will be derived from two sources in the rabbit: the fast-twitch, glycolytic muscle, tibialis anterior and the slow-twitch, oxidative muscle, soleus. Comparisons are to be made between the device designed and built in our laboratory with the NASA bioreactor.

II. Investigations of the mechanical and chemical factors that influence gene expression in developing and maturing skeletal muscle. Phase II involves investigations of how cellular signaling pathways may be involved in the modulation of skeletal muscle fiber type with changes in loading, activity, metabolic substrate and hormonal influences. Phase II of the task entails exploration of external mechanical and chemical factors (e.g., deformation magnitude and frequency, media conditions, growth factors, oxygen) and internal signaling molecules (cAMP, Type II adenylyl cyclase and protein kinase C theta (PKCθ), pH) triggered by external factors that may control the phenotype and metabolism of skeletal myocytes. Mature skeletal muscle fibers of a fast-twitch glycolytic fiber type will be subjected to various loading and mechanical deformation protocols. The effects of mechanical forces on skeletal muscle phenotype will be determined using the parameters outlined in Phase I. Mechanical forces will be measured, stress-strain relationships derived and correlated with changes in cellular signaling molecules and gene expression for signaling molecules (Type II adenylyl cyclase, PKCθ) and various markers of skeletal muscle fiber type. In like manner, cultured muscle fibers will be subjected to various conditions of oxygen tension, while controlling for mechanical forces, and changes in muscle fiber type will be determined.

Task Significance:
The development and further characterization of the in vitro culturing system described in this task and the technologies and techniques for monitoring and modeling the chemical and mechanical influences on skeletal muscle.
phenotype and function will provide insight into the cellular processes underlying maintenance of skeletal muscle function in normal and low gravity environments. Progress in describing the cellular signaling pathways involved in transducing mechanical signals generated by changes in activity to alterations in the intracellular milieu, as described in this proposal, may help provide clues to better countermeasures and therapeutic interventions for maintaining skeletal muscle function during spaceflight.

Progress During FY 1995:

Progress on the grant has been made on several fronts. A large quantity of skeletal muscle cells (myoblasts) have been isolated from both fast-twitch and slow-twitch skeletal muscles of adult rabbits. These cells have been expanded and characterized, and a substantial number of aliquots have been stored frozen for later use. The device utilized to support the growth of these cells and to induce mechanical deformation has been built, and is undergoing characterization. Specifically, a number of computer programs that control the stretch parameters have been written and tested. Currently, work is underway to characterize the silicone membrane on which the cells are seated, to test its uniformity under stretch parameters. A variety of substrates utilized to coat the membrane have also been assessed. These include gelatin, matrigel, and reduced-growth factor matrigel. Lastly, the bioreactor has recently been received, and experiments will now be initiated to test the efficacy of myoblast growth and differentiation of different sizes of macrocarrier beads, and various substrate coatings. Experiments to trouble-shoot myoblast culture in the bioreactor environment will be conducted first with the murine skeletal muscle cell line, C2C12, and then be followed by work with the isolated rabbit myoblasts.

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Neuro-endocrine Organoid Assembly In Vitro

Principal Investigator: Dr. Peter I. Lelkes
University of Wisconsin, Milwaukee

Co-Investigators:
Unsworth, B.R.
Marquette University

Task Objective:
The specific aims of our project, entitled "Neuro-endocrine Organoid Assembly In vitro" are as follows:
1. To assemble adrenal medullary endothelial and parenchymal cells into functional organoids. Progress will be monitored by evaluating, a) morphology (light microscopy and ultrastructure), b) intercellular communication (immunocytocchemistry), c) functional maturation and its hormonal control by corticosteroids, d) the expression of phenotypic, biochemical, and molecular markers.
2) To compare the usefulness and efficacy of NASA vessels with conventional co-culture systems (monolayer culture, suspension culture and 3-dimensional gel assembly). The time course, and the extent of neuro-endocrine cell differentiation, under the different culture conditions will be evaluated (as in 1).

Task Description:
Our long term research goal is to understand the fundamental mechanisms of neuro-endocrine gland assembly and differentiation. In our particular model system, the adrenal medulla, neural crest-derived cells of the sympathoadrenal lineage differentiate into neuro-endocrine chromaffin cells. We are particularly interested in the role of the microenvironment in this process. We have previously shown that during assembly and maturation of the adrenal medulla, as in other endocrine organs, parenchymal cells and endothelial cells interact through reciprocal, intercellular signals. Such signals may constitute soluble factors, heterotypic cell contacts, or may be derived from organ-specific extracellular matrix components. These cues comprise part of the epigenetic repertoire, which induces the ordered differentiation of both cell types into what is known as the 'endocrine structure'.

Using conventional 2-D culture techniques, we have shown that in co-culture with adrenomedullary endothelial cells, chromaffin-cell-derived pluripotent PC12 cells differentiate towards the neuroendocrine phenotype. We, therefore, hypothesize that capillary endothelial cells in the adrenal medulla provide some of the organ-specific, differentiative cues which contribute to the neuroendocrine differentiation of the chromaffin precursor cells.

In this project we extend our on-going in vitro studies on the mechanisms of organ-specific differentiation by using alternate methods of co-culture. We are exploiting the enhanced potential offered by the NASA vessels to analyze the temporal assembly of co-cultured adrenomedullary endothelial and parenchymal cells into functional, organelle-like structures (organoids). The simulated microgravity environment of the NASA vessels has been shown to randomize gravitational vectors and minimize detrimental shear forces routinely encountered in conventional three-dimensional suspension cultures in spinner flasks. We anticipate that the favorable culture conditions in the NASA vessel will accelerate differentiative, heterotypic cell-cell contacts and thus lead to differentiated organoid-assembly in vitro.

Task Significance:
In this study, we attempt, for the first time, to generate neuro-endocrine organoids in vitro by co-culturing different cells isolated from the same organ under simulated microgravity conditions. By using the NASA cell culture vessels as a novel, alternate approach to conventional suspension culture in spinner flasks, we are participating in NASA's assessment of simulated microgravity conditions for organ-specific culture and cellular differentiation. As previously shown, a major advantage of using the Rotating Wall Cell Culture Vessels (RWVs) developed by NASA is the enhanced cell viability and tissue differentiation under conditions of simulated microgravity and minimized shear stress.

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The unique conditions in the NASA vessels are believed to enhance cellular interactions and thus differentially accelerate and/or facilitate these processes. In complementing a number of other parallel studies within this program, our project is the first one specifically designed to examine the effects of microgravity on heterotypic interactions between parenchymal cells and microvascular endothelial cells isolated from the same organ, namely the rat adrenal medulla. Such intercellular interactions are believed to be of importance during all phases of the development of endocrine organs, from the earliest stages of embryonic genesis through postnatal maturation.

By using NASA's RWVs as the prime cell culture environment, we will be able to assess whether the spatial arrangement of the functional organ is affected by gravitational forces/vectors. Based on our preliminary observations, we hypothesize that by using the RWVs, we will be able to eliminate gravitational vectors, such as those present in static two-dimensional monolayers or in suspension cultures, and thus obtain a more realistic representation of tissue assembly, as seen in vivo.

Our co-culture system is ideally suited for dissecting the microenvironmental effects (heterotypic cell interactions, microgravity, etc.) on neuro-endocrine differentiation of adrenal medullary chromaffin cells, since many of the phenotypic and genotypic markers for organ specific differentiation of these cells are well characterized. Furthermore, our model system of heterotypic co-culture of adrenal medullary cells is of particular relevance in view of our recent finding that microgravity specifically alters the expression of pivotal enzymes in the catecholamine synthesizing cascade. Thus, our model system is also suitable to explore the cellular and molecular basis for (micro)gravity sensitivity, e.g., of signal transduction mechanisms involved in neuroendocrine hormone synthesis and secretion.

Based on the first two years of practical experience with one of the RWVs, we are confident that the enhanced cellular viability and differentiation achieved by culturing the cells under similar microgravity conditions will be of general advantage for developing new concepts for tissue culture and tissue engineering. Specifically, we anticipate that this novel environment will be beneficial for culturing and/or co-culturing fragile cell types, e.g., when isolated at early stages of embryonic development. We anticipate that within a reasonable time period, we will be able to test our model for organogenesis and differentiation during one of the future Space Shuttle missions.

Progress During FY 1995:

As indicated in last year's progress report, we spent the second year of this grant on establishing the cellular and molecular tools for our evaluating the effects of simulated microgravity on tissue-specific differentiation of PC12 cells and endothelial cells, in mono- and co-cultures. Using these tools, we have made considerable progress during the past year, which can be summarized as follows:

1. In mono-cultures of PC12 pheochromocytoma cells, we have identified several genes which are selectively upregulated by exposure for 20 days to simulated microgravity conditions in the NASA bioreactors. These genes include some of the catecholamine synthesizing enzymes, such as phenylethanolamine-N-methyl transferase (PNMT) and aromatic dopa-decarboxylase matrix proteins, such as fibronectin, laminin and SPARC. Thus, in line with our original hypothesis, we find that the favorable culture conditions in the NASA reactors provide microenvironmental stimuli constraints (e.g., randomized gravitational vectors, reduced fluid shear stress) suitable for the induction of tissue-specific differentiation. Importantly, the induction of PNMT signals a significant shift of the differentiation of these cells towards the neuroendocrine phenotype.

2. Interestingly, mono-cultures of rat adrenal medullary endothelial (RAME) cells were less susceptible to microgravity influences. In studying extracellular matrix expression, we fund no changes for collagen and fibronectin expression. By contract, we found significant changes in the expression of laminin. Importantly, these studies suggest that for both cell types, the laminin gene seems to be susceptible to modulation by the culture conditions in the NASA bioreactors.
3. Under co-culture conditions, PC12 cells and RAME cells were found to form large three-dimensional aggregates which structurally resemble the assembly of acini in the adrenal medulla: nests of parenchymal PC12 cells surrounded by capillary-like structures. Remarkably, such a complex three-dimensional assembly was only seen under co-culture conditions, never in the respective mono-cultures. The accelerated organ-specific differentiation of PC12 cells in the NASA vessels under these co-culture conditions was confirmed immunohistochemically, by the enhanced expression of another neuroendocrine marker, chromogranin B.

4. In continued collaboration with Dr. Timothy Hammond (UW-Madison), we have used the NASA rotating vessels to study the tissue-specific differentiation of human kidney approximal tubule epithelial cells. (For details, see the report of Dr. Hammond.) Importantly, our preliminary studies have resulted in a funded proposal, enabling us to study in detail the effects of simulated microgravity conditions in the NASA rotating vessels on the induction of tissue-specific traits in these cells, alone and in co-culture with organ-derived endothelial cells.

In summary, the third year of the program was very successful in advancing our basic knowledge on the differentiative effects of simulated microgravity in the NASA reactor as proposed in the original proposal. Much more detailed work needs to be done to further characterize the phenotypic and genotypic changes in organoid-like assemblies generated in the NASA vessels. Specifically, we will use our available cellular and molecular tools to examine the mechanisms by which these differentiative effect occurs. In preliminary studies, we have identified a possible mechanism, namely, changes in protein phosphorylation which occur solely under simulated microgravity conditions.

Prospect for 1996: In accordance with our specific aims, we will continue to evaluate the differentiative effects of simulated microgravity on (co-)cultures of adrenal medullary parenchymal PC12 and microvascular endothelial cells. We will assess the organ-specific (neuro-endocrine) differentiation of these cells cultured in the NASA vessels molecular biological and immunological techniques. We will refine our tools for assessing tissue-specific endothelial cell differentiation by developing species-specific molecular probes for use in Northern blots, RNase protection assays and for in situ hybridization. Finally, we will expand our observations as to the involvement in specific signal transduction pathways in the mechanisms by which cells might sense microgravity. In parallel to our biochemical and molecular biological approaches, we will continue to collaborate with our colleagues from the biotechnology group at JSC in evaluating the (co-)cultures by biochemical and immunohistochemical means on the light and electron microscopic level. We anticipate that within the next year we will be able to test our model for neuroendocrine organogenesis and differentiation during one of the future Space Shuttle missions.

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BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals


Presentations


**Formation of Ordered Arrays of Proteins at Surfaces**

**Principal Investigator:** Prof. Abraham M. Lenhoff  
University of Delaware

**Co-Investigators:**
No Co-I's Assigned to this Task

**Task Objective:**
The goal of the project is to investigate the formation and ordering of arrays of proteins at solid surfaces. Two parallel approaches are employed:

1. An empirical study, by scanning probe microscopy (SPM), of the effects of protein and surface parameters on array formation;

2. A fundamental examination, by molecular simulations and surface forces apparatus measurements, of the protein - protein and protein - surface interactions that give rise to protein adsorption and ordering.

Although our primary interest is in proteins, insight can be provided in both the simulations and experiments by corresponding experiments on spheres. For this purpose, the task includes study of charged polystyrene latex particles.

**Task Description:**
The FY95 effort concentrated on atomic force microscopy (AFM) of adsorbate particles and molecules, and associated simulations. AFM experiments were performed on adsorption and structuring of arrays of proteins and latex spheres on surfaces, complementing the purely protein-related work performed and proposed previously. Use of AFM makes possible the investigation of behavior at non-conducting surfaces, specifically mica, while examination of latex spheres makes possible discrimination among multiple possible explanations for changes in adsorption extent as a function of salt strength. The associated calculations seek to predict adsorption coverage of particles at charged surfaces. The initial computations covered energetics of ordered arrays of spherically symmetric particles, but for arrays lacking two-dimensional order, a Brownian dynamics simulation approach was used to allow configurational exploration.

**Task Significance:**
Ordered arrays of proteins are of interest for two main reasons:

1. **Protein structure determination.** X-ray crystallography is still the workhorse for determination of protein 3-D structures, but other techniques have begun to emerge. One class of methods that emerged during the 1980's is that of structure determination on two-dimensional crystals, with techniques such as electron microscopy employed to probe the structures of the constituent molecules. The 2-D arrays of interest to us are suitable for such investigations. Furthermore, it is possible that such arrays can serve as templates for the epitaxial growth of 3-D crystals.

2. **Synthesis of novel materials.** The complex structures of proteins give them functional properties that are also complex, and in addition can be produced reproducibly. Given proteins with the appropriate functions, ordered monolayers of protein molecules at solid surfaces may represent materials with useful properties that make various applications possible. Examples include electronic devices and "biocomposite" materials.

Apart from these applications, our studies of self-assembly have a further, more fundamental purpose: the colloidal forces driving 2-D self-assembly are the same as those giving rise to 3-D crystal growth, so understanding these forces and their interactions is expected to lead ultimately to insights into manipulation of both 2-D and 3-D...
II. MSAD Program Tasks — Ground-based

Discipline: Biotechnology

II. MSAD Program Tasks — Ground-based

Ground-based Discipline: Biotechnology

It is also the isolation of these colloidal forces from other disturbances that provides the incentive for microgravity-based research in this area, as in protein crystal growth.

Progress During FY 1995:

We have completed and submitted for publication the work begun in FY94 on kinetic and equilibrium aspects of adsorption of positively charged latex particles on mica. Our key experimental accomplishment was demonstrating the use of liquid tapping mode AFM to image adsorbed particles in situ, allowing elimination of any possible artifacts due to drying the samples prior to imaging. The structures observed show glass-like radial distribution functions (i.e., crystalline order is not observed). However, imaging in liquid shows that mobility of adsorbed particles is negligible on the time scale of the experiments, suggesting that the structures observed are not equilibrium structures. The mean interparticle spacing decreases, and hence the surface coverage increases, with increasing ionic strength, a trend that is opposite to that observed for proteins. The reasons for this behavior are elucidated by our simulations, of which we have performed two kinds: a random sequential adsorption approach for "soft" particles, and a hybrid Brownian dynamics-grand canonical Monte Carlo (BD-GCMC) method. The computations show that beyond a certain surface coverage, electrostatic repulsion between adsorbed particles and those approaching the surface gives rise to a kinetic barrier preventing surface coverage from reaching levels expected from equilibrium considerations. Such repulsive interactions are much weaker for smaller proteins, hence this behavior is not observed there. Smaller proteins would also be expected to be more mobile on the surface, thus explaining their ability to attain high degrees of two-dimensional periodicity. Work is continuing on applying to proteins the methods developed for latex particles; initial experiments on ferritin have demonstrated the ability to observe adsorbed molecules, but a systematic study has not yet been carried out.

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Multidisciplinary Studies of Cells, Tissues, and Mammalian Development in Simulated Microgravity

Principal Investigator: Dr. Elliot M. Levine

Co-Investigators:
Herlyn D.V.M., Prof. M.
Spain, Dr. L.
Panettieri, Dr. R.
Litt, Prof. M.
Pollack, Dr. S.
Meany, Dr. D.

Task Objective:
The Wistar Institute of Anatomy and Biology, in collaboration with The Hospital of the University of Pennsylvania, and the Department of Bioengineering of the University of Pennsylvania, is undertaking a study entitled "Multidisciplinary Studies of Cells, Tissues, and Mammalian Development in Simulated Microgravity". The joint research team (called WISTAR/PENN) combines cutting-edge tissue culture and molecular technology with advanced bioengineering analyses and expertise. WISTAR/PENN, consisting of eight principal investigators (and their research teams) and one scientific executive administrator, will develop nine different cell, tissue, and embryonic systems to be cultured in low shear bioreactors which simulate microgravity. The WISTAR/PENN group will employ these model systems to achieve several goals:

a. Develop optimum cell culture conditions for the propagation of a range of three-dimensional complex tissues in current bioreactors (with the recognition that bioreactor culture may have positive or negative effects, or both, on some of the model systems).

b. Pursue a comprehensive and ground-breaking investigation of the effects of microgravity simulated in current bioreactors on the model systems at the molecular, biomechanical, cellular and embryonic levels.

Combine the experience gained in (a) and (b) with theoretical modeling and predication to:

c. Produce, within a short time, advanced bioengineering enhancement of bioreactor technology.

d. Set the ground-work for future flight proposals.

Task Description:
We propose a broad program in which multiple culture systems will be evaluated in depth, providing a unique opportunity to draw general conclusions about the effects of low shear and simulated microgravity and the utility of bioreactor technology. The hypothesis inherent in our approaches and proposed research is that biomechanical forces, such as gravity and shear, stimulate responses from cells, tissues and embryos. The initial transduction of mechanical forces into the cells involves components such as surface and adhesion receptors and signaling mechanisms including protein kinase C and calcium fluxes. These then are translated into alterations in cell association, matrix synthesis, proliferation, migration, differentiation and gene expression. Each project will test one or more of the components of this hypothesis. As we achieve our goals, we will be able to directly compare microgravity effects on, for example, calcium fluxes in different model systems.

The cell and tissue types being studies are: endothelial and smooth muscle, breast and skin, airway smooth muscle, bone, thymus, and renal epithelial.
II. MSAD Program Tasks — Ground-based

Discipline: Biotechnology

Previous studies with NASA bioreactors often compared results obtained under simulated microgravity conditions in bioreactors with those obtained under gravity in static culture. We propose an approach in which, except for the gravity vector, the experimental conditions are more nearly equivalent. We will compare results obtained from cells or embryos in reactors operated at 18 rpm ("simulated microgravity") with results obtained in reactors operated at 1.5 rpm. As previously shown, particle motion with reference to the bioreactor wall is minimal at an operating speed of 18 rpm; we refer to this mode as "0G". In contrast, at a lower rotation speed of 1.5 rpm, gravitational forces are greater than the angular momentum. Consequently, particle motion with reference to the bioreactor wall is appreciable, although particles remain suspended; we refer to this mode as "1G".

This "0G" vs. "1G" approach permits another important aspect of NASA bioreactor technology to be considered. With few exceptions, cells and tissues in NASA bioreactors have been studied only under simulated microgravity conditions, i.e., in the "0G" mode. The consistently better growth and differentiation obtained under these conditions compared to static cultures may be a consequence of bioreactor attributes other than that of simulated microgravity. Low shear, minimal turbulence, and high oxygenation are examples of improved culture conditions also obtainable in bioreactors operated at "1G". The effect of simulated microgravity per se will be determined by comparison of the "0G" vs. "1G" modes proposed above. We will be mindful, however, that slightly greater shear may exist in "1G" conditions due to the particle motion through the culture medium as described above.

Conclusive studies of simulated microgravity and bioreactor technology will require a systematic approach. To meet the major challenge of applying and enhancing this technology, we believe it essential to rigorously characterize the biomechanical environment of cells, organ systems or embryos in the bioreactor. Such characterization is necessary for a quantitative understanding of the altered mechanical stimulus/cellular response studies in all the proposed projects. Therefore, the synopses of the projects' individual objectives begins with that of the biomechanical analysis project. The output of this project will influence ongoing experimental design and interpretation of data in all the other project which relate gravity-related stress stimuli to the transduction, biochemical, and morphogenetic pathways of cells, organs and embryos.

Task Significance:

The hypothesis inherent in all our experimental approaches and proposed research is that biomechanical forces, such as gravity and shear, stimulate responses from cells, tissues and developing embryos. The initial transduction of mechanical forces into the cells involves components such as surface and adhesion receptors and signaling mechanisms including protein kinase C and calcium fluxes. These then are translated into alterations in cell association, matrix synthesis, proliferation, migration, differentiation and gene expression. Each project will test one or more of the components of this hypothesis. As we achieve our goals, there will be the opportunity to directly compare microgravity effects on, as one example, calcium fluxes in different model systems.

Although not always recognized, the usual static or suspension culture conditions also exert significant biomechanical forces on cells. We hypothesize that, because of unidirectional gravity vector and/or high shear in such situations, bioreactor-grown cultures will more closely resemble in vivo tissue than cultures grown under ordinary conditions. By changing bioreactor rotation speed, as explained below, we will be able to dissociate the effects of simulated microgravity from the effects of low shear.

Progress During FY 1995:

Funding for this program began August 15, 1995, and the initial four month period has been devoted to hiring and training personnel, obtaining the necessary equipment, including 5 NASA bioreactors and motor bases, and initiating preliminary experiments with several different cell types. For instance, we are developing culture conditions for the growth of human vascular endothelial and smooth muscle cells as suspended aggregates in the NASA bioreactor. Studies are also in progress on the growth of human skin constructs and the growth and development of murine embryos.

Even before funding began, the program director and all principal investigators of our group presented and discussed our research plans in a one-day mini-symposium at NASA organized by us in cooperation with NASA/JSC. In

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addition, monthly program meetings are being held on our campus to discuss and coordinate progress in the various projects.

We also have initiated additional collaborations with Dr. Daniela Santoli (The Wistar Institute) working with human T-cells, Dr. John Hoyer (Children's Hospital of Philadelphia) on human renal epithelial cells, Dr. Paul Ducheyne and Portonovo Ayyaswamy (U. Pennsylvania School of Engineering) on the biophysical aspects of cellular growth in the bioreactor, and Drs. Boris Yaffe and Gretchen Darlington (Baylor University School of Medicine) on the growth of human liver cells.
Analysis of Electrophoretic Transport of Macromolecules using Pulsed Field Gradient NMR

Principal Investigator: Dr. Bruce R. Locke Florida State University

Co-Investigators:
Moerland, Dr. T.S. Florida State University

Task Objective:
The scientific objective of the present work is to apply pulsed field gradient nuclear magnetic resonance (PFGNMR) spectroscopy to the analysis of electrophoretic and diffusive transport of biological macromolecules in solutions of polymers, in gels and in gel-like media. PFGNMR offers the major advantage of allowing for measurement of the rates of transport of a wide range of molecules in media that is not necessarily optically transparent. A limited amount of work has been reported in the literature to show that PFGNMR can be applied to diffusion and electrophoresis; however, further development and study is necessary to apply the method to transport of larger macromolecules in a wider range of materials.

Task Description:
The goal for the first year of this project is to use PFGNMR, which is a well established method for measuring diffusion coefficients, to measure diffusivity of small molecules in gels and in gel-like media. In addition, small probe compounds will be used to investigate structural aspects of the polymer solutions, the gels, and gel-like media through measurement of the hindered diffusion in such media. Since the project began on August 30, 1995, the first years worth of work will continue into FY96.

Task Significance:
The production of biological macromolecules for medical, consumer, and industrial applications is often limited by the degree of purity to which these compounds can be manufactured. Electrophoresis is a purification technique that utilizes an applied electrical field to separate macromolecules on the basis of the charge and size of the macromolecule. Electrophoresis is performed in solutions of polymers or gels and microgravity conditions. The studies in the present work involves the use of pulsed field gradient nuclear magnetic resonance (PFGNMR) to study the motion of biological macromolecules in gels and solutions in order to improve our understanding of how these molecules can be separated by electrical fields.

Progress During FY 1995:
This project was initiated on August 30, 1995. The first month of research has focused on the design and specification of the probe insert that will be used in the NMR instrumentation for studying electrophoretic transport and electroconvection. In addition, preliminary studies of the self diffusion of water in acrylamide gels of various concentrations has been performed in order to assess the technique and to determine structural features of these gels.

STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 8/95 EXPIRATION: 8/96
PROJECT IDENTIFICATION: 962-24-08-18
RESPONSIBLE CENTER: MSFC

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II. MSAD Program Tasks — Ground-based

Discipline: Biotechnology

Ground-Based Program for the Physical Analysis of Macromolecular Crystal Growth

Principal Investigator: Prof. Alexander J. Malkin
University of California, Riverside

Co-Investigators:
McPherson, A.
Kuznetsov, Y.G.
Koszelak, S.
DeYoreo, J.J.
Land, T.
University of California, Riverside
University of California, Riverside
University of California, Riverside
Lawrence Livermore National Laboratory
Lawrence Livermore National Laboratory

Task Objective:
The general objective of this research is to gain a better understanding of the physics of macromolecular crystallization. These include development and application of the in situ high resolution techniques for both studies of the growth phenomena and mass transport in protein and virus crystal growth.

Task Description:
To achieve the objectives of this investigation, we are applying a combination of physical in situ techniques such as atomic force microscopy, conventional Michelson and phase shift Mach-Zehnder interferometry and inelastic light scattering for the quantitative investigation of the nucleation and crystallization phenomena for different proteins and viruses. The specific aims of this investigation are:

• determination of the sources of step generation for different macromolecular systems
• investigations of the surface morphology and kinetics of macromolecular crystallization
• determination of the fundamental kinetic and thermodynamic parameters, which govern macromolecular crystallization
• studies of the defect structure and mechanisms of impurity action
• investigation of the relative importance of transport processes versus surface kinetics
• determination of the concentration gradients around growing and dissolving crystals

Task Significance:
Macromolecular crystallization currently is highly problematic, to such an extent that it has become the rate limiting step to the broader application of x-ray diffraction analyses in molecular biology and biochemistry, which in turn has significant adverse effects on the progress of biotechnology in general. This is true, in a large part, because little is known of the physics of macromolecular crystallization. Crystallization experiments in space provide a significant improvement in crystal quality for a number of protein and virus crystals. In order to evaluate the impact of microgravity in macromolecular crystallization, it is necessary to compare the physics of crystallization in both environments. Increased understanding of the mechanisms of macromolecular crystallization might be expected to result in the development of new growth techniques and contribute to the medical and pharmaceutical applications of biological macromolecules.

Progress During FY 1995:
This grant was awarded in June 1995. During the reporting period, in situ atomic force microscopy was applied to study growth mechanisms of canavalin and satellite tobacco mosaic virus (STMV). In the case of canavalin, growth occurs by step flow on complex dislocation hillocks, while all STMV crystals investigated so far were dislocation-free and two dimensional nuclei were the sources of the growth steps. Step advancement rates were used to determine the kinetic coefficient, $b$. Sizes of the critical nuclei in case of two-dimensional nucleation provided an estimate for the free energy of the step edge, $\alpha$. Both parameters are two-to-three orders of magnitude lower than those estimated for inorganic crystals grown from solutions. Individual molecules were resolved both on the (111)
faces of STMV crystals and (100) faces of canavalin crystals. Defect formations due to the incorporation of derby particles were also obtained.

In situ Michelson interferometry was utilized to investigate growth kinetics and surface morphology in canavalin crystallization. Interferometric patterns and kinetic measurements from growing macromolecular crystals as small as 20 μm were obtained. The kinetic coefficient $B$ for canavalin crystallization was estimated to be consistent with one determined from AFM studies. Dislocations were the sources of growth steps and change in activities of dislocation sources under different growth conditions were analyzed.

For the studies of concentration gradients around the growing and dissolving crystals, we have designed and constructed a real-time phase shift Mach-Zehnder interferometer. We carried out preliminary experiments on crystallization of several macromolecular systems.

**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Journals**


**Presentations**


Task Significance:

A reductionist model - one in which a human thyroid organoid can be constructed from its separate elements - will allow disease-oriented biological investigations, e.g., the study of disease relevant lymphocytes in the human autoimmune thyroid diseases or growth patterns of malignant thyroid tumors.

Progress During FY 1995:

The NASA-developed rotary cell culture system provides a unique, low-shear environment conducive to three-dimensional (3D) cell growth. To explore its usefulness for artificial thyroid neo-organ (organoid) construction, the SV40 transformed, immortalized human thyroid cell line TAD2 was grown in a high-aspect ratio vessel (HARV) for optimum oxygenation in medium RPMI 1640, supplemented with 10% FBS and 1% antibiotics with or without basement membrane extract (BME, 1%) at an initial inoculation concentration of 1 X 10^6 cells/ml. In the presence of BME, new cell structures several mm in size formed within a 9-day growth period and were fixed in 10% formalin for light microscopy and in 3% glutaraldehyde for electron microscopy (EM) whereas, in the absence of BME, there was no cluster formation and cell numbers declined. Hematoxylin/eosin staining revealed undifferentiated spindle and epithelialoid cells. Immunohistochemistry showed no staining for human thyroglobulin. Likewise, RT-PCR for human thyroglobulin did not demonstrate mRNA for human thyroglobulin. The absence of...
human thyroglobulin mRNA was consistent with the negative gene regulation exerted by SV40 large T antigen. However, scanning EM demonstrated cell/cell contact of cell aggregates and transmission EM showed desmosomes and/or tight junctions and cell cisternae filled with amorphous fibrillar material. The cells appeared to be polarized as evidenced by the appearance of microvilli. In addition, vesicles and lipid droplets were visible. TAD2 cells grown under the same defined medium conditions (1% BME) in monolayer cultures and at the same cell density did not form 3D structures although they exhibited contact inhibition. Culture under simulated microgravity favored formation of 3D structures even in transformed thyroid cells in the presence of low concentrations of BME. These observations suggest the use of this system for studies of normal thyroid cell/cell interaction, as well as human thyroid autoimmune disease and human thyroid cancers.

STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 9/95  EXPIRATION: 9/99
PROJECT IDENTIFICATION: 962-23-01-17
NASA CONTRACT NO.: NAS9-816
RESPONSIBLE CENTER: JSC
II. MSAD Program Tasks — Ground-based

**Biological Particle Separation in Low Gravity**

Principal Investigator: Dr. D. J. Morré

Co-Investigators:
- Van Alstine, J.M.
- Morré, D.M.

Discipline: Biotechnology

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**Task Objective:**

Subcellular particles play key roles in normal health, as well as the course and treatment of a variety of diseases. Most subcellular particles are not homogeneous but exist in distinct subpopulations with specific functional characteristics. Functional heterogeneity is especially evident in cellular endosomes. Research on such subpopulations is currently limited by a paucity of effective separation methods. Those commonly used (e.g., Phase Partition and Free-Flow Electrophoresis) have both been shown to benefit from low gravity. The objective of this research is to prepare to test the hypothesis that "the low gravity environment of space will allow unparalleled separations of subcellular particles," resulting in enhanced knowledge concerning endosomal subpopulations.

**Task Description:**

The ground-based research necessary to responsibly evaluate this promising hypothesis and, possibly, test it in low gravity is being undertaken by Purdue University in conjunction with the University of Alabama in Huntsville (UAH). Purdue has expertise on subcellular particle separation by Free-Flow Electrophoresis (FFE) and Phase Partition (PP). UAH has expertise related to flight experimentation involving FFE and PP, as well as the use of polymer coatings and polymer-linked affinity ligands to enhance separations obtained by these methods.

**Task Significance:**

Physicians and scientists are now in need of a better understanding of what subpopulations of particles exist in cells, how they might contribute to disease, and how they point to new methods to treat disease. Our research is aimed at providing the ability to use space to satisfy this need. The research goal is similar to that for protein crystal growth experiments. Much of the value obtained from protein crystals grown in space is related to new knowledge of the protein structure. So, too, the immediate value in being able to isolate defined endosomal subpopulations will be in the knowledge of their existence and how this enhances current medical hypotheses and treatments. Secondary value will come from obtaining even small samples of purer materials as they may allow us to develop or improve methods for their large scale isolation on earth.

**Progress During FY 1995:**

1. Development of rapid, nondestructive, method to electrokinetically quantitatively chemical and polymer groups on native and modified biotechnical apparatus surfaces.
2. Developed enhanced biocompatible polymeric coatings for use in partition and FFE separation methodologies.
3. Refined theory of operation of PEG-affinity derivatives used to enhance both FFE and PP separations.
4. Above theory expanded to include direct comparison to polymer "coated" colloids and flat surfaces.
5. Methodology for endosome isolation and characterization.
6. Isolation and characterization of rat muscle endosomes carrying glucose transporters as a potentially useful ground-based model.
7. Detergent-evorted plasma membrane vesicles as endosome models.
II. MSAD Program Tasks — Ground-based

STUDENTS FUNDED UNDER RESEARCH:

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals

Presentations


II. MSAD Program Tasks — Ground-based

Continuous, Noninvasive Monitoring of Rotating Wall Vessels and Application to the Study of Prostate Cancer

Principal Investigator: Prof. David W. Murhammer
University of Iowa

Co-Investigators:
Arnold, M.A.
Cohen, M.B.
University of Iowa

Task Objective:
The primary hypotheses that will be tested in this research are (1) NIR spectroscopy can be used for the real-time, non-invasive monitoring of glucose, glutamine, ammonia, and lactate in a rotating-wall vessel (RWV) and (2) cellular adhesion molecular (CAM) expression by prostate cancer cell lines under microgravity conditions better simulate in vivo conditions than routine tissue culture. The specific objectives that will be undertaken to test these hypotheses are (1) develop calibration models for glucose, glutamine, ammonia, and lactate in increasingly complex mixtures of components found in prostate cancer cell culture medium, (2) develop an on-line monitoring system for the rotating-wall vessel (RWV), and (3) simulate in vivo prostate cancer expression of cellular adhesion molecules (CAMs) by growing prostate cancer cells in rotating-wall vessels (RWVs).

Task Description:
The first phase of this research will involve the development of calibration models for the off-line determination of glucose, glutamine, lactate, and ammonia concentrations in prostate cancer cell medium from a single near-infrared (NIR) spectrum. We will begin with a simple matrix and then systematically evaluate more complex matrices, allowing for potential interferences to be identified and for the development of rational procedures to eliminate the adverse effects of these interferences. In the second phase of this research, methodologies will be developed to interface the NIR spectrometer with a perfusion RWV such that the glucose, glutamine, lactate, and ammonia concentrations within the vessel can be non-invasively monitored. This will involve the use of bifurcated fiber optic probe to deliver NIR light to the bioreactor contents and to collect reflected spectra for analysis. In addition, this phase of the proposed research will utilize luminescence quenching and indicator absorption spectroscopy to non-invasively monitor the dissolved oxygen concentration and pH, respectively. The information obtained through this monitoring system will be utilized to control the pH and nutrient concentrations within the RWV so that the behavior of prostate cancer cell lines can be investigated under conditions closely resembling those found in vivo. Specifically, the expression of CAMs, which are important for invasion and metastases of this and many other types of cancer, will be evaluated.

Task Significance:
Successful development of non-invasive NIR spectroscopic sensors to monitor critical analyzes used in conjunction with non-invasive pH and dissolved oxygen monitoring will clearly revolutionize the operation of bioreactors in general and RWVs in particular. We propose to focus attention on selective monitors for glucose, glutamine, ammonia, lactate, pH, and dissolved oxygen because these are the most critical factors in animal cell cultures and affect the overall production efficiency (quality and quantity) of bioreactors in general. Although our work centers around development of monitors specific for prostate cancer cell lines operated in the perfusion mode, it is important to note that the analytical methods developed here will be applicable to nearly all fermentation processes with minor modifications. Successful completion of this work is expected to have a major impact in the general field of biotechnology because the availability of continuous monitors will enable the development of active feed-back control strategies which will dramatically improve production efficiency of nearly all recombinant products. In addition, such NIR monitors will be valuable basic research tools capable of providing chemical information that can be used to study the complex biological reactions associated with recombinant processes. Overall, the proposed...
NIR spectroscopic monitors will be an enabling technology with many applications throughout the field of biotechnology. This technology will also greatly benefit future microgravity research since it can be applied to continuously monitor and control bioreactors operating in space.

In addition to the wide ranging benefits resulting from the non-invasive monitoring technology, this research will also address questions regarding the mechanisms involved in invasion and metastasis of prostate cancer. This research phase will greatly benefit from the non-invasive monitoring technology by allowing more accurate simulation of in vivo conditions resulting from environmental control. It is anticipated that the knowledge obtained from these studies will allow for a better understanding of the biology of prostate cancer.

Progress During FY 1995:

Simultaneous measurement of glucose and glutamine. As a first step toward establishing the analytical utility of using NIR spectroscopy for bioreactor monitoring, we have used NIR spectroscopy to measure glucose and glutamine simultaneously in binary mixtures. Our results demonstrate that glucose and glutamine can be measured independently at millimolar concentrations in aqueous solutions composed of glucose and glutamine. The ability to differentiate glucose and glutamine comes from the differences in their NIR spectra. Basically, glucose is characterized by three unique absorption bands centered at 4300, 4400, and 4710 cm\(^{-1}\), while glutamine possesses bands centered 4390, 4580, and 4700 cm\(^{-1}\). A total of 72 standard solutions were prepared and 215 spectra were collected. It is important to note that there was no detectable correlation between the concentrations of glucose and glutamine in these standards. Spectra corresponding to a fraction of these samples were used to develop calibration models while the remaining spectra were used to judge the validity of these models by testing their ability to accurately predict glucose and glutamine concentrations. Unique calibration models were developed for glucose and glutamine based on a combination of Fourier filtering and partial least squares (PLS) regression. The best calibration models were able to measure glucose and glutamine with standard errors of prediction (SEPs) of only 0.32 mM for glucose and 0.75 mM for glutamine over concentration ranges from 1.66 to 59.91 and 1.10 to 30.65 mM, respectively.

Simultaneous measurement of glucose, glutamine, glutamate, ammonia, and lactate. We have also established that glucose, glutamine, glutamate, ammonia, and lactate can be measured independently and simultaneously by NIR spectroscopy. Again, differences in the NIR spectra for these compounds permit the selective analysis. Even for compounds as similar as glutamine and glutamate, spectral differences are apparent. Calibration models have been developed based on Fourier filtering coupled with PLS regression. These calibration models were then used to predict the concentration of each analyte from the remaining spectra. In all cases, the mean percent errors were less than 10% and the SEPs were under 0.65 mM. Glucose and glutamine, in particular, can be measured with a SEP of 0.54 and 0.24 mM, respectively, in solutions with changing concentrations of glutamate, lactate, and ammonia.

NIR Spectra for Amino Acids. Our work has demonstrated that the key to building successful and selective calibration models is the presence of unique spectral information for each analyte in the sample. The ability to selectively monitor glucose and glutamine in a bioreactor requires unique NIR spectra for these compounds relative to all other matrix components. Clearly, amino acids represent a matrix component that we must be able to differentiate with the NIR spectral information. NIR spectra for 18 different amino acids have been collected and compared to see if the NIR spectral features for glucose and glutamine are unique. Inspection of these data reveals that each amino acid possesses a unique NIR spectrum. The differences between glutamine and asparagine are particularly noteworthy because of the structural similarity of these two amino acids. As stated before, glutamine possesses bands centered at 4390, 4580, and 4700 cm\(^{-1}\), while asparagine has bands at 4380, 4580, and 4740 cm\(^{-1}\). Also, these spectral features differ in relative absorptivities and band shapes. Our previous experience suggests that glutamine and asparagine can be differentiated spectroscopically and selective calibration models can be developed for glucose and glutamine in the presence of millimolar concentrations of all these amino acids.
II. MSAD Program Tasks — Ground-based

**STUDENTS FUNDED UNDER RESEARCH:**

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**TASK INITIATION:** 8/95  **EXPIRATION:** 8/99

**PROJECT IDENTIFICATION:** 962-23-01-24

**NASA CONTRACT No.:** NAG9-824

**RESPONSIBLE CENTER:** JSC

**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Journals**


II. MSAD Program Tasks — Ground-based

Discipline: Biotechnology

Crystallization Studies in Microgravity of an Integral Membrane Protein: The Photosynthetic Reaction Center

Principal Investigator: Dr. James R. Norris
Argonne National Laboratory

Co-Investigators:
Deisenhofer, J.
Thurnauer, M.
Tiede, D.
Thiyagarajan, P.
Schiffer, M.
Furrer, R.
Howard Hughes Medical Institute
Argonne National Laboratory
Argonne National Laboratory
Argonne National Laboratory
Argonne National Laboratory
Free University Berlin

Task Objective:
The objective of this project is to investigate mechanisms and pathways for membrane crystallization using small angle neutron scattering (SANS).

Task Description:
Advances in the understanding of membrane protein crystallization are important from both biological and pharmaceutical viewpoints. Membrane proteins are responsible for many of the major biological processes such as vision, nerve conduction, cell differentiation, photosynthesis, and respiration. The unique aspect of membrane protein crystallization compared to other protein crystallization is the required presence of the detergent for protein solubilization in the case of membrane protein crystallization. The presence of the detergent greatly modifies the phase map for membrane protein crystallization. This task is using SANS to analyze the physical chemistry and structures of components used in membrane protein crystallization.

The bacterial photosynthetic reaction center is being used as a test membrane protein. This integral membrane protein has been successfully crystallized in two different detergents: lauryldimethylamine-N-oxide (LDAO), and n-octyl-B-D-glucoside (OG). Conditions required for crystallization of the reaction center protein differ significantly for these two detergents. The difference in crystallization requirements shows the influence of the detergent in changing the pathways or intermediates in reaction center crystallization. In FY94 this task completed a characterization of LDAO and OG micelle structure and micelle-micelle interactions under solution conditions used for reaction center crystallization. This work showed how conditions used to optimize reaction center crystallization modify the structure and physico-chemical properties of the detergent micelles.

In this fiscal year, this task used SANS to examine structures in reaction center solutions as a function of detergent, temperature and ionic strength. This work characterized how the detergent changes the solubilization state of the reaction center. This work provides the information necessary for identifying the physical chemistry that serves as the basis for crystallization, and for developing rational schemes for optimizing membrane protein crystallization.

Task Significance:
Current results on crystallization in microgravity have shown that the minimization of convection and sedimentation in space leads to improved size and/or ordering of crystals for many water-soluble proteins. In several cases, the resolution of the x-ray diffraction was better than any crystal produced on Earth. In cases where the x-ray diffraction quality did not exceed the best obtained on Earth, the space-grown crystals were still better than those in the control experiment. These results indicate the need for optimization of space hardware compared to the results achieved in the laboratory, but still point to the beneficial effects of microgravity on the crystallization process.

Printed: 28 FEB 1996
Progress During FY 1995:

This fiscal year our small angle neutron scattering studies of intermediates in crystallization pathways were extended to include characterization of structures of a detergent-solubilized membrane protein, the photosynthetic reaction center, as a function of detergent, ionic strength and temperature. The results obtained in this task documented how the physical-chemical properties of the detergent-solubilized reaction center change with detergent. This work has provided unique information that is critical for evaluating mechanisms and pathways of detergent-solubilized membrane proteins.

One significant accomplishment was the development of a computer algorithm that calculates solution scattering profiles based upon protein crystal coordinates. This program allowed us to analyze structures of protein solutions more quantitatively. SANS measurements were made using fully-deuterated protein and protonated detergents. A neutron contrast-matching technique was used to suppress the neutron scattering from the protonated detergents and to selectively detect scattering exclusively from the deuterated protein in these complex mixtures.

This task found that scattering profiles for LDAO solubilized reaction centers could be fit with a solution scattering profile calculated from the reaction center crystal structure. This provided proof that: i) the reaction center is monodispersed in LDAO; ii) the reaction center structure in solution is consistent with the crystal structure; and, iii) the contrast matching techniques have successfully suppressed scattering from the detergent. This work served as a starting point for comparing structures in solutions of reaction center under other conditions, and established the validity of our strategy to use isotopic labeling to selectively identify scattering from the reaction center in complex crystallization mixtures.

Scattering profiles for reaction centers in OG solutions were found to deviate from calculated scattering profiles for monodispersed reaction centers. Comparisons with scattering profiles calculated for reaction dimers show that the reaction centers in OG are likely to be a mixture that is predominately dimeric with some higher order aggregates. An examination of reaction center SANS as a function of ionic strength and temperature lead to the following conclusions:

a. Reaction centers have a propensity to dimerize. This tendency appears to be a property of the reaction center protein and not the detergent. The tendency can be masked by the solubilizing detergent. The dimerization is minimized in LDAO, and is most evident in OG. It is diminished by high ionic strength in both detergents.
b. Temperature in the range 6° to 23° C does not affect the LDAO solubilized reaction center. In contrast, cooling reaction center in OG to 6° C caused the formation of tetramers (dimerization of dimers).
c. Freezing in water and glass-forming solutions caused extensive three-dimensional aggregation in which the detergent was excluded from the protein-protein interface contacts.
d. Hysteresis in response to changes in temperature caused variability in reaction center dispersity detected at room temperature.

This work identified fundamental features of reaction center physical chemistry that is likely to play a role in determining protein-protein interactions in crystallization conditions. Preliminary measurements were also made on reaction center structures in the presence of the crystallizing agent polyethylene glycol 4000, PEG. This work provided the first examination of reaction center structures as a function of position in the soluble domain of the crystallization phase map. The results show the surprising result that there is diminishing monomer concentration as crystallization mixtures approach the crystallization threshold, and that there is increasing concentrations of soluble small aggregates. Although the structure of the aggregates have not yet been analyzed in detail, this project is finding that structures of reaction center solutions in the soluble domain are more complex than had been anticipated from idealized views of protein crystallization.
BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals

Presentations


Insect-Cell Cultivation in Simulated Microgravity

Principal Investigator: Prof. Kim O'Connor
Tulane University

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
As discussed in the 1995 report on our NASA grant (NAS9-773), we found distinct differences in the growth and metabolic properties of Spodoptera frugiperda insect cells cultivated in simulated microgravity within the High Aspect Rotating-Wall Vessel (HARV) as compared to conventional bioreactors such as shaker-flasks. Over this first month of the renewed grant, our objectives are two fold.

The first objective is to extend our comparison of HARV and shaker-flask cultures of S. frugiperda cells to stationary phase and death phase, characterizing differences in cellular properties and investigating the mechanisms underlying these changes.

It was also evident from our earlier work that HARV cultivation may improve recombinant protein production from stably transformed insect cells. During this one-month period, our second objective was to quantify recombinant protein expression in shaker-flask control cultures.

Task Description:
Our current research involves two cell lines: IE1FB, a clone derived from S. frugiperda, and S2LO-VCAM, Schneider II cells derived from Drosophila melanogaster expressing recombinant human vascular cell adhesion molecule (VCAM) upon induction with copper ions.

In comparing HARV cultures of insect cells with those produced in conventional bioreactors, we selected a shaker flask for the control. Relative to other conventional bioreactors, shaker flasks provide an extremely gentle culturing environment. Yet, hydrodynamic forces in this control are nearly twice the magnitude of those encountered in the HARV. Differences observed between HARV and shaker cultures of insect cells should be applicable and magnified when comparisons are made between the HARV and more turbulent bioreactors.

Task Significance:
Insect cells are invertebrate animal cells that show great potential as hosts for the commercial production of recombinant proteins. This is a relatively new phenomenon, beginning in the mid-1980's. Since this time, there has been an explosive growth in the number of recombinant proteins that have been expressed in insect cells with less than 10 by 1984 to over 100 by 1990. The reason for this growth is the ease with which recombinant proteins can be expressed in and purified from insect cells relative to mammalian cells.

Recombinant proteins are playing an increasingly greater role in medicine, basic research and agriculture for use as protein therapies, diagnostic agents and insecticides to name a few. The sale of recombinant proteins makes a substantial contribution in the U.S. economic market. In the pharmaceutical industry alone, biotherapies, which are primarily recombinant proteins, generated $1.2 billion in U.S. sales in 1991 and are predicted to generate nearly $8.0 billion in 2001.

With current technology, commercial production of recombinant proteins from insect cells is hindered since they are more sensitive to hydrodynamic forces in a bioreactor and have a greater oxygen uptake rate than most vertebrate cells. This combination places severe constraints on cell oxygenation during large-scale cultivation: sufficient oxygen must be transported to the insect cells for respiration without inducing hydrodynamic damage to the cells.
Conventional methods of oxygenation, such as sparging, supply sufficient oxygen but do so by increasing mixing of growth medium and gas flow rate, resulting in cell destruction from hydrodynamic forces in the medium and at the gas/liquid interface.

The HARV provides a low-turbulence environment in which cells grow suspended in culture medium through end-over-end mixing and are oxygenated via a silicon membrane. In this environment, hydrodynamic forces are greatly reduced over those in conventional bioreactors while mass transfer rates for oxygen remain high.

Our research evaluates the potential of the HARV for cultivation of insect cells and production of recombinant proteins derived from these cells.

**Progress During FY 1995:**

Since the renewed grant, in late summer, we have been conducting an in-depth study of *S. frugiperda* cells in stationary and death phase. HARV cultures remain in stationary phase 2 to 3 times longer than shaker-flask cultures. This increase is attributed to reduced turbulence and lower concentration of metabolic wastes in the HARV. Typically, stationary phase lasts 10 days in the HARV and is followed by gradual cell death. During this 10-day period, there is a noticeable reduction in cell size in HARV cultures; the number of cells with diameters under 10 microns rises from less than 2% to greater than 15%. This trend becomes even further magnified during death phase. Shaker-flask cultivation has the opposite effect; cells larger than 30 microns account for less than 5% of newly inoculated cultures but greater than 20% after 12 days of cultivation.

These changes in cell size could be indicative of different mechanisms of cell death in the two vessels: apoptosis in the HARV versus necrosis in the shaker flask. To test this hypothesis, we are characterizing DNA fragmentation and quantifying the formation of apoptotic bodies in the two vessels.

In addition to the research described above, we are investigating recombinant protein production in HARV cultures of insect cells. During this first month of the grant period, we have been working with stably transformed *Drosophila* cells expressing VCAM. This study is being done in collaboration with Dr. Alain Bernard at Glaxo in Switzerland. With Dr. Bernard's help, we are learning the procedures to detect the recombinant protein and measuring its expression in control *Drosophila* cultures grown in shaker flasks.

Besides our work with insect cells, we are studying the effect of simulated microgravity on DU 145 human prostate adenocarcinoma cells.

From our previous research with this cell line, simulated microgravity significantly alters cell aggressiveness, resulting in a slower growing and more differentiated culture than can be obtained in either a spinner flask or T-flask. For additional information on these studies, see the 1995 report on our NASA grant (NAG9-733).

Currently, our focus is to investigate the mechanisms that underlie this phenomenon. During this first month of the grant period, we are attempting to correlate the decrease in cell aggressiveness in HARV cultures with changes in the expression of growth factors, growth factor receptors and extracellular matrix components synthesized by DU 145 cells.

We speculate that the reduction in cell aggressiveness can be attributed to three-dimensional growth in the HARV. Three-dimensional growth allows for increased cell-cell and cell-matrix interaction relative to monolayer culture and to a rich interstitial fluid containing growth factors and other biological effectors at concentrations found in intact tissue.
II. MSAD Program Tasks — Ground-based

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals


Presentations
Insect-Cell Cultivation in the NASA High Aspect Rotating-Wall Vessel

Principal Investigator: Prof. Kim O'Connor

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
This project was designed to broaden the application of rotating-wall vessels and, in particular, the High Aspect Rotating-Wall Vessel (HARV) to insect-cell cultivation and the production of recombinant proteins from these cultures. During this period, we expanded our research into the areas of tissue modeling with HARV cultures of DU 145 human prostate adenocarcinoma cells and bovine corneal cells.

Insect Cells
With Sf9 cells derived from Spodoptera frugiperda, we assessed whether the HARV had a beneficial effect on insect-cell cultivation relative to conventional bioreactors. To this end, one of our objectives was to compare the growth and metabolic profiles of Sf9 cells in these two reactor environments.

Based on the positive results from this comparison, we began investigating the mechanisms by which the HARV affects insect-cell cultivation. We hypothesized that reduced turbulence in the HARV relative to conventional bioreactors may alter stress protein synthesis in Sf9 cells. Since very little is known about stress protein induction in this cell line, another objective during this phase of research was to characterize the phenomenon.

It was also evident from the comparison that HARV cultivation may improve recombinant protein production from stably transformed insect cells. To this end, a third objective was to initiate research in this area.

DU 145 Cells
Our objectives are five-fold, the first of which is to develop cell models of human prostate cancer that can be used to identify more accurate markers of metastatic potential than are currently available. Second, we will ascertain whether cell models generated with the HARV have substantially different metastatic potentials from those produced with conventional cultivation chambers. And, we will attempt to gain insight into the mechanisms which underlie these differences.

Another objective is to quantify the effects that the HARV has on metastatic potential which others have described qualitatively. Our fifth objective is to compare cell cultures grown in the HARV and spinner flask. The latter is a popular bioreactor for bench-scale research in bioengineering. A favorable comparison for the HARV will aid in its acceptance by the engineering community.

Corneal Cells
During the grant period, we initiated a study to develop tissue models of the cornea with the HARV. Our objectives for this preliminary work were to select cell lines and characterize the growth and morphology of our initial models.

Task Description:
Insect Cells
Our research involves three cells lines: Sf9, a popular host for recombinant protein production; I31FB, a clone derived from Sf9 cells that continuously expresses recombinant beta-galactosidase; and S2LO-VCAM, Schneider II cells derived from Drosophila melanogaster expressing recombinant human cell adhesion molecular upon induction with copper ions.

In comparing HARV cultures of insect cells with those produced in conventional bioreactors, we selected a shaker
flask for the control. Relative to other conventional bioreactors, shaker flasks provide an extremely gentle culturing environment. Yet, hydrodynamic forces in this control are nearly twice the magnitude of those encountered in the HARV. Differences observed between HARV and shaker cultures of insect cells should be applicable and magnified when comparisons are made between the HARV and more turbulent bioreactors.

In our study of stress protein induction, we identified a subset of stress proteins that are responsive to multiple forms of stress. The subset will be the focus of our future work with the HARV.

DU 145 Cells
This study explores the dependence of metastatic potential on the design of the cultivation chamber for growth of DU 145 cells. Three chambers were used in this study: the HARV, spinner flask, and T-flask. DU 145 cells grew in the HARV and spinner flask in suspension as aggregated, three-dimensional cultures of Cytodex-3 microcarrier beads and as cell monolayers in the T-flask. While both the HARV and spinner flask produced three-dimensional cultures, fluid turbulence, which can damage animal cells, was greater in the latter.

Metastatic potential was characterized in terms of cell growth and differentiation. Slower growth is indicative of reduced metastatic potential. By itself, however, growth is not an accurate measure of metastatic potential; it could reflect, for example, poor cell health. Therefore, another indicator, cell differentiation, was investigated. In solid tumors, growth is often inversely related to differentiation. Slower growth coupled with enhanced differentiation would demonstrate cell health and be a strong indicator of reduced metastatic potential.

Corneal Cells
We started this project with Dr. Glenn Spaulding at NASA. At that time, our corneal cells were purchased as a mixed population of rabbit endothelial cells, fibroblasts and epithelial cells in fifth passage. The fibroblasts quickly dominated the culture. In fact, the endothelial cells had become senescent within two passages after inoculation in the HARV.

It was evident from this study that we needed to work with younger cultures and pure cell types. To achieve this, we established corneal cell lines ourselves from bovine eyes obtained at a local slaughterhouse.

Task Significance:
Insect Cells
Insect cells are invertebrate animal cells that show great potential as hosts for the commercial production of recombinant proteins. This is a relatively new phenomenon, beginning in the mid 1980's. Since this time, there has been an explosive growth in the number of recombinant proteins that have been expressed in insect cells with less than 10 by 1984 to over 100 by 1990. The reason for this growth is the ease with which recombinant proteins can be expressed in and purified from insect cells relative to mammalian cells.

Recombinant proteins are playing an increasingly greater role in medicine, basic research and agriculture for use as protein therapies, diagnostic agents and insecticides to name a few. The sale of recombinant proteins makes a substantial contribution to the U.S. economic market. In the pharmaceutical industry alone, biotherapies, which are primarily recombinant proteins, generated $1.2 billion in U.S. sales in 1991 and are predicted to generate nearly $8.0 billion in 2001.

With current technology, commercial production of recombinant proteins from insect cells is hindered since they are more sensitive to hydrodynamic forces in a bioreactor and have a greater oxygen uptake rate than most vertebrate cells. This combination places severe constraints on cell oxygenation during large-scale cultivation; sufficient oxygen must be transported to the insect cells for respiration without inducing hydrodynamic damage to the cells.

Conventional methods of oxygenation, such as sparging, supply sufficient oxygen but do so by increasing mixing of growth medium and gas flow rate, resulting in cell destruction from hydrodynamic forces in the medium and at the gas/liquid interface.
The HARV provides a low-turbulence environment in which cells grow suspended in culture medium through end-over-end mixing and are oxygenated via a silicon membrane. In this environment, hydrodynamic forces are greatly reduced over those in conventional bioreactors while mass transfer rates for oxygen remain high.

Our research evaluates the potential of the HARV for cultivation of insect cells and production of recombinant proteins derived from these cells.

**DU 145 Cells**
The prostate is the most common site of cancer in men. In the U.S. last year, approximately 200,000 men were diagnosed with prostate cancer; another 38,000 died from this disease. A unique feature of prostate cancer is its prevalence at autopsy in men with foci of carcinoma cells confined to the prostate but who have died from other causes. This discrepancy between prevalence and mortality confounds treatment decisions. Because death from prostate cancer ultimately results from metastasis, the ability to predict the metastatic potential of localized prostate cancer will greatly aid in treatment design.

Currently, choice of treatment in clinically localized prostate cancer is based largely on phenotypic properties of the diseased tissue such as tumor volume and DNA ploidy. These markers only indirectly measure metastatic potential, and thus, their ability to accurately predict a patient's prognosis is limited. Monitoring proteins and/or genes that regulate cell growth should more accurately distinguish between tumors with a potential for slow progression and those that progress rapidly, leading to early death.

Identification of useful markers of metastatic potential of prostate cancer has been hindered in part by a lack of human cell models that mimic the diseased prostate. With the HARV, we have been able to generate such models. Specifically, we have regulated the metastatic potential of DU 145 cells by the choice of cultivation chamber.

This work is done in collaboration with researchers in the Department of Pathology at Tulane Medical School. This group has a drug development effort for prostate cancer and has strong ties to the pharmaceutical industry. It is headed by Professor Sanda Clejan, Director of Chemistry, Special Chemistry and Endocrinology for Clinical Pathology.

**Corneal Cells**
The need for corneal tissue models is great. They are used to develop novel treatments for corneal disorders such as infection with herpes simplex virus. It is estimated that 90% of U.S. adults have antibodies to herpes and 10% of these give clinical manifestations of the disease. Herpes infection is the leading cause of corneal blindness. Tissue modeling can eventually regenerate tissue for transplantation. Corneal transplants are the most frequently performed human transplant procedure. Each year, there are in excess of 40,000 such surgeries. At any given time, there are nearly 5,000 people in the U.S. on waiting lists for transplants because of tissue shortage. Regenerated tissue would eliminate this shortage.

The objective of tissue modeling is to develop a tissue structure that mimics its native counterpart. The majority of cornea models have been cell monolayers; however, they preclude an accurate representation of three-dimensional phenomena such as would healing and nutrient mass transport. Another popular model is donor tissue. Its use is limited by availability and reproducibility. These limitations can be overcome with three-dimensional tissue supported by extracellular matrix synthesized by the cells themselves.

**Progress During FY 1995:**

**Insect Cells**
A methodology was developed to culture Sf9 cells in the HARV. In this vessel, the growth and metabolic profile for these insect cells were profoundly different that those obtained in shaker-flask culture. Specifically, stationary phase in the HARV was extended from 24 hr to at least 7 days, while cell concentration and viability remained in excess of $1 \times 10^7$ viable cells/ml and 90%, respectively.
Measurements of glucose utilization, lactate production, ammonia production and pH change indicate that the HARV had a two-fold effect on cell metabolism. Less nutrients were consumed and less wastes were produced in stationary phase by as much as a factor of 4 over that achieved in shaker culture. Those nutrients that were consumed in the HARV were directed along different metabolic pathways as evidenced by an extreme shift in glucose utilization from consumption to production in lag phase and a decrease in yield coefficients by one half in stationary phase. These changes reflect a reduction in hydrodynamic forces from over 1 dyne/cm² in shaker culture to under 0.5 dyne/cm² in the HARV.

These results suggest that cultivation of insect cells in the HARV may reduce production costs of cell-derived biologicals by extending production time and reducing medium requirements. In addition, this research represents the first use of the HARV for cultivation of freely suspended and non-mammalian animal cells.

For the work described in the previous paragraphs, we received a NASA Tech Brief Award.

In our characterization of stress protein induction, we observed that anoxia transiently induces a cluster of heat shock proteins at 71 and 72 kDa in Sf9 cells. This is a subset of a larger group of stress proteins induced by heat shock. Several heat shock proteins reported in this study were previously undetected in Sf9. With these additional proteins, the stress response of hyperthermic Sf9 closely resembles that of Drosophila melanogaster. Prior investigations of stress protein induction during oxygen deprivation focused on mammalian cells. In sharp contrast to these cells, anoxic Sf9 cells neither induce glucose-regulated proteins nor suppress the heat shock family of 71/72 kDa proteins.

We are collaborating with Professor Don Jarvis at Texas A&M University and Dr. Alain Bernard at Glaxo in Switzerland to study recombinant protein production from stably transformed insect cells in the HARV. With Dr. Jarvis, we have determined that the growth and metabolic profiles of IElFB cells in the HARV and shaker flask are similar to those of Sf9 cells. We have extended the duration of these experiments to determine how long the stationary phase in the HARV is stable. Also, we are measuring recombinant beta-glactosidase production in terms of the intracellular and extracellular concentration of this protein present in medium as well as its rate of synthesis.

Our collaboration with Dr. Bernard on S2LO-VCAM cells is in its initial stages. We have mastered the culturing procedures for shaker and T-flask. The cells are smaller than Sf9 cells and have far faster doubling times. We are currently learning the procedures to detect the recombinant cell adhesion molecule.

DU 145 Cells
Cultivation of DU 145 cells in the HARV, spinner flask and T-flask generated cultures with distinctly different metastatic potentials.

HARV cultures were the least aggressive; they were characterized by slower growth and enhanced differentiation relative to T-flask controls. Slower growth was reflected in reduced doubling times, glucose consumption and percentage of S-phase cells. The extent of differentiation was monitored by the staining intensity of select cytoskeletal proteins: cytokeratins 8 and 18, actin and vimentin.

This reduction in metastatic potential was attributed to three-dimensional growth in the HARV. Three-dimensional growth allows for increased cell-cell and cell-matrix interaction relative to monolayer culture and to a rich interstitial fluid containing growth factors and other biological effectors at concentrations found in intact tissue. Also, it should be noted that genetic mutations and mass-transport limitations cannot account for the changes induced in the HARV cultures.

The metastatic potential in the spinner flask was slightly higher than in the HARV, particularly with respect to cell differentiation. Other differences between the HARV and spinner flask pertain to aggregate morphology. Scanning electron microscopy revealed that aggregates were larger and their surface structures were more pronounced in the HARV. In comparing these two vessels, the lower metastatic potential in the HARV could have reflected more extensive three-dimensional growth and reduced turbulence.
This is the first quantitative study of the effects of HARV cultivation on metastatic potential and the first comparison of the HARV and spinner flask.

**Corneal Cells**
With the aid of Professor Diane Blake of the Departments of Ophthalmology and Biochemistry at Tulane Medical School, we have developed protocols to establish pure cultures of corneal endothelial cells and fibroblasts from bovine eyes. Culture purity was verified with immunocytostaining.

While the fibroblasts can proliferate for many passages, the endothelial cells grow for only a limited number of passages. After second passage, maximum cell density is reduced in half as average cell size increases, and doubling time slows slightly in third and fourth passage. With a week between each passage, it is possible to conduct a three-week experiment with proliferating endothelial cells despite their limited life span.

Currently, HARV cultures of these cell lines are under investigation.

**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Journals**


**Presentations**


Use of Rotating Wall Vessel (RWV) to Facilitate Culture of Norwalk Virus

Task Objective:
The objective of the project is to use the NASA-developed slow-turning lateral vessel (STLV), a type of rotating-wall vessel (RWV) used for differentiation of tissue cultures, to facilitate culture of Norwalk virus. We will culture and confirm differentiation of various human intestinal cell lines or populations. With this completed, stool specimens derived from Norwalk virus-infected volunteers will be used to infect selected human intestinal cell lines obtained from RWV tissue culture differentiation studies. The infected cell cultures will be characterized for evidence of Norwalk virus replication by immunochromical, biochemical, ultrastructural, histological, and infectivity studies, and neutralization assays. Finally, we plan to generate tissue culture adapted strains of Norwalk virus with enhanced replication properties in conventional tissue culture lines and systems.

Task Description:
This project will use the RWV as a means to develop a tissue culture system for Norwalk virus, a gastrointestinal pathogen. In collaboration with Dr. M.P. Moyer and T. Goodwin, we have gained the expertise of tissue culture establishment of human intestinal derived cell populations in the RWV system. This system is being refined for our purpose of propagating Norwalk virus. The project utilizes the collected specimens from our human volunteer Norwalk virus challenge studies (Dr. P.C. Johnson) as an inoculum. Evidence will be accumulated, as discussed in objectives, to confirm replication of the virus in the RWV system. Once samples enriched for putative progeny virus populations have been identified, further characterization of the material will occur. These include gradient purification, biochemical analysis, and electron microscopy. As a conclusive test, the progeny virus from the cultures will be passed several times into new RWV cultures. The confirmation of replication of Norwalk virus will be undertaken as occurred previously with the primary passages. Eventually, the RWV-derived Norwalk strains will be adapted to conventional culture systems, which will enable tests such as neutralization assays to be performed to evaluate the immunogenic efficacy of future Norwalk virus vaccine preparations.

Task Significance:
The successful propagation of Norwalk virus may require a differentiated intestinal tissue culture system. A well known Norwalk virus inoculum, 8Pflls, is infectious in human volunteer studies, but has not been demonstrated to replicate in vitro. Approximately 60% of volunteer individuals challenged with Norwalk virus do experience gastrointestinal, corroborated with studies at this institution by Johnson and other investigators. Disease occurs in the upper small intestine. Since the original inoculum used for human volunteer studies is being depleted, other sources of infectious virus need to be generated for studies in basic virology, vaccine development and infectious dose volunteer challenge studies. It is our contention that mature three-dimensional culture of human intestinal tissue will support Norwalk virus growth, whereas undifferentiated or unrelated tissues are inadequate to do so. Progeny virus generated by these studies will be used in future testing of vaccine trial efficacy through demonstration of neutralizing antibodies of immunized individuals.

Progress During FY 1995:
One of the first well-documented confirmations of the enhanced tissue culture system provided by the RWV was reported by Goodwin, Moyer and coworkers, (P.S.E.B.M., 202: 181, 1993). Human mesenchymal cells were established on microcarrier beads in the RWV, followed by addition of small intestinal epithelial cells. The cell cultures developed into a three-dimensional array containing differentiated cellular markers that were not detectable or lowly expressed in conventional monolayer cells. Their report exemplifies the possibility of completely new
aspects of biological investigations, including applications in the private sector.

In collaboration with Dr. Mary Pat Moyer at the University of Texas Health Science Center at San Antonio, we initiated pilot studies to propagate Norwalk virus. We have preliminary evidence that Norwalk virus can be cultured utilizing this system. Using the cell preparations reported in her article, we infected these and other cultures with a Norwalk inoculum derived from human volunteer studies.

Preliminary ultrastructural analysis of these mesenchymal/epithelial human intestinal cells infected with Norwalk virus was undertaken. At day 0 prior to infection, the ultrastructure appears without obvious pathology. However, at day 6 post-infection, cytoplasmic vacuoles are noted. This pathology progressed to frank cytoplasmic degradation and nuclear condensation by day 8 post-infection.

Immunohistochemical staining of Norwalk-infected formalin fixed and paraffinized tissue from the RWV has been pursued. Acute and convalescent (>3 weeks after illness) paired sera from two of our Norwalk-challenged volunteer donors as well as hyperimmune rabbit anti-recombinant Norwalk virus (rNV) capsid serum (supplied in collaboration by Dr. M.E. Estes) have been used in an immunoperoxidase staining and ELISA procedures.

Our further studies support the contention that there is some level of Norwalk virus replication in the human mesenchymal/epithelial cells grown in the RWV. Low levels of detectable antigen by solid phase and in situ immunocytochemistry were noted in these cultures. In addition, glucose consumption fell during days 6 and 8 post-infection. The evidence of Norwalk virus replication within the primary human intestinal cell cultures grown in the RWV was quite encouraging. However, the difficulty and reliability in obtaining these primary cell populations has necessitated a search for alternative tissue culture sources. Accordingly, we have been investigating several established human intestinal cell lines for adaptation within the RWV.

The human intestinal tissue culture cell lines were established in a culture on microcarrier beads in the RWV. At two weeks of culture, the cells were beginning to cover the microcarrier beads as seen by phase contrast microscopy. Evidence of three dimensional structure was evident. Initial differentiation studies have detected keratin within these microcarrier RWV cultures. The cells appear to be arranging themselves in a pattern similar to gut epithelium.

The search for an optimal human intestinal cell line is ongoing for our purposes. Initial studies have been encouraging. Samples were harvested daily for the first four days, and every other day thereafter. Using hyperimmune sera, obtained from immunized animals with rNV capsid stocks, infected cells were analyzed for evidence of Norwalk virus replication. Cell samples were fixed in formalin and paraffinized for immunocytochemical analysis. Cytoplasmic reactivity was noted in rare cells within one day post-infection. The total number of cells displaying immunoperoxidase reactivity was found to increase several-fold with cell samples from subsequent days post-infection. The immunostaining reactivity is blocked when the hyperimmune serum is preincubated with rNV capsid preparations, supplied by Dr. Estes. Borderline levels of Norwalk virus were detected by the polyclonal anti-rNV ELISA, but this must be confirmed by quantitative PCR studies. The histological and ultrastructural investigation of this work is underway. This is an important first step in establishing a characterized cell culture system within the RWV for our Norwalk virus propagation work.
BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals
II. MSAD Program Tasks — Ground-based

Shear Sensitivities of Human Bone Marrow Cultures

Principal Investigator: Dr. Bernhard O. Palsson
University of California, San Diego

Co-Investigators:
Wang, Dr. H.
University of Michigan

Task Objective:
The objective of this research is to reconstruct human bone marrow tissue ex vivo using the NASA bioreactor to provide the culture environment for three-dimensional growth.

Task Description:
The specific aims of this program are three:
1) To find optimal growth conditions for human bone marrow as a function of the supplied growth factors;
2) To develop a shear stress chamber that measures the shear stress sensitivity of human bone marrow cells;
3) To use the information gained from 1) and 2) to implement long-term, continuous bone marrow cultures in the RWV.

Task Significance:
This study will focus on the elucidating role of three-dimensionality in bone marrow stem cell differentiation. The development of a three-dimensional in vitro cell model will permit investigation into the biochemical signals that trigger cell differentiation into various stages and subtypes of human blood cells. It also will permit the investigation of the role of three-dimensionality in extensive cell to cell contact and exposure to growth factors.

Progress During FY 1995:
Conditions for the expansion of adult human bone marrow mononuclear cells in the rotating wall vessel (RWV) have been established. Total cell numbers and the number of progenitor cells increase over time by a factor of 10-20 fold. Shear sensitivity of the cells does not hamper growth of these cells. At present, it is unknown whether stem cells are maintained or if they expand in number in the RWV. This issue will be investigated in the coming year.

Furthermore, we have shown that the composition of the basal medium has a significant effect on the growth performance. Elevated amino acid and vitamin concentrations improve growth by 50-100 percent. Optimal amino acid and vitamin concentrations will be determined in the coming year using a factorial experimental design. The interactions between the stirring rate and the inoculum density have been investigated. These two factors interact and we have determined the critical inoculum densities for a range of rpms. Optimal bioreactor operational procedures will be developed based on this data.

Students Funded Under Research:

Task Initiation: 11/92  Expiration: 11/95
Project Identification: 962-23-01-09
NASA Contract No.:  NAG9-652
Responsible Center:  JSC

Printed: 28 FEB 1996
Microgravity and Immunosuppression: A Ground-Based Model in the Slow Turning Lateral Vessel Bioreactor

Principal Investigator: Dr. Neal R. Pellis
NASA Johnson Space Center (JSC)

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
This investigation tests the hypothesis that simulated and true microgravity modulate immune function by affecting lymphocyte locomotion through intercellular matrix.

Task Description:
The investigation addresses three specific aims that encompass the use of the Slow Turning Lateral Vessel (STLV) as a model for microgravity. In the first aim, the basis of the loss of locomotion in the STLV is sought using video and biochemical analysis of the critical components of lymphocyte locomotion. The second seeks the basic mechanisms to be targeted in an attempt to ameliorate the loss of lymphocyte locomotion in the STLV and in microgravity. Finally, the third aim is to determine if the loss in locomotion is related to actual decline in immunity.

Task Significance:
The ability of the immune system to maintain hemeostasis is predicated on many different lymphocyte activities. A critical aspect in immune function is the movement of lymphocytes through interstitium to engage and destroy antigenic stimulus. Both clinostatic rotation and actual microgravity of space inhibit the locomotion of lymphocytes through Type I collagen. Therefore, it is essential to understand how alterations in gravity modify immune function, not only in the context of space travel, but also to apply the microgravity tool to modulate immune function.

Progress During FY 1995:
Clinostatic rotation in the STLV inhibits the locomotion of resting lymphocytes through Type I collagen. Activation by polyclonal stimulation increases the proportion of locomotory cells in the STLV. Treatment of lymphocytes with transferrin-iron complex significantly promoted locomotion in stationary and rotated lymphocyte cultures. An immunological adjuvant, lipophilic muramyl-tripeptide (MTP-PE, had similar activity. It is tentatively concluded that each of these processes may result in the production of chemokinetic substances that participate in the locomotory process. Using molecular genetic strategies, we will investigate the changes in the expression of the gene that encode lymphokinetic and chemokinetic substances in lymphocytes.

STUDENTS FUNDED UNDER RESEARCH:

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TASK INITIATION: 9/95  EXPIRATION: 8/99
PROJECT IDENTIFICATION: 962-23-01-38
RESPONSIBLE CENTER: JSC

Printed: 28 FEB 1996
BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals


Presentations

Presentation "Establishment of 3-dimensional primary hepatocyte cultures in microgravity environment." Amer. Assoc. for the Study of Liver Diseases, Chicago, IL, Nov. 3-7, 1995.


II. MSAD Program Tasks — Ground-based

**Isolation of the Flow, Growth and Nucleation Rate, and Microgravity Effects on Protein Crystal Growth**

**Principal Investigator:** Dr. Marc L. Pusey  
**NASA Marshall Space Flight Center (MSFC)**

**Co-Investigators:**

No Co-I's Assigned to this Task

**Task Objective:**

The purpose of these studies is to determine how \( \mu_g \) affects the protein crystal growth process and how that effect can be optimized for the production of higher diffraction quality protein crystals. In pursuit of this goal, the research will comprise a ground and a \( g \)-based program. The research will consist of:

- the development of new proteins for use as models in protein crystal growth studies;
- determinations of protein solubilities as a function of solution parameters;
- the determination of protein crystal nucleation rates;
- the effects of low velocity solution flow on protein crystal nucleation and growth rates;
- the effects of low velocity solution flow on protein-protein and protein-solvent interactions; and,
- the effects of protein crystal nucleation and growth rates on Earth and in \( \mu_g \) on crystal diffraction quality.

At the conclusion of this effort several new proteins will have been developed as models for protein crystal growth. This will entail acquiring a detailed background knowledge about their crystallization conditions and solubility behavior, to a level of detail approximating that currently available for hen egg-white lysozyme. This data will be invaluable for the subsequent research to be carried out in this and other laboratories.

**Task Description:**

For the first year, the efforts will be centered upon developing methods of measuring crystal nucleation rates and reconciling these methods with each other and with more traditional "bulk solution" type measurements. Instrumentation will be acquired to build a means of following the aggregation, by light scattering and scintillation measurements, along a low velocity flow cell. Measurements will be made of crystal growth rates in a flowing solution. The effects of variations in the nucleation and growth rate on lysozyme crystal quality will be determined, based upon experiments done with a long duration dialysis system developed in this laboratory (McDUCK, or Multiple-chamber Dialysis Unit for Crystallization Kinetics). We will also begin the screening process for development of several new proteins as potential candidate model proteins for use in protein crystal growth studies.

**Task Significance:**

Evidence shows that high purity and a typically narrow range of solution conditions are important to the growth of high x-ray diffraction quality protein crystals. Intuitively, one would assume that careful control of the processes, resulting in the slow growth of carefully nucleated crystals would result in the highest quality crystals. To date this has not been demonstrated and the comparative effects of nucleation rate, growth rate, and \( \mu_g \) on the obtained protein crystal quality are not known. Also absent are any clear explanations for the effects of solution flow on protein crystal growth, often cited as the rationale for the use of \( \mu_g \). This research will show, using several model proteins, how each factor (i.e., nucleation rate, growth rate, solution flow, and \( \mu_g \)) affects protein crystal growth. These results can then be incorporated into the subsequent design of protein crystal growth systems for use both on Earth and in \( \mu_g \).

**Progress During FY 1995:**

This work is a new start for FY95. Preliminary results have been obtained for the McDUCK apparatus. After a three-month long equilibration period, crystals from each of the chambers were mounted and tested for x-ray
II. MSAD Program Tasks — Ground-based

Discipline: Biotechnology

diffraction quality. While all crystals diffracted to \( \approx 1.6 \) \( \AA \) resolution with similar data quality, there was a marked variation in the quality of the low resolution data. In general it was found that the longer the nucleation rate/slower the growth rate the higher the quality of the low resolution data. More experiments are underway to verify and extend these results.

We have purified ca 30 grams of concanavalin A, and are now doing preliminary screening studies to identify useful crystallization conditions. Crude glucose isomerase (ca 15 gms) was donated to this laboratory by NOVO Nordisk. Conditions have been identified which yield small bipyrimidal crystals by sitting drops and by dialysis, and solubility studies will be initiated with this material.

**STUDENTS FUNDED UNDER RESEARCH:**

**TASK INITIATION:** 6/95  **EXPIRATION:** 6/99  
**PROJECT IDENTIFICATION:** 962-23-08-38  
**RESPONSIBLE CENTER:** MSFC

**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Journals**


Microgravity Crystallization of Avian Egg White Ovostatin

Principal Investigator: Dr. Marc L. Pusey
NASA Marshall Space Flight Center (MSFC)

Co-Investigators:
Carter, D.
NASA Marshall Space Flight Center (MSFC)

Task Objective:
The research objective is designed to study the ovostatin from the standpoints of its suitability as a model protein for protein crystal nucleation and growth mechanisms, the bio-mechanical movements which apparently form the basis of its inhibitory activity, and using it as a means of studying the a2M group of proteins. Further, the intention is to develop the crystallization conditions to be tried based upon studies of the physico-chemical parameters, such as solubility phase behavior and observed nucleation kinetics. This will be our first demonstration protein for the application of what is being learned about the crystal growth of macromolecules.

Task Description:
The study of ovostatin is of interest for several reasons. This protein is attractive as a model for protein crystal nucleation and growth studies. Crystals can be easily and rapidly grown in bulk solution. With a minimum step height of 25-34 nm, surface features should be readily detectable in real-time using interferometric techniques, allowing direct observation of the crystal growth process. The large size would facilitate protein crystal nucleation studies using light scattering methods. Practically, ovostatin can be easily purified in large quantities, requiring about six dozen hen egg whites/gram of protein. Material purified has been stable for prolonged periods (over four months) during crystallization trials and has been kept for over one year as a lyophilized powder. Finally, there is the similarity between it and a2M. Currently, the only structural information extant is from electron microscopy studies of isolated molecules.

Task 1: Preparation of ovostatin for use in experiments. Large scale protein purification will be continuous for the duration of the project. This material will be used to establish the reference ground-based crystal quality against which future flight crystals will be measured. Ovostatin prepared in this laboratory will be made freely available to other researchers wishing to study it.

Task 2: Screening of ovostatin crystallization conditions. The best crystallization conditions determined at this time will be used for future flight experiment(s). These conditions will then be used for the final solubility diagram determinations to be used in designing the flight experiments.

Task 3: Establishing baseline crystal quality. Multiple data sets will be acquired using the x-ray diffractometer. These will be used to establish the baseline ground-grown crystal quality.

Task 4: Studies of ovostatin. These will last for the duration of this project. These will be done using the instrumentation developed in the laboratory for the study of the protein crystal nucleation and growth processes.

Task Significance:
Any knowledge gained about ovostatin based on crystal structure analysis would advance the overall knowledge of this family of proteins.

Progress During FY 1995:
Previous microgravity protein crystal growth experiments have used vapor diffusion, free interface diffusion, and dialysis techniques with most being done using vapor diffusion. Littke and John, using free interface diffusion,
reported a $10^3$ volume increase for beta-Galactosidase over crystals grown on Earth. In vapor diffusion experiments, at least three proteins have shown significantly improved diffraction resolution over any of the crystals grown on Earth, despite the fact that, because of short flight durations, crystallization conditions have had to be optimized for rapid nucleation and growth, i.e., the temporal conditions were more favorable for lower quality crystals. Current experiences are that ovostatin crystals nucleate and finish growing within a one- to three-day period. The process will have to be slowed down to fit better within the timeframe for a typical microgravity PCG experiment, as opposed to the usual case where the crystal growth process must be speeded up. Based on the ovostatin crystal currently grown, there is considerable room for improvement in both crystal size and diffraction resolution.

Ovostatin purified in this laboratory has now been supplied to four other research groups as a test material. Dr. William Wilson (Mississippi State University) has found a dramatic dimerization-tetramerization behavior at low ionic strength. This has led us to an investigation of ovostatin activity vs. structure which has been carried out by a mummer faculty (Dr. D. Moriarity, UAH). Ovostatin crystallizability has been found to be very sensitive to the purification procedure and materials employed. We are currently supplying ovostatin to the laboratory of Dr. David Blow (Imperial College) for extensive crystallization trials which we hope will lead to a preliminary structural determination. However, the protein sent to Imperial College has yet to crystallize, even when material from the same preparation gives crystals in our laboratory. Also, we have yet to find any new crystallization conditions for ovostatin beyond those first described in the initial proposal.

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Printed: 28 FEB 1996
II. MSAD Program Tasks — Ground-based

Discipline: Biotechnology

Stem Cell Expansion in Rotating Bioreactors

Principal Investigator: Dr. Peter J. Quensenberry
University of Massachusetts

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:

Task Objective #1: This is to develop improved methods for cytokine supported in vitro expansion or maintenance of engrafting lympho-hematopoietic progenitor/stem cells using the non-shear rotating three-dimensional culture vehicles developed within the NASA research program.

Task Objective #2: This is to evaluate the culture of purified stem cell populations in the non-shear rotating three-dimensional culture vehicles and compare this with conventional culture vehicles.

Task Objective #3: This is to determine if co-culture of isolated irradiated stromal cells or a stromal cell line in the non-shear rotating three-dimensional culture vehicles would improve expansion of stem cells and maintenance of primitive engrafting stem cells.

Task Description:

Task Description #1: We have previously found that cytokine exposure in conventional culture vehicles results in expansion of progenitor cells assayed in vitro, induction of cell cycle progression but the development of a profound defect in engraftment in in vivo transplantation models. In our first series of experiments, we will compare conventional culture vehicles to the non-shear rotating three-dimensional culture vehicles for their ability to support both in vitro progenitor cells and in vivo engrafting cells in vitro after exposure to a variety of cytokines including Interleukin-3, Interleukin-6, steel factor, GM-CSF, basic fibroblast growth factor and Interleukin-1 in various combinations. We will carry out cytokine dose response and time course evaluate the impact of various cell concentrations and monitor in vitro progenitors, their cell cycle status and engrafting ability. In all the studies in Task Description #1, whole unseparated murine marrow cells will be cultured. Further, in these experiments, we will assess cytokines in optimal doses for their capacity to expand stem cells in vitro and we will also access different conditions and different cytokine doses for their ability to maintain without induced cell proliferation progenitor/stem cells in vitro. These later studies are particularly important in that we feel that this may be the culture condition in which engrafting stem cells can be maintained and induction of cycling may impair engraftment in this setting. There are many important considerations with regard to the culture of hematopoietic stem cells in vitro and approaches to expand stel cell populations. As noted above, our data suggests that cycle activation actually prevents cells from engrafting. However, cycle activation is necessary for expansion of stem cell populations and for retroviral integration in various gene therapy approaches. Therefore, a very important aspect of these studies would be to determine whether stem cells can be induced to expand with associated proliferation and activation of cell cycle and then this state reversed so that an expanded population of stem cells or a gene carrying population of stem cells may now return to the phenotype of a primitive engrafting stem cell. Accordingly, we will assess various conditions to see if we can reverse the cytokine induced engraftment defect. Once again, we will compare the non-shear rotating three-dimensional culture vehicles with standard tissue culture flasks. In these experiments, stem cells will be induced to cycle by cytokine exposure and then these populations will be subjected to either growth in selected low levels of cytokines, hematopoietic stem cells inhibitors such as transforming growth factor beta, or growth on various stromal populations. The goal here would be to induce quiescence in the previously stimulated stem cell populations and in these studies we will assess the cells for cycle status, progenitor content and an ability to engraft in in vivo models.

Task Description #2 is as outlined under #1 for whole marrow, but will be carried out on highly purified populations of murine hematopoietic stem cells. Purifications will consist of depleting lineage positive cells and
then selecting cells which stain very weakly with the dyes Rhodamine and Hoechst. Alternative purifications utilizing Sca antigen or c-Kit are also possible. The assessment of growth and comparison between systems will be as described in Task Description #1.

Task Description #3 stromal cells will be established in the culture vehicles and then co-cultured with either whole marrow or purified marrow cells. Outcomes would be assessed as outlined under Task Description #1.

Task Significance:
Task significance #1: Successful accomplishment of this Task Objective could lead to superior methods of maintaining hematopoietic stem cells in vitro in various proliferative states. This in turn could form a basis for more effective approaches to marrow transplantation and to gene therapy.

Task significance #2: The accomplishment of the goals outlined in Task Objective #1, but utilizing purified hematopoietic stem cells, would both open insights into basic stem cell biology but would also potentially reduce costs in marrow expansion and gene therapy approaches substantially making developed technology much more feasible for clinical application.

Task significance #3: It is possible that superior results with regard to stem cell maintenance and gene transfer will be obtained in the presence of stromal cell co-culture using the non-shear rotating three-dimensional culture vehicles. Success here would, again, increase efficacy of approaches for both marrow transplant and gene therapy.

Progress During FY 1995:
We have now obtained the cylindrical cell culture vessel and have begun our preliminary experiments with the culture vessel. We have not gone through a full detoxification and conditioning of the vessel. We are presently assessing growth of WEHI (a leukemia mouse cell line) cells in the vehicle. We are assessing the growth of WEHI cells in RPMI-1640 with 10% fetal calf serum. The initial experiments are without antibiotics. We initiated cultures with a total of 5.2 X 10^6 cells in approximately 80 ml. After two days of growth, we obtained 138 million cells in the same volume. Viability of the initial starting cells was 85.6% and after two days was 88.5%. Due to the machine being inadvertently shut off, we also were able to evaluate non-rotary conditions briefly and found that we could resuspend the cells and obtain good on-going viability. We are at present obtaining micro-carrier beads to initiate co-culture operations and have ordered two more vessels in order to begin comparisons with multiple conditions in the rotary vessels and with stationary flask cultures.

**STUDENTS FUNDED UNDER RESEARCH:**

**TASK INITIATION:** 9/95  **EXPIRATION:** 9/99

**PROJECT IDENTIFICATION:** 962-23-01-20

**NASA CONTRACT NO.:** NAG9-819

**RESPONSIBLE CENTER:** JSC
II. MSAD Program Tasks — Ground-based

Study of Crystallization and Solution Properties of Redesigned Protein Surfaces

Principal Investigator: Prof. David C. Richardson
Duke University Medical Center

Co-Investigators:
Gemert, K.
Richardson, J.
Duke University Medical Center

Task Objective:
The objective of this work is to determine what combinations of protein surface features and solution factors best promote the growth of highly ordered protein crystals and to seek strategies for improving control over the process. The experiment design is to make detailed analysis of crystal contacts in Cytochrome B562, construct a planned series of mutations at those crystal contacts, study crystal growth and solubility properties of the mutant proteins, study resultant crystal order and use the results to further refine analyses, hypothesis, and test mutants.

Task Description:
High-resolution x-ray crystal structures of proteins are the backbone of academic and industrial research efforts to understand and control the detailed functional properties of these important molecules. Many such studies are stymied, either by lack of crystals altogether or by crystals whose degree of order is inadequate to show details at the level of resolution needed.

Two sets of factors jointly influence the growth of ordered protein crystals: the atomic-level details of the protein surface, including flexibility and bound waters; and the solution conditions, including concentration and identity of precipitants and other components, pH and ionic strength, temperature, interactions at solid or liquid interfaces, vibration, convection currents, etc. Traditionally, the protein surface was not variable except by trying different species, and the only strategy for solution conditions was simply trying as many variations as possible. The most general conclusion was that what worked for one protein was likely to be different than what worked for another. Recently, however, there has been support and encouragement, largely led by NASA, for scientific study of the process of crystallization. The most notable single result so far has been the demonstration that growth in microgravity can produce significantly better-ordered crystals for many proteins, presumably because of the absence of convection currents at the crystal surface. The absence of convective currents, in turn, allows for the increased effect of random diffusion of the protein molecules at the crystal surface and for the increased effective binding energy of the protein molecule to the growing crystal. Both of these are presumed to lead to more accurate and more stable attachment of the protein molecules to the crystal, and thus a better-ordered crystal.

Three-to-four mutants will be studied per year. Each study will involve computer-aided graphics studies of crystal contacts and design of mutants, genetic engineering of mutants, protein purification, crystallization experiments, crystal solubility determinations, face growth rate measurements, calorimetric measurements of crystal growth, and evaluation of crystal diffraction.

Task Significance:
The research aims to contribute to the scientific understanding and the practical improvement of protein crystal growth by tying together a series of designed mutations at known crystal contacts with the changes in crystallization behavior and parameters. There are several logical levels at which the results of this research should be useful.

A research study of the relative strengths of the binding of protein molecules into their crystal is important for understanding which factors are improved in microgravity, whether the growth cessation phenomena can be alleviated in normal gravity, and how changes in crystal contacts can improve the overall order of protein crystals.
II. MSAD Program Tasks — Ground-based

A detailed study of protein crystal contacts and their specific effects may also help in the future to sort our influences on nucleation of protein crystals versus later growth. This research is designed to collect two overlapping but distinct types of information: what specific side chain changes will strengthen or weaken a contact for a particular crystallization media; and what contact strength is optimum, relative both to the other contacts and to the diffusion and convection conditions. Such information will surely aid in future rational control of the crystallization process. Crystal contacts are one type of protein-protein interaction. Related work in this laboratory studies the full range of such interactions with eventual goal of design and control of protein structure formation as well as crystallization.

Progress During FY 1995:

Diffraction quality crystals have been grown of seven different mutants of B562, which vary three residues of crystal contact loop E 81, G 82, K 83 (thus B562-EGK is native). B562-AVK, B562-ISA, B562-RGM, and B562-LGR crystallized under conditions similar to that of the native B562 conditions (3.5 M PO4, pH 7.0), while B562-LAA, B562-SLS, and B562-SSR crystallized under conditions of higher PO4 and lower pH (3.75 M PO4, pH 6.0). The remainder of the mutants studied formed needles or microcrystalline precipitate. The space groups of individual crystals were determined for four crystals (B562-AVK, P21; B562-LAA and B562-SLS, C2221; B562-SSR; and, P212121). All of which are a different space group than the wt_B562 (P1), suggesting something different about the packing of the molecules within the crystal but, fortunately, not precluding the particular target contact region being similar enough for meaningful comparison. Diffraction data has been collected for B562-AVK and B562-LAA, and is being processed to determine the molecular structure and the actual protein contacts in these crystals. Various mutants as well as B562-wt are being tried at different crystallization conditions to find pairs of crystals (wt and mutant) which can be directly compared. Computer graphics studies of crystal contacts and protein-protein interactions continue to be done to provide context for the results of the specific crystal structure experiments.

STUDENTS FUNDED UNDER RESEARCH:

| BS Students: | 3 |
| MS Students: | 0 |
| PhD Students: | 0 |
| BS Degrees: | 1 |
| MS Degrees: | 0 |
| PhD Degrees: | 1 |

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals


Presentations
Convective Flow Effects on Protein Crystal Growth and Diffraction Resolution

Principal Investigator: Prof. Franz E. Rosenberger  University of Alabama, Huntsville

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
This research aims at developing a detailed understanding of:

• the nucleation and growth mechanisms involved in the crystallization of globular proteins;
• the formation mechanisms of structural and compositional nonuniformities (defects) in protein crystals and their dependence on growth conditions; and,
• the dependence of x-ray diffraction resolution of protein crystals on defect types and concentrations and, thus, on crystallization conditions.

Task Description:
We seek to establish, for select proteins, a correlation of well-defined solution conditions (purity, pH, buffer, precipitant, supersaturation, temperature, and bulk transport) with nucleation and growth behavior during crystallization, and the x-ray diffraction resolution of the resulting crystals.

Towards the above goals we are pursuing the following tasks:

• Characterization of the purity of protein solutions by gel electrophoresis, immunoblotting with comparison to authentic protein standards, and Fast Protein Liquid Chromatography (FPLC).
• Preparation of highly homogeneous protein samples by FPLC.
• Studies of protein interactions and aggregation in under- and supersaturated solutions by static and dynamic light scattering.
• Determinations of the precipitant and protein impurity repartitioning between solutions and growing protein crystals, using atomic absorption and optical spectroscopy, ion selective potentiometry, and gel electrophoresis.
• In situ studies of the protein growth morphology and kinetics, with and without forced solution flow, by high-resolution microscopic interferometry.
• Numerical modeling of diffusive-convective mass and momentum transport, and the resulting interface morphology, in geometries characteristic of protein crystallization on Earth and in space, using experimentally determined precipitant repartitioning and growth kinetics data.
• Measurements of the kinematic viscosity of supersaturated protein solutions, with a capillary flow technique, in support of the modeling efforts.
• Measurements of the dependence of the refractive index of solutions on salt and protein concentration, in support of the light scattering studies.
• Measurements of protein solubilities as a function of precipitant concentration, pH and temperature, using a miniaturized optical scintillation technique.
• Characterization of the structural quality of selected crystals by x-ray diffraction and topography to reveal the influence of kinetics and transport effects.

Task Significance:
The pharmaceutical industry needs protein structure information to facilitate rational drug design. However, many of the currently available protein crystals are too imperfect to yield detailed structure information. The reasons for this low crystal perfection are not well understood. Interestingly, crystallization experiments in space have led to significant improvements in crystal perfection for some proteins. Again, the physical mechanisms for this are not clear. Our research aims at clarifying the connection between the magnitude of gravity present during protein
crystallization and the resulting crystal quality. This insight is expected to lead to the design of protein crystal growth techniques that result in larger and more perfect crystals.

**Progress During FY 1995:**

We have identified and quantified the protein impurities in hen-egg-white lysozyme (HEWL) obtained from the three major commercial suppliers. Depending on the source, the protein heterogeneities were found to be 1-6% (w/w). Furthermore, we have obtained gram quantities of electrophoretically homogeneous (>99.9% w/w) HEWL by single step semi-preparative scale cation exchange FPLC with a yield of about 50%.

The extensive body of simultaneous static and dynamic light scattering data obtained for under- and super-saturated HEWL solutions was quantitatively analyzed in terms of a colloid model potential. The net protein charge and Hamaker constant obtained by fitting of the potential to the scattering data agrees well with earlier results. This, together with the low polydispersity of the dynamic scattering data excludes the presence of HEWL aggregates prior to nucleation.

The high-resolution microscopic investigations of HEWL growth morphology and kinetics without forced flow were continued. Most importantly, growth from the highly purified material (see above) showed neither growth sector boundary delineations nor striations resulting from kinetics fluctuations. Furthermore, temperature-induced striations appeared only on extreme temperature changes in growth from this material. This emphasizes the importance of purification of the starting material for achieving low-defect-density protein crystal growth.

The repartitioning of Na+ and Cl- ions between HEWL solutions and crystals was concluded. It was unambiguously shown that the salt-corning is associated with the preferential incorporation of the higher molecular weight protein impurities into the crystals. No salt corning occurred in growth from the purified material. On re-contamination of HEWL with avidin, salt corning was as pronounced as in the least pure source material. This further emphasizes the importance of pure starting materials.

The numerical model for diffusive-convective transport in HEWL crystallization was expanded. On inclusion of a meso-scale model for the growth-step motion and its dependence on transport-conditioned protein and impurity concentrations, both the convex and concave growth morphologies observed earlier were quantitatively reproduced.

<table>
<thead>
<tr>
<th>STUDENTS FUNDED UNDER RESEARCH:</th>
<th>TASK INITIATION: 2/93</th>
<th>EXPIRATION: 1/96</th>
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<td>BS Students: 0</td>
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**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Journals**


**Presentations**


Printed: 28 FEB 1996
II. MSAD Program Tasks — Ground-based

**Discipline: Biotechnology**

Muschol, M. "Light scattering from lysozyme solutions: Monomeric interactions or aggregation?" ACS Southeast Regional Meeting, Birmingham, Alabama, October 1994.

Muschol, M. "Static and dynamic light scattering from protein solutions: aggregation or monomeric interactions?" Protein Crystal Growth Conference, Panama City Beach, Florida, April 1995.


Rosenberger, F. "How can crystals grow with faceted habits when corners and edges get more readily supplied with nutrient?" Physics Department, Michigan Technological University, March 1995.

Rosenberger, F. "Monte Carlo studies of the interdependence of crystal growth morphology, interface kinetics and bulk transport." Physics Department, Michigan Technological University, March 1995.

Rosenberger, F. "Lysozyme, a simple system, why bother?" Protein Crystal Growth Conference, Panama City Beach, Florida, April 1995.


Nucleation and Convection Effects in Protein Crystal Growth

Principal Investigator: Prof. Franz E. Rosenberger  
University of Alabama, Huntsville

Co-Investigators:
Vekilov, Dr. P.  
Muschol, Dr. M.  
University of Alabama, Huntsville

Task Objective:
The objectives of this research are:

- understanding of the nucleation and crystal growth mechanisms to facilitate a rational approach to protein crystallization;
- determination of the factors that currently limit the x-ray diffraction resolution of protein crystals, and their correlation to crystallization conditions; and,
- development of novel technologies to study and monitor protein crystal nucleation and growth processes, in order to increase the reproducibility and yield of protein crystallization.

Task Description:
Studies of the aggregation state in purified, compositionally characterized, under- and supersaturated protein solutions, to identify the conditions leading to the formation of crystalline nuclei versus undesirable precipitates. For this task, we are developing a novel methodology/instrumentation for simultaneous multiangle light scattering (SMALS) studies, and apply it to select proteins. The SMALS approach will, for the first time, allow for unambiguous aggregation studies to be conducted at the high supersaturations typical of protein crystallization processes. For low molecular weight proteins, the light scattering studies will be supplemented by neutron diffraction investigations of the aggregation behavior.

Studies of the effects of defined solute and impurity transport on morphology and kinetics in protein crystallization and on resulting crystal perfection, to test our hypothesis that the limited structural perfection of Earth (and some space) grown crystals is partly due to instabilities in the transport and interfacial processes involved. The growth morphology of select, well characterized proteins, will be monitored by high-resolution, in situ interferometry for a wide range of crystallization conditions. In particular, we will study the effects of forced solution flow/convection on kinetics and structural perfection. The structural quality of crystals will be evaluated by x-ray diffraction and topography.

Task Significance:
The pharmaceutical industry needs protein structure information to facilitate rational drug design. However, many of the currently available protein crystals are too imperfect to yield detailed structure information. The reasons for this low crystal perfection are not well understood. Interestingly, crystallization experiments in space have led to significant improvements in crystal perfection for some proteins. Again, the physical mechanisms for this are not clear. Our research aims at clarifying the connection between the magnitude of gravity present during protein crystallization and the resulting crystal quality. This insight is expected to lead to the design of protein crystal growth techniques that result in larger and more perfect crystals.

Progress During FY 1995:
The design of the simultaneous multiangle light scattering unit has been completed and acquisition of the main components has begun.
In preparation for detailed impurity analyses and purification work, a systematic comparison and quantification of the sensitivity of staining techniques of SDS PAGE gels was conducted. An enhanced silver staining technique, with a sensitivity of 0.1 ng of protein, was shown to be ten times more sensitive than normal silver stain and 100-1000 fold more than coomassie blue.

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Presentations


Rosenberger, F. "Protein crystallization." Instituto de Fisica, Universidad Nacional Autonoma de Mexico, Mexico City, September 1995.

II. MSAD Program Tasks — Ground-based

Discipline: Biotechnology

Enhancement of Cell Function in Culture by Controlled Aggregation Under Microgravity Conditions

Principal Investigator: Prof. W. M. Saltzman
Johns Hopkins University

Co-Investigators:
Leong, Prof. K.
Johns Hopkins University

Task Objective:

Cell-cell contact within tissues is fundamental to the regulation of cell differentiation and function. Cell aggregates, formed in vitro and maintained in culture, have been shown to maintain many of the functions of the native tissue. The main objectives of this program are: i) development of methods for controlling cell aggregation using bioactive polymers and polymer microspheres and ii) systematic study of the function and behavior of suitably aggregated cells in culture. The discovery of new methods for improving cell growth and function in culture is critical to the development of hybrid artificial internal organs and mammalian cell bioreactors.

Task Description:

To achieve these goals, we have formulated the following specific objectives for a three-year period of study:

Objective 1: Synthesis of water soluble polymers for controlling cell aggregation. We will synthesize water soluble polymers with bioactive groups that are specifically recognized by certain cells and use these multifunctional polymers as molecular nuclei to initiate and control cell aggregation. We will use these polymers to control aggregation by adding them to gently agitated or quiescent suspensions of single cells; this technique will produce small cellular aggregates (<10 cells, diameter<100 mm). By altering the properties of the polymers and the conditions of aggregation, we will identify approaches for obtaining cell aggregates of different size, polydispersity, and morphology. We have demonstrated the feasibility of this approach using N-acetyl glucosamine (specific for chicken hepatocytes) attached to vinyl polymers.

Objective 2: Fabrication of polymer microspheres for carrier-assisted cell aggregation. We will fabricate microspheres containing encapsulated, soluble mediators of cell growth and function. We will use the microspheres to create larger aggregates with a central polymer core. We have also demonstrated the feasibility of this approach using microspheres composed of vinyl polymers and cultured hepatocytes.

Objective 3: Development of methods for culturing cell aggregates under unit gravity. In preliminary studies, we have encapsulated hepatocytes and hepatocyte aggregates within gels of type I collagen, cultured these encapsulated cells, and examined subsequent cell growth, function, and viability. We will test gels of collagen under different hydration conditions to find an optimal experimental system for maintaining cells in an aggregated and suspended state in the laboratory. The gels will be used to suspend aggregates created with water-soluble polymers and microspheres. To develop model culture systems representing both liver and neuronal tissues, we will use primary cultures of hepatocytes, primary cultures and fetal brain cells, neuroblastoma cell lines, and the PC12 cell line.

Objective 4: Measurement of cell function under different aggregation conditions in gel culture. We have already developed methods of monitoring cell growth and metabolism in culture by measuring cellular protein, DNA, and lactate dehydrogenase (LDH) content. We have also developed methods for monitoring cell function in culture by following albumin and uric acid secretion (for hepatocytes) and expression of specific enzymes and responsiveness to nerve growth factor (for PC12 cells). Using the optimal experimental system, defined in specific objective 3, we will systematically examine the function of cell aggregates in culture. Aggregates will be cultured under a variety of conditions including i) conventional static culture, ii) static culture with aggregates suspended within gels of extracellular matrix molecules, and iii) microgravity culture of aggregates within the NASA rotating-wall bioreactors.
II. MSAD Program Tasks — Ground-based

Discipline: Biotechnology

Objective 5: Preparation of experiments for evaluation under microgravity conditions. In specific objectives 1 through 4, we will have identified the important variables for controlling cell aggregation and function in aggregate culture. In the final stages of this project, we will design methods for developing physiologically realistic cell aggregates under microgravity conditions and for testing the influence of aggregation on cell function in suspension culture under microgravity in space.

Task Significance:

These studies are uniquely suited for study in microgravity. First, cell aggregation in zero gravity will be driven by migration and diffusion rather than by forced collisions as is necessary on earth. The resulting aggregates may be closer to those found in tissues, since tissues are formed by migration and selective adhesion. Second, gentle suspension culture techniques can be used to culture the aggregates in microgravity. By the end of the period of laboratory study proposed here, we will have developed cell aggregation techniques appropriate for testing under microgravity conditions in space.

Progress During FY 1995:

Objective 1: We have synthesized several water-soluble molecules as molecular nuclei for cell adhesion. These molecules are based on poly(ethylene glycol) with bioactive peptides, NH$_2$-Gly-Arg-Gly-Asp (GRGD) and NH$_2$-Gly-Tyr-Ile-Gly-Ser-Arg (GYIGSR), grafted to the termini. We have developed covalent coupling methods for attaching these peptides to PEG with a variety of molecular weights (5,000 to 30,000) and to polystyrene microspheres (0.1 to 10 mm). We have used quantitative cell adhesion assays to determine the best peptides for conjugation. We have examined the kinetics of cell aggregation of a number of important cell types (including neural cells and cell lines, fibroblasts, and genetically engineered fibroblasts). We are developing methods for examining cell function for aggregates maintained in culture. The first report in this work was published this year (Dai, Belt, and Saltzman, 1994) and other publications are in progress.

Objective 2: We have synthesized polystyrene polymers modified with carbohydrates and peptides at the surface. A manuscript describing this work was published this year (Gutsche, et al., 1994). These unique polystyrene-based polymers can be formed as microspheres, and cells attach and grow avidly to these novel microcarriers. We have developed similar methods for coupling peptides to polystyrene supports with a range of sizes.

Objectives 3 and 4: We have developed methods for culturing and forming aggregates under unit gravity. A manuscript that was recently accepted for publication is appended. (Krewson, C.E., Chung S.W., Dai, W., and Saltzman, W.M. Biotechnology and Bioengineering 43:555-562 (1994). We have adapted these methods for producing aggregates within the NASA rotating-wall vessels.

Objective 5: Only applicable to the third project year.

STUDENTS FUNDED UNDER RESEARCH: 

TASK INITIATION: 11/92  EXPIRATION: 11/95
PROJECT IDENTIFICATION: 962-23-01-10
NASA CONTRACT NO.: NAG-654
RESPONSIBLE CENTER: JSC

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals


Printed: 28 FEB 1996

II-270
II. MSAD Program Tasks — Ground-based


Presentations
Culture of Porcine Islet Tissue: Evaluation of Microgravity Conditions

Principal Investigator: Dr. David W. Scharp
Washington University School of Medicine

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
Porcine islet tissue is receiving new attention as an attractive, potential tissue for application in clinical islet transplantation in patients with Type 1, Insulin-Dependent Diabetes Mellitus (IDDM). Recent clinical success of islet transplantation into patients with renal grafts using human islets and standard immunosuppression demonstrated that greater than one year islet graft function can be achieved off insulin therapy. As additional patients are being transplanted to establish how many patients can achieve insulin independence and for what duration, investigators are anticipating that the 4,000 human organ donors a year will be insufficient for the numbers of potential transplants that can be achieved. Thus, adult porcine islet isolations are being developed. We have developed an adult porcine islet procedure but realize that the use of neonatal porcine islet tissue is more suited for clinical application from a cost production viewpoint as well as from a safety viewpoint, since the neonatal porcine islet tissue could be obtained from gnotobiotic donors. While this seems ideal, there has not been any reliable way to isolate neonatal islet tissue in any quantity nor any method to culture these islet cells. We have recently developed a markedly improved method for the isolation and purification of neonatal porcine islet tissue, but, have no reliable method to culture islet cells that can not only produce new islets, called pseudoislets, but also take advantage of their inherent growth and maturation potential prior to consideration of transplantation.

Learning of the microgravitational culture system with low shear rate and the proven importance in producing other tissue types from single cells developed by Dr. Glenn Spaulding (NASA JSC), he and I have established a new collaboration that would combine our islet tissue.

Task Description:
To accomplish these three objectives, we propose the following specific aims for the investigations:

1. To culture neonatal islet tissue by rotational, microgravitational and static methods to determine optimal ways of formation and preservation of functional pseudoislets.

2. To examine the ability of cultured neonatal porcine islet tissue to develop and maintain differentiated islet functional characteristics.

3. To determine the ability of optimally cultured porcine islet tissue to be successfully transplanted into diabetic recipients.

4. To examine the replication potential of neonatal porcine islet tissue through culture manipulations. These proposed studies combine the islet expertise with a method of rotational islet culture that has successfully formed neonatal porcine pseudoislets with the microgravitational expertise with a specific low shear culture system that seems ideally suited to the fragile neonatal islet cells. Successful completion of these proposed studies should provide important results that will have considerable application in the islet field as well as in the field of microgravitational studies important to future NASA objectives.

Task Significance:
The results of these studies will provide new information to three areas. The results will be important to: 1) a better understanding of the development of neonatal islet tissue, 2) NASA considerations of islet tissue as a potential type of tissue for their microgravitational studies, and 3) islet transplantation for developing an effective culture system for this promising new source of islet tissue.
Progress During FY 1995:

This project continues to utilize islet tissue isolated from 14 to 29 day neonatal pigs which have been surgically altered at three days of age and given feed enriched with potential growth factors to increase the beta cell production and maturation for the 10-day to two week period prior to harvest. So far this year, we have performed twelve separate experiments, each closely monitored at different stages by sampling for DNA extraction, insulin production, glucose utilization rates, insulin extraction, and histology evaluation.

Results show a two- to seven-fold increase in the number of aggregates at two weeks of culture. Although glucose utilization is fairly constant, there seems to be a maximum output of insulin at one week of culture. Final viability testing shows functional islet tissue and a 95% viability by fluorescein diacetate staining. Recent studies have been focused on varying the culture medium used, as well as varying the tissue and bead ratios. One of our latest developments has to do with increasing the ability to process more tissue at a time, therefore cutting down on the variabilities and increasing the yield per grams of tissue, allowing us to culture more aggregates from each experiment.

Although in vitro testing has been a successful tool in monitoring the progress of our tissue survival and growth, we have just recently begun to study the potential of this project with in vivo growth of our neonatal tissue. We are using the SCID (sever-combined immune deficient) mouse as a recipient model, in order to avoid any rejection problems. The results of these experiments are too recent to draw conclusions but our expectations for this series are high.

STUDENTS FUNDED UNDER RESEARCH:
TASK INITIATION:  1/93  EXPIRATION:  1/96
PROJECT IDENTIFICATION:  962-23-01-08
NASA CONTRACT NO.:  NAG-653
RESPONSIBLE CENTER:  JSC
Robotic Acquisition and Cryogenic Preservation of Single Crystals of Macromolecules for X-Ray Diffraction

Principal Investigator: Craig D. Smith, Ph.D. University of Alabama, Birmingham

Co-Investigators:
DeLucas, L. University of Alabama, Birmingham (CMC)

Task Objective:
Experiments are aimed at the development of new or better methods of preserving protein crystals at ultra-low temperatures. Cryogenically preserved crystals can be used for immediate enhanced x-ray crystallographic data collection or stored indefinitely until data collection facilities become available.

Task Description:
The initial focus is on cryofluid/protein crystal interactions, thermal processes which affect the success of freezing protein crystals, and the development of rapid physical handling methods of these small fragile crystals to enhance success rates of cryofreezing. Understanding the freezing process on a molecular scale may provide more guidance to the current empirically derived methods.

Task Significance:
Protein crystals used for x-ray crystallographic experiments usually are very fragile and short-lived. Data from these crystals produce three-dimensional structures of proteins important to many biochemical processes. Freezing protein crystals can greatly enhance the structural information obtained by crystallography by reducing their fragility and dramatically increasing their lifetimes. Experimental data also show that a clearer picture of the protein structure can result when the crystal is frozen.

Progress During FY 1995:
This grant has been funded for only a few weeks and experiments are currently in progress.

STUDENTS FUNDED UNDER RESEARCH: TASK INITIATION: 10/95 EXPIRATION: 10/96
PROJECT IDENTIFICATION: 962-23-08-47 NASA CONTRACT NO.: NAG8-1193 RESPONSIBLE CENTER: MSFC

Printed: 28 FEB 1996
Influence of Microgravity Conditions on Gene Transfer Into Expanded Populations of Human Hematopoietic Stem Cells

Principal Investigator: Dr. F. M. Stewart

Co-Investigators:
Stein, Dr. J.
Lawrence, Dr. J.

University of Massachusetts Medical Center

Task Objective:
We plan to evaluate the ability of the HARV (high aspect rotating-wall vessel) three-dimensional culture system to optimize engraftment conditions of human hematopoietic cells into SCID-NOD mice by evaluating: a) its effects on purified stem cells and the development of stromal cells, b) cytokine stimulation in this system and expansion of stem cells, c) repetitive transplant, and d) cell cycle manipulation of stem cells. These conditions set the stage for subsequent gene transfer studies with neomycin-resistance gene NEO\(^{R}\) transfection into primitive lymphohematopoietic stem cells.

Task Description:
We propose to use the HARV three-dimensional culture system and microgravity concepts to evaluate marrow cells from normal donors transduced with NEO\(^{R}\), assessing long-term gene transduction frequency and gene expression, the effect of 5-FU pre-treatment (a method of cell cycling) on transduction frequencies in this system, and engraftment (with minimal or no myeloablation) into SCID-NOD animals. The results of these studies may show that the microgravity culture approaches may improve gene transfer, stem cell expansion and engraftment into SCID-NOD animals. From these studies and others beyond the scope of this proposal, the potential benefits (and risks) of transferring other genes such as MDR1 into normal cells (or into cancer cells) may be assessed. Ultimately, as part of the future clinical gene therapy protocol, we plan to evaluate MDR1 gene transfer into normal marrow from patients with ovarian carcinoma in an effort to escalate doses with p 170 pump drugs such as Taxol, thereby potentially increasing cure rates, while minimizing toxicity.

Task Significance:
Hematopoietic stem cells, because of their ability to repopulate bone marrow of myeloablated animals, have been an attractive vehicle for various gene therapy approaches. Work in different animal species and humans indicates an ability to transfer genes to hematopoietic stem cells using a variety of approaches including retroviral vectors. Genetically transduced hematopoietic stem cells have been transplanted into animals and humans, and the expression and long-term persistence of the genes demonstrated in vivo. Unfortunately, the level of expression and percent cells carrying the transferred gene have been disappointingly low in expression of the transferred gene, especially with progressive cell differentiation into different lineages. Alternate possibilities include a failure to transfer the gene of interest into long-term hematopoietic repopulating cells and/or a failure of these cells to engraft. Current culture systems are limited in supporting long-term preservation and expansion of stem cells. Since co-culture with gene-carrying viral supernatants appears at present to be the most effective method of gene insertion in human cells, optimizing culture conditions may be important for maximizing the efficiency of gene transfer into hematopoietic stem cells.

Progress During FY 1995:
We plan to evaluate the ability of the STLV three-dimensional culture system to optimize engraftment conditions of human hematopoietic stem cells into SCID-NOD mice by evaluating a) its effects on purified stem cells and the development of stromal cells; b) cytokine stimulation in this system and expansion of stem cells; c) repetitive transplant; d) cell cycle manipulation of stem cells. These conditions set the stage for subsequent gene transfer studies with the neomycin-resistance gene (NEO\(^{R}\)) and other genes.
Since our grant funding period commenced in September, 1995, we have initiated the process of titering amphotropic MDR1-vector supernants with the assistance of Ingenex. We have also obtained from Genetics Therapy, Inc., the appropriate vectors carrying the neomycin-resistance gene (LNL-6 and G1NA). We have purchased a total of three STLV bioreactors. Due to a backlog of ordering, we were only able to obtain one bioreactor but expect to receive two bioreactors in January '96. We have hired a technician to assist with the culture studies and SCID-NOD mouse experiments. We visited the Johnson Space Center in December, 1995. During our visit, we were able to accomplish the technical aspects of the STLV and HARV bioreactors.

We have also procured a number of human cord blood specimens that have been cryopreserved and will be used in additional future experiments. We sent 10 cord blood samples to NASA/JSC for human stem cell work as part of our continued collaborative interaction with scientists at JSC/Houston.

**STUDENTS FUNDED UNDER RESEARCH:**

**TASK INITIATION:** 9/95  **EXPIRATION:** 9/99

**PROJECT IDENTIFICATION:** 962-23-01-27  
**NASA CONTRACT NO.:** NAG 9-820  
**RESPONSIBLE CENTER:** JSC
II. MSAD Program Tasks — Ground-based

Mechanisms for Membrane Protein Crystallization: Analysis by Small Angle Neutron Scattering

Principal Investigator: Dr. David M. Tiede
Co-Investigators: No Co-I's Assigned to this Task

Task Objective:
The goal of this project is to characterize structural intermediates in pathways for membrane protein crystallization. This project will use small angle neutron scattering, SANS, to characterize detergent and protein structures that exist in solution as a function of initial crystallization conditions, and this project will examine how these structures change as a function of time and trajectories that move the crystallization mixtures into metastable, crystallization phases.

Task Description:
This project will use the bacterial photosynthetic reaction center as a model for examining mechanisms for membrane protein crystallization, and for investigating the influence of the detergent on crystallization. Membrane protein crystallization differs from other protein by requiring the presence of a solubilizing detergent. The reaction center has been successfully crystallized in two types of detergent, zwitterionic lauryldimethylamine-N-oxide, LDAO, and non-ionic n-octyl-B-D-glucoside, OG. Conditions required for reaction center crystallization change depending upon the detergent used for solubilization of the reaction center. This demonstrates that the solubilities of both detergent and protein must be manipulated in order to successfully crystallize membrane proteins.

Protein crystallization is typically described in terms of three discrete phases: a solution phase where the detergent-solubilized protein complexes are soluble, a metastable crystallization phase where crystal nucleation and growth occur in the presence of solubilized protein, and finally a precipitation phase in which there is no solubilized protein in solution.

This project will use SANS to characterize how detergent and protein structures vary as a function of position in this phase diagram. Neutron scattering is sensitive to isotopic substitution. This project will use isotopic labeling to selectively extract neutron scattering contributions from the detergent and the reaction center structures in crystallization mixtures. These structures will be time-resolved during the time-course of crystallization.

Task Significance:
Protein crystallization is frequently a rate-limiting step in the analysis and understanding of the molecular basis for disease and drug therapy. This is particularly true for membrane proteins that are responsible for many major biological processes such as vision, nerve conduction, photosynthesis and respiration. So far there has been relatively little success in the crystallization of membrane proteins compared to water-soluble proteins. This project will provide unique, first-of-the-kind data on structural intermediates in pathways leading to the crystallization of the bacterial photosynthetic reaction center, which is one of the few membrane proteins that has been successfully crystallized. This information is necessary for the development of more general, rational strategies for enhancing the efficiency of membrane protein crystallization.

Progress During FY 1995:
Funding was just received at the end of fiscal year 1995. No progress to date.
II. MSAD Program Tasks — Ground-based

Discipline: Biotechnology

STUDENTS FUNDED UNDER RESEARCH: TASK INITIATION: 10/95 EXPIRATION: 9/99
PROJECT IDENTIFICATION: 962-23-08-39
RESPONSIBLE CENTER: MSFC
Preparation and Analysis of RNA Crystals

Principal Investigator: Dr. Paul Todd
University of Colorado, Boulder

Co-Investigators:
Kundrot, C.E. University of Colorado
Schultz, S.C. University of Colorado

Task Objective:
Grow crystals of specific RNA's and nucleic-acid peptide complexes that diffract x-rays to high resolution.

Identify pathways that lead to highest-quality crystals of RNA's and complexes.

Detail the mechanism of the role of inertial acceleration (gravity) in high-resolution RNA crystal growth.

Task Description:
1. Preparation of high-quality crystals and their complexes.

2. Pathway to high-quality RNA crystals.

3. Mechanism of action of inertial acceleration (gravity) on crystal growth.

Task Significance:
There is a rising interest in the use of specially-tailored RNA sequences as pharmaceutical and diagnostic chemicals. These sequences must have a three-dimensional structure that allows them to interact with target molecules, usually proteins. One example is a 28-base RNA sequence that inhibits the reverse transcriptase of HIV-1; this target molecule is required for the replication of the AIDS virus in human cells. Another is a longer RNA that behaves like an enzyme and enzymatically cleaves virus RNA in cells. To assess the ability of these RNA's to interact with their targets and to possess chemical stability, their three-dimensional structure needs to be determined by x-ray crystallography, which requires high-quality crystals at least 0.5 mm long. However, there is very little experience, worldwide, in crystallizing RNA's. Therefore the goals of this project are to provide high-quality RNA crystals and a paradigm for their reliable preparation.

Progress During FY 1995:
The following progress has been made in addressing the tasks listed above:

1. Preparation of high-quality crystals of RNA's and their complexes: Oligonucleotide "UU-dodecamer" and nucleic acid-protein complex crystals were grown in Fluid Processing Apparatus (FPA) and Materials Dispersion Apparatus (MDA) on Shuttle mission STS-69. At the time of this writing additional samples are awaiting the launches of Shuttle mission STS-73 and METEOR commercial experiment carrier (unmanned orbiter). The methods of double diffusion, step diffusion, and osmotic dewatering were employed on all three flights, and crystals were formed by these methods on STS-69, including one diffraction-quality crystal of the UU-dodecamer and a few diffraction-quality crystals of nucleic acid-protein complexes.

2. Pathway to high-quality RNA crystals: An osmotic crystallizer with 24 reaction chambers has been built and tested. A successful transport model has been applied to the control of solute concentration rate. A transport rate measuring device has been built and tested. A 12-base RNA "UU-dodecamer" has been synthesized by in vitro transcription; its phase diagram in methylpentane diol and ammonium acetate at 37° C has been determined, and crystals of this material have been grown by osmotic dewatering in the laboratory.
3. Mechanisms of action of inertial acceleration (gravity) on crystal growth: Crystal/nucleus sedimentations as a factor has been studied by growing crystals with the above crystallizer upside-down in the laboratory. Under this condition, crystallization seldom proceeds beyond the nucleation stage, and the concentration of solutes at the "top" of the chamber leads to a high frequency of continuous nucleation. Low-gravity experiments have been performed as mentioned above.

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**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Proceedings**

**Presentations**

Development of Microflow Biochemical Sensors for Space Biotechnology

Principal Investigator: Dr. Bruce Towe
Arizona State University

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
The objective of this work is to develop biochemical sensors and associated hardware for space bioreactor applications. Two different methods are being explored consisting of flow injection analyzers (FIA) of small format and low reagent volume demands, and micro-flow type fluorescent biochemical sensors. Each of these two types of sensors are being implemented in hardware that have configurations which are specific to the environment and construction of the space shuttle bioreactor. Together, the two systems offer the opportunity to measure the concentration of basic culture nutrients of glucose and oxygen as well as the potential to measure other biochemicals of future interest such as galactose, urea, creatinine, an other cellular products released into the cell culture medium.

Task Description:
A microflow colorimetric sensor for glucose is being constructed based on the use of a predissolved single reagent system. The Sigma, Inc., (St. Louis, Mo.) 'Trinder test"employs peroxidase and glucose oxidase enzymes with another dissolved reagent to produce, in the presence of glucose, stoichiometric amounts of a colored dye that is measured photometrically.

The basic glucose sensing strategy is to pump submicroliter quantities of Trinder reagent and dialyzed sample of filtered bioreactor media into a common mixing plenum and photometrically recording color changes. The project requires the development of a fluid handling system that is resistant to bubble formation and other problems brought on by the forces of liftoff and zero gravity operation.

The fluorescent oxygen sensor under development uses an oxygen sensitive dye trapped in a micro-flow recirculating loop using a length of thin silicone tubing that is immersed in the bioreactor solution or media flow loop. The portion of the sample exiting from the bioreactor media is subjected to a fluorescence test by an in-line optical cell. The dye is then recirculated back to a small, several milliliter, holding reservoir for reuse.

The important problems and issues under research concern the best way to implement the biochemical sensor's flow system so that it can self-check and recalibrate itself periodically. Another important issue is determining methods to minimize the quantity of reagents that are used over two week to one month missions in order to make the sensor small and compact. Research is being conducted to find ways to improve the shelf life of reagents so that over one month sensor operational lifetimes can be achieved.

The project consists of developing the principles and manufacturing approaches to microflow injection analysis for application to space bioreactors. Specifically we plan to:

1. Test microdialysis methods of sample withdrawal from the bioreactors as a way of avoiding clogging and bubble entrainment.

2. Develop more optimal techniques for the improved storage of the Trinder reagents without refrigeration. We are specifically investigating the augmentation of the Trinder with additional quantities of enzyme (glucose oxidase, peroxidase) prior to charging the sensor storage reservoirs in order to help offset losses of their activity with time.

3. Identify ways to fabricate the sensors in a reliable and maintenance-free configuration.
II. MSAD Program Tasks — Ground-based

4. Develop a testing protocol that conforms to the operating conditions the sensor will experience in space bioreactors. This will allow final shakedown and identification of possible problems with final sensor integration into the configuration used for flight.

Task Significance:

Maintenance of cell culture environments in terms of glucose and oxygen tension is of particular importance to the successful growth of mammalian cells. The range of permissible biochemical variation in culture media, however, is very narrow and specific to a given cell type. For this reason, a critical requirement of successful space bioreactor operation is the measurement of the concentration of biochemicals in cell growth media. This information would then be used by a control system to maintain cell culture chemical balances. Unfortunately, the rigorous demands placed on biochemical sensors by space biotechnology reactors precludes the use of commercially available biochemical sensors that meet the size, weight and bulk requirements of flying in a space shuttle locker. As a result, a good argument could be made that lack of suitable biosensors is one of the primary factors presently limiting the success of biotechnology reactors in space.

Progress During FY 1995:

We have found that significant improvements in the useful lifetime of the sensors, up to 30 days, can be achieved by application of anti-bacterial techniques in handling of the integral reagent supplies. We are presently testing versions of the sensor hardware that have a potentially improved reliability through the replacement of micropumps with fluorocarbon pressurized reagent-delivery reservoirs.

STUDENTS FUNDED UNDER RESEARCH: TASK INITIATION: 9/95 EXPIRATION: 8/99

PROJECT IDENTIFICATION: 962-23-01-33

NASA CONTRACT NO.: NAG9-815

RESPONSIBLE CENTER: JSC
Experimental Studies of Protein Crystal Growth Under Simulated Low Gravity Conditions

Principal Investigator: Dr. Eugene H. Trinh  
Jet Propulsion Laboratory (JPL)

Co-Investigators:
DeLucas, Dr. L.J.  
University of Alabama, Birmingham
Rhim, Dr. W.K.  
Jet Propulsion Laboratory (JPL)

Task Objective:
The goal of this ground-based experimental research task is to better establish the role of convective flows in the determination of the characteristics of crystals grown from protein solutions. The primary objectives are first to inhibit natural convective flows around isolated crystals immersed in single levitated solution droplets, and then to either allow natural convection to occur or to carefully increase the magnitude of an artificially-induced convective flow. The secondary objective is to develop a new experimental approach for the detailed characterization of the crystal environment while providing simulated low gravity conditions in 1-g by using freely levitated solution droplets rotating along a horizontal axis.

Task Description:
A specific objective is to obtain a quantitative measure of the effects of varying the flow velocity around a crystal by at least one order of magnitude while maintaining all the other relevant parameters constant. The specific tasks are to measure the face growth rate and to monitor the depletion layer around a growing crystal while inhibiting convection. We will first attempt to visualize it in the simulated low-gravity condition, and then perturb it by the induction of controlled flow fields.

Task Significance:
The influence of gravitational effects on the evolution of living and inorganic crystalline materials has been recently underscored by investigations performed in microgravity. In particular, strong circumstantial evidence has been gathered pointing to the marked improvement in the characteristics of protein crystals grown in low Earth-orbit. The importance of the understanding of the structure of the assembly of protein molecules for application to the biotechnology and pharmaceutical fields makes the identification of the specific gravity-related processes of particular significance. Understanding and perhaps eventually controlling the right parameters on Earth will significantly impact our ability to improve our methods to grow proteins to extract more valuable information to be used for the manufacture of drugs and to better understand biochemical processes affecting human health.

Progress During FY 1995:
No significant activity has taken place in FY95 except for the assembly of the laboratory set-up. Funding will be officially in place at JPL at the beginning of November 1995.

STUDENTS FUNDED UNDER RESEARCH:  
TASK INITIATION: 11/95  
EXPIRATION: 9/99  
PROJECT IDENTIFICATION: 962-23-08-40  
RESPONSIBLE CENTER: MSFC
II. MSAD Program Tasks — Ground-based

Task Objective:
The primary objective of our program is to develop extensive insight into the biophysical chemistry of protein crystallization, with simultaneous emphasis on developing new approaches for catalyzing three-dimensional protein crystal growth. Genetic engineering techniques and powerful in situ optical imaging and spectroscopy methodologies will be used to dissect the biomolecular structure-function relationships governing crystallization. The site-directed mutagenesis of streptavidin will allow the mapping of specific protein side-chains to specific physico-chemical roles in crystallization. These physico-chemical roles will be experimentally elucidated using an array of cutting-edge surface analytical techniques.

Task Description:
Our first two tasks are (1) to study the effects of reduced biotin-binding affinity on the two-dimensional crystallization process by the use of site-directed streptavidin mutants, and (2) to characterize how site-directed side-chain alterations of the lateral protein-protein contact zones affect two-dimensional crystallization (i.e., crystal growth, size, and morphology).

Modern in situ surface analytical techniques will be employed and further developed to probe (a) the surface binding of the model protein, (b) the two-dimensional crystallization processes, and (c) the growth rates. The surface binding, as well as the size and shapes of resulting crystals, will be characterized as a function of the binding affinity to the surface anchored ligands. The crystallization of streptavidin underneath biotinylated surfaces will serve as our first model system.

Task Significance:
Despite the importance of protein crystallization to many fields in the biological sciences, the biotechnology field, and the pharmaceutical industry, there remains a striking paucity of fundamental principles describing protein crystal nucleation and growth. Our program is aimed at filling this gap, by providing molecular insight into the biomolecular structure-function relationships governing protein crystallization in the model streptavidin two-dimensional system that is amenable to in situ physico-chemical characterization. Site-directed mutagenesis experiments allow us to alter selected protein side-chains. We will probe the resulting changes in physical parameters of the crystallization process as a function of these changes using a combination of surface analytical techniques.

In situ monitoring of crystallization processes is an important need for microgravity environment research, as very little information is available for how the dynamics compare to ground-based growth. Thus, although our first generation of proposed experiments are designed for ground-based research, the optical and mass sensitive techniques to be utilized in these experiments should be readily adapted to future in-space applications.

Progress During FY 1995:
Our program got started officially only on September 1, 1995. In preliminary experiments we could already demonstrate that the protein surface density can be quantified by Brewster angle microscopy and that the difference in optical thickness between the noncrystalline and the crystalline layer is sufficient to provide for contrast rich images. We determined a critical surface density for commercial streptavidin that is required to induce the...
two-dimensional crystallization of streptavidin. Several mutants have been produced to alter (a) the binding affinity of streptavidin to biotin, and (b) the protein-protein contacts. These mutants are currently under investigation with respect to their crystallization behavior.

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Automation of Protein Crystallization Experiments: Crystallization by Dynamic Control of Temperature

Principal Investigator: Dr. Keith B. Ward

Co-Investigators:
Zuk, Dr. W.M.
Perozzo, M.A.

Task Objective:

The goal of this research program is to develop a dynamically-controlled crystallization system (DCCS) in which protein supersaturation is controlled by varying the temperature while crystallization is monitored by optical means. This device will also be capable of being controlled telerobotically. The program intends to extend its accomplishments in this area by continuing to enhance the DCCS, expanding the system to include multiple crystallization chambers and incorporating more efficient and versatile systems for monitoring the progress of nucleation and crystallization. A final goal of this project is to ascertain to what extent the technique of temperature-controlled crystallization is applicable for protein crystallization.

Task Description:

A study of a representative sample of well-characterized proteins that have been successfully crystallized using other methods is proposed. The temperature coefficient of solubility will be measured using the DCCS, and attempts to prepare crystals in this apparatus will allow us to judge the general usefulness of this approach.

The proposed methods of research include further modifying the current design to incorporate video monitoring to provide visual observation of growth volumes, to introduce dynamic light scattering, and to expand the system to include multiple crystallization cells, each with separate temperature controls. Telerobotic control experiments will continue using enhanced control software, and the results of the experiments will be aimed at defining the capabilities and limitations of remotely-controlled crystallization protocols on space platforms in microgravity. Collection of protein temperature solubility data will be enhanced by the development of more fully automated software algorithms. The temperature of a sample is slowly changed step-wise until the level of scintillation signal indicates that the crystallization phase boundary has been crossed. The temperature will then be recycled using a finer step size until the solubility temperature of the sample is determined to within 0.1°C. Although currently some of this process is conducted manually, further development of the control software will completely automate the process.

Task Significance:

This research is important in continuing the development of dynamically-controlled crystallization systems, proving the usefulness of dynamic control in conducting protein crystallization experiments in microgravity. This work will also aid the current effort of other NASA-funded Principle Investigators in designing advanced crystallization apparatuses. This system will also be used, while it is being developed, to explore whether temperature control of supersaturation is a technique that can have wide applicability in laboratory-based protein crystallization.

Progress During FY 1995:

The miniaturized dynamically controlled temperature device has been used to collect preliminary solubility data on hen egg-white lysozyme, a-chymotrypsin, ferritin, ribonuclease A, and catalase. Some success was achieved in crystallizing lysozyme and ferritin through temperature control. The control software which operates the crystallization device is being redesigned to permit simultaneous multiple experiments and to enhance the analysis of intensity data. We have also initiated the integration of dynamic light scattering equipment into the apparatus to study the effects of temperature on protein aggregation.
II. MSAD Program Tasks — Ground-based

STUDENTS FUNDED UNDER RESEARCH: TASK INITIATION: 1/93 EXPIRATION: 1/96
PROJECT IDENTIFICATION: 962-23-08-24
NASA CONTRACT NO.: H-07975D
RESPONSIBLE CENTER: MSFC

BIBLIOGRAPHIC CitATIONS FOR FY 1995:

Presentations
Thermal Optimization of Growth and Quality of Protein Crystals

Principal Investigator: Dr. John M. Wiencek
University of Iowa

Co-Investigators:
Arnold, E.
Rutgers University

Task Objective:
The overall goal of this project is to control supersaturation at constant values during protein crystal growth by varying the temperature in a predetermined (by simple theory) manner. Applying the theory requires knowledge about specific physicochemical properties of the protein solution including the effect of supersaturation on growth rates and the effect of temperature on protein solubility. Our specific goals are:

- Application of a temperature control strategy which maintains constant supersaturation to the growth of lysozyme crystals and comparison to traditional isothermal strategies.
- Investigation of batch isothermal calorimetry as a tool to determine lysozyme solubility as a function of temperature by employing the Van't Hoff equation.
- Construction of a video microscopy apparatus for determination of crystal growth rates and terminal size.

Work for the coming year will focus on the measurement of solubility and crystal growth rate for human serum albumin as well as the assessment of the crystals grown by the developed strategies via x-ray diffraction.

Task Description:
Three protein systems have been chosen for study: lysozyme, human serum albumin (HSA), and HIV reverse transcriptase (RT). Each of these proteins have unique features that make them interesting. Lysozyme and HSA represent fairly inexpensive and readily available proteins and will be model systems for investigations elucidating the effects of growth rates on crystal quality. Once strategies that are optimal are available, such strategies will be applied to the RT system as a realistic test case.

Experimental evidence suggests that larger and higher quality crystals can be attained in the microgravity environment of space. Fundamental studies have attempted to measure and model the effects of gravity-induced convection and sedimentation on the crystal growth process. However, the effect of growth rate on protein crystal quality is not well documented. If the growth rate is controlled, how much time is required to allow for interfacial attachment of the large protein molecule? What is the impact of this "attachment time" on crystal quality? This research effort is directed at measuring the effects of crystal growth rate on the ability of crystals to diffract x-rays.

We hope to link crystal quality to slow growth rates and discern "how slow is slow enough." In addition, processing strategies will be developed which x-ray crystallographers can use to grow larger, high-quality crystals. The investigation requires the measurement of protein (lysozyme, HSA and RT) solubility at two or more different temperatures (typically 4°C and 25°C) and the growth rate at two or more different supersaturations.

Microcalorimetry is a potentially powerful technique to measure these and other (e.g., nucleation) physical parameters of the protein systems.

Task Significance:
Development of a systematic method of protein crystallization may lead to crystallization of previously uncrystallizable proteins and add to current knowledge of protein structure/function relationships. Knowledge of detailed protein structure is essential for rational design of therapies and small molecule pharmaceuticals.
Progress During FY 1995:

Work is progressing on the use of predictive temperature control algorithms to maximize crystal size and quality.

Our previous efforts have shown that the predictive temperature profiles for lysozyme can lead to fewer, larger crystals with notable improvement in crystal quality. Current efforts have focused on the extension to other protein systems. The physico-chemical parameters required to implement such temperature profiles include the heat of crystallization, growth rate functionality in terms of supersaturation, and the location of the labile zone. We are deriving theoretical as well as practical methodologies based on microcalorimetric measurements to obtain such information. The results to date with lysozyme have proven our theoretical expressions correct provided care is taken when determining terminal crystal sizes. Using these parameters, we are concurrently growing crystals of Equine Serum Albumin via our temperature control algorithm. Work for the next year will continue along these lines.

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BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals


Presentations

Wiencek, J.M. "Production of protein crystals suitable for x-ray diffraction analysis: controlling growth rates via temperature manipulation." Fall Colloquia, Chemistry and Chemical Engineering, Polytechnic University, Brooklyn, New York, October 1994.
A Rational Approach for Predicting Protein Crystallization

Principal Investigator: Dr. W. W. Wilson

Mississippi State University

Co-Investigators:

No Co-I's Assigned to this Task

Task Objective:

The primary objective is to begin light scattering measurements on the protein α-chymotrypsin to obtain second virial coefficient values in a variety of solvent conditions. The experimental data obtained will later be compared to theoretical results.

Task Description:

Static laser scattering (SLS) will be used to determine osmotic second virial coefficients, $B_{22}$, for the protein in solvents which vary in ionic strength, pH and temperature.

Task Significance:

The proposed work will determine the feasibility of theoretically predicting the solution conditions that are favorable for protein crystallization.

Progress During FY 1995:

A graduate student has been identified to begin the task, and the materials for Phase I of the work have been procured.

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TASK INITIATION: 8/95  EXPIRATION: 8/99

PROJECT IDENTIFICATION: 962-23-08-34

RESPONSIBLE CENTER: MSFC
Search for a Dilute Solution Property to Predict Protein Crystallization

Principal Investigator: Dr. W. W. Wilson Mississippi State University

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
The primary objective of the research is to discover a unique dilute solution parameter that universally and unambiguously predicts protein crystallization.

Since most crystallographers will not have access to sophisticated laser scattering instrumentation, a secondary objective of this research is to construct a simple laser scattering device that determines the universal predictor values. The device will be miniaturized to work with sub-milliliter volumes and incorporate the latest optical fiber technology for beam delivery and signal detection.

Task Description:
Static, dynamic and electrophoretic laser scattering techniques will be used to carefully measure an array of thermodynamic and hydrodynamic (not kinetic) solution parameters for each of a group of selected proteins dissolved under crystallizing as well as non-crystallizing solvent conditions. The proteins chosen will have a wide variation with respect to molecular weight and crystallizing conditions such as temperature, pH and crystallizing agent type (inorganic salts, PEG's and other organics). The laser scattering solution parameters will be measured in the dilute protein concentration regime, often 10-20 times below protein saturation.

The research approach is to obtain comprehensive measurements of the SLS, DLS and ELS parameters from a set of selected proteins under both crystallizing and precipitating conditions with particular attention given to the dilute solution regime. The selection of the proteins is significant, and some collaboration with protein crystallographers will be required to totally define the set. Prior verbal agreement for such advisory collaboration has been obtained from Marc Pusey and Dan Carter at Marshall Space Flight Center in Huntsville, Alabama, Pat Weber at DuPont in Wilmington, Delaware, Alex McPherson at the University of California, Riverside, Franz Rosenberger at the University of Alabama, Huntsville, and Charlie Bugg and Larry DeLucas at the University of Alabama, Birmingham. Use will also be made of the Biological Macromolecule Crystallization Database compiled by Gary Gilliland at the Center for Advanced Research in Biotechnology in Rockville, Maryland. Based on years of experience in performing laser scattering measurements and on the manpower requested in the budget, a target number of twenty proteins is projected for the set, corresponding to roughly one complete set of measurements per protein per month. This amount of time accounts for protein purification procedures as well as repetitions for each of the SLS, DLS, and ELS experiments.

Task Significance:
It is anticipated that a particular solution parameter (or combination of parameters) will be discovered that has quantitative values within a reasonably narrow range for crystallizing conditions and values significantly outside that range for non-crystallizing or precipitating conditions. If such a universal predictor can be proven, then its use will have an immediate impact in the protein crystal growth community in general and microgravity research in particular. The solution conditions for protein crystallization in a microgravity environment should be maximized during ground testing so that a high probability for crystallization is achieved. Having a universal predictor will allow crystallographers to fine tune existing crystallization protocol or discover new conditions to crystallize difficult proteins.
Progress During FY 1995:

A predictor for protein crystallization has been defined in terms of a crystallization slot represented by values of a dilute solution parameter called the osmotic second virial coefficient, B_{22}. The slot includes values of B_{22} between about -2 to -8 x 10^4 mol ml g^{-2}. We have also found that B_{22} is a qualitative predictor for protein solubility for both normal and retrograde solubility behavior.

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**TASK INITIATION:** 7/93  **EXPIRATION:** 6/96

**PROJECT IDENTIFICATION:** 962-23-08-2!

**NASA CONTRACT NO.:** NAG8-965

**RESPONSIBLE CENTER:** MSFC

**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Journals**


**Presentations**


Wilson, W. "The second virial coefficient as a predictor in protein crystallization." Protein Crystal Growth Conference, Panama City, Florida, April 1995.
Phase Shifting Interferometric Analysis of Protein Crystal Growth Boundaries and Convective Flows

Principal Investigator: Mr. William K. Witherow

NASA Marshall Space Flight Center (MSFC)

Co-Investigators:
Sibille, Dr. L.  
Smith, D.

Universities Space Research Association (USRA)

NASA Marshall Space Flight Center (MSFC)

Task Objective:
The objective of the proposed study is to obtain experimental evidence of several characteristics of a crystallizing protein solution and model their effects on the crystal growth process. The characteristics to be studied include: the presence of concentration gradients during the crystal growth, the extent of the boundary layer from the crystal face, and the effect of buoyancy driven convection on the growth. Phase shifting interferometry (PSI) can provide significant insight to this issue. PSI will produce a visual confirmation of the concentration profile at the boundary layer and concentration values within the depletion region. PSI has the potential to offer a direct visualization of convective flows within proteinic crystallizing solutions.

Task Description:
Mach-Zehnder and Reflection Interferometry with a magnification of up to 1000x will be used to examine growing protein crystals. Phase shifting will be accomplished using an electro-optic phase modulator or a piezo electric mirror. Abel inverse transforms will be used on the resulting phase maps to provide planar phase information.

Task Significance:
Protein crystals are grown to determine the three-dimensional structure of proteins. By utilizing x-ray or neutron diffraction, the collected information allows the direct identification of active sites of the macromolecule, its conformation, and sequence of the amino acids. Sections of very large assemblies of proteins such as structural proteins or viruses can also be crystallized. Crystallization is therefore the starting point of any study aiming at the development of new drugs and the understanding of viral diseases. Crystallization techniques for proteins are now well known, but a biophysical understanding of the growth mechanisms is underdeveloped. This aspect of protein research needs to be expanded as the proteins being studied are more complex and their purification more costly. Since the beginning of the 1980's, tens of proteins have been crystallized in microgravity. Microgravity-grown crystals of several proteins were found to be larger or diffracted with higher resolution than ones previously grown on the ground. These results tend to demonstrate that a reduction of gravity affects the interfacial growth mechanisms which are directly dependent upon the mass transport regime. Convection is known to play a significant role in the growth kinetics of inorganic crystals but its importance is still debated in relation to the crystallization of biological macromolecules. There is little doubt about the existence of convective flows in proteinic solutions, but the flow rates they generate close to the crystal/solution interface and their effect on growth kinetics have not been quantified experimentally. The technique of PSI will allow us to determine these values and provide direct comparison between solutal flows in crystallizing solutions under various levels of gravity. An examination of the flows in the fluid and its correlation with crystal growth will strongly depict the role of microgravity in protein crystal growth.

Progress During FY 1995:
A phase shifting interferometer has been constructed and a computer program modified to collect the data and produce a phase map of the regions around growing protein crystals. A special optical quality test cell has been designed and built to grow the protein crystals. Work is progressing on collecting data from growing protein crystals and improvements are being made to the optical system.
II. MSAD Program Tasks — Ground-based

Discipline: Biotechnology

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PROJECT IDENTIFICATION: 962-23-08-44

RESPONSIBLE CENTER: MSFC

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Presentations
Sibille, L. "Work-to-date (poster)." 6th International Conference on Crystallization of Biological Macromolecules, Hiroshima, Japan, November 12-17, 1995.
II. MSAD Program Tasks — Ground-based

Characterization of Solvation Potentials Between Small Particles

Principal Investigator: Dr. Charles F. Zukoski
University of Illinois, Urbana-Champaign

Task Objective:
A combined experimental and modeling effort is used to characterize how variations in solvent chemical potential alter the states of aggregation of colloidal particles.

Task Description:
The objective of the FY95 program is to develop a combined density fractional theory and Monte Carlo simulation technique to describe the phase behavior of particles where interactions are dominated by solvation forces. In addition, the role of solvent/particle interactions in creating hydrophobic attractions is explored. On the experimental side, direct links are made between interparticle forces and phase behavior through measurements of protein second virial coefficients by light scattering and phase behavior as protein concentration is increased.

Task Significance:
Developing methods which reliably result in high quality protein crystals is of major technological significance in the development of fundamental understanding of biochemical phenomena and the expression of genetically altered therapeutic proteins. In the work carried out here, new methods of controlling protein crystallization are explored. The modeling effort seeks to guide the experimental program by providing understanding of the role of solvation interactions in controlling the state of protein aggregation. The modeling effort has shown that the rarely recognized variable of solvent chemical potential can be used to control protein crystallization behavior. The experimental program is aimed at developing methods of characterizing protein interactions as solvent chemical potential is altered and demonstrating links with crystallization behavior.

Progress During FY 1995:
Three major steps forward have been made. First, a model for electrostatic interactions between changed surfaces has been developed where the chemical potential of the solvent as well as the ions between the two approaching surfaces is held constant. This model captures both classic descriptions to double layer interactions as well as providing evidence for electrostatic attractions between like surfaces. The second step has been the development of analytical scanning probe microscope techniques for probing strength, range, and solvent dependencies of hydrophobic interactions. Thirdly, we have shown that the second virial coefficient can be used to predict the solubility of proteins and other globular macromolecules. Our work suggests that if compared on an equal footing, broad classes of globular macromolecules will have the same solubilities.

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Project Identification: 962-23-08-27
NASA Contract No.: NAG8-976
Responsible Center: MSFC

Bibliographic Citations for FY 1995:

Journals
Presentations


II. MSAD Program Tasks — Ground-based

Discipline: Combustion Science

Effects of Energy Release on Near Field Flow Structure of Gas Jets

Principal Investigator: Prof. Ajay K. Agrawal
University of Oklahoma

Co-Investigators:
Gollahalli, Dr. S.R.
University of Oklahoma

Task Objective:
The primary objective of this research is to understand how buoyancy affects the structure of the shear layer, the development of fluid dynamic instabilities, and formation and characteristics of the coherent structures in the near-nozzle regions of burning gas jets. The secondary objective is to understand the role of buoyancy in the flame lifting and reattachment process, evaluate the scaling behavior of diffusion flames, and aid in the development of a theoretical model by providing quantitative temperature data throughout the flame in the absence of buoyancy effects.

Task Description:
The initial phase of this project involves the visualization of near-field flow structures in cold jets and nonsooting flames at the same flow conditions or the same jet exit Reynolds number, allowing the effects of energy release on these structures to be identified by comparison. The experiments will use hydrogen and hydrogen-inert gas mixtures as the fuel with air as the oxidizer. Energy released in the flame will be controlled by varying the hydrogen mole fraction in the jet stream. Attached flames, lifted flames, and flames in the transition region between these two extremes will be studied. Experiments for all cases will be conducted in both normal and reduced gravity.

Task Significance:
The phenomena occurring near the exit of a gas jet nozzle determine burning characteristics and the rate of pollutant generation. The effect of buoyancy on these processes is poorly understood and hence it is difficult to model when designing commercial combustors. This project will study the fluid dynamics of turbulent gas jet combustion, applicable to commercial combustors of this type and may lead to higher efficiencies and lower pollution generation levels.

Progress During FY 1995:
Laboratory experiments were conducted using hydrogen at atmospheric pressure. The objective of these experiments was to determine flame characteristics including the Reynolds number at lift-off for nozzle diameters from 0.25 mm to 3.00 mm.

Reduced pressure laboratory facilities were developed. They included a vacuum pump rated at 117 CFM at an absolute pressure of 0.1 inches of mercury, and a stainless steel test chamber with appropriate optical access. The custom test chamber includes a diffuser section around the inlet nozzle to provide a uniform air flow around the fuel nozzle and a honeycomb diffuser at the outlet to provide a smooth transition to the outlet. Additionally, there is a remote igniter that retracts following ignition.

Analytical work was initiated to predict characteristics of the hydrogen jet. This effort involved numerical simulations of burning and non-burning diffusion jets at various operating conditions. A general purpose, commercial computational fluid dynamics code (CFD), PHOENICS, was used to compute the flow field. In the future, the energy release mechanism will be modeled using detailed chemical kinetics. Preliminary calculations were performed by CHEMKIN, a chemical kinetics code developed at the Sandia National Laboratory. The PHOENICS code was interfaced with the CHEMKIN code to simulate hydrogen jets with and without chemical reactions. We have also held discussions with the Naval Research Laboratories to use some of their simulation codes.
The PI and Co-I traveled to NASA Lewis and conducted the tests in the 2.2 Second Drop Tower. The test rig DIANA was used for these experiments. DeVon Griffin of LeRC provided the necessary training and support for using the rig and the drop tower; the rainbow schlieren filters used were developed by Paul Greenberg, also of LeRC. The experiments involved hydrogen flames at low cold flow Reynolds numbers (up to 400) from 0.58 mm and 1.2 mm ID fuel nozzles. Color schlieren images at normal gravity and microgravity were recorded in S-VHS format. Analysis of these tapes revealed that the flame anchored upstream of the fuel tube exit at both normal and low gravity. However, the upstream distance of 2 O.D. at normal gravity increased to approximately 6 O.D. at low gravity. Discussions were held with NASA scientists to develop techniques for quantifying the rainbow schlieren data.

These tests revealed that a more intense light source would be required to temporally resolve turbulent structure in the flames. Hence, a high intensity strobe with a xenon flash lamp was later modified to mate with the drop tower fiber optics. This lamp operates at 30 Hz, has a pulse energy of 1.3J with a duration of 6.5 microseconds. The lamp should have enough intensity to allow the 30 Hz video system to open its shutter for a duration of 200 microseconds. While this system appears adequate for current needs, we are also exploring the arc lamps sold by Oriel Corporation.

Ground experiments were conducted at atmospheric pressure for various fuel tubes and flow rates. A symmetric rainbow filter was created and used for some of the experiments. For a 0.58 mm I.D. tube, the flame appeared laminar at a Reynolds number of 540. At higher Reynolds numbers the laminar and turbulent zones were distinguished by the flame necking. The necking distance decreased with increasing Reynolds number as the flame became fully turbulent, lifted-off and eventually blew-off. The test facility for low pressure experiments was fabricated and has undergone validation tests. The entire test assembly was mounted on a swiveling bracket to facilitate orienting the flow direction at any angle with the gravity vector.

The rainbow filter generation computer programs, originally developed for a Unix machine, were ported to a personal computer. The filter data are stored in TIF format. Rectangular and circular filters of any RGB distributions can now be created readily. We are producing symmetric linear and exponential filters which will be used to evaluate sensitivity of the rainbow system. We acquired a digital film recorder with cost-sharing using internal funds. The recorder allows generation of a large variety of filters quickly and locally.

To complete the proposed reduced gravity test program, large hydrogen flow rates are required. Using the system currently in place at the 2.2 Second Drop Tower would require a large amount of onboard fuel to insure a steady flow rate. Due to safety concerns a balloon-based fuel supply system was developed. A fuel-filled balloon is contained inside a pressure vessel filled with an inert gas. The balloon inflates to a volume of approximately 100cc, with the fuel exiting at a constant flow rate due to the high pressure of the surrounding inert. Using this technique, the fuel would be completely consumed at the end of the drop. Preliminary tests on a demonstration unit were encouraging and we are now developing a procedure to systematically fill the inert and the fuel.

We identified and ordered a high intensity, short arc strobing white light source to maximize throughput in the single-mode fiber used in the 2.2 Second Drop Tower. This xenon light source from Oriel Corporation provides 0.32 J/pulse at up to 100Hz. The arc dimensions are 3mm x 2.5 mm with a pulse duration of 1.6 microsecond. A 1:1 condenser lens assembly is used to collect and reimage light onto a fiber bundle focusing assembly which could be linked to the transmitting fiber. The strobe can be pulsed manually or triggered externally by a TTL signal.

Preliminary experiments with an existing xenon strobe indicated a need to synchronize light source with the video camera. This was accomplished by an electronic circuit consisting of a sync separator, an adjustable time delay, and a trigger. The video signal was sync separated at either 30 or 60Hz. Thus, the light source could be triggered at 30Hz to synchronize with each video frame or at 60Hz to synchronize with each video field. The light source and the camera could be synchronized online by the adjustable time delay. Because we have not yet received strobe from Oriel, the synchronization circuit board was tested using an existing strobe.
The rainbow filter generation computer programs were extended to create filters of any RGB distributions. These distributions could be prescribed analytically or numerically. The revised program was used to create a variety of symmetric and exponential filters. Experiments with exponential filters improved resolution and visual appearance of the color schlieren images. We have acquired and installed the Polaroid Model HR 6000 Digital Palette Film Recorder. We have chosen TIFF format for all of our imaging work.

Experimenting with image acquisition, we found that 640x480 RGB schlieren images captured at 30Hz could not be transferred to the computer memory in real time. The limitation was imposed by the PC EISA-bus used by the frame grabber. The frame acquisition program was modified to trim the image such that small images could be stored in real time. A small image does not necessary lose useful data because the schlieren images often have a large background.

We evaluated a general purpose software MATLAB with its Image Processing tool box and determined that this software would limit our imaging capabilities. Thus, we have developed a set of subroutines in Fortran which could be integrated with public domain image read/write functions written in "C". These subroutines were developed for both the PC and Unix platforms. The subroutines have allowed us to develop image processing tools in Fortran.

The primary image processing task is to invert the schlieren images into refractive index distributions. A computer code was developed to curve-fit the angular deflection data from schlieren images using Hermite polynomial of some order. The deflection data could then be inverted to the refractive index distributions analytically without computationally intensive numerical integration. The computer codes to curve-fit and to invert deflection data have been validated using known distributions.

The vacuum chamber was reassembled and a shake-down test with hydrogen flames was conducted. The overall system performed well. The tests indicated needs to achieve a uniform airflow around the fuel nozzle and to simultaneously regulate airflow and the chamber pressure. Thus, the fuel-tube mounting system and the honeycomb were modified, and a flow control valve was placed downstream of the chamber. With these modifications, the airflow characteristics improved considerably. Adding the downstream valve not only provided independent control of the chamber pressure and airflow rate but also provided cold air to quench the exhaust gases. Thus, the temperature of gases into the vacuum pump was lowered. Recently, we used a smoke machine to visualize airflow in the chamber at cold flow conditions. We are in the process of instrumenting this test facility.

We have acquired and installed a Sun Sparcstation 20 which will be dedicated to the computational work. The PHOENICS and CHEMKIN coded have also been installed on this workstation. We have also acquired and installed another general purpose computational fluid dynamics software CFD-ACE by CFDRC.

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BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Presentations
II. MSAD Program Tasks — Ground-based

Discipline: Combustion Science

Radiative Extinction of Diffusion Flames

Principal Investigator: Prof. Arvind Atreya
University of Michigan

Co-Investigators:
Wichman, Prof. I.S.
Michigan State University

Task Objective:
The objective of this program is to quantify the conditions under which a stabilized, laminar diffusion flame will be extinguished by radiative heat losses from flame-generated particulates (e.g., soot) that drain the chemically released energy from the flame. These tests must be conducted in microgravity because radiation-induced extinction may not be possible under normal-gravity conditions where buoyancy-generated convection would sweep the radiating sources upward and away from the flame.

Task Description:
The program is to have simultaneous experimental and theoretical efforts. Experimentally, normal-gravity tests using a quasi-one-dimensional counterflow diffusion flame burner will be studied to quantify soot production and oxidation rates and their optical properties. These data are needed both for the formulation of the reduced-gravity testing and for the development of theoretical models.

Subsequent reduced-gravity testing is to be pursued in the 2.2 Second Drop Tower at NASA Lewis Research Center, where a laminar diffusion flame is to be stabilized about a spherical porous burner. In these tests the local fuel concentrations will be varied by the introduction of inerts into the fuel-flow stream. Measurements of flame temperature and radiation flux will be used for comparisons with theory.

A numerical model will be developed to simulate the reduced-gravity experimental configuration, and will include a chemical-kinetics-model and an empirical model of the production and consumption of soot particulates. A model of the radiant emissions from the flame, associated with the particulates, will be developed.

Task Significance:
The microgravity flames will demonstrate the concept of radiative extinction in stabilized flames, distinct from the case of spreading diffusion flames. This mechanism of extinction is likely unique in microgravity, and thus may have application to fire safety aboard orbiting spacecraft.

Progress During FY1995:
This report describes the final efforts of this program, completed during FY95.

1) Several weeks of 2.2 Second Drop Tower testing were conducted to refine the spherical burner design and ignition techniques. The porous ceramic material from which the burners have been made (to minimize heat losses to the burner) have a non-uniform microstructure that results in a flow that is not spherically symmetric, especially during the pressure pulse experienced during transition from the hydrogen ignition flame to the main fuel flow. Manufacturing techniques have been developed by the PI to mitigate these effects, demonstrated by the achievement of spherical blue flame surfaces. Additionally, the radiation detector system developed by the PI have shown that the dynamic range of emissions from the flame exceed that of the standard drop tower data acquisition system (10 bits). However, since the burner technology has been established, a limited number of drop tower tests have indicated that the time profile of the radiant emissions is repeatable where the system is not saturated.

In the last series of tests, an innovative method of measuring flame radiation using a trio of independent band limited photo-diode detectors (with different band-pass frequencies) was successful, and combined with thermocouple measurements provide time varying histories of radiant emissions, temperature and visible flame radii for spherically
expanding diffusion flames in quiescent microgravity environments for methane, ethylene, and acetylene all in air at normal atmospheric pressure. These results have been used to infer the history of soot formation, residence time and oxidation in these flames and are being compared to the numerical model developed earlier.

STUDENTS FUNDED UNDER RESEARCH:
BS Students: 0
MS Students: 2
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TASK INITIATION: 4/91 EXPIRATION: 3/95
PROJECT IDENTIFICATION: 962-22-05-29
NASA CONTRACT NO.: NAG3-1460
RESPONSIBLE CENTER: LeRC

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Presentations

Multicomponent Droplet Combustion in Microgravity: Soot Formation, Emulsions, Metal-Based Additives, and the Effect of Initial Droplet Diameter

Principal Investigator: Prof. C. T. Avedisian
Cornell University

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
The objective of the proposed research is to provide insights and data that supports the role of immiscible (e.g. emulsion) and miscible metal-containing additives, and to examine the influence of the initial droplet diameter on the combustion of fuel droplets in microgravity. In addition, the feasibility of using a laser-based light scattering technique for detecting soot formed in spherically symmetric droplet flames in microgravity will be examined.

Task Description:
The burning of multicomponent droplets is complicated by the influence of composition, which can effect the formation of soot, lead to multistage combustion in which one component preferentially vaporizes from the droplet during combustion, or possibly result in the droplet exploding during combustion due to achieving an internal superheat condition. These problems are extremely complicated, especially that of soot formation. A CCD camera coupled with an image intensifier for the purpose of recording UV light emissions from a burning droplet will be measuring the droplet flame diameter and a laser light scattering technique will be set-up to provide a quantitative measure of soot formation. The experiments will be conducted in a drop tower at Cornell that provides approximately 1 second of reduced gravity.

Task Significance:
Little data have been provided regarding the spherically symmetric burning of unsupported droplets of multicomponent fuels or emulsions. The importance of examining fuel blends is that most practical fuels which are burned in combustion-powered devices or incinerators are multicomponent in nature, usually miscible, but sometimes immiscible (emulsions).

If the mechanism for an influence of initial droplet diameter on spherically symmetric droplet burning can be understood and predicted for the "simple" spherically symmetric droplet flame, this information may be useful for providing insights into the influence of initial droplet size on soot formation in a convective environment. Metal additives can create the potential for significant reductions in particulate emissions by the possible effect of the ions produced on the nucleation and agglomeration of soot particles, and their possible oxidation; their effect in the droplet flames, especially in the spherically symmetric configuration, is unknown.

Progress During FY 1995:
There is no progress to report at this time since work will not start until September 1995.
BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Proceedings

Presentations
Avedisian, C.T. and Shaw, B.D., "Droplet combustion and soot formation in microgravity." 1994 Fall technical Meeting The Eastern States Section of the Combustion Institute, pp.19-26, 12/05/94.
II. MSAD Program Tasks — Ground-based

Discipline: Combustion Science

Development of Advanced Diagnostics for Characterization of Burning Droplets in Microgravity

Principal Investigator: Dr. William D. Bachalo
Aerometrics, Inc.

Co-Investigators:
Sankar, S.V.
Aerometrics, Inc.

Task Objective:
This research is intended to develop rainbow thermometry for quantitative radial temperature measurements in burning droplets and use Morphologically-Dependent Resonances for quantification of the radial regression rate. Both of these techniques will be developed using devices amenable for use in one of NASA's reduced gravity aircraft; this hardware will be delivered to LeRC at the end of the project.

Task Description:
Rainbow thermometry utilizes the angular dependence of rainbow location. In other words, given a known droplet composition, the location of the rainbow generated by that drop when illuminated with a monochromatic source will give the refractive index of the drop, and thus, its temperature. Instrumentation and software will be developed that will permit quantitative droplet temperature measurement.

Morphologically-Dependent Resonances occurs when the droplet diameter is an integral number of wavelengths of the illumination source. As the droplet size regresses due to combustion, this condition will be repeatedly met, causing the droplet to periodically "shine." The rate at which this occurs will give the diameter regression rate and will be accomplished using the same hardware as for the rainbow thermometry.

Task Significance:
Droplet combustion is an economically vital phenomenon occurring in liquid-fueled engine combustion, which includes both mobile and stationary combustors. Optimization of such designs has been hindered due to insufficient knowledge about the nature of combusting droplets. This project will develop analytic instrumentation that will allow measurement of the radial temperature distribution and size of combusting droplets. While the tools to be developed can be used in either normal or reduced gravity, the latter admits the use of much larger droplets and dramatically increases the information yield of the experiment.

Progress During FY 1995:
Efforts on this project have been focused on both modeling and hardware capabilities. On the modeling side, the initial efforts centered on extending the Lorenz-Mie theory to include scattering from droplets that were radially inhomogeneous. The goal was to develop a software package that integrated light scattering and combustion models to produce a theoretical description of the droplet size and burning rate throughout the combustion process. An additional challenge was to develop scattering theories for droplets with diameters on the order of millimeters, since droplets previously modeled by this community were much smaller.

As the theoretical effort progressed, the software was used to determine the size of droplets produced by a drop-on-demand generator to within ± 1 micrometer. Computer analysis also revealed that only rays passing through the outer 50% of the drop radius contribute to the rainbow signal. While disappointing for those wishing to measure the complete radial temperature of the drop, most reduced gravity measurements restrain the drop on fiber tethers. Hence, the measurement technique will not be perturbed by the method of anchoring the drop.

Two different techniques were developed for measuring droplet size regression during burning. The first, known as the phase method, relies on processing two frames of the rainbow thermometry signal to extract components whose contributions vary rapidly with time. These processed signals are then correlated to determine the phase difference

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between the two. This difference gives a rate of diameter change; the method is capable of detecting diameter changes as small as 2-5 nanometers but has difficulty following a droplet whose size is varying rapidly.

In the frequency method, a photodetector is placed at the rainbow angle and the fluctuations in light intensity are recorded. Using computer models, the time required for one cycle of intensity oscillation may be determined and a droplet regression rate then calculated from the time data. The resolution with this technique is approximately 200 nm. Hence, for droplets with a fast regression rate this would be the superior technique, whereas droplets that are regressing more slowly would be more amendable to the phase method. Both capabilities will be included in the system to be delivered to LeRC.

Hardware development included laboratory breadboarding to prove the technique outlined in the proposal, integration of software to automatically collect the data, a drop-on-demand generator to produce droplets for laboratory study and the hardware required to acquire and process the rainbow thermometry signals. Since deliverables on this task include hardware that could be flown on NASA's reduced gravity aircraft, LeRC personnel have provided the PI's team with mechanical drawings of an existing aircraft rig that would be compatible with the proposed effort.

The theoretical analysis and computations performed during the current reporting period raised several issues that need to be satisfactorily addressed before the design of the deliverable system can be undertaken. The most important of these pertain to the measurement of the fuel droplet regression rate. The need to record the high frequency oscillations of the rainbow signature imposes certain requirements on the optical system: high laser power, polarized light source, coherent light source, short exposure timing for the CCD, and high rep rates. In principle, all of these specifications can be met by incorporating advanced components such as gated intensified CCD cameras, dual cameras, and feedback control of laser power and camera gain. However, the limited financial resources available in the current Contract does not permit the development of such a sophisticated system. In light of these limitations, we are currently discussing different options with NASA so that a satisfactory system may be delivered by us at the end of this Contract. The possibility of using a high powered, fiber coupled diode laser (available at NASA) was discussed. However, the fact that the laser is incoherent and randomly polarized raises doubts with regard to its suitability for measuring the droplet regression rates. This issue needs to be resolved as soon as possible so that we may proceed with the design and fabrication of the deliverable system.

In the area of software development, efforts have been taken to develop LabView based data acquisition and processing for the DataRaptor frame grabber board. The development and testing of this software will continue for the next few months.

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**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Presentations**

II. MSAD Program Tasks — Ground-based

Discipline: Combustion Science

Ignition and Combustion of Bulk Metals

Principal Investigator: Prof. Melvyn C. Branch
University of Colorado, Boulder

Co-Investigators:
Daily, Prof. J.W.
Fiechtner, Dr. G.J.
University of Colorado, Boulder

Task Objective:
This project is an analytical and experimental investigation of the mechanisms of ignition and combustion of metal pellets under low-pressure oxygen atmospheres. Innovative features of the research apparatus and procedures are a non-disturbing radiative-ignition source and improved diagnostics for determination of surface and gas temperatures, flame characteristics, and metal-oxide surface morphology.

Task Description:
The study explores the entire process of metal combustion, starting from heat-up and ignition. The metal fuels are in the form of bulk pellets rather than the typical non-representative powder forms. A continuous, high-heat-flux radiant heat lamp is the ignition source, which eliminates the contamination of the reaction zone caused by the usual promoted igniters. Other innovative features of the experiment are in the diagnostics for the determination of temperatures of each phase, flame characteristics, fuel mass loss, and surface structure. The initial phase of the research is normal-gravity testing to evaluate the performance of the ignition system and to obtain reference data on ignition criteria, flame characteristics, metal-surface morphology, and heat and mass-transfer mechanisms for a number of pure metals. Upon completion of the normal-gravity tests, the tests continue at elevated gravity in the Geotechnical Centrifuge of the University of Colorado, using the same compact combustion chamber with its optical access and diagnostics but modified for remote operation.

Task Significance:
The study is the first approach to the investigation of ignition and combustion of bulk pure metals under conditions that facilitate the interpretation of the temperature and compositional histories. The practical application of the results promises improvement in the handling and fire safety of oxidizer pumping and storage systems in spacecraft and industrial applications.

Progress During FY 1995:
The normal-gravity experiments were continued to supplement the temperature measurements and visual observations obtained in the preceding year. Tests on eight different pure metals provided a selection of fuels with ignition temperatures above, at, and below the vaporization temperatures. The specimen pellets, 4-mm diameter by 4-mm high, were slightly smaller than those used in the preceding years. The pellets were ignited by a 1000-W xenon short-arc lamp, and the reaction was terminated at various stages of heat-up and combustion. Diagnostics included a spectrograph-array flame analyzer, scanning-electron-microscope (SEM) metallography analyzer, and an X-ray spectrometer chemical analyzer.

The time-resolved measurements of gas-phase emission spectra in the reaction zone were conducted with magnesium (Mg) specimens. A spectrograph with a diode-array detector measured the color temperature from the broad-band continuum spectrum. Prominent lines in the narrow-band spectra established the abundance of Mg and its MgO product through identified electronic and vibration resonances. This information will be very useful in determining the effect of gravity-induced convection in gas-phase reactions of bulk metals.

The SEM surface-morphology and chemical analyses of quenched specimens were conducted with copper (Cu) specimens. The oxidation history of Cu during the heating and combustion was determined from the identification
of surface and internal reactants and products by quantitative electron-microprobe analysis. It is anticipated that in microgravity the chemical nature of the oxide layer will be altered by the lack of convective transport of oxygen into the metal surface.

Elevated-gravity measurements were conducted with titanium (Ti) specimens over an acceleration range of 2 to 20 g in the University of Colorado Geotechnical Centrifuge. The complete experimental system was mounted on swiveling baskets at both ends of the rotating arms of the Centrifuge. Power, control, and data signals were transmitted through slip rings. An additional silicon photodiode was installed to monitor any changes in the Xe arc lamp output due to the increased acceleration environment. Results of the testing, in terms of the time-related temperature responses, indicated a distinct change in the heating rate of the Ti samples. At 3 and 5 g, the heating rate decreased and the ignition delay increased, although the ignition temperature remained comparable to that at normal gravity. At higher accelerations, however, the trend reversed and ignition delay decreased. In fact, at 15 and 20 g, the heating rate was greater that at normal gravity and the ignition temperature appeared to be higher. This behavior is due apparently to the enhanced transport of oxygen to the metal surface overcoming the negative effects of increased convective heat losses from the flame zone.

The project concluded on schedule at the beginning of December 1995. The final activities were the reconfiguration for low-gravity testing of the basic apparatus used on the Centrifuge. The low-gravity investigation is conducted in the DC-9 Airplane Facility at the NASA Lewis Research Center. A new data-acquisition-and-control system and electrical power supply and control were specified, secured, and checked. The apparatus is mounted in two frames: one for the combustion chamber, lamp, and diagnostics, and the other for the gas and vacuum storage and handling system.

A proposal for the follow-on microgravity study (a ground-based experiment) was submitted in response to a NASA Research Announcement in Microgravity Combustion Science and was selected for support. The new project, NASA Grant No. NAG 3-1685, continues the efforts of the completed subject project without interruption.

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**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Journals**
II. MSAD Program Tasks — Ground-based

Discipline: Combustion Science

Ignition and Combustion of Bulk Metals in Microgravity (Ground-Based Experiment)

Principal Investigator: Prof. Melvyn C. Branch
University of Colorado, Boulder

Co-Investigators:
Daily, Prof. J.W.
Fiechtnier, Dr. G.J.
University of Colorado, Boulder
University of Colorado, Boulder

Task Objective:
This project is an analytical and experimental investigation of the mechanisms of ignition and combustion of metal pellets under low-pressure oxygen atmospheres in a low-gravity environment. Innovative features of the research, continued from a preceding normal-gravity and elevated-gravity study, are a non-disturbing radiative-ignition source and improved diagnostics for determination of surface and gas temperatures, flame characteristics, and metal-oxide surface morphology.

Task Description:
The experimental study uses the apparatus and techniques developed in the preceding normal-gravity project to explore the entire process of metal combustion, starting from heat-up and ignition. The metal fuels are in the form of bulk pellets rather than the typical non-representative powder forms. Two unique features of the apparatus are the 1000-W, short-arc-lamp ignition source, which eliminates the contamination of the reaction zone and ignites all metal specimens in a clean and reproducible manner, and the compact combustion chamber with full optical access for non-disturbing measurements. The observation of the metal surface during ignition and combustion is achieved through high-speed photography, using a camera on loan from the Lewis Research Center. Metal-oxide combustion specimens are recovered after each test and analyzed physically and chemically by electron microscopy.

Elevated-gravity measurements, already underway, are to be completed in the Geotechnical Centrifuge operated by the University of Colorado. The compact, remotely operated apparatus used on the Centrifuge is modified for the microgravity experiments, to be conducted in the Lewis Research Center airplane facility. Metal combustion is strongly affected by gravity level, particularly in microgravity, because the changed mobility of the liquid phase in a varying convective environment affects the surface exposure of the burning metal.

The analytical study involves numerical modeling of the metal heating, ignition, and combustion processes based on information from the experimental studies. The modeling calculations cover the effects of gravity level and oxygen pressure. The initial findings were verified by the normal-gravity measurements of metal temperature rise, with excellent agreement. The extended modeling incorporates the variations in heat loss by natural convection, induced by the various gravity levels, as validated by the microgravity and elevated-gravity test results. The experimental results also contribute information necessary to add surface and gas-phase reaction rates into the modeling.

Task Significance:
The study is the first approach to the investigation of ignition and combustion of bulk pure metals under conditions that facilitate the interpretation of the temperature and compositional histories. The practical application of the results promise improvement in the handling and fire safety of oxidizer pumping and storage systems in spacecraft and industrial applications.

Progress During FY 1995:
For the low-gravity investigation to be conducted in the DC-9 Airplane Facility at the NASA Lewis Research Center, the apparatus was mounted in two frames: one for the combustion chamber, lamp, and diagnostics, and the other for the gas and vacuum storage and handling system. A new electrical system was designed, with a 120-VAC...
source for the lamp, pumps, spectrograph, computers, and other major diagnostics, and a 28-VDC source for the camera, shutters, and solenoids. The mechanical and electrical system designs were approved by NASA Lewis Ground-Based Facilities Branch. A new data-acquisition-and-control system for the low-gravity test program was specified by the Principal Investigator with NASA approval. In addition, after investigation of other diagnostics requirements, the Principal Investigator selected a new diode-array detector/spectrograph for the low-gravity apparatus. This instrument will improve the time- and space-resolved measurements of the emissions from the vapor-phase flames in the metal-oxygen reaction. The spectrograph has been acquired and installed.

Following the approval of the Test Request and Safety Request, the apparatus was shipped to NASA Lewis on August 23 for experiments on board the DC-9 Reduced-Gravity Research Aircraft. Because of an oil-pump problem on the airplane, the testing was delayed for three weeks. Starting on September 12, the student investigators conducted three days of experiments under multiple parabolic trajectories, covering checkout tests and combustion tests on magnesium and titanium pellets. The apparatus was returned to the University of Colorado on September 15.

Interpretation of the low-gravity test results from the data record of high-speed motion-picture photographs and thermocouple indications is now in progress.

**STUDENTS FUNDED UNDER RESEARCH:**

- BS Students: 2
- MS Students: 0
- PhD Students: 1

**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Proceedings**

**Presentations**
Modeling of Microgravity Combustion Experiments - Phase II

Principal Investigator: Prof. John D. Buckmaster
University of Illinois, Urbana-Champaign

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
Modeling of existing microgravity experiments for improving our understanding of the fundamental physics. Specific areas of interest include: smoldering combustion, premixed flammability studies, radiation effects, and diffusion flame instabilities.

Task Description:
Microgravity combustion experiments amenable to modeling are identified, and analytical and numerical models are developed from first principles for various systems and configurations. Model predictions are compared with experimental results and necessary adjustments and enhancements are incorporated into the models. Precise quantitative comparisons are made using numerical simulations where possible.

Task Significance:
Modeling provides invaluable physical insights into the experimentally observed behavior. This study is done in close collaboration with experimentalists with the goal of providing a clear understanding of the underlying physics.

Progress During FY 1995:
A paper entitled "Unsteady Spherical Flames in Dusty Gases," by J. Buckmaster and A. Agarwal is published by Combustion Science and Technology. The Abstract of the paper reads: "We examine an expanding spherical premixed flame propagating in a mixture containing inert dust. The presence of the dust generates a significant radiative energy flux that influences the flame speed. With the adoption of a hydrodynamic description in which stretch effects are accounted for using an empirical formula, and with the use of the Eddington approximation to describe the radiation, a simple numerical problem is formulated valid for that initial time interval in which the flame temperature changes by O(e) amounts where e is the small inverse activation energy. Solutions are constructed using parameter values appropriate to lean CH4/air mixtures, and for modest particle loadings. We describe the competition between the Zeldovich-Spalding effect in which radiative losses on the diffusive scale tend to quench the flame, and the Joulin effect in which radiative preheating on the scale of the Planck length tends to strengthen the flame."

The paper entitled "The role of slip in the generation of acoustic instabilities in gas turbine combustion systems," by M. DiCicco and J. Buckmaster, which was completed in December 1993 and submitted to the Journal of Propulsion and Power, is accepted for publication. The Abstract of the paper reads: "Slip affects the response time of fuel sprays to acoustic fluctuations in a gaseous flow field. This paper describes how gaseous fuel is released by evaporation as an oscillating response to the acoustic velocity fluctuations, and so contributes to acoustic instability. The paper discusses the differences due to the evaporation characteristics of various fuels (JP-4, JP-5, and D-2) as well as the effect of droplet size, inlet air temperature, air speed, and Reynolds number on mass response. For example, it is shown that instability will be driven harder at lower frequencies, higher gas velocities, and increased liquid volatility."

The paper entitled "The effects of radiation on the thermal-diffusive stability boundaries of premixed flames," by J. Buckmaster and T. Jackson was published in Combustion Science and Technology. The Abstract of the paper reads: "We examine the stability of premixed flames in mixtures containing significant amounts of fine dust whose sole impact is upon the radiative transport. By using well-established modeling strategies together with a simple..."

Printed: 28 FEB 1996
radiation model which preserves much of the essential physics, it is possible to explore to what extent radiative transport displaces the classical non-hydrodynamical stability boundaries of the plane deflagration. Analysis is possible for arbitrary values of both the Planck length and the Boltzman number. It is shown that the pulsating-traveling wave instability is strongly enhanced by the presence of radiation, and can be present even if \( Le<1 \). On the other hand, radiation tends to suppress the cellular instability normally associated with values of \( Le \) less than 1. The latter is consistent with preliminary experimental observations of Abbud-Madrid and Romney.

The paper entitled "Quenching of reverse smoulder," by D. Lozinski and J. Buckmaster is accepted for publication by Combustion and Flame. The Abstract of the paper reads: "A simple model of reverse smoulder in a porous medium is analyzed using asymptotic methods. When the only chemical reaction is exothermic oxidation, the burning rate is a single-valued function of the blowing rate, increasing from zero to a maximum, and then returning to zero. When endothermic pyrolysis is added to the description, the burning rate is double-valued for blowing rates less than some maximum. Beyond this maximum there are no solutions. The upper branch of the double-valued solution is the physically relevant one. On it, for certain choices of parameters, the burning rate increases from zero to a maximum, and then decreases until quenching occurs at the maximum blowing rate. This behavior mimics experimental observations by Torero, Fernandez-Pello, and Kitano (1993)."

The paper entitled "Some topics in reverse smoulder," by J. Buckmaster and D. Lozinski, is published in the book entitled Modelling in Combustion Science, Eds. J. Buckmaster and T. Takeno, Springer, Lecture Notes in Physics 449, p. 308, 1995. The Abstract of the paper reads: "The role of thermal non-equilibrium and endothermic pyrolysis is discussed in the context of a simple model of reverse smoulder combustion. It is shown that non-equilibrium has little qualitative effect on the nature of the solutions, but endothermic pyrolyzes can lead to quenching at sufficiently large blowing rates."

The paper entitled "An elementary discussion of forward smouldering," by J. Buckmaster, C. Fernandez-Pello, and D. Lozinski is accepted by Combustion and Flame. The Abstract of the paper reads: "We describe an elementary model of one-dimensional unsteady forward smouldering, purged of all unnecessary physics. Following work of Dosanjh and Pagni, a late time solution is constructed, characterized by two reaction fronts - an exothermic oxidation front, an unusual kind of diffusion flame; and an endothermic pyrolysis front. It is shown that the flame temperature and the ratio of the speeds of the two fronts relative to the solid are independent of the blowing rate, in agreement with data obtained by Ohlemiller and Lucca. The structure of the oxidation front is described in the context of 1-step Arrhenius kinetics, and it is shown that leakage of solid reactant through the front is possible, but not leakage of oxygen. An elementary pyrolysis structure is also examined which reduces to the frontal model in a certain limit, and clarifies its nature."

A paper entitled "A Theory of Shallow Smoulder Waves," by J. Buckmaster is submitted for publication. The Abstract of the paper reads: "A smoulder wave traveling through a porous solid slab is analyzed as a small Stefan number free-boundary problem, assuming that the wave depth is small compared to its length. It is shown that the wave aspect ratio \( AR=(\text{length/depth}) \) depends on two parameters: the Peclet number \( PeL/D \) where \( D \) is the air density, \( U \) the wave-speed, \( L \) the length, and \( D \) is the diffusion coefficient of oxygen; and the stoichiometric volume ratio of solid/air, which is small on account of the large solid/air density ratio. Assuming Peclet numbers near 1, it is shown that when free-stream convection is modest the aspect ratio will be large. When convection is large, however, no general statement is possible, although shallow waves can occur for realistic parameter values. Wave shapes are calculated, as are the oxygen and temperature fields. For large convection velocities the results are in qualitative agreement with numerical simulations, and with experimental observations.
II. MSAD Program Tasks — Ground-based

Discipline: Combustion Science

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals


Lozinski, D. and Buckmaster, J., Quenching of reverse smoulder. Accepted by Combustion and Flame, (May, 1995).

Books


Presentations


II. MSAD Program Tasks — Ground-based

Discipline: Combustion Science

Buoyancy Effects on the Structure and Stability of Burke-Schumann Diffusion Flames

Principal Investigator: Prof. L.- D. Chen
University of Iowa

Co-Investigators:
Stocker, D.P.
NASA Lewis Research Center (LeRC)

Task Objective:
The general goals of the proposed research are to improve our understanding of (1) the influence of buoyancy on co-flow diffusion flames, e.g., Burke-Schumann diffusion flames, and (2) the effects of buoyancy on vortex-flame interactions in co-flow diffusion flames. The overall objectives of the proposed work are to:
1. Verify the zero-gravity Burke-Schumann model and the gravity-dependent Hegde-Bahadori extension.
2. Investigate the flame stability in a buoyancy-dependent flow-field as affected by the co-flow oxidizer.
3. Examine the state relationships of co-flow diffusion flames.
4. Study flow vortex and diffusion flame interactions.

Task Description:
In order to meet the objectives, the phenomena will be investigated by:
1. Microgravity testing.
2. Normal-gravity testing (e.g., at reduced pressure to emulate reduced gravity).

D. Stocker (NASA LeRC) will have the lead responsibility for the microgravity testing, which will be generally conducted in the 2.2-second Drop Tower. The tests will be conducted in the rig fabricated for Stocker’s precursor studies of Burke-Schumann diffusion flames. Also, D. Stocker and L.-D. Chen will consider testing in the Zero-G Facility and in research aircraft (e.g., DC-9) if a need is identified. In either case, the tests will most likely be conducted in rig(s) built for the Gas-jet Diffusion Flame (GDF) Experiment, since only limited modification would be required.

Normal-gravity tests will be conducted under the direction of Prof. Chen at the University of Iowa and possibly at the Wright Laboratory (at WPAFB, in Dayton, OH), since the WPAFB facilities have been made available to this project at no cost. The normal-gravity experiments will complement the microgravity experiments, but will include tests that cannot be readily conducted in the microgravity facilities. For example, some instrumentation (e.g., NOx analyzer) that will be used in the normal-gravity testing cannot readily be used in the microgravity tests due to various constraints (e.g., time required for measurement, size of instrument, "ruggedness" of instrument, etc.).

Prof. Chen will direct the numerical modeling which will be conducted at the University of Iowa. An existing, semi-implicit, transient, axisymmetric code will be modified to include gas-phase radiation, state relationships or reduced mechanisms for major species, and finite chemistry for NOx. The predictions will be verified by the experimental measurements.

Task Significance:
The 1928 Burke-Schumann diffusion flame analysis is of fundamental and historical interest, and can be found in many, if not all, introductory combustion textbooks in the U.S. Experimental validation of this zero-gravity model will be an important contribution to combustion science, and it is likely to be referenced in future textbooks. Additionally, the study results could lead to the long-term to improved engine or furnace efficiency and reduced pollutant emissions.
Progress During FY 1995:

Experimental Testing:
During FY95, frequency measurements were made to determine the spectrum of the dynamic response of a pulsed methane-air Burke-Schumann Diffusion flame (BSDF). The data showed that there existed a higher frequency component between 20 to 30 Hz when the BSDF was driven by a frequency of 1 to 4 Hz. This high frequency component was reproduced in the numerical simulation, and the high frequency component disappeared in the simulation when gravity was set to zero in the simulation. The driven BSDF was reported in two papers presented at the Iowa Academy of Science annual meeting (Bang and Chen - Experimental; Sheu and Chen - Numerical).

Experiments were also conducted to determine the flame length and maximum radius of a free-jet laminar methane-air diffusion flame in reduced pressures. The results were compared with Roper's theory, which extended the classical analysis of Burke and Schumann. We also extended Roper's analysis to account for varying molecular diffusion coefficient resulting from the pressure variation. The comparison of the measurements and analysis showed good agreement when the maximum flame length was used as the scaling length. The results were presented in a poster presentation at the Joint Technical Meeting of the Central States, Western States, and Mexican Sections of The Combustion Institute and The American Flame Research Conference, 1995. Preparatory experiments for automated temperature traversing were also made for the free-jet methane-air laminar diffusion flame in reduced pressures using a coated fine-wire thermocouple. Temperature profiles showed that the maximum temperature lies outside the luminous boundary with a distance on the order of 1 to 2 mm. The separation distance appeared to increase with decreasing system pressures. Experiments are in progress to characterize the frequency response and velocity characteristics of the proportional valve that will be used in planned Drop-Tower experiments of the pulsed BSDF, as well as to determine the stability limits of a methane-air laminar jet diffusion flame in cross flows.

Detailed design of a new burner was initiated at NASA Lewis for planned testing in the Drop Tower. The new burner is being designed for flow visualization, and measurements of temperature, pressure, species, and thermal radiation. Furthermore, the burner is being designed for both circular and slot geometry, and co-flow and cross-flow configurations. A glovebox investigation was proposed in February 1995 for the study of the stability of ducted jet diffusion flames. The proposal was approved for a 6-month proof-of-concept period. The detailed designs of the prototype hardware will be completed in September 1995 at NASA Lewis. The experiment will consist of a fuel nozzle centered in a fan-driven flow duct, instrumented with an astronaut-positionable temperature rake. The rake will include an array of five thermocouples and numerous SiC fibers (used to provide temperature isotherms). The duct will also be instrumented with a hot-element anemometer, a pressure transducer, fuel flow controller, and additional thermocouples. Nitrogen-diluted methane and/or ethane will be used as the fuel. The operational variables are fuel flow, air velocity, the temperature rake position, and possibly the fuel.

Numerical Modeling:
During FY95, the time-accurate numerical model was extended from the fast chemistry (i.e., the flame-sheet model) to include the finite-rate chemical reactions for the simulation of laminar hydrogen-air and methane-air BSDF with or without external forcing of the fuel jet. The numerical model was successfully tested to include (a) a global 1-step a detailed reaction for CH4-air, (b) a global 4-step reaction for CH4-air without an external forcing of the fuel jet, (c) a detailed mechanism for H2-air (i.e., testing the H2-O2 mechanism) without the external forcing, (d) a 4-step reduced mechanism for CH4-air without the forcing. Currently, the numerical model is tested for the nitrogen chemistry for prediction of the NO emission from CH4-air BSDF. A reduced mechanism has been derived which includes the thermal NO, prompt NO, NO2 and N2O formation, destruction and transport in the BSDF studied. The N2 reduced mechanism solution will be compared with that based on a detailed N2 chemistry for a stationary BSDF in normal-gravity and zero-gravity conditions. The numerical simulation based on 1-step global reaction and 4-step reduced mechanism showed that the effects due to differential diffusion of species, non-unity Lewis number (or preferential diffusion in energy and species), and thermal/Soret diffusion are more pronounced in microgravity environment. The 1-step global model is currently being tested for its prediction of the stabilization location of the BSDF (i.e., flame base location) as a function of the velocity, while keeping an identical velocity for the fuel and air at the inlet. The preliminary results showed that the flame base moved to downstream locations with increasing velocities, although the prediction has not shown the rapid increase of the flame base when a lifted condition is approached. The simulation does correctly predict a triple-flame structure at the flame base.

One-dimensional laminar flamelet modeling was also made to assess the characteristic times that are required for the
II. MSAD Program Tasks — Ground-based

Discipline: Combustion Science

flamelet to reach steady-state response, for example, due to the time-varying flow conditions as reflected in
flame-vortex interaction or increasing the flow velocity. The flamelet calculation will also be tested to compare the
NO predictions based on our derived reduced mechanism with those based on a detailed mechanism.

STUDENTS FUNDED UNDER RESEARCH:

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PROJECT IDENTIFICATION: 962-22-05-63

NASA CONTRACT NO.: NAG3-1592

RESPONSIBLE CENTER: LeRC

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Proceedings

Presentations


Printed: 28 FEB 1996

II-315
Gravitational Effects on Premixed Turbulent Flames

Principal Investigator: Dr. Robert K. Cheng

Lawrence Berkeley Laboratory

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
The overall objective of this experiment is to investigate experimentally the dynamics of low Reynolds number premixed turbulent flames in a microgravity environment.

Task Description:
The emphasis of this experimental program will be on measuring flame wrinkle scales and imaging mean flowfield properties of conical Bunsen-type flames and rod-stabilized \( v \)-flames. Laser diagnostics will be used to obtain statistical scalar and velocity information to evaluate parameters suitable for predicting the effects of gravity on turbulent premixed flames. A 2.2. Second Drop Tower experiment, and Lear jet parabolas will be performed as well as parallel laboratory experiments.

Task Significance:
This research effort will provide insight to determine significant processes through which gravity affects flame properties such as flame speed and flame structures. The results will be used to determine the gravity-influenced limit for premixed turbulent flames. Such knowledge is valuable for guiding the development of turbulent combustion models to include the effects of gravity. This experimental study will contribute to resolving the inconsistencies that exist between experiments and theories and help couple chemical and fluid mechanical processes found in many turbulent combustion environments. If better understood, turbulent combustion can be exploited to enhance burning rates and volumetric power density in many heating and power generating systems.

Progress During FY 1995:
The investigation of the laminar and turbulent stabilization limits for two flame configurations (i.e., conical and \( v \)-flames) in \(+g\) and \(-g\) have been completed. This study provided the necessary foundation for interpreting the results obtained in microgravity and can be applied to selecting the proper operating conditions for future microgravity experiments. For \( v \)-flames, reversing the gravitation vector extends the stabilization limit to leaner equivalence ratios. For conical flames, changes in the stabilization limits are not as drastic due to the effectiveness of the stabilizer ring (patent pending). Analysis of the \( v \)-flame angle in normal gravity for a large range of equivalence ratios and flow rates were performed. The results are used to compare with those determined under \(-g\) and microgravity. Such comparison will help to explain the stabilization limits.

During the course of the flame stabilization studies it was discovered that buoyancy can be used to stabilize laminar flat flames. These flames occur in \(-g\) when the exit flow velocities are close to the laminar burning speed. The most important stabilization mechanism is the balance between the flow momentum generated by combustion and buoyancy forces of the products. The discovery of these flames is significant to this research because they provide a convenient means to characterize the scaling between momentum and buoyancy for premixed flames.

In preparation for the use of the planar imaging technique in microgravity, preliminary tomographic study of conical flames was conducted. Tomography involves seeding the premixed flow with a silicone oil aerosol that burns and evaporates at the flame front. When illuminated with a laser sheet, the flame wrinkles are delineated. This technique has been successfully applied in a laboratory to study premixed turbulent flames. Uniform seeding is one of the most important criteria for tomography and difficult to achieve using existing hardware. Alternative techniques and design changes are being explored to overcome this problem.
**II. MSAD Program Tasks — Ground-based**

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**PROJECT IDENTIFICATION:** 962-22-05-32  
**NASA CONTRACT NO.:** C-3200012  
**RESPONSIBLE CENTER:** LeRC

**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Journals**  
Gravitational Effects on Premixed Turbulent Flames: Microgravity Flame Structures

Principal Investigator: Dr. Robert K. Cheng
Lawrence Berkeley Laboratory

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
The overall objective of this experiment is to investigate the influence of gravity on premixed turbulent flame propagation.

Task Description:
The emphasis of this experimental program will be on measuring flame wrinkle scales and imaging mean flowfield properties of conical Bunsen-type flames and rod-stabilized V-flames. Laser diagnostics will be used to obtain statistical scalar and velocity information to evaluate parameters suitable for predicting the effects of gravity on turbulent premixed flames. DC-9 parabolic flights will be performed as well as parallel laboratory experiments. The research effort is an extension of a previous NRA effort entitled "Gravitational Effects on Premixed Turbulent Flames."

Task Significance:
This research effort will provide insight to determine significant processes through which gravity effects flame properties such as flame speed and flame structures. The results will be used to determine the gravity-influenced limit for premixed turbulent flames. Such knowledge is valuable for guiding the development of turbulent combustion models to include the effects of gravity. This work will contribute to resolving the inconsistencies that exist between experiments and theories. If better understood, turbulent combustion can be exploited to enhance burning rates and volumetric power density in many heating and power generating systems.

Progress During FY 1995:
Work continued on investigating laminar flat flames stabilized by buoyancy to determine the appropriate scaling parameters to describe the ratio between flame generated flow momentum and buoyancy forces. This knowledge is essential for evaluating the effects of gravity on more complex turbulent flame situations.

LDA was used to measure the axial and radial velocity components. Centerline and traverse velocity profiles were obtained for five different conditions. For one of these flames, the flowfield was scanned to obtain a velocity vector map that showed flow divergence and overall characteristics.

Implicit in buoyancy stabilized flames is a balance of momentum and buoyancy forces within the products. The important parameter in our analysis is the Richardson Number, Ri, which is the ratio of momentum to buoyancy. It was found that an important length scale for defining the Ri for this problem is the thickness of the product zone. This thickness is defined by the distance between the flame front and the stagnation point where the velocity of the product decreases to zero due to buoyancy. The Ri defined for all cases is about one. This is in accord with the notion that in order for these flames to exist, the momentum generated by combustion should balance the buoyancy force. The results also show that information obtained along a center can be used to describe the dynamics of the entire flame flowfield.

Work will continue with LDA measurements of -g v-flames and conical flames. These results are needed to help interpret the schlieren observation in microgravity. Once the overall characteristics of turbulent v-flames and conical flames are determined, they will be used to guide the selection of the experimental conditions for the next microgravity parabolic flight campaign. Central to the next campaign is the use of planar imaging technique. Also planned is the use of tomography if an appropriate laser source is available.
II. MSAD Program Tasks — Ground-based

Discipline: Combustion Science

STUDENTS FUNDED UNDER RESEARCH: STANDS FUNDED UNDER RESEARCH: TASK INITIATION: 3/95 EXPIRATION: 3/97
PROJECT IDENTIFICATION: 962-22-05-32 RESPONSIBLE CENTER: LeRC

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Proceedings
Combustion of Interacting Droplet Arrays in a Microgravity Environment

Principal Investigator: Dr. Daniel L. Dietrich
NASA Lewis Research Center (LeRC)

Co-Investigators:
Kitano, K.
Hokkaido National Industrial Research Institute

II. MSAD Program Tasks — Ground-based

Task Objective:
This research program involves the study of one-dimensional and two-dimensional arrays of droplets in a buoyant-free environment. The purpose of the work is to extend the data base and theories that exist for single droplets into the regime wherein droplet interactions are important.

Task Description:
The emphasis of the present investigation is experimental, although comparison will be made to existing theoretical and numerical treatments when appropriate. Both normal-gravity and low-gravity testing will be employed, and the results compared.

The normal-gravity testing will utilize the classical suspended droplet technique; single droplets and droplet arrays will be supported on 125 µm optical fibers in a combustion chamber where the ambient environment can be controlled. The low-gravity testing will employ droplets suspended on 15 µm Si-C fibers, a new technique developed during the past year, again in a combustion chamber where the ambient environment can be changed.

Task Significance:
The eventual goal will be to use the results of this work as inputs for models on spray combustion, wherein droplets seldom burn individually (the combustion history of a droplet is strongly influenced by the presence of the neighboring droplets).

Progress During FY 1995:
A significant number of improvements to the normal gravity testing apparatus were implemented this year. These include: 1) the purchase and use of a long distance working distance microscope to obtain detailed magnified images of the droplet, 2) a computer system (based on the Apple Macintosh) to control the dispensing, deployment and ignition of the droplet(s), 3) improved deployment system, 4) computer based data acquisition of analog pressure transducer, radiometer and thermocouple data, and 5) automated computer data analysis. The data acquisition is now performed real-time to the computer, which can also be analyzed in several minutes for the droplet size history.

Single droplet combustion and two-droplet array data were taken with the improved normal gravity test apparatus. The single droplet combustion data were for chloroheptane and chlorodecane in support of the work on Laser-Induced Incandescence (LII). The burning rate constant for both fuels was approximately 0.87 mm²/sec for 1.4 mm initial sized droplets burning in 1 atm air.

In collaboration with R. Vander Wal, data was obtained for the burning of heptane, decane and hexadecane. The data was simultaneous measurements of the natural flame, a backlit view of the droplet and radiometric measurements of the flame.

Single and two droplet array testing of decane droplets in air (normal gravity), at atmospheric and subatmospheric pressures started this month. The initial droplet sizes were on the order of 1-1.5 mm. As of the writing of this report, inter-droplet separation distances of 4, 6, and 8 mm were tested and analyzed at pressures of 1, 1/2 and 1/4 atm (air ambient).
Flight hardware for the Fiber Supported Droplet Combustion (FSDC) glovebox experiment was assembled and delivered to KSC. Everything is on schedule for the USML-2 mission in September 1995.

**STUDENTS FUNDED UNDER RESEARCH:**

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**TASK INITIATION:** 10/89  **EXPIRATION:** 12/94

**PROJECT IDENTIFICATION:** 962-22-05-33  **RESPONSIBLE CENTER:** LeRC

**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Journals**

**Presentations**

Dietrich, D.L., and Vander Wal, R., "Relative soot volume fractions in droplet combustion via laser-induced incandescence." Presented at the Eastern States Section of the Combustion Institute, Fall meeting.
Internal and Surface Phenomena in Heterogeneous Metal Combustion

Principal Investigator: Dr. Edward L. Dreizin
AeroChem Research Laboratories, Inc.

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
This project is an experimental and analytical study of condensed and vapor-phase combustion and oxidant penetration in burning metal droplets. The emphasis is on the determination of metal combustion rates and temperature histories and the explanation of disruptive burning, such as in microexplosions.

Task Description:
The study uses a novel apparatus, developed by the Principal Investigator, that generates and ignites metal droplets through electrical-arc discharges with consumable, pure-metal electrodes. This insures repeatable formation and ignition of uniform metal droplets with a controllable initial temperature and velocity. The burning droplets are subsequently quenched to terminate the reaction at prescribed stages of the combustion. Test variations include those of temperature, droplet size, and atmospheric oxygen concentration for at least three different metals. The experimental data cover droplet observations, droplet temperatures and diameters as functions of time, and chemical and metallurgical analyses of the inner composition of the quenched, solidified specimens.

In microgravity, the absence of settling forces permits the precise control of the droplet motion to improve the interpretation of the droplet-diameter and droplet-temperature histories. Therefore, a subsequent set of experiments will be conducted in low gravity using the NASA Lewis Research Center parabolic-trajectory airplane facility.

Concurrent with the experimental studies, analytical studies verified by the experimental results will model the processes occurring in metal droplet combustion to provide a theoretical foundation of surface and internal transport and reaction-rate phenomena in the metal droplets.

Task Significance:
The generation of monodisperse metal droplets creates a set of microgravity metal-combustion experiments analogous to those of microgravity fuel-droplet combustion. In contrast to the essentially homogenous liquid-fuel droplet, however, the internal structure of the metal droplet can be altered by controlled quenching and rapid freezing. The results of the study have practical application to the safety and improved performance of metallized fuels, such as those used in solid rocket motors.

Progress During FY 1995:
The first-year effort of a planned four-year project is complete. The metal-droplet generation and ignition apparatus was already available, and only small modifications, principally in the electrode feed and positioning unit, were required to initiate the studies. The normal-gravity research in the first year covered the investigation of the temperature and compositional histories of burning droplets of aluminum (Al), zirconium (Zr), and titanium (Ti).

The experiments on Al droplets concentrated on 190 mg initial-diameter particles burning for periods up to 100 ms in air atmospheres. While several methods of terminating the combustion were investigated, the most repeatable results were obtained by rapid quenching through impingement onto an aluminum plate. The Al studies complemented studies on this metal that were initiated prior to the NASA-supported research. The results have contributed new understanding of the observed phenomena of discontinuous temperature histories and "jumps". The analyses of quenched droplet cross-sections were also revealing. For example, after about 50 ms of combustion, the
recovered particles consisted of a spherical segment composed of stoichiometric aluminum oxide overlaying the bulk particle composed of Al with dissolved oxygen. For longer periods of combustion, the volume of the oxide segment increased with time to encompass nearly all of the droplet.

The experiments on Zr droplets were facilitated for good repeatability by using an electrode of Zr wire with a very thin coating of copper. The study concentrated on 240-µm initial diameter particles. Spectroscopic measurements showed a strong radiation return from a cloud surrounding the droplet, implying a vapor-phase combustion, an unexpected result for Zr combustion. The burning Zr particles were quenched rapidly by impingement on aluminum foil. Both oxygen and nitrogen from the air atmosphere dissolved into the droplets during combustion. The rate of nitrogen uptake decreased with time after an initially strong value, but the rate of oxygen uptake was essentially constant with time. Analysis of the quenched droplets showed that a Zr-O-N solution formed first. Stoichiometric ZrO₂ formed at a later time during the combustion process, but the solid oxide remained dissolved in the liquid matrix. The droplet became supersaturated when its temperature decreased after about 150 ms of combustion time. Eventually, the oxide precipitated rapidly with a sudden evolution of heat, a phenomenon proposed as the cause for temperature jumps during combustion and inhomogeneities in the quenched droplets. In tests where the droplets fall into an oxygen atmosphere before quenching, disruptive burning occurred. This also was attributed to the sudden release of the heat of precipitation.

The experiments on Ti droplets concentrated on 220-µm initial diameter particles. These studies and their interpretations are in process.

With the completion of the normal-gravity experiments, the project efforts will turn to the redesign of the apparatus for tests on zero-initial-velocity droplet trajectories and in microgravity environments.

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**STUDENTS FUNDED UNDER RESEARCH:**

- **Task Initiation:** 7/94
- **Expiration:** 6/98
- **Project Identification:** 962-22-05-46
- **NASA Contract No.:** NAS-3-27259
- **Responsible Center:** LeRC

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**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Journals**


**Proceedings**


**Presentations**

II. MSAD Program Tasks — Ground-based

Flame-Vortex Interactions Imaged in Microgravity

Principal Investigator: Prof. James F. Driscoll
University of Michigan

Co-Investigators:
Dahm, Prof. W.J.A.
Sichel, Prof. M.
University of Michigan
University of Michigan

Task Objective:
The objective of this research is to investigate a method that will provide high quality, quantitative, color enhanced digital images of a vortex exerting aerodynamic strain on a flame under microgravity conditions. This will be used to:
1. Quantify how the vortex distorts the flame;
2. Define the degree of flame curvature;

Task Description:
The fundamental interaction between turbulence and chemistry in a combustion process will be studied by interacting a repeatable, well-defined vortex, or variable size and strength vortex, with premixed and nonpremixed flames. The complicating effects of buoyancy will be eliminated by employing the NASA LeRC 2.2 Second Drop Tower. High quality, quantitative, color enhanced 2-D images will be obtained of the temperature field, the flame emission, and the mixture fraction (nonpremixed case). Results will be compared to full Navier-Stokes direct numerical simulations and to the classical theory of stretched flames.

Task Significance:
Images of the flame shape and curvature will be used to assess numerical simulations which neglect buoyancy, and to deduce universal, buoyancy free scaling relations showing the effects of vortex size and strength. Understanding of turbulent combustion, applicable to all practical combustion, will be enhanced.

Progress During FY 1995:
A graduate student (Alan Tuikki, a Ph.D. candidate in Aerospace Engineering) was hired for the project. Equipment information and catalogs were collected from vendors of the components needed for the drop experiment. Blueprints for the modified flame-vortex interaction experiment were drawn up on CLARIS CAD and given to give to the machinist. A Xenon arc lamp white light source was borrowed from an existing research project and was tested to see if a white light sheet of the proper thickness (1mm) and intensity can be obtained. A satisfactory 1mm thick sheet was obtained that has a 7.5 cm confocal length, which is sufficient. The energy per pulse in the sheet was measured using a pulsed energy meter. Optimization of the sheet optical arrangement is continuing. Updated PIV data processing software was obtained from Larry Goss at Wright Patterson AFB. A student is beginning to test the software on an in-house computer. It is planned to use this software to obtain velocity field images and local strain rates on the flames in the drop experiment. The synchronization of the experiment was tested using an existing setup. A NASA drop frame was received. It is planned to set up the experiment in this frame in the next month as the equipment is received.

A writeup of the proposed experiment was assembled, with drawings of the arrangement and photographs of the equipment that was purchased. A proposed electronic wiring diagram was outlined (in a general sense) and the commands that need to be programmed on the Tattletale timing controller are listed. Most of the equipment to be used on the drop experiment has been received. Tests were conducted to check the operation of the video camera, light source, atomizer, and optical system and some solenoid valves. The entire air supply system and the fuel
supply system components were ordered. Some potential problems with handling propane were solved. A small tank must be filled properly, allowing for several safety features to prevent overfilling with liquid. The propane distributor recommended several fittings which were ordered. The thin filament array mount was designed. It is planned to mount 50 filaments 1 mm apart between two stiff wires and then insert this into the combustion chamber. The components were designed and the mount is being constructed by the departmental machinist.

All of the plumbing required for the drop package was received and was partially assembled on a benchtop rig. Some remaining optical components also were received. It was found that the condenser lens that originally was suggested by Oriel Optics did not provide a uniform light sheet so it was replaced with an acromat which was satisfactory. An optical shelf and DDAC controller were provided by NASA and integrated into the experiment. The Xenon light source was synchronized with the video camera. J. Driscoll and two students visited NASA to discuss safety aspects. It was decided to eliminate the fuel and air tanks and fill the experiment with a mixture prior to the drop. The power distribution circuit diagram that was designed by NASA was assigned to an electronics technician for construction. The ignition system was designed utilizing an electric match used in model rockets will be used.

Some one-g experiments were conducted to test out the PIV system that will be used in the drop experiment. Approximately 50 PIV images were obtained for different flame-vortex interactions. A new seeding method was developed so that particles can survive the flame and yield velocity in the products. The new flame-vortex interaction chamber was designed and machined. It consists of three sections: an upper rectangular section with the ignition wires and controllable relief ports, a central rectangular section with optical windows, and a lower rectangular section with the loudspeaker, sharp edged orifice, and fuel inlet ports. Each section has flanges and gaskets so that the three sections bolt together for easy access and to allow the central section to be replaced for future measurements. The DDAC was programmed and will be tested upon completion of the power distribution box. An additional electronics box was designed by NASA to allow the video camera 60 Hz framing signal to drive the light source. A graduate student has assembled and used the necessary PIV velocity field imaging equipment needed to analyze the PIV images that will be obtained in the drop tower experiments. Exceptionally high quality PIV velocity field images for the one-g flame-vortex interaction were obtained. The vorticity field has been measured and several important scientific findings have been made. The flame generates vorticity that was not expected and a physical reason for this finding has been identified. Vortices of different strengths cause different "flame-generated turbulence". The necessary software was written (and commercial software was interfaced) so that velocity vectors can be properly plotted and vorticity contours plotted.

The Michigan flame-vortex drop package was prepared for an anticipated late April drop in the NASA 2.2 second drop tower. The optics were optimized such that the light source and the video camera are properly synchronized. The fuel-air loading system, which is separate from the drop package, was further modified and tested to provide a homogeneous fuel-air charge. The port system that exhausts the product gases was tested and modified. The PIV data analysis system was improved and tested at one-g. In the future, PIV velocity field images will be obtained in microgravity with the drop package, and a major part of this effort is the data reduction and interpretation of the velocity field images. Therefore it is important to have the PIV data analysis system operating to its full potential. One-g images were obtained and analyzed. These images will be presented at the April microgravity conference to illustrate the type of information that PIV provides. A second drop package was designed and is being prepared; it is a study of nonpremixed flames and vortices, and differs from the first package, which is a study of premixed flames. Different fuel and air management systems are required. The second drop package is being assembled in UM laboratories.

Several problems with the drop rig were identified and corrected. The fuel-air charging system was modified in order to meet NASA safety standards. It was decided to not drop any bottles of pure fuel but to charge the system with an external fuel bottle. Plumbing and construction of the charging system was completed. The ignitor first used was modified to make it easy to replace between drops. The DDAC computer was programmed. A safety switch was added which prevents ignition from occurring unless the exhaust ports are open. The light sheet used for imaging was improved by modifying the lens positions and one lens was replaced with a shorter focal length to achieve a thinner light sheet.
II. MSAD Program Tasks — Ground-based

Discipline: Combustion Science

Another graduate student (Charles Mueller) has been developing the PIV hardware and software for use on both the one-g experiment and the future microgravity PIV measurements. Several hundred one-g PIV images have been recorded and analyzed. The flame-generated vorticity, stretch rate of the flame, and other properties of flames that are being wrinkled by vortices have been obtained. Software was written to deduce the stretch rates, vorticity and plot the contours.

A graduate student (Fernando Costa) spent several months in Washington DC at the Naval Research Lab working with Dr. K. Kailasanath. Professor Sichel at Michigan is developing a code which will simulate the experiments to be performed in the drop tower. The NRL code FLAMEID is superimposed on a flame interface that is convected by the vortex induced velocity field. This code simulates complex chemistry, realistic diffusion coefficients, and unsteady effects. The advantage of such simulations are that several important physical processes can be turned on and off in order to understand which physical process is the cause of an experimental observation. For example, when local flame extinction occurs, radiative heat losses can be removed from the simulation and it typically is found that extinction no longer occurs, indicating the importance of quantifying and properly simulating heat losses.

Professor Dahm has received a second drop rig frame and has been adding the necessary DDAC, power distribution box and optics. This drop rig is designed to study the interaction of a nonpremixed flame and a vortex, which is an important problem that affects the flame blowout limits, NOx generation, soot generation and radiation losses in turbulent nonpremixed flames.

Two papers have been prepared for journal publication and conference presentation, "Time-Evolution of the Vorticity Field as a Vortex Interacts with a Flame" by Mueller, C. J., Driscoll, J.F., Reuss, D., Drake, M.C., and Rosalik, M. to be submitted to Combustion and Flame. "Analysis of Unsteady One-Dimensional Homogeneous Diffusion Flames at Constant Pressure", Costa, F.S., Kailasanath, K., and Sichel, M. to be presented at the ICDERS Meeting and then submitted for publication.

Some additional problems associated with the drop rig were solved. The electrical discharge of the Xenon light source disturbed the operation of the DDAC computer. Proper shielding and relocation of the light source was found to eliminate the problem. The software for the DDAC was refined and works well. Some components of the fuel-air charging system were added. A pressure gauge was added to measure the pressure downstream of the choked orifices to insure that choking occurred when the seeder is added downstream. The operation of the seeder was tested. Runs were made under several conditions to calibrate the system and determine the best running conditions. The operation of the safety switch was tested.

A paper that describes the one-g PIV baseline tests was written and is now ready to be submitted for journal publication. It was found that baroclinic torques due to gravity have a large effect on the wrinkling and stretch rates of typical flames. These effects were quantified in the one g experiments. An important research issue is how the flame wrinkling process differs in microgravity when these baroclinic torques are not present. Turbulent flame properties such as local extinction, blowout, and burning velocity are very sensitive to the flame wrinkling process and may be significantly changed by subjecting a flame to a microgravity environment.

The drop experiment was modified and tested to satisfy the recommendations of the safety committee. A switch was added that will not allow the ignitor to fire unless the exhaust ports are open. The previous switch provided this capability but was controlled by the DDAC. Runs were made with the exhaust ports closed at the normal lean conditions (equivalence ratio of 0.6) and at the worst-case scenario of equivalence ratio of 1.0. The blowout patch operated correctly and the chamber maintained its integrity. Numerous runs of the experiment were made to check out the timing and repeatability of the flame. There was a problem with some air leaking into the chamber which renders the mixture inflammable. Efforts were made to correct all leaks. The DDAC program was upgraded and improved.

Marty Sichel met with Dr. Kailasanath at NRL in Washington and is planning to do direct numerical simulations of the experiment. Comparison of measurements with simulations will provide quantities (such as chemical species concentrations) that cannot be measured.

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Experiments were prepared for drop tower tests and drops were made during the week of July 17. These drops were of a preliminary nature and the purpose was to pass the NASA safety inspection, test the system integrity, and identify any potential problems that need to be solved before the next set of drops, which will be data-gathering drops. The following was learned from the July 1995 drops:

1. The experiment passed the NASA safety committee inspection.

2. There is a 20 minute time period between the charging of the combustion chamber and the actual drop. Our design does not maintain the seeding particles in a uniform concentration during that time; some air leaked in and some particles settled out. Changes need to be made to get better visualization using these seeding droplets.

3. The spark ignitor did not properly ignite the fuel-air mixture in the tests conducted just prior to the NASA drop tower tests. The existing ignitor has to be replaced with a new ignitor that is lightweight and does not interfere with the DDAC controller, yet it must be energetic enough to consistently ignite the lean fuel-air mixture.

4. The optics, video camera, transmitter, DDAC controller and all valves and safety interlocks operated correctly on the drops that were made.

5. A fuel charging room in the basement of the drop tower facility is needed to safely charge this experiment. Plans were made by both Michigan and NASA personnel to insure that the necessary items will be available for the next set of drops.

A new ignitor was ordered and received. The new ignition system consists of a dc to dc voltage amplifier, a high voltage relay, and a high voltage capacitor. New electrodes were also added. The particle seeding was not acceptable because of leaks in the chamber exhaust ports, which remain closed for 20 minutes prior to each drop. Steps were taken to seal such leaks. New gasket material and a stronger spring tension to seal the metal end plate against the gaskets were added.

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II. MSAD Program Tasks — Ground-based

Aerodynamic, Unsteady, Kinetic, and Heat Loss Effects on the Dynamics and Structure of Weakly-Burning Flames in Microgravity

Principal Investigator: Prof. Fokion N. Egolfopoulos
University of Southern California

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
To obtain quantitative information (flame speed, velocity field, temperature) about the characteristics of weakly burning flames in microgravity, and to use this information to analyze near-limit phenomena at normal gravity conditions.

Task Description:
The experiments will employ a counter-flow burner geometry to provide a nearly adiabatic flame with well controlled strain, for conditions that cannot be achieved in normal gravity. Both premixed and diffusion flames will be studied at various pressures, and Laser Doppler Velocimetry, Particle Image Velocimetry and high-speed cinematography will be used to measure the flow fields. Models will be developed for these cases for comparison purposes. In addition, flame interactions will be studied, and numerical simulations of flame propagation in tubes in spherical vessels will be performed. Experiments will be conducted at normal gravity, in the 2.2 Second Drop Tower, and aboard the DC-9 aircraft.

Task Significance:
The research will allow for the first time a study of flames burning near their flammability limit (the concentration of fuel in air that just forms a flammable mixture) without the complications of buoyancy, wall heat losses or time-varying strain. It will provide fundamental data about the flammability limits that can be used to verify theoretical calculations, and has applications to turbulent combustion such as that found in engines.

Progress During FY 1995:
Progress this year consisted mainly of building up the drop rig and performing initial drops. The ignition process was tested by using two different approaches, namely a hot wire and a spark igniter. Results show that clearly the use of hot wire leads to larger ignition time, so it was decided to use a spark for ignition during the drop tests. The flow oscillator, to be used in unsteady experiments, was constructed and extensively tested. After initial LDV measurements, we are now in the process of modifying the shape of the oscillator in order to come as close as possible to a sine wave.

The drop rig was completed. It consists of two opposed jet burners with co-flow (30 mm Φ), on board flow control, data acquisition system, camera to record flame shape, spark ignitor on retracting arm, and photo diode to record extinguishment. In addition, a PIV seeder was completed at Lewis for bench tests, and will be incorporated into the rig for the next drops.

The rig was completed at USC and shipped to NASA Lewis for drops in April, 1995. Unfortunately, the rig had a number of deficiencies which were identified by the NASA technicians and major reconstruction is being done at Lewis. However, after rework some drops with successful ignition were accomplished, but parameters were not set correctly to obtain flame extinguishment during the short drop time.

Meanwhile, a new approach is being considered for the determination of laminar flame speeds by utilizing the nearly-ideal plug flow exit velocity profile of the present burners. This has not been done before, and it allows the approach of the flame very close to the burner exit. Under such conditions the flow divergence can be minimum and the nozzle exit velocity will give us the true laminar flame speed.
Substantial numerical work has been completed on stretchless curved flames (spherical and cylindrical) in an attempt to assess the effect of curvature and radiation on the burning of weak flames. In addition to steady calculations, unsteady calculation has also been done in order to assess the combined effect of radiation, curvature and unsteadiness.

**STUDENTS FUNDED UNDER RESEARCH:**
- BS Students: 0
- MS Students: 1
- PhD Students: 1

**TASK INITIATION:** 5/94  **EXPIRATION:** 4/98

**PROJECT IDENTIFICATION:** 962-22-05-50

**NASA CONTRACT NO.:** NAG3-1615

**RESPONSIBLE CENTER:** LeRC

**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

*Proceedings*

Effects of Gravity on Sheared and Nonsheared Turbulent Nonpremixed Flames

Principal Investigator: Prof. Said E. Elghobashi
University of California, Irvine

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
To answer fundamental questions regarding the effect of buoyancy on turbulent combustion. These questions include:

1) How does buoyancy affect the small-scale structure of the scalar and velocity fields?
2) How does buoyancy affect the distribution of scalar dissipation near the reaction zone?
3) Is the flamelet model valid for buoyant turbulent diffusion flames?
4) How does flame extinction depend on the appropriate non-dimensional numbers (Fr, Da, Re, etc.), the magnitude of the reaction energy, and the direction of gravity relative to the fuel-air interface?

Task Description:
This research is entirely computational. It will focus on direct numerical simulation (DNS) of two cases of flow: an initially-homogeneous turbulent flow without shear, and a flow with initially uniform shear. In both cases separate, parallel streams of fuel and oxidizer will enter the computational domain and react. Numerical models that have already been developed by the Principal Investigator will be extended to these cases with principal additions to include finite rate chemistry and non-zero gravity. The results will be presented in the form of diagrams based on the appropriate nondimensional numbers.

Task Significance:
This work will answer fundamental questions about how buoyancy induced by gravity affects turbulent combustion. In particular, gas, temperature, and velocity distributions can be very different without the flows caused by gravity, and this research will focus on revealing the small-scale structural differences in the flame, as well as probing the dependence of flame extinction on the alignment between gravity and the fuel-air interface. With this information, mathematical models of turbulent diffusion flames can be modified to better account for buoyancy effects. Better understanding of the physical processes will lead to improved combustor design and enhanced predictive capabilities regarding fires on Earth and in a space environment.

Progress During FY 1995:
The code was successfully ported to the C90 at NASA Ames, which offers better performance than the Y-MP previously used. However, tests using four-dimensional arrays to make the code more user-friendly proved to slow it down, so the original 1-D arrays were kept. The energy and species equations (presented in the original proposal) were rederived from first principles to ensure their consistency with the assumptions. No errors were found. We normalized these equations and formed the dimensionless parameters, e.g. Froude and Damkohler numbers. Using the code we obtained homogeneous isotropic fields of velocity and species concentration in fully-developed isotropic turbulence without chemical reaction. These fields were then used as initial conditions for the reacting flow simulation. The code was tested using a one-step reaction with finite rate including source terms (heat release and chemical reaction rates) in the governing equations. We also tested the code with low, moderate, and high Damkohler numbers to examine the reaction zone structure and resolution requirements. Good preliminary results were obtained for a coarse grid (N=64³ points), and later with a finer grid (96³ points), and a Reynolds number = 25 (based on Taylor microscale) and two different Damkohler numbers (1 and 10).
In order to study the effects of the finite rate chemistry on the flow behavior, the input/output and restarting modules were modified in the code. The input and restarting procedures were modified to allow for flexibility in changing time step sizes between runs. The output is enhanced for the additional quantities related to the newly introduced scalar (fuel mass fraction). A post-processing program was developed for reading the output data of the numerical simulation, calculate additional quantities (e.g., pdf of angles between scalar gradients and the principal axes of strain rate), then provide output files that are ready for visualization using Tecplot graphics.

The effect of gravity was studied by introducing a gravitational acceleration of magnitude $0.1g$ ($0.981 \text{ m/sec}^2$) to the original fast chemistry code. The boundary condition in the direction of gravity is modified to account for the non-symmetric flow behavior. Primary results show dramatic enhancement in turbulent activities as can be seen in skewness, turbulent intensity, and vorticity.

Test runs (for the finite rate case) are now being performed to determine the parameter ranges (Damköhler number, Zeldovich number, and the heat release parameter) that can be resolved by the grids now available ($96^3$ and $128^3$). The effect of gravity is studied by examining various quantities in the wavenumber space, including the energy spectrum, the transfer function, and the source/sink term due to gravity (buoyancy).

We are currently working toward understanding the fundamental physics of the multi-way interaction between turbulence, chemical reaction and buoyancy in a nonpremixed flame. The reaction rate is finite (Damköhler number < infinity) and the flow is without shear (equivalent to grid-generated turbulence). The results were obtained from a $96^3$ mesh and initially homogeneous isotropic turbulence with $Re_\theta = 25$, and the nondimensional energy release parameter, $Ce$, is prescribed such that the ratio $T_{flam}/T_\theta = 6$. These conditions insure the resolution of the Kolmogorov scale motion throughout the simulation. The effects of buoyancy on turbulence were studied by examining the spectra of turbulence energy in horizontal planes (i.e., normal to the gravity direction), as well as distributions of vorticity, enstrophy, temperature, variance of scalar (mixture fraction) and scalar dissipation. Discussion of these results is available in the cited paper.

**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

*Proceedings*
II. MSAD Program Tasks — Ground-based

Combustion of Electrostatic Sprays of Liquid Fuels in Laminar and Turbulent Regimes

Principal Investigator: Prof. Alessandro Gomez
Yale University

Co-Investigators:
Long, Prof. M.B.
Smooke, Prof. M.D.
Yale University

Task Objective:
The focus of this project is the study of the formation and burning of sprays of liquid fuels at both normal and reduced gravity. The objective is to synthesize gradually the complexity of practical combustion systems, using well-defined sprays to generate controlled clouds of droplets to be burned in well-characterized combustion environments.

Task Description:
The program is strategically articulated in three parts: an experimental one at normal gravity, in the combustion laboratories at Yale; an experimental part at reduced gravity, in the 2.2s drop tower at NASA-Lewis, and a computational part that parallels the experimental components. The microgravity experiments are aimed at studying the same types of sprays that have been previously examined at normal gravity in an environment free of natural convection effects. The numerical modeling is pursued to facilitate the interpretation of the experimental results and to complement the measurements, especially in the microgravity phase of the project, in which the constraints on available diagnostic techniques are severe.

Electrostatic sprays and ultrasonic nebulizers are used as atomizers. Experiments are conducted first in counterflow and co-flow laminar diffusion flames.

Task Significance:
To improve the understanding of spray combustion using well-defined sprays and combustion environments, which will ultimately lead to improvements in the practical implementation of liquid fuel atomization, for example in diesel or liquid-fueled rocket engines.

Progress During FY 1995:
1) The experimental study on the structure of an axis-symmetric laminar spray flame using the electrospray was completed. The flame appearance and temperature field showed little or no evidence of individual droplet burning. Rather, internal group combustion was experienced, in which the bulk of droplet evaporation occurred in an inner core that provided vapor diffusing into a flame enveloping the bulk of the droplet cloud. Droplet life-histories showed that the droplets follow the d-square law during a significant part of their lives with relatively constant evaporation coefficients on the order of 0.6 mm²/s. The experimentally-inferred evaporation coefficient is within the margin of error of that calculated in the case of isolated droplet evaporation in a convective environment. We conclude that, although the interactive effect between the droplets results into a flame enveloping a droplet cloud, individual droplet evaporation still applies within the cloud, that is the droplet interaction is not sufficiently intense to cause a departure from the d-square law.

2) After retrofitting the co-flow axisymmetric spray diffusion flame burner to a drop-module for the Lewis 2.2s Drop Tower, we performed seventeen drop tests to investigate the effect of gravity on the structure of these flames. Co-flow flames are significantly affected by gravity. Flames are (between 40% and 100%) longer and wider in microgravity as compared to normal gravity, similarly to what was observed for gaseous diffusion flames and despite the fact that previous gas-phase velocity measurements at normal gravity indicated that the spray flames, at least initially, are not buoyancy controlled.
3) One of the short-term objectives of this project was that of replacing the elecrospray in a counterflow spray burner with an ultrasonic nebulizer. With such device the available range of flow rates would be sufficient to explore regimes of flame extinction and droplet-droplet interactions under conditions of small slip between the two phases that are amenable to numerical modelling. After adapting a Sono-Tek nebulizer to the counter-flow flame burner, we succeeded in establishing a self-sustained flat flame after numerous modifications to the burner. Tests are now being performed on flames operated at a liquid fuel flow rate of 2 cc/min and at a strain rate of 30 s⁻¹, i.e. roughly one order of magnitude above what could be achieved with the electrostatic spray.

4) The computational modelling of the spray flames is progressing. After some unsuccessful attempts at modelling heptane counterflow diffusion flames, we concluded that the detailed chemistry mechanism that we were using was inadequate. We then tried methanol spray flames whose chemistry had already been validated in vapor diffusion flames studies. The counterflow spray diffusion flame code promptly converged using as boundary conditions input from the experimental portion of the program. Good agreement with the experimental data was obtained provided that the liquid fuel fraction (the percentage of liquid in the liquid phase at the burner mouth) was kept (artificially) low.

5) In preparation for experiments on turbulent spray combustion, single point Raman thermometry for turbulent spray diffusion flames was developed with the objective of measuring mean and fluctuating components of the temperature field pointwise and in the presence of droplets. The technique entails using the ratio of the spectrally integrated Stokes/Antistokes Raman scattering intensity, as detected by two photomultipliers interfaced with boxcar integrators. A counterflow gaseous diffusion flame burner was also constructed to provide a well-defined environment in which the Raman thermometry is calibrated against accurate thermocouple measurements. Preliminary experiments suggest that we can measure average temperature with an accuracy better than 2%. Interference from elastic light scattering contaminating the signal at Raman wavelengths can be eliminated using optical filters and some signal conditioning. Current efforts are focused to minimize laser-induced gas breakdown in the most dense region of the spray.

6) We completed a manuscript on high resolution measurements of droplet size by laser light scattering. The technique entails imaging on a photodiode array the angular scattering intensity collected by a lens and applying a Fast Fourier Transform (FFT) to the array serial output to recover an effective frequency and phase corresponding to the number and position of the scattering lobes. The method yields the determination of size with an accuracy better than 1%, if the index of refraction is known and constant. The technique can be in principle applied in real time at data rates as high as tens of KHz with modest equipment investment. Errors associated with particle trajectory effects and changes in the index of refraction were also considered. Results appear not to be affected by the former, whereas variations of the refractive index over a range typical of an evaporating droplet cause a deterioration of the sizing accuracy to approximately 3%. An article was submitted to Applied Optics. Further details on both the fundamentals of the electrospary and its application to combustion are given in the two Ph. D. dissertations that have resulted from this project.

**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Journals**


II. MSAD Program Tasks — Ground-based

Discipline: Combustion Science

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Presentations


II. MSAD Program Tasks — Ground-based

Discipline: Combustion Science

Three-Dimensional Flow in a Microgravity Diffusion Flame

Principal Investigator: Prof. Jean R. Hertzberg
University of Colorado, Boulder

Co-Investigators:
Linne, M.A.
Colorado School of Mines

Task Objective:
The objective is to study the influence of buoyancy on the three-dimensional fluid dynamics of the interaction between a diffusion flame and the elliptic vortex rings formed by pulsations of the fuel flow issuing from an elliptic nozzle.

Task Description:
A methane diffusion flame burning in atmospheric conditions is investigated. Elliptic vortex rings are generated by a combination of periodic flow perturbations (active forcing) and the elliptic geometry of the fuel nozzle (passive forcing). The fuel flow is forced by an in-line 100 watt loudspeaker system at frequencies up to 1 Khz. Phase-locked flow visualization and particle image velocimetry (PIV) are the primary diagnostics.

Task Significance:
Techniques of active and passive control of the fluid dynamics in diffusion flames have the potential to enhance combustion efficiency and reduce pollutant formation. Potential applications include industrial and residential burners and gas/liquid fueled engines.

Progress During FY 1995:
The overall objective of this work is to understand the fluid dynamics of the interaction between large scale, three-dimensional vortex structures and a transitional diffusion flame in the microgravity environment. The vortex structures are used to provide a known perturbation of the type used in active and passive shear layer control techniques. Active forcing refers to techniques which add acoustic or kinetic energy to the flow in order to influence the shear layer vortex dynamics. Passive forcing relates to the manipulation of geometry to influence the three-dimensional vortex dynamics.

A combustor incorporating these techniques was designed and completed during the past year. Passive forcing is provided by a 2:1 aspect ratio elliptic cross-section nozzle. The elliptic nozzle is designed to minimize flow separation while providing the three-dimensional flame. Active forcing is provided by a loudspeaker in the combustor plenum. A flow seeding technique was developed that uses aspirated vegetable oil to visualize the flowfield. Canola oil was found to be most effective.

A preliminary combustor using an actively forced, axisymmetric flow has been under investigation during the past year. Experiments have shown that under specific forcing conditions the yellow diffusion flame splits into two or more flames that are shorter and mostly blue in color. This behavior has also been observed in the preliminary tests of the microgravity combustor. The flame exhibits both splitting and bifurcation behavior. At high forcing levels the flame is split 100 percent of the time. At lower forcing levels the split state is intermittent with a classical diffusion flame state, indicating a bifurcation.

Video imaging was used on the preliminary combustor to characterize the flame splitting phenomenon as a function of flowrate, forcing frequency, and forcing amplitude. The frequency response of the preliminary combustor was measured with a sound pressure level (spl) meter. The frequency response of the combustor when filled with methane was found to be significantly different than when it was filled with nitrogen due to the differences in the speeds of sound in the two gases. Preliminary results indicate that flame splitting behavior coincides with peaks in
the sound pressure field suggesting that the phenomenon may be found at other frequencies if the amplitude could be driven high enough. However, the amplitude is not the only controlling factor because flame splitting was not observed above 300 Hz even though the frequency response indicated the existence of amplitude peaks comparable to those found below 300 Hz.

A three-dimensional visualization technique is currently under development which will provide a better understanding of the flame splitting phenomenon as well as the three-dimensional microgravity flame. A Nimslo photographic camera has been adapted for close-up stereoscopic use. An ultrasonic flow seeder is being developed that will provide effective seeding independent of fuel flowrate.

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BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Proceedings
Unsteady Numerical Simulations of the Stability and Dynamics of Flames in Microgravity

Task Objective:
Study the structure (multidimensional) and dynamics (propagation, stability and extinction) of premixed flames in micro- and normal-gravity environments.

Task Description:
Perform detailed numerical simulations using time-dependent, one- and two-dimensional flame models. These models solve the multispecies coupled partial differential reactive-flow equations, including fluid, thermodynamic and transport properties, chemical kinetics, radiative heat transfer, buoyancy and boundary heat loss effects. The investigator will a) systematically isolate and evaluate the relative importance of various physical and chemical processes that may be controlling the structure and dynamics of premixed flames, and b) compare simulation results qualitatively and quantitatively with experimental observations.

Task Significance:
Small amounts of fuel leaking from fuel cells could pose a fire hazard on the space-shuttle and similar vehicles. Therefore, understanding the burning and extinguishment characteristics of flames are important for the safe operation of space vehicles. The knowledge gained from such studies is also useful for the safe and efficient use of fuels on Earth. Computer simulations are a cost effective way to obtain data to corroborate limited experimental observations and provide additional details which are difficult to obtain from space-based experiments.

Progress During FY 1995:
The newly developed parallel version of the flame code is being used to study the detailed dynamics of the extinguishment of downward-propagating methane-air flames. The plan is to compare these observations to those made in the earlier NRL study of the extinguishment of hydrogen-air flames. Such a comparison will be helpful in isolating effects that are specific to the highly diffusive and easily combustible hydrogen-air mixtures from those that are more general and applicable to a variety of fuel-air mixtures. Furthermore, it will also provide some information on the relative role of conductive and radiative heat losses in extinguishing flames propagating in tubes. The earlier NRL studies of one-dimensional, zero-gravity flames in lean methane-air mixtures showed that radiative losses alone can extinguish some flames. In this study of two-dimensional flames, radiative and conductive losses will be considered together and separately to isolate their effects.

The cellular structures observed in lean methane-air mixtures (5-6%) were very weak when compared to those in lean hydrogen-air flames of comparable burning velocities. Therefore, after discussions with Professor Paul Ronney, a CO₂ diluted methane-oxygen mixture, with an effective Lewis number of about 0.70, was chosen for studying a case which should exhibit strong cellular structures. Both the simulations and Ronney's experiments indeed indicate well defined cellular structures. However, the detailed structures are not quite the same in the two cases. Computed burning velocities are larger than the experimental ones; however, the flame temperatures are about the same. Three-dimensional effects, insufficient numerical resolution and inadequacy of the chemical kinetics scheme to adequately capture the effects of CO₂ dilution are possible explanations for the observed differences.
The variation of the burning velocity with dilution is being studied to see if the "skeletal" mechanism used in the computations is adequate to represent the chemical effects of dilution with CO2. This work is hampered by the lack of experimental data on CO2 dilution effects. Further tests with N2 dilution are underway to check if the reaction scheme is adequate for any dilute mixture. Tests in which the equivalence ratio was varied showed that the scheme was adequate for those cases.

Currently, studies of near limit downward-propagating methane flames are continuing. Discussions with Prof. Paul Ronney and others at the International Combustion Symposium identified the third-body efficiencies in the chemistry scheme as a possible source of the differences observed earlier between Paul's results and ours for the extinguishment of CO2 diluted methane flames. It was decided to pursue this matter further using the FLAME1D code before performing more detailed multidimensional simulations of the extinguishment of methane-air flames. Different values for the third body efficiencies suggested in the literature have been incorporated into the codes. A repeat of stoichiometric methane-air flames shows that these parameters do not make any difference in the results. However, a repeat of the simulations of the CO2 diluted mixtures used in Ronney's experiments shows that these parameters have a strong influence on the flame temperatures and velocities. In fact, it has been difficult to even ignite one of these mixtures. NRL results to date show that the burning velocities of near-limit CO2 diluted mixtures are now lower than Ronney's. Furthermore, more than one combination of third-body efficiencies could give the same overall results. Further simulations have shown that various intermediate values for the burning velocities can be obtained with different choices for the individual third-body efficiencies. Now, it is clear that the exact values of the third body efficiencies could play a crucial role in obtaining accurate results for near limit mixtures. There is sufficient variation in Ronney's experimental data that it by itself cannot be used to determine the required third-body efficiencies. Discussions with other scientists are currently underway to determine how accurately these parameters are known. In order to determine if the problems encountered above with the third-body efficiencies is primarily restricted to CO2 mixtures, flames in lean methane-air mixtures were recomputed with the new values for the efficiencies. These studies show that they do affect the results. Calculations with a 5% methane-air mixture now show that it is not possible (for the range of parameters tried) to obtain a steadily propagating flame in this mixture when radiation and non-unity third-body efficiencies are taken into account. The lean-limit obtained with the current values for the third-body efficiencies is between 5.1 and 5.2% methane in air. Further one-dimensional simulations show that the zero-gravity limit is around 5.15% and the downward-propagating limit is around 5.20%. These numbers are in agreement with drop-tower and KC-135 results of Professors Streibow and Ronney.

Two-dimensional studies of the flame structure have resumed. A 56.5% CO2 diluted mixture was chosen for further studies since the burning velocity of a planar flame in this mixture is about 8 cm/s and the burning velocity of near-limit mixtures in Ronney's experiments were about 8 cm/s. What NRL wants to focus on here is to examine if the computed structure of the flame is similar to the one observed in experiments if near-limit mixtures with comparable burning velocities are chosen. Note, however, that the actual dilution of the near-limit mixtures in the experiments varies between 57.44 and 58.34% CO2. As discussed previously, this difference is attributed to the uncertainties in the exact values to be used for the third body efficiencies. NRL's current simulations show a nearly straight flame with multiple cells and is similar to those observed in the experiments. Longer calculations show that the size of the cells is comparable to those observed in the experiments. We observe 5 complete cells in a 5.1 cm channel and 10-11 cells are observed in the experiments using a 10 cm diameter tube. These results are presented at the 1995 AIAA Aerospace Sciences Meeting.

The focus of the current research is on developing a three dimensional version of the flame code. This is expected to take several months. Since NRL has adopted the time-step or process splitting approach to solve for the various physical and chemical processes, the code is very modular. The various processes can be worked on individually and generalized into three dimensions. The first process to be tackled is convection. A three-dimensional version of the basic Flux-Corrected transport algorithm has been implemented. Currently, the BIC (Barely Implicit Convection) part of the convective transport is being worked on.
II. MSAD Program Tasks — Ground-based

Discipline: Combustion Science

STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 1/95   EXPIRATION: 1/98
PROJECT IDENTIFICATION: 962-22-05-22
NASA CONTRACT NO.: NASA-DPR
RESPONSIBLE CENTER: LeRC

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals


Presentations


Soot and Radiation Measurements in Microgravity Turbulent Jet Diffusion Flames

Principal Investigator: Prof. Jerry C. Ku
Wayne State University

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
The objectives of this study are to determine modeling coefficients from measurements of soot morphology and radiation in both normal and reduced-gravity turbulent gas jet diffusion flames, and to further refine models for soot formation and spectrally dependent radiation properties.

Task Description:
In the area of experimental measurements, thermophoretic particle sampling and subsequent transmission electron microscopy analysis are used for soot particle size and analysis of aggregate morphology. Laser light absorption imaging provides for the determination of soot volume fractions. Emission imaging using isolated bandpass filters will be used to measure spectrally dependent soot radiation properties and possibly temperature. Rapid insertion of fine-wire thermocouples will also be used for the determination of temperature.

In the area of modeling, Favre-averaged boundary layer equations with a k-e-g turbulence model and conserved scalar approach with an assumed probability density functions (pdf) are used to predict flow field and gaseous species mole fraction profiles, respectively. The soot formation model has been modified and tested to predict soot volume fraction and number density. The energy equation is included to provide a full coupling between flame structure and radiation analysis. A third-order spherical harmonics approximation and the YIX method have been applied to solve the radiative transfer equation. In the proposed study, soot formation and radiation models will be improved, and methods for efficient spectral integrations and iteration between the solutions for the flame structure and the radiative transfer equation will be sought.

Task Significance:
Microgravity Combustion is not only relevant to fire safety on board a spacecraft, but also provides a unique condition for a better understanding of combustion fundamentals of this common type of combustion.

Progress During FY 1995:
There is no progress to report at this time as the project has not started yet.
II. MSAD Program Tasks — Ground-based

Sooting Turbulent Jet Diffusion Flames

Principal Investigator: Prof. Jerry C. Ku

Wayne State University

Co-Investigators:
Greenberg, P.S.

NASA Lewis Research Center (LeRC)

Task Objective:
The objectives of this study are to model soot formation and radiation for turbulent jet diffusion flames and to determine the modeling coefficients from measured data under both normal and reduced-gravity conditions.

Task Description:
In regard to experimental measurements, thermophoretic particle sampling and electron microscopy are used for soot particle size and aggregate morphology analysis. Laser light absorption imaging provides for the determination of soot-volume fractions, and emission imaging and thermocouple measurements will be used for soot thermometry. Laser Doppler Velocimetry may possibly be employed to measure velocities and turbulence intensities, but is beyond the stated deliverables of this effort.

In the area of modeling, Favre-averaged boundary layer equations with a k-e-g turbulence model and the conserved scalar approach with an assumed pdf (probability density function) are used to predict flow field and gaseous species mole fraction profiles, respectively. Transport of soot particles is described by equations for volume fraction and number density using rate equation models. The energy equation is included to provide coupling between flame structure and radiation analyses. The radiative flux is solved from the radiative transfer equation (RTE).

Task Significance:
Microgravity combustion is not only relevant to fire safety on-board a spacecraft but also provides a unique condition for better understanding of fundamentals of this common type of combustion.

Progress During FY 1995:
The YIX method was applied to solve radiative heat transfer in a finite cylindrical enclosure with a nonhomogeneous, nongray, emitting, and absorbing soot/CO2 mixture. Numerical results were obtained for radiative heat transfer within a turbulent ethylene jet diffusion flame from precalculated flame temperature, soot volume fraction, and CO2 concentration maps. Soot only, CO2 only, and combined cases were examined over the spectral range of 1-20 microns. Soot absorption coefficient spectra were calculated from soot volume fraction and refractive indices according to the Drude-Lorentz dispersion model based on the three frequently cited dispersion parameter sets. Results from these three dispersion parameter sets show that the difference in maximum flux is 51%. Thus, current uncertainties about soot spectral refractive indices are the main limitation on accurate estimates of the radiative heat transfer from sooting combustion systems. The exponential-wide-band model is used to calculate the CO2 absorption coefficient spectrum. Overall, the contribution of soot radiation is only two to three times that of CO2. Consequently, both soot and CO2 contributions are significant, and must be accounted for. For CO2 gas, only contributions around absorption bands at 2.7 and 4.3 microns are significant. It seems that spectral contributions from the range above 5 microns may be neglected without any significant loss of accuracy in evaluating total radiation properties. This work was submitted to the ASME Journal of Heat Transfer. In addition, an improved method for handling the discontinuity between the velocity of fuel and co-flow has been implemented. The agreement between model prediction and Prof. Santoro's co-flow soot volume fraction data (already in the literature) is very good now.

The fabrication of a coaxial, piloted burner for turbulent gas jet diffusion flame studies has been completed. Extremely small tubing diameters (n 0.75 mm inner diameter for the inner jet) were employed to obtain large
II. MSAD Program Tasks — Ground-based

Reynolds numbers while retaining a reasonable overall flame height compatible with the dimensions of the combustion chamber on the present 2.2 second laser rig. The major challenge was to provide a stable, coaxial arrangement of the inner jet and pilot nozzles. This was accomplished by the staff of the LeRC instrumentation shop, who employed an arrangement of laser-welded thin wire spacers to accurately control the relative position of the inner and outer tubes. After welding the spacers in place, the thin wires were then ground to the appropriate diameter to fit accurately into the pilot tube. The spacers also serve as flow straighteners for the pilot flow. Initial tests indicate that adequate symmetry has indeed been achieved.

A series of drops were conducted in the NASA LeRC 2.2-Second facility to obtain the soot volume fraction in reduced gravity laminar jet diffusion flames. Tests indicated a reduction in local volume fraction for 1.5 mm jets using ethylene as a fuel at flow rates between 1.0 and 3.0 cc/sec. This resulted in volume fraction signals (obtained via light extinction measurements) with poor SNR's. Larger diameter burners were fabricated to provide longer optical path lengths, thus increasing signal strengths. Limitations on flow rate due to the capacity of the rig and the resulting flame lengths proved to be disadvantageous because stable flames could not be achieved in the available test time. Apparently Reynolds number considerations separate two different regimes wherein these flames are either diffusion or momentum dominated. The former evolve too slowly to be studied in the 2.2 second facility; the latter produce insufficient volume fractions for tractable flow rates and flame heights. Switching to acetylene as the fuel increased the soot production to the level wherein reasonable SNR's could be obtained. Some issues relating to the stability of these flames as a function of burner diameter and flow rate under reduced gravity conditions remain, and are presently being pursued. The hardware has been performing consistently well, as has the facility's newly refurbished air-bag deceleration system.

Test were performed relating to the input/output properties of the Betacam video recording system being utilized for reduced gravity soot absorption measurements. In particular, the absolute gain and offset of the recorded signal was examined to a resolution of 8-bits. It was determined that this fidelity could be achieved through appropriate calibration and attention to procedural details. The resulting performance negates the requirement for on-line digitization in the 2.2 second facility, insofar as 8-bit, monochromatic signals are concerned.

To establish the validity of data reduction methods presently being used in soot volume fraction measurements via laser light extinction, Professor J.C. Ku et al at Wayne State University have approached this problem by synthesizing forward-propagated data for a 2.3 cc/sec laminar ethylene diffusion flame. A soot volume fraction map was generated by numerical simulation; this result was then employed to generate simulated 2-D projection data. Spatially random noise was added to this data to replicate the effect of spatial noise found in our actual data sets. Noise amplitudes representing 5, 10, and 20% of the original signal were added before tomographically inverting the 'noisy data.' Agreement with the original soot volume map was obtained in all cases to within 5%.

As a further cross check on soot volume fraction data obtained via imaging laser light extinction measurements, the test conditions reported by Santoro, et al in their 1983 Combustion and Flame paper were repeated. After correcting the results to account for the wavelength dependence of the absorption cross sections, excellent agreement was obtained with Santoro's original data. The spatial smoothing algorithms being utilized are being investigated in more detail. The IMSL lower-order B-spline polynomial is observed to preserve the shape and magnitude of the absorption peaks, but to bias their spatial locations. Linearly weighted spatial convolutions of various sizes appear to handle this aspect more favorably, and continue to be pursued. The effect of constricting the angular collection specification has produced no measurable effect on 1-g laminar ethylene diffusion flames, as was anticipated.

Experiments to fully demonstrate and characterize the technique of soot volume fraction extinction imaging have been completed. The range of topics investigated includes: 1) a study of data inversion methods, including angular sufficiency criteria, spatial smoothing algorithms, and noise tolerance, 2) temporal averaging vs. single-shot data, 3) the specification of forward-scattered angular acceptance, and 4) the use of a moving spatial diffuser to corrupt coherent interference effects. Data was also obtained to compare to previously published values for ethylene/air coflow-stabilized flames. These results are being summarized in a manuscript for journal submission.

Having successfully addressed issues relating to both data acquisition and fuel delivery systems, 2.2 second drop
tower tests to study soot volume fractions in laminar jet diffusion flames were repeated. Tests were conducted for both pure acetylene and acetylene diluted with nitrogen. While soot volume fraction data was successfully acquired and analyzed, these tests will be repeated and studied for repeatability. The low pressures required by Safety for acetylene mixtures has been shown in previous drops to require finesse in mixing, filling, and igniting. Analogous 1g tests have also been completed.

The analysis of initial soot volume fraction data comparing 1g and 0g laminar, acetylene/nitrogen (50/50) jet diffusion flames was then undertaken. A number of interesting features are observed: 1) the 1g flame is non-smoking, whereas the corresponding 0g flame smokes heavily; 2) the 0g flame generates more soot overall; 3) the 0g flame generates more soot on a per volume basis, and 3) the soot shell occurs at significantly greater radial locations in the 0g flame. Prior to publication, it is felt by the investigators that complementary soot temperature data is required. The collection of this data is presently in progress.

The piloted, turbulent diffusion flame burner designed to conduct normal and reduced gravity experiments relating to sooting turbulent jet diffusion flames has been successfully demonstrated. In these tests, ethylene was used as the fuel for both the main fuel jet as well as the coaxial pilot jet. Extremely stable operation was obtained for a primary/pilot mass flow rate of 100:1. It is not possible to initially light the pilot flame at this low flow rate, which corresponds to 0.1 cc/sec. The pilot flow must be initiated at twice this rate and then be reduced to the final value after stabilizing the primary jet. The Reynolds number of the primary jet is presently on the order of 3000.

The soot formation model used by Prof. J. Ku and co-workers at Wayne State University has been shown to accurately fit a volume fraction map when experimental data has been used to provide rate formation coefficients. The ability of the model to correctly predict volume fractions for differing flow rates using the same rate coefficients is not as successful. Modifications to the existing formation model, and alternate models are being investigated. In other results, the YIX method for calculating radiative transfer has successfully shown to demonstrate convergence on the iterated temperature field between the energy and radiative transfer equations when the Roseland mean (i.e. averaged over the wavelength spectrum) is employed.

Modeling analyses for soot formation and radiation in gas jet diffusion flames have been completed for both laminar and turbulent cases. Rate-equation models are used to model detailed soot transport. Local radiation fluxes from soot and gases are solved using the YIX method (which has been demonstrated to be as accurate as the Monte Carlo method) with the Rossland mean absorption coefficients. Soot radiation and flame structure analyses are coupled through an iteration process with convergence based on flame temperature. So far, the major conclusions are: 1. By optimizing the rate coefficients in soot formation models, good agreement are found between model predictions and published data for a laminar and a turbulent ethylene flames. 2. Compared to the data we obtained using the imaging absorption technique for acetylene laminar and turbulent flames, the model made reasonably good predictions with only some slight adjustments of ethylene rate coefficients - indicating that the model has pretty good generality. 3. Based on 1g vs ug comparisons based on our data, using the same rate coefficients for 1g and ug flames of the same fuel and flow rate, the model again made reasonably good predictions - again indicating that the model has pretty good generality in terms of its accountability for buoyancy effects.

**STUDENTS FUNDED UNDER RESEARCH:**
- BS Students: 0
- MS Students: 1
- PhD Students: 3

**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Journals**

Printed: 28 FEB 1996
II. MSAD Program Tasks — Ground-based

Discipline: Combustion Science


Proceedings

Presentations
Studies of Flame Structure in Microgravity

Principal Investigator: Prof. Chung K. Law
Princeton University

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
The objectives of this work are to understand and quantify the structure, stabilization mechanisms, soot formation in, and extinction of one-dimensional premixed and non-premixed laminar flames.

Task Description:
This program comprises two main elements:

1. A numerical simulation of one-dimensional laminar flames is to be developed that, in addition to including the usual fluid mechanical and heat transfer mechanisms, will include detailed chemical kinetic mechanisms for comparison with the unique experimental results.

2. A drop-tower text apparatus is to be used to observe premixed laminar flames stabilized about cylindrical and spherical porous burners to distinguish heat loss and flow divergence influences on flame stabilization and flame front stability.

Task Significance:
The one-dimensional adiabatic laminar unstretched premixed flame is a fundamental precept of combustion science, but cannot be stabilized in normal gravity because of the straining or asymmetrical influences of gravitationally induced buoyant convection. In microgravity experiments, this program demonstrates this combustion paradigm and provides a capability for probing the structure, chemistry, soot dynamics, and flame propagation speed of these fundamental flames.

Progress During FY 1995:

1) The theoretical modeling of spherical burners for premixed and non-premixed flames was developed that provided predictions for behavior expected from drop tower tests. This model includes the rotation of the spherical burner creating a rotating flame with variable burner radii. The theoretical conclusions show that based upon variations in the mixture Lewis number (the ratio of thermal to mass diffusivities) from unity value, the flame response to burner rotation, namely either elongation or flattening with respect to the sphere rotation axis is changed in extent or direction.

2) A spherical burner capable of rotation was built for use in the 2.2 Second Drop Tower apparatus previously used for cylindrical burner tests. Tests with this burner were first conducted to consider premixed flames stabilized about a spherical porous burner, adding a degree of curvature to that of the cylindrical premixed flames previously studied. In addition, the spherical burner system will be rotated during tests to introduce controllable body forces into the flame during the drop tests. At year's end the test results were being compared to the completed theoretical model results.

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TASK INITIATION: 5/90 EXPIRATION: 11/97
PROJECT IDENTIFICATION: 962-22-05-37
NASA CONTRACT NO.: NAG3-1713
RESPONSIBLE CENTER: LeRC

Printed: 28 FEB 1996
II-345
II. MSAD Program Tasks — Ground-based

Discipline: Combustion Science

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals


Presentations
II. MSAD Program Tasks — Ground-based

Chemical Inhibitor Effects on Diffusion Flames in Microgravity

Principal Investigator: Dr. Gregory T. Linteris
National Institute of Standards and Technology

Co-Investigators:
Hochgreb, S.
Massachusetts Institute of Technology (MIT)

Task Objective:
The objectives of the proposed research are as follows:

1. To determine the effects of flame inhibitors on the physical characteristics (height, shape, color, and luminosity) and stability limits (ignition, extinction, lift-off and blow-off) of gaseous diffusion flames in the presence of halogenated fire suppressants in microgravity.

2. To develop quantitative analytical models for the observed behavior, including chemical kinetic effects, in order to understand the mechanisms of inhibition of halogenated compounds in gaseous diffusion flames.

Task Description:
The experiments will consist of normal and microgravity studies of laminar jet and co-flow diffusion flames inhibited by halogenated suppressant addition to the air or fuel stream. In microgravity, visual and temperature diagnostics will be utilized to detect flame shape, dynamics and stability limits. Normal gravity experiments will make use of additional chromatographic and spectroscopic diagnostics in addition to visual and temperature measurements.

Analytical and computational work will be performed using existing general 2-D codes with chemical kinetic models for comparison with the observed results. The calculations should serve for comparison with observations, explanation of observations and selection of additional experimental conditions of interest.

Task Significance:
Fire suppression in space is one of the main reasons to investigate combustion phenomena in microgravity. Since halogenated compounds will remain as the fire suppressant of choice in many space missions, there is clearly a need for experimental evidence on the effectiveness of these compounds under zero buoyancy conditions.

The final product of the experimental and analytical work is a comprehensive and quantitative understanding of the physical and chemical phenomena involved in the suppression of diffusion flames through the addition of chemical halogenated inhibitors. This understanding is expected to be very useful both from a fundamental viewpoint as well as for the practical utilization of chemical fire suppressants in space.

Progress During FY 1995:
During the first quarter, a combustion bomb at MIT was used to perform experiments and numerical modeling was performed on the laminar flame speeds of inhibited methane/air premixed flames at elevated temperatures and pressures. The work contributed with additional data for the verification of current halogenated inhibitor chemical models.

The first series of microgravity experiments was initiated in January 95, to investigate the effects of chemical inhibitors on methane laminar gas-jet diffusion flames. These experiments have covered a wide range of conditions, including variation in inhibitor, inhibitor concentration, pressure, oxygen concentration, fuel, and fuel flow rate.
Counterflow Diffusion Flame Experiments
Investigatory counterflow diffusion-flame experiments involving methane, halogenated inhibitors and oxidizer mixtures were performed at NIST in March. The apparatus consisted of opposed jets of fuel and oxidizer each about 20 mm in diameter and separated by about 15 mm. The velocity profiles of the jets were straightened by a series of stainless-steel screens in the inlet tubes. Methane and carbon monoxide/hydrogen were the fuels used, and CF3Br, CF3H, and C2F5H were the inhibitors added to the fuel or oxidizer sides of the flame. Hydrogen was added to the CO in quantities of around 1-2% to allow moist (rather than dry) CO oxidation. Strain rates from 45 to 300 1/sec were used, but most experiments were done at a strain rate of 75 1/sec.

The experiments showed that the addition of CF3Br produced a reddish zone offset from the flame by nearly a millimeter on the side to which the inhibitor was added. This zone is thought to originate from the decomposition of the inhibitor with the reddish color resulting from the emission spectrum of atomic bromine. This hypothesis is reinforced by the experiments with the fluorinated inhibitors, CF3H and C2F5H, which showed no secondary zone or reddish color. The addition of CF3Br also enhanced soot production while the fluorinated inhibitors tended to reduce soot production. In fact, sooting methane flames with CF3Br added showed three distinct regions—a thin blue flame, a slightly thicker red CF3Br zone on whichever side the inhibitor was added, and a thick bright yellow soot zone on the fuel side. As expected, extinction of the flame required about 3-4 times as much CF3H as CF3Br, and about twice as much C2F5H. The extinction strain rates of these experiments were lower than those of Seshadri.

Drop Rig / MIT Laboratory Experiment Construction
During April and May, a low pressure jet diffusion flame apparatus was built at MIT. It was designed for easy installation into a drop rig. Experiments should begin within a week; we will first duplicate the conditions of tests performed previously in the 2.2 Second Drop Tower, and then use the rig to explore regimes for study at low gravity. Future experiments will employ nonsooting fuels (CO and H2) as well as additional diagnostics.

2.2 Second Drop Tower Experiments
A second set of microgravity experiments were performed at NASA-Lewis during the first two weeks of June. These experiments were designed to fill in gaps in the experimental test matrix of the first visit to Lewis last spring, and to expand the test to regimes which appeared to be interesting based on the previous results. One subset of this session was aimed at studying the apparent suppression of soot with the addition of concentrations of 8% CF3H to the oxidizing mixture, compared to an uninhibited flame.

Experiments performed at 5% and 6% CF3H (intermediate between the previous results at 4% and 8%) show that there is a progressive decrease in the amount of soot observed at concentrations around 4-5%. In order to investigate whether the decrease in soot was primarily an effect of the corresponding decrease in O2 due to the addition of inhibitor, experiments were also performed with 8% N2 dilution, and also with oxygen enrichment (30% O2). The reduction in the sooting tendency was not as large with N2 as the diluent.

Another subset of experiments was aimed at determining the structure and extinction limits for methane diffusion flames with CF3Br addition to the oxidizer. Experiments were performed at 0.5% and 3% CF3Br, and 18% and 30% O2. The 3% CF3Br and the 1% CF3Br/18% O2 tests both showed extinction. Both flames lit, developed into the previously observed two-zone structure, then shrunken and faded away within about 0.5 seconds. The 18% O2 test was added to investigate the reduction of soot in the CF3Br inhibited flame, and provided information on the structure of the flame before it extinguished. The 3% CF3Br/30% O2 flame was stable, but had a long thin inner luminous structure which was unexpected based on previous results. A total of five 30% O2 flames were tried, but more are needed to draw more definitive conclusions. Finally, a subset of low-pressure (0.25 atm) flames were tested. Addition of 4% and 8% CF3H showed a marked increase in flame size. The low-pressure, 8% CF3H, 30% O2 flame exhibited a large blue reaction zone around a bright core.

Numerical Flame Structure Calculations
As a first step in understanding the structure of the inhibited flames, we have been using a counterflow diffusion flame code (obtained from M. Smooke) to obtain flame structure solutions which simulate conditions approximating those of our experiments. We hope to obtain solutions for both CHF3 and CF3Br inhibited flames for a range of strain rates. To date, efforts have been concentrated on CHF3.
Minor modifications of an existing chemical kinetic mechanism to make it suitable for use in the counterflow code were made; we have obtained a converged solution for a somewhat reduced mechanism and are now attempting to build to a solution using the complete mechanism for CHF3.

Fourth Quarter:
During July the low-pressure jet diffusion flame apparatus was completed here at MIT, and a set of experiments were performed. The experiments were designed to mimic the microgravity tests, using methane as the fuel, and having a 1.7 mm diameter nozzle and a Reynolds number based on nozzle diameter of 98. These experiments were aimed at determining a reasonable test pressure range for ground-based experiments, and finding limiting concentrations of CF3H for these conditions. A test pressure of 25 kPa was determined to be appropriate for these tests since it is low enough to avoid soot luminosity and buoyant instability, while staying significantly above the pressure at which the flame becomes unstable. Lower pressures would reduce buoyancy further, but would result in a weaker flame. The limit concentrations of CF3H for a laminar jet at Re=98 at 25 kPa were 4% with air, and 27% with 30% O2.

In August, a third set of microgravity experiments were performed at the NASA-Lewis 2.2 Second Drop Tower. This set of experiments was designed to clarify a few points from previous experiments and investigate potential effects of higher flow rates and CO fuel. The first important piece of information found was that the methane flame could be made open-tipped by lowering the oxygen concentration. We have found no mention of this effect in the literature.

Additional, better images were recorded of the flame behavior with 1% CF3Br by moving the camera closer to use more of the pixels, and setting the gain of the camera more accurately, aided by experience from previous experiments. Higher flow rate experiments were conducted at double the flow rate of the initial base case, corresponding to a cold-flow Reynolds number based on nozzle diameter of 196. These flames showed similar characteristics with respect to addition of inhibitor as the lower flow rate flames, but required more inhibitor to get the same effect. For example, the Re=98 flame showed the two-zone structure at 1% CF3Br, while AT Re=196 the two-zone structure was not evident at 1%, but only at 2% CF3Br. In addition the low flow rate (Re=98) flame extinguished at 15% CF3H, while the Re=196 flame persisted.

A number of experiments were also performed with a mixture of 99% CO / 1% CH4 as the fuel. Diffusion flames with this fuel at the same volume flow rate as methane (154 sccm) were very small, so the flow rate was doubled in subsequent tests(308 sccm). These flames proved to be easily extinguished by the inhibitor, blowing out at 3% CF3H, or 0.5% CF3Br. At these low inhibitor concentrations, no significant changes in structure could be observed. In later tests, the lower flow rate (154 sccm) flame appeared to be more stable, persisting at 0.5% CF3Br, whereas the higher flow rate flame would not, which is opposite the behavior observed for methane. However, no significant structural changes were observed.

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BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Proceedings


Printed: 28 FEB 1996
Structure and Dynamics of Diffusion Flames in Microgravity

Principal Investigator: Prof. Moshe Matalon
Northwestern University

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
The objectives of the work are to gain insight into diffusion flames by studying simple combustion systems. The emphasis of the work will be to understand the structure and dynamics of stationary spherical flames, and the coupled processes occurring in the liquid and gas phases associated with the burning of liquid fuels.

For gaseous flames, the Principal Investigator will investigate the processes that lead to extinction of these flames either by the process of blowoff or by radiative losses. He will also examine the nature of the interactions between two diffusion flames, the mechanisms leading to the generation of flame front instabilities, the formation of cellular flames and the effect of residual gravitational acceleration on microgravity diffusion flames.

For liquid fuel burning, the Principal Investigator will examine the possible generation of instabilities that are intrinsic to the vaporization process and identify the importance of thermocapillary motion on the overall burning process.

Task Description:
The program is entirely theoretical in nature. The Principal Investigator will work closely with related experimental investigations in the microgravity combustion science program to identify problems of specific interest.

The porous sphere will be the model problem for gaseous diffusion flames. A standard formulation of the conservation equations in spherical coordinates forms the base case. The model will then be extended to incorporate finite rate kinetics through activation energy asymptotics, enabling details of the flame structure to be elicited. The model can then be extended to include a standard form of a radiative loss to determine the extinction limits, blowoff and radiative, of the combustion system. Finally, the model will be extended to determine the effects of interaction by modeling two spherical diffusion flames in close proximity to each other employing bi-spherical coordinates in the approach outlined above.

As a next step the core model will be extended to incorporate stability theory and determine the conditions for the onset of flame-front instabilities similar to those that have been observed experimentally. Similarly, the effect of a low-magnitude, time-varying body, force (g-jitter) on the flame will be examined, by casting the disturbance as a sinusoidal term in the governing equations.

The ideas developed above will then be extended to liquid fuels. The major change to the model will be the incorporation of the liquid phase, the conservation equations for the liquid phase, and the coupling of the gas and liquid phases. A major addition will be the incorporation of thermocapillary motion in the liquid phase equations.

Task Significance:
The proposed research will lead to a greater understanding of diffusion flames in general. By working closely with experimental studies in the microgravity science program, the investigator will be able to give a theoretical foundation to many experimental observations of diffusion flames in microgravity.
II. MSAD Program Tasks — Ground-based

Discipline: Combustion Science

Progress During FY 1995:

Near Limit Oscillations of Diffusion Flames

An analysis has been carried out to explain the observed oscillations that occurred during the last stages of burning of a candle flame in a microgravity environment. The analysis is based on the assumption that at this stage the oxygen concentration is low and consequently the burning intensity is weak. We were able to establish that, in the presence of heat loss, oscillations similar to what has been observed in the experiments develop once the Damkohler number is reduced below a critical value. By further reducing the Damkohler number, the amplitude of these oscillations continue to grow presumably leading to extinction.

Annular Pool Fires

Annular pool fires admit various possible "stationary" combustion states. There is always the possibility of a quenched state and a pool burning; but occasionally one finds front propagation in a circulatory fashion. An analysis has been carried out in an attempt to explain the possibility of front propagation. Although an instability has been detected for a sufficiently large value of the Lewis number associated with the fuel and for an appreciable amount of heat loss, the instability found corresponds to pulsations and does not identify a non-zero wave number that would have characterized the front propagation.

Flame Front Instabilities in Diffusion Flames

Studies of intrinsic instabilities in flames have been predominantly concerned with premixed systems. A manifestation of an intrinsic instability is the spontaneous development of cellular structures, which is commonly observed in premixed flames. The competing effects of thermal and mass diffusivities play a central role in the development of cellular flames. One might therefore expect that they play a similar role in diffusion flames as well. However, very little has been done on the subject. The lack of experimental work in this area may be attributed to the fact that it is difficult to generate in the laboratory a one-dimensional diffusion flame free of the stabilizing influence of stretch and curvature. Using a spherical burner in microgravity a one dimensional spherical flame of sufficiently large size can be produced. A theoretical study has thus begun to examine possible thermal-diffusive instabilities in diffusion flames. The first most important step has been completed; that is a general model has been developed to describe the dynamics of a diffusion flame of general (three-dimensional shape covering the whole range of parameters: from the equilibrium limit down to extinction.

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PROJECT IDENTIFICATION: 962-22-05-47
NASA CONTRACT NO.: NAG3-1604
RESPONSIBLE CENTER: LeRC

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals


Presentations
Filtration Combustion for Microgravity Applications: (1) Smoldering, (2) Combustion Synthesis of Advanced Materials

Principal Investigator: Prof. Bernard J. Matkowsky
Northwestern University

Co-Investigators:
Bayliss, A.
Volpert, V.A.
Northwestern University

Task Objective:
Investigating combustion in porous media with applications to 1) smoldering and 2) combustion synthesis of advanced materials, also referred to as self-propagating high-temperature synthesis (SHS).

Task Description:
1) Proposing/developing theoretical models describing the fundamental mechanisms for the phenomena under consideration, 2) performing complementary analytical and numerical work on the proposed models and mechanisms, and 3) comparing the results of these analyses to experiments.

Task Significance:
Both smoldering and combustion synthesis of advanced materials (SHS) contribute to fundamental science, and also represent important applications for microgravity combustion science; in smoldering to fire safety in both normal and microgravity, and in SHS to the determination of optimal synthesis conditions in both normal and microgravity environments. Combustion synthesis appears to compete favorably with conventional technology, by achieving shorter synthesis times, and at lower cost, by employing the internal energy of the combustion reactions rather than the costly external energy of a furnace, employed in conventional technology.

Progress During FY 1995:
A paper entitled "Combustion of Porous Samples with Melting and Flow of Reactants," by A. Aldushin, B. Matkowsky, K. Shkadinsky, G. Shkadinskaya, and V. Volpert is accepted for publication by Combustion Science and Technology. The Abstract of the paper reads: "We formulate and analyze a model describing the combustion of porous condensed materials in which a reactant melts and spreads through the pores of the sample. Thus there is liquid motion relative to the porous solid matrix. Our model describes the cases when the melt either fills all the pores or spreads through only some of them. In each case the melt occupies a prescribed volume fraction of the mixture. We employ both analytical and numerical methods to find uniformly propagating combustion waves, to analyze their stability and to determine behavior in the instability region. The principal physical conclusion which follows from our analysis is that the flow of the melted component can result in nonuniform composition of the product. Unlike models which do not take into account the relative motion of the components, this model exhibits a dependence of the structure of the product on the mode of propagation of the combustion front. Thus, if the initial mixture is uniform, models which do not allow for relative motion necessarily lead to uniform structure of the product, while in the model employed here the structure can be nonuniform. We observe that the structure of uniformly propagating combustion waves depends on whether the refractory or melting component is in excess in the initial mixture. We determine how various parameters of the system affect stability and find a pulsating instability of the uniformly propagating solutions. We also perform numerical simulations in order to (i) study the dynamical behavior of the combustion wave in the instability region, (ii) obtain a description of the melt flow on the scale of the entire sample rather than on the scale of the combustion wave, i.e. to study the evolution of the size of the liquid melt layer which may occupy only a part of the product region. We show, in particular, that a transition to relaxation oscillations may occur closer to the threshold of instability than in gasless solid fuel combustion. Our numerical and analytical results are in qualitative agreement."

Printed: 28 FEB 1996
A paper entitled "Propagation and Extinction of Forced Opposed Flow Smolder Waves," by D.A. Schult, B.J. Matkowsky, and V.A. Volpert and A.C. Fernandez-Pello is accepted to Combustion and Flame. The Abstract of the paper reads: "Smoldering is a slow combustion process in a porous medium in which heat is released by oxidation of the solid. If the material is sufficiently porous to allow the oxidizer to easily filter through the pores, a smolder wave can propagate through the interior of the solid. We consider samples closed to the surrounding environment except at the ends, with gas forced into the sample through one of the ends. A smolder wave is initiated at the other end and propagates in a direction opposite to the flow of the oxidizer. We employ large activation energy asymptotic methods to find uniformly propagating, planar smolder wave solutions. We determine their propagation velocity, burning temperature, final degree of fuel conversion, and extinction limits. We also determine spatial profiles of gas flux, oxidizer concentration, temperature, and degree of conversion of the solid, including the burning temperature and final degree of conversion."

A paper entitled "Forced Forward Smolder Combustion," by D.A. Schult, B.J. Matkowsky, and V.A. Volpert and A.C. Fernandez-Pello is accepted for publication by Combustion and Flame. The Abstract of the paper reads: "We consider porous cylindrical samples closed to the surrounding environment except at the ends, with gas forced into the sample through one of the ends. A smolder wave is initiated at that end and propagates in the same direction as the flow of the gas. We employ asymptotic methods to find smolder wave solutions with two different structures. Each structure has two interior layers, i.e. regions of relatively rapid variation in temperature separated by longer regions in which the temperature is essentially constant. One layer is that of the combustion reaction, while the other is due to heat transfer between the solid and the gas. The layers propagate with constant, though not necessarily the same, velocity, and are separated by a region of constant high temperature. A so-called reaction leading wave structure occurs when the velocity of the combustion layer exceeds that of the heat transfer layer, while a so-called reaction trailing wave structure is obtained when the combustion layer is slower than the heat transfer layer. The former (latter) occurs when the incoming oxygen concentration is sufficiently high (low). Reaction trailing structures allow for the possibility of quenching if the gas mass influx is large enough; that is, incomplete conversion can occur due to cooling of the reaction by the incoming gas. For each wave structure there exist stoichiometric, and kinetically controlled solutions in which the smolder velocity is determined, respectively, by the rate of oxygen supply to the reaction site and by the rate of consumption in the reaction, i.e. by the kinetic rate. Stoichiometric (kinetically controlled) solutions occur when the incoming gas flux is sufficiently low (high). For each of the four solution types, we determine analytical expressions for the propagation velocities of the two layers, the burning temperature, and the final degree of solid conversion. We also determine analytical expressions for the spatial profiles of temperature, gas flux, and oxygen concentration. Gravitational forces are considered and are shown to have a minimal effect provided the ambient pressure is large compared to the hydrostatic pressure drop. The solutions obtained provide qualitative theoretical descriptions of various experimental observations of forward smolder. In particular, the reaction trailing stoichiometric solution corresponds to the experimental observations of Ohlemiller and Lucca, while the reaction leading stoichiometric solution corresponds to the experimental observations of Torero et. al."

Professor Matkowsky's request to rebudget the grant in order to accommodate subsistence support for his post-doc with no extra cost to NASA has been approved.

In an effort to foster international collaboration, Professor Matkowsky have gotten together with John Lee from McGill University (funded by the Canadian Space Agency) within the last few months of 1994. He has also contacted other SHS experimentalists such as Prof. J. Moore from Colorado School of Mines (funded by ourselves), and Carlos Fernandez-Pello on smoldering research in order to be knowledgeable about the latest work of the experimentalists.

Professor Matkowsky has responded to the Glovebox solicitation with international partners. His collaborators are J. Lee, S. Goroshin and D. Frost of the Mechanical Eng. Dept. at McGill University in Montreal, Canada, who are funded by the Canadian Space Agency. The proposal is about the SHS of ZnS to learn more about the kinetics of the process.
### II. MSAD Program Tasks — Ground-based

**Discipline:** Combustion Science

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**PROJECT IDENTIFICATION:** 962-22-05-55

**NASA CONTRACT NO.:** NAG3-1608

**RESPONSIBLE CENTER:** LeRC

### Bibliographic Citations for FY 1995:

**Journals**

**Presentations**
Combustion of PTFE: The Effect of Gravity on Ultrafine Particle Generation

Principal Investigator: Prof. J. T. McKinnon
Colorado School of Mines

Co-Investigators:
Todd, P.W.
Oberdorster, Prof. G.
University of Colorado
University of Rochester

Task Objective:
This project is an experimental and analytical study of the fundamental chemical and physical mechanisms and the influence of gravity on the ultrafine particles and gases generated by the thermal breakdown of polytetrafluoroethylene (PTFE) wire insulations. PTFE is a widely used spacecraft material that resists ignition and fire spread, but it can degrade when stressed by thermal and electrical overloads to release ultrafine (10 to 100 nm) particles as aerosols or smoke.

Task Description:
The experimental study will be performed in three tasks.

1. Normal-gravity tests, conducted at the Colorado School of Mines, investigate PTFE powder, or a reference perfluorinated alkane, in a flow-tube reactor under three atmospheres—wet air, dry air, and nitrogen. The reaction-temperature range is from 380 to 700 C. The gas products are collected and analyzed using gas chromatography and mass spectrometry. The concentration of ultrafine particles in the effluent gas stream (smoke) is measured using optical absorption. The particles are also collected on filters and analyzed chemically and physically by Fourier transform infrared spectroscopy (FTIR) and electron microscopy.

2. Normal-gravity tests, conducted at the Colorado School of Mines, investigate the smoke emitted from a PTFE-insulated copper wire thermally overloaded in a closed chamber under wet air, dry air, and nitrogen. In these tests, smoke density is measured by light scattering, and representative samples of the particles are collected by thermophoresis.

3. Microgravity tests, conducted in the NASA Lewis Research Center drop-tower and airplane facilities, investigate the effect of gravity on the degradation reaction and product evolution. The test apparatus and operating plan for the microgravity experiments will be established from the results of the Task 1 and 2 studies.

The accompanying analytical study develops a comprehensive model to predict ultrafine-particle production, given inputs of heating rate, polymer quantity, degree of forced convection, and other factors. This analysis is based on elementary reaction rates from established databases, the Principal Investigator's previous studies on soot formation, and the interpretations of the results of the experimental tests.

Task Significance:
The study will examine the phenomenon of ultrafine-particle generation by the thermal degradation of a widely used spacecraft wire insulation. The results contribute to the knowledge of the physical and chemical mechanisms of this process and offer practical applications in the reduction of health hazards in spacecraft and in the early-warning detection of fire incidents by atmospheric smoke sampling.

Progress During FY 1995:
The tube-furnace flow apparatus for the Task 1 studies was constructed and used for testing. The degradation studies were conducted under three atmospheres—dry air, air saturated by bubbling through water, and nitrogen. The
atmospheric exhaust from the furnace passed through a densely packed glass wool filter to trap the aerosol particles, with gas samples removed for off-line analysis.

The tube-furnace tests heated a gram of PTFE powder. The analyses showed that there was negligible degradation below a temperature of 390°C. Above that threshold temperature, the evolution of reaction products increased rapidly with temperature; this rate could be modeled by an Arrhenius-type dependency. The atmospheric composition had a significant effect on the nature of the degradation products. Under nitrogen, no smoke was observed, i.e., the only products appeared to be gases. Under dry air, the effluent had a large quantity of smoke (an aerosol of particulates). Under wet air, the effluent had a light quantity of smoke. Under all three atmospheres, the analyses showed that the principal gaseous product was perfluoroethylene, C6F4.

Preliminary FTIR analyses of the composition of the collected smoke particles showed that the composition under wet air was not the same as that under dry air and both differed from the PTFE reactant. The chemical identification of the FTIR peaks is still in process, however.

Transmission electron-microscopy analyses of the collected particles were also made. Both individual particles and agglomerated clusters were noted. The analysis of individual particles was aided by dispersion of a small sample in a solvent, then vibrating and evaporating to isolate the particles. For the degradation under dry air, primary particles were in abundance, and they ranged in nominal size from 7 to 15 nm. Cluster sizes were in a nominal range from 100 to 400 nm. For the degradation under wet air, primary particles were scarce. The nominal size range under this atmosphere was from 20 to 40 nm for the primary particles and from 250 to 400 nm for the clusters.

The wire-insulation degradation apparatus for the Task 2 studies was constructed and used in tests on copper wires insulated with polyethylene and PTFE. The initial observations for the PTFE wires agreed with those made in the tube-furnace tests. Considerable smoke was evolved under dry- and wet-air atmospheres, but none under nitrogen. The heat-up time before visible degradation was of the order of 12 sec. Obviously, the experimental conditions must be accelerated for success in the planned microgravity drop-tower testing. The continuing wire-insulation-degradation studies are to use video imaging to observe the dynamic progress of the reaction and effluent release.

Preliminary designs have evolved for microgravity testing. The Principal Investigator has assembled a prototype apparatus in a frame modeled after the NASA Lewis Drop Tower frames. Preliminary tests of apparatus integrity are being performed in a makeshift, but serviceable, 1.5 Second Drop Tower at the Colorado School of Mines.

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BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Proceedings
Premixed Turbulent Flame Propagation in Microgravity

Principal Investigator: Prof. Suresh Menon
Georgia Institute of Technology

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
The overall objective of this research is to characterize the behavior of turbulent premixed flames and to use the experimental data to validate a novel computational method to simulate accurately the behavior of premixed flames. In order to understand the behavior of turbulent flames, access to all the length scales (from the device size to the Kolmogorov scale) in the flow field is required. A possible method to achieve this is to reduce the flow velocity. However, in normal-gravity this is not possible due to gravitational acceleration and the turbulent stresses are overwhelmed by buoyant stresses. Therefore, experiments in microgravity will be carried out that will allow the study of low-speed turbulent flames without buoyancy effects.

Task Description:
Premixed combustion of hydrogen-air mixtures will be studied in a Couette flow configuration which is essentially a flow between two parallel plates moving opposite to each other. The turbulent flow field will be characterized with the flame speed being the primary variable of interest. Additional data on pressure rise and temperature will also be measured.

Task Significance:
Improved understanding and modeling of practical turbulent flames will lead to increased efficiency and reduced pollutant formation. Practical applications of technology to industrial combustors and high throughput engines may be anticipated.

Progress During FY 1995:
The experimental device for the cold-flow tests was designed, constructed and assembled. The entire Couette flow set-up is enclosed in a glass box whose dimensions are 889 mm x 737 mm x 356 mm, and 10 mm thick. The Couette flow channel is made of infinite-belt type, where both walls move at the same speed, but in opposite directions. The belt, made of Mylar, is 0.1 mm thick and 305 mm wide and is supported and driven by two solid aluminum cylinders which are each 76 mm in diameter. A DC motor is used to drive one of the cylinders while the other cylinder has adjustments available to adjust the tension of the belt. The belt speed is determined using a proximity switch that measures the rpm of the driving shaft. A maximum belt speed of 2 m/s is possible with the current configuration and the belt spacing can be varied from 10 mm to a maximum of 76 mm.

Preliminary LDV measurements of the Couette flow in the test facility were initiated. Some minor problems with the belt and the drive system were encountered but they now appear to be working adequately. The design requirements for the hot flow facility are being evaluated; however, there is no plan to carry out the construction until the cold flow experiments have been completed. Direct numerical simulations of the Couette flow were also initiated. These utilize a 64x96x64 grid.

Preliminary cold flow results for the mean velocity profile have been obtained using both a constant coefficient and a dynamic sub grid model for the sub grid kinetic energy. Comparison with available experimental data shows that the present scheme is capable of resolving mean motion.

The model for thin flames has also been implemented and it is planned to initially carry out parametric studies for constant density flames. The heat release model is already implemented but it will require further testing.
II. MSAD Program Tasks — Ground-based

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TASK INITIATION: 5/94  EXPIRATION: 5/98

PROJECT IDENTIFICATION: 962-22-05-48
NASA CONTRACT NO.: NAG3-1610
RESPONSIBLE CENTER: LeRC

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Proceedings
A Fundamental Study of the Combustion Syntheses of Ceramic-Metal Composite Materials Under Microgravity Conditions - Phase II

Principal Investigator: Prof. John J. Moore

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
Obtaining an improved understanding of the effect of gravity on the combustion synthesis of ceramic matrix-metal infiltrated composites; develop new and improved-property materials.

Task Description:
Combustion synthesis reactions are conducted both under normal gravity (various orientations of samples with respect to gravity) and microgravity conditions to generate either porous or dense ceramic matrices in the presence of varying amounts of excess Al₂O₃ (diluent) and/or excess aluminum, which will be available to fill residual pores by capillary action. The effects of gas generation during reactions on the porous matrix formation are studied under different levels of gravity. The dense composite materials are produced using a one step, low cost simultaneous combustion-consolidation process. The microstructure and properties of both dense and porous product composite materials are also characterized.

Task Significance:
An improved understanding of the role of gravity on the combustion synthesis of ceramic-ceramic and ceramic matrix-metal composite materials allows for new approaches for the development of specified microstructures, thereby resulting in improved material properties. One can also explore ways to develop brand new materials by microgravity processing (e.g. high surface area expanded or foamed ceramics, and fully dense interpenetrating phase composites). The expected benefits of such inexpensive, light, strong, and either highly porous or dense composite materials range from construction-support systems to liquid-metal, filtering-systems applications.

Progress During FY 1995:
Professor J. Moore has been pursuing the B₄C/Al₂O₃ system using the reaction:

\[ 2B₂O₃ + C + (4+x)Al + yAl₂O₃ -> B₄C + (2+y)Al₂O₃ + xAl \]  

for further in depth analysis with graduate student, Brooke Whitehead. The selection of this system is based on the observation that it showed expansions of approximately 150% under 2-g, and over 400% under 0-g conditions (Lear Jet), as opposed to 300% under n-g, after the combustion synthesis process. The student has concentrated her efforts on the more accurate control of the combustion temperature to avoid melting Al₂O₃, with the expectation that, if successful, this would provide increased surface area in the expanded ceramic composite.

Two modes of combustion are being studied: 1) simultaneous, where the mixture is ignited by heating the whole pellet at once, and 2) propagating, where the ignition is at one end of the pellet. Depending on the value of \( y \) and combustion mode, the reactions are observed to be quenching, unstable, or stable, and the product has different pore sizes, distribution and expansion.

Brooke focused mostly on the 10 wt% excess Al₂O₃ (diluent) samples, with some additional 5 wt% and no excess Al₂O₃ samples in argon gas (inert atmosphere). A trend of continuous reduction in combustion temperature with increasing diluent is observed. The SEM images of all samples show the formation of shells within the sample. The determination of the interior structure and composition of the shells by using electron back-scatter imaging, as
II. MSAD Program Tasks — Ground-based

well as the measurement of the surface area will be conducted by the next student (Tania Mattor).

The vacuum pump is in place to complete reactions utilizing 15-20 wt% Al_2O_3 as a diluent. The goal is to decrease the combustion temperature of the reaction system below the melting point of Al_2O_3 so that the reacted samples exhibit a higher surface area/weight ratio.

Brooke Whitehead's samples for the B4C/Al_2O_3 system with 0, 5 and 10 wt% excess Al are flown on the Lear Jet by LeRC personnel for a total of seventeen trajectories during the last week of October 1994. Problems with the aircraft electrical system during flights indicate that at least some of the temperature measurements will not be reliable for some tests, which is quite disappointing.

Data from the last Lear Jet flight experiments was analyzed. Brooke defended her MS Thesis in December, 1994. Major conclusions from Brooke Whitehead's MS Thesis are summarized below: Expansion increased when graphite was used as the reductant instead of lampblack. Reactions using graphite resulted in higher combustion temperatures (T_c) than those that used lampblack.
- Increased T_c resulted in increased expansion.
- Conducting the reaction in low gravity (Lear Jet) resulted in significant increases in porosity, T_c, ignition temperature (T_i), and expansion.
- Increased amounts of Al_2O_3 diluent did not increase expansion, but did increase the amount of unreacted products.
- Conducting the reaction in the propagating mode resulted in near-unidirectional expansion in the vertical direction.
- Conducting the reaction in the simultaneous combustion mode resulted in near-3-dimensional expansion.

Tania Mattor has replaced Brooke Whitehead as an MS student. Tania is focusing on using both graphite and lampblack as the carbon source. She is conducting the SHS reactions in the propagating mode under vacuum, near atmospheric and an intermediate subatmospheric pressure. Under vacuum conditions both graphite and lampblack reactions proceed in an unstable spiral mode. If the power supply is turned off immediately after ignition, the reaction would quench out. However, if the power supply remains on until the reaction progresses partially through the pellet, the reaction would go to completion in what appears to be a stable manner. Recorded combustion temperatures has varied between 1490C and 1813C if lampblack is used, and between 1696C and 1962C if graphite is used as the carbon source. Measured expansions so far have varied between 235% and 511%. Expansions under normal gravity but vacuum conditions can be even larger than low gravity but atmospheric conditions.

Professor Moore's post-doc, Chris Feng, is processing the TiC/Al2O3/Al system using the reaction:

\[ 3TiO_2 + 3C + (4+x)Al + yAl_2O_3 \rightarrow 3TiC + (2+y)Al_2O_3 + xAl \]  (2)

under normal gravity conditions using a hot press. He is studying the effect of process parameters, such as reactant stoichiometry, green density, pre-ignition conditions, thermodynamics (thermochemistry) as well as consolidation conditions, on the kinetics and stability of combustion, microstructural uniformity and morphology, and resulting mechanical properties of synthesized TiC/Al2O3/Al products.

The effect of excess Al (xAl) and diluent Al_2O_3 (yAl_2O_3) on the combustion temperature (T_c) has been examined both theoretically (using thermochemical enthalpy-temperature relationships) and experimentally. Both additions, i.e. xAl and yAl_2O_3, lower T_c and decrease the grain growth of Al_2O_3. The TiC particles are much smaller than the Al_2O_3 particles, but even these TiC particles decrease slightly in size with increased additions of xAl and yAl_2O_3 (i.e. by increasing x from 0 to 2, and/or increasing y from 0 to 2, the size of the TiC grains decreases from 2 microns to 1 microns, which is insignificant for this size range to affect properties). This increased refinement in structure on lowering T_c can contribute to increased fracture toughness.

A major effort has been to use these data to produce dense ceramic (TiC-Al_2O_3) and ceramic-metal (TiC-Al_2O_3-Al) composites using reaction (2), by simultaneously coupling the consolidating (hot press) load to observing the initiation of the combustion synthesis reaction. (The initiation of the combustion synthesis reaction was achieved...
using a rf induction coil in which the sample was placed, and the temperature was measured by means of a thermocouple embedded in the wall of the graphite die. Once the exotherm was observed on the chart recorder that as interfaced to the thermocouple, the compaction/consolidating load was applied. Quite often, the initiation of the exothermic combustion synthesis reaction was also observed by a sudden change in pressure registered within the hot press system. Both of these techniques were used to identify the initiation of the reaction. The effect of reaction stoichiometry (xAl, yAl2O3), pressure applied to the reacting pellet, and the time and temperature at which the consolidating load was applied have been examined in order to correlate these processing parameters with the resulting microstructures, product density and mechanical properties (using four point bend modulus of rupture (MOR) and four point bend chevron notch fracture toughness tests). These correlation studies are currently underway. Some initial data indicates that, as would be expected, both flexural strength (MOR) and fracture toughness increase with increase in consolidating load applied at 1600°C. Although these initial data indicate a slightly lower MOR than perhaps would be expected according to the rule of mixtures, the MOR approaches the predicted values (320 MPa) as the consolidating load is increased above 30MPa. On the other hand, extremely high fracture toughness values (6 to 10 MPa.m1/2) have been recorded. These values are as good as some recent published work from Japan in which SiC whiskers were used to reinforce Al2O3. Since the use of whiskers presents considerable health hazards, the technique that Chris Feng is exploring may provide some significant advantages. A full evaluation and analysis of these data is currently underway and should be completed in the next few weeks.

Chris has also been using differential thermal analysis (DTA) to identify the controlling reaction mechanism for reaction (2). The sequence of the combustion reaction is investigated through studying the various combination of the reactants. These data confirmed that the reaction sequence for reaction (2) is: 1) aluminothermic reduction of TiO2, i.e. 
\[ 3\text{TiO}_2 + 4\text{Al} \rightarrow 3\text{Ti} + 2\text{Al}_2\text{O}_3, \]
followed by 2) the synthesis of TiC, i.e. 
\[ 3\text{Ti} + 3\text{C} \rightarrow 3\text{TiC}. \]

Professor Moore visited Professor B. Matkowsky at Northwestern University for a day discussing how the two groups may interact in the future on microgravity combustion synthesis. Professor Moore believes that they have some important experimental observations which can benefit from some form of mathematical modeling.

Professor Moore's request for international collaboration with GIL of Newfoundland, Canada, as part of his new NRA grant which started January, 1995, was approved verbally by Dr. Mickey King on the condition that no NASA money or facilities are used. Professor Moore has recruited an MS student, Travis Woodger, who went to Newfoundland in May, 1995 to conduct SHS experiments with acoustically levitated samples until December as part of this collaboration.

In an effort to establish international collaboration, Professor Moore contacted Professor Alexander Merzhanov at ISMAN in Russia, who is the pioneer of the SHS process, to develop a joint research program. Merzhanov's microgravity SHS proposal has been approved for funding by the Scientific and Technologic Advisory Committee of the RSA. The combined collaborative proposal currently includes a triangular approach involving the US, Russians and the Canadians.

### Students Funded Under Research:

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**Task Initiation:** 1/95  **Expiration:** 1/98

**Project Identification:** NAG3-1698  **NASA Contract No.:** 962-22-05-38

**Responsible Center:** LeRC

### Bibliographic Citations for FY 1995:

**Journals**

II. MSAD Program Tasks — Ground-based

**Discipline: Combustion Science**


**Proceedings**


**Presentations**


Mattor, T., "Combustion synthesis of porous ceramics." Student Poster (Second Prize), Rocky Mountain Section AVS Meeting, Dever, CO, August, 1995.


Flow and Ambient Atmosphere Effects on Flame Spread at Microgravity

Principal Investigator: Prof. Paul D. Ronney
University of Southern California

Co-Investigators:
No Co-Is Assigned to this Task

Task Objective:
This program is a three-year experimental and theoretical study of the effects of ambient atmosphere on the properties of flame spread over thin and thick solid fuel beds. In particular the effect of the type of inert gas, which affects the Lewis numbers of fuel and oxidant, and the effect of the addition of sub-flammability-limit concentrations of gaseous fuels to the oxidizing atmosphere will be studied. The effect of convection will be studied through one-g and mg experiments with and without a forced flow. Moreover, the influence of thermal radiation, whose effect is known to be markedly different depending on the convection level, will be addressed.

Task Description:
The emphasis of this study is on thermally thin fuels because of the limited μg test time available in ground-based facilities, and preliminary scaling analyses suggest that thermally thick fuels can be examined as well when gaseous fuel is added to the oxidizing atmosphere.

The experiments will be conducted in a combustion chamber in which a convective flow of a few cm/sec can be imposed in the direction opposite the flame spread. The oxidizing atmosphere will be mixed by the partial pressure method. For tests of Lewis number effects, inert He, Ne, N2, CO2 and SF6 will be used since they provide Lewis numbers from about 0.3 to 1.4. CO and CH4 will be used for the gaseous fuels. Thin fuel samples will be ashless filter paper and thick fuel samples will be PMMA. Fuel samples of varying thickness will be ignited by the heat generated by a current passed through a coiled nichrome wire coated with nitrocellulose.

The primary diagnostics are video and an array of fine-wire thermocouples to measure the temperature simultaneously at several locations. The video records provide information on the spread rate and flame shape. The thermocouples give an independent check of the spread rates and the existence (or lack thereof) of a separate flame front in the case of added gaseous fuel. The temperature data may also be used to determine the heat flux from the gas phase to the fuel bed, which can be related to the spread rate.

Task Significance:
The understanding and control of accidental fires is a critical safety issue in both terrestrial and space-borne environments. The proposed work would provide insight that could be used to assess the fire hazards associated with non-standard atmospheres that might be employed in future manned spacecraft. Also, fires in enclosures produce a considerable amount of unburned vaporized fuel and partially combusted gases such as CO. One-g experiments have shown that the addition of combustible gases such as CO to the oxidizing atmosphere may increase the flame spread rate substantially. This study could provide information to improve models of fire development and spread in enclosures at one-g and μg.

The influence of weak forced convection is particularly important for studies of flame spread at μg because there is very little buoyancy-induced flow at μg. Experiments by Olson and collaborators shows that the presence of forced convection currents (for example due to ventilation systems in manned spacecraft) can have a profound effect on the spread rate and extinction conditions. Consequently, the understanding of these effects is critical to understanding how fires might start, spread, and be extinguished at μg conditions.
Progress During FY 1995:

To improve the sensitivity of the video imaging system for the very weak near-extinction flames, a shearing interferometer has been designed and is being constructed for evaluation. The shearing interferometer has no parts that have critical alignment requirements, and thus may be especially suitable for drop tests. The interferometric measurements may also be useful to supplement the thermocouple temperature measurements.

As a precursor to the experimental study, an analytical study has been initiated in conjunction with Dr. Mike Delichatsios of Factory Mutual Research Corporation in Norwood, MA. The goal of the work is to extend Dr. Delichatsios's previous (1986) exact solution of flame spread over a pyrolyzing fuel bed to consider the effects Lewis number, finite-rate chemistry, and gaseous fuel addition on flame spread rate. A preliminary theory of Lewis number effects on flame spread over thin fuel beds, including finite-rate chemistry, has been obtained and compared with prior one-g data from Zhang et. al., (1992).

An apparatus for conducting microgravity experiments in the NASA-Lewis 2.2 Second Drop Tower under quiescent flow conditions has been completed. This facility consists of a combustion chamber with quartz windows, shearing interferometer system, video cameras and a data and control acquisition system. This system has recently been used successfully by the PI for experiments on radiation-driven flows in gases. For the flame spread experiments, the radiation-driven flow test section has been removed and replaced by a sample holder, mixing fan, flame spread initiation system and thermocouple rake. In the past quarter all of these components have been built, integrated into the chamber and functionally tested. Initial drop tests are scheduled for October of 1995.

A model for the effect of gravity on upward-spreading flames has been developed by the PI. For flames spreading upward over thin fuels under the influence of buoyancy, a model for the flame spread rate (Sf(upward)) normalized by the spread rate for downward propagation (Sf(downward)), as a function of the Grashof number (Gr = 3D gW^3/n^2), where g is the gravitational acceleration, W the sample width and n the kinematic viscosity, was developed. The predicted expression for the spread rate over narrow samples (low Gr) was Sf(upward)/Sf(downward) ~ Or. The predicted spread rate agrees well with the experimental results at low Gr. At higher Gr, turbulent flow and radiative loss from the fuel surface alter the behavior and reduce the predicted exponent on Gr to 12/7 and 0, respectively.

\[ Sf(upward)/Sf(downward) \sim Or \]

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

**Proceedings**

**Presentations**
Delichatsios, M.A., Ronney, P.D., "Horizontal and lateral flame spread on solids: closure and diffusional Lewis Number effects." Fall Technical Meeting, Combustion Institute, Eastern States Section, Clearwater Beach, FL., December 5-7, 1994.

Combustion Research

Principal Investigator: Dr. Howard D. Ross

NASA Lewis Research Center (LeRC)

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
The task objective is to advance the understanding of fundamental combustion phenomena and/or processes that are affected by the presence or absence of gravity.

Task Description:
The research approach is to provide for limited precursor studies by external investigators and for the engineering and fabrication of hardware needed to conduct in-house research and assist in the research efforts conducted on-site at LeRC in support of Code UG-sponsored Principal Investigators (PI's) and National Research Council (NRC) graduate student researchers. Funds for facility overhead charges are provided through separate Research and Technology Operations Plans (RTOP) resources.

Subtasks are funded in part by this task, or are included for completeness at the request of NASA Headquarters.

Task Significance:
Significance varies with the various research efforts, and therefore, more focused, effort-specific, statements are given below.

Progress During FY 1995:

Binary Azeotropic Droplet Combustion Experiment - Dr. R. Colantonio

Objective:
Non-ideal mixtures that exhibit a relative extremum in their vapor pressure and "volatility reversal" at a particular composition (azeotropic point) are classified as azeotropic mixtures. This investigation will observe the liquid droplet and flame histories and location of the azeotropic point in reduced gravity for a number of binary azeotropic mixtures.

The fluid motion within a burning droplet is of great importance for practical applications involving droplet transport enhancing both heat and mass transport. Advanced laser diagnostics will be utilized to observe internal recirculation patterns that will be used to determine its affect on preferential vaporization and extinction characteristics of azeotropic droplets and non-azeotropic (zeotropic) droplets.

Radiation heat flux from flames to the surrounding objects determines the degree of ignition, burning rate and extinction characteristics of burning droplets. Radiation flux measurements will be performed to quantify the radiation heat loss fraction during the burning histories and assist in locating the critical points for a number of binary azeotropic mixtures.

Description:
Droplet burning of azeotropic will be performed at the NASA 2.2 Second Drop Tower. A demonstrated droplet deployment and ignition system will be used. The droplet deployment system consists of two deployment needles that merge together via rotary motors. A fuel dispenser will form the droplet between the two needle tips. Once the droplet is formed, the rotary motors withdraw the needles. Two hot-wire hoops will be used to ignite the fuel droplet once the needles have been withdrawn and the droplet is suspended during reduced gravity.
Both a color CCD and high-speed 16 mm B&W cameras will be used to observe the burning characteristics of azeotropic fuel mixtures. Particle Tracking Velocimetry (PTV) will be used to observe internal recirculation within burning and non-burning droplets. A thin laser sheet will cut through a plane of a seeded droplet with the particle's motion with a CCD camera. Two thermopile radiometers will be used to measure radiation emissions emanating from a burning droplet. One radiometer will have a low-pass UV filter and the other will have no filter.

Significance:
Many hazardous liquid wastes (e.g., aromatics, polyhalogenates, alcohols and hydrocarbons) can form azeotropes. The unique feature about the behavior of azeotropic multicomponent liquids is the modulating of relative volatilities by suppressing the gasification of the lower or higher boiling point component beyond a critical composition. This feature is very important in the disposal/incineration of azeotropic mixtures and in the chemical distillation industry. Azeotropes can also be formed with commonly burned fuels. Water when added to ethanol will form an azeotropic mixture and is very soluble in ethanol. Ethanol is an alternate fuel being considered by industry to reduce emission levels exhausted from automobiles. Limited data exists on the burning of azeotropic droplet mixtures. By observing the burning behavior of a fully symmetric, stationary droplet a greater fundamental understanding of the volatility reversal and burning characteristics unique to azeotropic mixtures will be gained. Also, most droplet combustion models ignore any form of radiation emission and internal droplet recirculation or assume a quantitative value. This research effort will confirm previous modeling assumptions.

Progress:
The experimental hardware has been built and operational since May 10, 1995. Over 35 drops have been completed with the burning of the azeotropic mixture propanol/tetrachloroethylene. The data is currently being analyzed. Preparations are underway to burn ethanol/methylcyclohexane. Modifications to the rig will be made in August 1995 to accommodate two thermopile radiometers. PTV laser equipment and associated hardware are on order and will be fitted to the rig in October 1995.

Radiative Flame Extinction at Large Droplet Radius - P. Struk (GSRP)

Objective:
To verify experimentally the existence of a theoretically predicted large droplet radius above which a spherical steady flame cannot be sustained due to gas-phase thermal radiative heat loss. Large fuel droplets are simulated using a wetted porous sphere continuously supplied fuel by a syringe pump.

Description:
The research consists of two phases: a ground-based phase and a low-gravity phase (using the NASA LeRC DC-9 aircraft). The ground-based phase involves the development of porous spheres, hardware buildup, as well as ascertaining the feasibility and techniques of using porous spheres to simulate droplet burning. In the low-gravity phase, wetted porous spheres are ignited in various atmospheric conditions. The radius of the droplet is varied by adjusting the fuel flow rate into the porous sphere.

Significance:
This experiment will provide information on large droplet burning using porous spheres in low gravity. The information gathered may provide evidence of a droplet diameter limit above which steady droplet burning is not possible. If extinction is observed, it will suggest that gas phase thermal radiative heat loss can cause diffusion flame extinction in low gravity as theoretically predicted.

FY95 Progress:
A droplet combustion experiment using wetted porous spheres being fed fuel from a syringe pump was flown aboard the NASA LeRC DC-9 aircraft in June 1995. The experiment was designed and built to use the flight hardware developed for the Gravitational Influences on Flammability and Flame Spread Test System (GIFTS) experiment. Porous spheres ranging in size from 4 mm to 6 mm were tested both in 21% oxygen at pressures of less than 1 atmosphere and 15% oxygen at pressures of less than 0.5 atmospheres. The experiment was conducted both attached to the aircraft and free-floating in the cabin.
After ignition, flames appeared near the droplet surface (on the order of a droplet diameter) and began to grow spherically outward sometimes reaching diameters near 10 times the size of the droplet. The large flames were susceptible to g-jitter, however, and many cases lost their spherical symmetry just after ignition especially when attached to the aircraft. Free-floating the experiment provided less g-jitter, however, for shorter durations ranging up to roughly 6 seconds (due to repositioning of the free-float package for safety which produced significant accelerations). All near spherically symmetric cases appeared to continuously grow and concurrently decrease in luminous intensity until disrupted by g-jitter (which typically enhanced the burning) or, as observed in a few cases, extinguish. However, no flames appeared to reach a steady state since the flames continuously moved radially outward as viewed by the human eye—a quantitative analysis is currently underway in order to verify this observation. In the coming months, a thorough analysis of the data will be done.

**Combustion Synthesis of Fullerenes - J. Brooker**

**Objective:**
The objective of this research is to produce, collect, and quantify fullerenes synthesized during combustion under normal gravity and microgravity and compare the results relative to: C60 and C70 composition, C60 and C70 percent of soot, and C70/C60 molar ratio. In phase one, acetylene will be burned in a gas-jet premixed flame; in phase two, benzene will be studied.

**Description:**
Flames will be used to generate fullerenes in a sub-atmospheric combustion chamber. Normal gravity tests will be performed to determine conditions favorable for fullerene production and to define a suitable method of collection and species separation/identification. Subsequent experiments will employ the 2.2 Second Drop Tower and possibly aircraft.

**Significance:**
The suggested potential applications for fullerenes include superconductors, lubricants, catalysts, high energy fuels, polymers, and biomaterials. Some of the emerging applications that are currently under investigation include the growth of diamond films on surfaces for protective coatings and electronic industries, their use as optical limiters to protect sensors from intense radiation sources, incorporation of fullerenes into photoconducting polymers with applications in light detection and electrostatic imaging, and the possible use of fullerene derivatives as drugs to combat AIDS by interacting with the active site of HIV.

**FY95 Progress:**
Mechanical/electrical design is continuing on the drop rig. The DACS, battery box and spark igniter have been completed. Preliminary calculations favor the flat-flame over the gas-jet burner due to the flow rates needed to achieve appreciable quantities of fullerene. Fullerene samples have been ordered along with an HPLC column and solvents to separate the extract the fullerene fractions. Design calculations showing the pressure rise in the chamber using the new flow requirements were completed. Pressure drop calculations for the acetylene flow stream were completed. These calculations were necessary to do the selection of the fluid components in the C2H2 stream, size the C2H2 supply tank, and estimate the operating time for one full tank of C2H2.

Investigation of a suitable chamber size continues so that a McKenna Flat-Flame burner can be used with minimal side effects. Fabrication of the drop frame will remain on hold until a chamber size is determined.

The one-g gas-jet burner has been assembled and tested with premixed methane/oxygen flames. Pressure regulation equipment was incorporated into the one-g laboratory experiment. A pressure of 1.1 torr was achieved with minimal leakage. Sierra mass flow controllers have arrived and will be connected to the MKS cluster gauge to complete the pressure / mass flow control system. The spark igniter was installed and successfully ignited a methane diffusion flame. The vacuum connector had to be relocated since the combustion chamber would not fit over the baseplate. Gas-jet acetylene diffusion flames have been established in the 12.7 cm OD tubular burner. A premixed acetylene/oxygen flame was also attempted, however, it was determined that the OD was too large for this...
II. MSAD Program Tasks — Ground-based

Discipline: Combustion Science

flame and a smaller tubular burner (OD = 3.175 cm) was constructed. Acetylene diffusion flames have also been established using the McKenna flat burner.

High Lewis Number Premixed Gas Flames -- Dr. H. Pearlman (NRC)

Objective:
The objective of this study is to develop a fundamental understanding of the inherent stability of premixed gas flames at earth and micro-gravity.

Description:
The research approach is to design and construct hardware used to study freely-propagating flames in tubes in both normal and microgravity. The experimental configuration is chose because it eliminates the additional complexities of heat loss and hydrodynamic effects common to burner stabilized flames.

Significance:
Understanding the intrinsic stability of premixed gas flames is essential to our fundamental understanding of heat and mass transport in the presence of chemical reaction. This work is applicable to our understanding of combustion phenomena as well as to our knowledge of a broader class of chemical and biological systems known as reactive-diffusive systems, which include such diverse phenomena as the patterns of spots on animals, the spread of infectious disease, the formation of spiral galaxies, as well as the rhythmic beating of a human heart and the solitary voltage pulses which travel along nerve fibers.

FY95 Progress:
A concentrated experimental effort was undertaken to conclusively demonstrate the high-Lewis number, diffusive-thermal instability in premixed gas combustion at normal and reduced-gravity. This work began three years ago and has recently culminated in some astonishing discoveries [1-2]. Most notable is that high-Lewis number, premixed gas flames exhibit spontaneous pattern formation and chemical oscillations similar to those observed in the liquid-phase Belousov-Zhabotinsky (BZ) reaction, as well as a host of biochemical and electrochemical oscillators. While the patterns formed in these systems are similar, the mechanisms which drive the instabilities are different. Our premixed flame oscillations are believed to be due to the high-Le of the mixtures which means that a sufficient imbalance between the rates of heat and mass diffusion in the presence of chemical reaction can cause such instability. Moreover, this combustion system is believed to be the first observation of a non-isothermal, gas-phase oscillator which exhibits spontaneous pattern formation and traveling waves where temperature plays the role of the autocatalyst and provides the essential feedback into the reaction to sustain the oscillations.

A multitude of experiments were performed at normal gravity (Ig) which concentrate on freely-propagating flames in tubes filled with a quiescent mixture of butane and oxygen diluted with helium or helium-inert diluents. These mixture are chosen to systematically vary the Le, defined as the ratio of the thermal diffusivity of the mixture (mostly diluent) to the mass diffusivity of the scarce reactant (butane) in the mixture. In these mixtures, the Le varies from 3.8 to 4.3. The tubes are typically open at the ignition end and closed at the opposite end. This geometry is selected to minimize the role of conductive heat loss and imposed velocity gradients (i.e., flame stretch), both of which complicate the behavior of burner-stabilized flames. The flames are recorded using two high-speed EktaPro image-intensified video cameras at framing rates of 1000 frames per second. These high framing rates are necessary because the characteristic frequency of the oscillations is on the order of 100-150 cycles per second; they are too fast to be seen with the naked eye or with standard video cameras (30 frames per sec).

Three fundamental modes of propagation were observed. They are classified according to the mixture stoichiometry and the Lewis number, where the stoichiometry of the mixture is varied by controlling the volumetric fuel fraction and the Lewis number is varied by controlling the inert gas(es) in the mixture. Using helium as the inert gas, variations in the stoichiometry (i.e., fuel fraction) reveal the following modes as the fuel fraction decreases from stoichiometry toward the lean flammability limit; (1) steadily-propagating, spatially uniform flames, (2) radially-propagating, concentric ring waves on the surface of a steadily-propagating flame (target patterns) and (3) combined concentric-ring waves (target patterns) and rotating spiral waves on the flame surface. In a similar
fashion, gradual reduction in the \( \text{Le} \) by replacing some of the helium with another inert (e.g., Ar), for fixed stoichiometry, stabilizes flames which are unstable at high-\( \text{Le} \) numbers. Hence, there exists a critical \( \text{Le} \), \( \text{Lec} \), below which the flames are stable irrespective of the stoichiometry.

In summary, these tests provide a wealth of irrefutable evidence to support the existence of the high-\( \text{Le} \) instability. Moreover, they are the first to suggest that a combustible system can also be excitable and spontaneously oscillatory. This comes as quite a surprise since excitability, spontaneous pattern formation and oscillations have never been observed in an exothermic, gas-phase reaction, let alone a combustion reaction. This system is attracting a great deal of interest on part of physicists, chemists and mathematicians for several reasons: (1) it may serve a model for biological systems which are also exothermic reactive-diffusive systems which display spontaneous oscillations, (2) it is nonlinear and autocatalytic and thus may provide new insight into chemical turbulence and chaos when coupled with diffusional and convective processes, and (3) it has never been seen before in a gas-phase chemical reaction.

Venting Extinguishment Experiment -- Jeffrey S. Goldmeer (G.S.R.P.)

Objective:
The Venting Extinguishment Experiment is examining the extinction of a solid diffusion flame at low pressures in reduced gravity. The experiments being conducted will yield data on the flammability of the fuel at various pressures and flow velocities. This data will provide information regarding the effectiveness of the depressurization process as a fire extinguishment technique in reduced gravity.

Description:
The Venting Extinguishment Experiment is examining the combustion and extinction behavior of a solid, horizontal, PMMA cylinder in a low-speed flow in reduced gravity. The depressurization process is being examined in three phases. The first portion involved 1-g testing to determine the appropriate test parameters. The second portion of the research examined the effect of reduced gravity on the selected combustion configuration utilizing the NASA Lewis 2.2 Second Drop Tower. The third phase of the research will examine the effects of flow and depressurization on the flame in 1-g and in low-gravity. The low-g depressurization experiments will be conducted on board a NASA reduced gravity aircraft. (This research is part of J. Goldmeer's doctoral program at Case Western Reserve University.)

Significance:
Combustion of solids in low-speed forced flows at reduced gravity is relevant to fire safety in spacecraft. In an emergency a compartment within a space vessel could be intentionally vented to space to extinguish a fire. However, the effect of the induced flow on the fire in the low gravity and low pressure environment is unknown.

Progress:
A solid-phase cylindrical conduction model was developed to use in conjunction with an existing gas-phase combustion code. The solid-phase model takes into account the reduction in the cylinder diameter with the use of a non-dimensional coordinate transform (Wise and Ablow, 1954). Solid-phase burning rates have been measured and compared to literature and numerical values.

A series of experiments were conducted on the NASA Lewis Lear Jet (March 1995) and on the NASA Lewis DC-9 (June 1995) utilizing the Spacecraft Fire Safety Facility. In the combustion tests PMMA cylinders (2.5 cm in length, 1.9 cm in dia) were ignited in 1g prior to the trajectory; the sample was allowed to burn through the high-g portion of the trajectory. As in previous experiments chamber pressure, solid-phase centerline temperature, acceleration levels, and forced flow rates were measured. Flame images were recorded on video for analysis.

The goal of the Lear Jet tests was to determine if the effects of g-jitter could be reduced. In previous tests, the g-jitter associated with the aircraft extinguished the flames during the transition from normal gravity to low-gravity. In these experiments, the forced flow rate was increased to minimize the effect of buoyant induced flows on the flame. The forced flow velocities examined were 10 to 20 cm/sec.
The goal of the DC-9 flights was to examine the flammability limit of the burning solid at low-pressure in low-gravity. These experiments utilized a vacuum pump to obtain chamber pressures below 0.1 atm. During these flights the trajectories were flown to minimize negative-g's during the period of low-gravity. In low-gravity as the solid-phase temperature increased, the extinction pressure decreased. This trend was similar to the results from previous normal gravity experiments. In both the normal and low-gravity experiments, the samples were able to burn at pressures below 0.3 atmospheres. This is significant, as the lower pressure limit for venting on the International Space Station is approximately 0.3 atmospheres.

**STUDENTS FUNDED UNDER RESEARCH:**

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**TASK INITIATION:** 1/91  **EXPIRATION:** 12/95

**PROJECT IDENTIFICATION:** 962-22-05-08

**RESPONSIBLE CENTER:** LeRC

**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Journals**


**Presentations**


Reduced Gravity Combustion with 2-Component Miscible Droplets

Principal Investigator: Prof. Benjamin D. Shaw
University of California, Davis

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
Determine the combustion of two-component miscible droplets in reduced gravity via experimental and theoretical analysis. Specific objectives were to gather data on transient liquid behaviors (e.g., diameter histories and disruption), transient flame behaviors (e.g., sudden flame contraction and extinction), and sooting (e.g., luminosity and transport of observable soot particles).

Task Description:
The experiments involved burning two-component miscible droplets in environments of differing pressures and compositions. Data was obtained by direct photography of droplets and flames. Droplets initially about 1 mm in diameter and with components that differ significantly in volatility were studied.

Hydrocarbon (e.g., heptane/hexadecane) or alcohol mixtures in oxygen mole fractions from about 0.1 - 0.5 and initial pressures from 0.5 - 2 atm were used. Different inerts in the gas phase enhance or inhibit certain phenomena; e.g., CO₂ inhibit soot formation, while helium may promote extinction by increasing gas transport rates. Theoretical and computational research includes: modeling of fundamental droplet combustion phenomena; studies of allowable acceleration levels and durations to define experiment requirements; and studies of issues such as allowable droplet oscillation amplitudes, oscillation decay times, and pre-ignition vaporization.

Task Significance:
The reduced gravity experiments should yield insight into the efficiency of liquid-phase species diffusion, since this diffusion may markedly influence combustion behaviors (e.g., two-staged combustion behaviors). By noting times for flame contraction to occur, calculations for effective liquid-phase species diffusivities may be made (e.g., with asymptotic or with numerical models). Finally, unique sooting behaviors may appear since gas-phase compositions may differ significantly with time. Differences in sooting may be observable photographically by noting behaviors of apparent flame luminosities and soot particles large enough to be observed.

The theoretical efforts will aid interpretation of the reduced-gravity experiments as well as improve understanding of fundamental phenomena that occur during combustion in reduced-gravity.

Progress During FY 1995:

Experimental
A new droplet combustion apparatus developed and built at the University of California, Davis was used for a large number of experiments. Use was made of the 2.2 Second Drop Tower at the NASA Lewis Research Center. From August 1994 to February 1995, 133 successful drop tower experiments were performed (out of 203 attempts). Ambient pressures were varied from 0.3 - 3 atm (abs), the diluents N₂, CO₂, Ar and He were used, and many different fuel mixtures (alkanes and/or alcohols) were investigated. The primary fuel mixtures studied were heptane/hexadecane, in air or O₂-He environments. About 40 experiments were conducted with heptane/hexadecane mixtures in O₂-He environments at 1 atm or below. The experiments were very successful, allowing many new phenomena to be observed as well as providing verification of theories related to flame contractions associated with combustion of two-component droplets.

Printed: 28 FEB 1996
II. MSAD Program Tasks — Ground-based

We are now in the process of analyzing the films from these successful experiments. Because of the large volume of data obtained, this process has taken several months; the data analysis will be completed this fall. An overview of the experimental results can be found in Ref. [1]. Results have indicated that liquid-phase species diffusivities (as estimated by noting times for flame contractions) varied significantly with the ambient pressure. As the ambient pressure was increased, values for effective liquid-phase species diffusivities increased as well. This is most likely related to the fact that droplet surface temperatures will increase with increasing pressures, which will increase liquid species diffusion rates. Burning rates, transient flame behaviors, extinction, and visible sooting behaviors varied strongly with ambient gas pressure and composition, as well as with the initial droplet diameter and initial droplet composition. Articles on these (and other) aspects of the experiments have been submitted for publication [2-5].

Computational:
Detailed computational modeling (in collaboration with Professor H.A. Dwyer) has focused upon predicting effects of capillary flows on transient droplet vaporization. A finite-volume method is used, and variable properties are allowed in the liquid and gas phases. Modeling of single-component droplet vaporization in a hot environment has clearly shown that capillary flows dramatically influence droplet vaporization and internal circulation behaviors under conditions representative of slowly-drifting droplets in reduced-gravity experiments as well as rapidly-translating droplets in practical high-pressure sprays (see [6] and the references contained therein).

The computer code was recently modified to allow for multicomponent liquids. Thermal and solutal Marangoni effects on transient droplet gasification are now being investigated.

Analytical:
To aid interpretation of experimental efforts, analytical studies of gasifying droplets in reduced-gravity environments have been performed. These efforts focused on determining: (1) effects of small gravitational levels on droplet gasification; (2) and hydrodynamic stability of two-component droplets. Each effort has considered evaporation; combustion will be considered later.

The gravity-level research [7] focused on how small gravitational levels affect droplet gasification characteristics. The (viscous) conservation equations were nondimensionalized, and a small parameter that accounts for the effects of gravity was identified. Asymptotic analyses were developed in terms of this parameter. Near a droplet, the flowfield is spherically symmetric to leading order. Outer zones exist, however, where buoyancy significantly influences the flow; a stagnation point is predicted to exist in an outer zone.

The stability analyses [8,9] addressed the problem of predicting when spherically-symmetrical gasification of two-component droplets can be expected to be hydrodynamically unstable from surface-tension gradients. It was found that with zeotropic mixtures and for $\partial s/\partial T < 0$ and $\partial y/\partial T < 0$ (where $s$ is surface tension, $T$ temperature, and $y$ the liquid mass fraction of the more volatile droplet component), the thermal Marangoni effect is stabilizing and the solutal Marangoni effect is destabilizing. Heptane/hexadecane mixture droplets were predicted to be hydrodynamically stable. For methanol/water droplets, there is a critical radius for stability. When a droplet is initially pure methanol and absorbs water from the ambient, the critical radius depends upon the relative humidity of the environment. The instability for water/methanol droplets stems from the fact that water has a very high surface tension relative to methanol.

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Printed: 28 FEB 1996
BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals


Presentations


II. MSAD Program Tasks — Ground-based

Quantitative Measurement of Molecular Oxygen in Microgravity Combustion

Principal Investigator: Dr. Joel A. Silver

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
There are two objectives of this task. The first is to develop diagnostics based on diode laser absorption spectroscopy for use in microgravity combustion research. The general technique is expected to be applicable to major gases involved in combustion and in spacecraft cabin environments. The specific application in this task is the quantitative determination of molecular oxygen. The second objective is to develop a better understanding of the relative roles of diffusion and reaction of oxygen in microgravity combustion. Oxygen has a major role in controlling flame properties such as flame front speed, extinguishment, flame size, and temperature.

Task Description:
This investigation involves the determination of molecular oxygen using diode laser absorption spectroscopy. This technique uses a compact, low power optical system to probe the near infrared bands of oxygen. Though these bands are relatively weak, high sensitivity is maintained by means of wavelength modulation of the diode laser with detection at twice the modulation frequency. A compact laser scanner permits the laser beam to traverse the entire flame region. This scanner eliminates the need to use optical fibers in the setup. A compact electronics package includes all control, detection, and data acquisition electronics.

Task Significance:
Oxygen, as the primary oxidizer in flames, controls the major flame properties such as flame spread, flame front speed, temperature, and extinguishment properties. Its quantitative determination therefore is necessary both for an improved understanding of flame behavior in general and for fire safety on board spacecraft. Oxygen measurement is also critical for ground-based applications such as engine efficiency, energy production, air quality monitoring, and industrial processes such as steel manufacture. The specific technique under development is a compact, lightweight, and rugged instrument offering a wide range of laboratory, field, and industrial process measurements.

Progress During FY 1995:
Optimum spectral lines for simultaneous measurement of temperature and O₂ concentration have been computed. Four pairs of lines have been identified which show appropriate temperature sensitivity. Expected temperature accuracy is ±25 K. Optimum lasers for these lines have been ordered.

The PI is investigating the possibility of using Vertical Cavity Surface-Emitting Lasers (VCSEL's) as a potential replacement for the conventional laser diodes. The VCSEL's show promise of increased long-term reliability, wider tuning range, better beam profile, and reduced mode-hopping.

Optical and mechanical design has been completed and components have been ordered, received, or fabricated. Assembly is underway. Various lens types have been tested for laser focusing.

The VCSEL lasers were purchased and characterized. The tuning range is large enough to allow the entire R-branch of the ν=0 band near 760 nm to be scanned. The temperature tuning rate was determined. The noise power density decreases with frequency to the shot noise limit at frequencies well below the 1 MHz detection frequency.

The scanning mirror system was built and demonstrated to move a collimated 1 mm-diameter beam across the 40 mm range at 20 Hz. Laser power on the detector was flat to better than 2%.

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The electronic scanning of the mirrors and the laser frequency is complete and both scan ramps are working simultaneously.

The data acquisition board was acquired and programmed to control all operating parameters and to collect data.

Integration of the system into solid-surface combustion experiments was started.

Fabrication was started on parts to integrate the optical setup into the solid-surface combustion chamber. Electronic design was completed.

A 40 Mb solid state hard drive based computer was received and tested. The control software was installed. The computer can communicate with a standalone computer so that experiment initiation can be performed remotely and data can be downloaded after a drop.

**STUDENTS FUNDED UNDER RESEARCH:**

**TASK INITIATION:** 7/94  **EXPIRATION:** 6/97

**PROJECT IDENTIFICATION:** 962-22-05-51

**NASA CONTRACT NO.:** NAS3-26553

**RESPONSIBLE CENTER:** LeRC

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**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Presentations**

Numerical Modeling of Flame-Balls in Fuel-Air Mixtures

Principal Investigator: Prof. Mitchell D. Smooke

Co-Investigators: No Co-I's Assigned to this Task

Task Objective:
The overall objective of the proposed work is to understand if and the mechanisms by which stationary, spherical flames, sometimes called flame balls, stabilize in a quiescent, premixed gaseous fuel–oxidizer environment. These flames have been observed experimentally shortly after ignition in microgravity, but their long-term persistence is as yet undetermined.

Task Description:
The approach to meet this objective is to modify an existing numerical model, which includes full chemical kinetics, to account for radiative heat losses. Parametric analyses of kinetic, transport, and radiative effects will then be performed. The predictions will then be compared to the experimental observations of Professor P. Ronney as well as theoretical results developed by Professor J. Buckmaster.

Task Significance:
The significance of this effort is to enhance the understanding of very basic combustion phenomena, such as: flammability limits and stabilization mechanisms, and the probability that it will find its way into combustion science textbooks.

Progress During FY 1995:
During the first year of the project our goals have included verifying the sensitivity of the flame ball radius and the value of the lean and rich flammability limits to 1) the hydrogen-air chemistry, 2) the transport model, and 3) the radiation approximation. Our previous hydrogen-air flame ball computations [1] employed a Curtiss-Hirschfelder transport approximation with a radiation model in which interpolated emissivity data was used to construct an approximation for the divergence of the radiative heat flux.

Utilizing the kinetic theory of dilute gas mixtures, we can write expressions for the species diffusion velocities. Using the recent theory of iterative transport algorithms, [2] rigorous kinetic theory expressions can be derived for all of the needed transport coefficients. In particular, the thermal conductivity, the species diffusion coefficients, and the thermal diffusion coefficients are obtained by solving constrained singular linear systems. These approximate expressions are accurate and computationally much more cost-effective than a direct numerical inversion of the associated linear systems. Moreover, the approximate species diffusion coefficients and thermal diffusion coefficients automatically satisfy the mass conservation constraint [3].

Since radiation plays a key role in defining the structure and stability of flame-balls, it is necessary to construct a careful estimate of the radiative contribution. We assume that for hydrogen-air mixtures the only significant radiating species is H₂O. (For methane-air systems additional terms from CO and CO₂ will be included.) By utilizing an optically thin limit in which self-absorption of radiation is neglected, the divergence of the net radiative flux is given by: \( \frac{1}{r^2} \frac{d}{dr} \left( \frac{d}{dr} q_{\text{rad}} \right) = 4\pi \sum \mu_i B_i(T) \) where \( B_i(T) \) is the Planck function evaluated at the band centers of the contributing vibration-rotation or pure rotational bands whose integrated intensities are given by \( \mu_i [3,4] \).

Utilizing a modified hydrogen-air reaction mechanism [5] with the transport model employed in [1], we computed flame balls as a function of the equivalence ratio. Temperature and radii results were compared with our original computations. While the temperature and the size of the flame ball at the rich limit show only minor changes.
there is a modest increase of the rich flammability limit with the new mechanism. Only very small changes occur on the lean side but they move in the direction of increased agreement with the experiments [1]. We have also compared the temperature and radii as a function of the equivalence ratio for the new mechanism/old transport calculations with calculations made with the new mechanism and the transport and radiation models discussed above. Essentially no major differences were found. Nevertheless, due to its more accurate formulation, the detailed transport and band radiation models will be utilized in all future flame ball calculations.

**STUDENTS FUNDED UNDER RESEARCH:**

**TASK INITIATION:** 5/94  **EXPIRATION:** 4/98

**PROJECT IDENTIFICATION:** 962-22-05-60

**RESPONSIBLE CENTER:** LeRC

**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Journals**

Interactions Between Flames on Parallel Solid Surfaces

Principal Investigator: Dr. David L. Urban
NASA Lewis Research Center (LeRC)

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
The objective of the proposed research is to study the interactions between flames which are spreading or established over parallel solid surfaces and to determine the relative importance of radiative and convective heat exchange versus the potential reduction of oxidizer due to the presence of the second fuel surface. The results from this work will be used to improve existing models of flame spread over solid surfaces and to improve our understanding of fire spread and growth for practical fuel geometries.

Task Description:
The research will be conducted under a ground-based program. Low gravity testing will be conducted at the NASA LeRC drop towers and on NASA's low-gravity research aircraft. The objective of this work will be to determine the dependence of flame spread over parallel sheets on geometric parameters such as height and width and to use this understanding to select the optimum configuration for more extensive tests. These tests will determine the dependence of spread rate for parallel surfaces on fuel thickness, separation distance, oxidizer flow direction and rate and gas phase radiation. In addition, the controlling factors in the unstable regime at close surface separations will be established. Tests will also be conducted to determine the flame structure and burning rate for flames established over parallel thick fuels with forced flow between the plates. Finally, these results will be combined with numerical/analytical results to determine the importance of radiative feedback, between interacting flames and surfaces, on flammability and spread and burning rates.

Task Significance:
The interaction between flames on solid surfaces is of considerable practical importance since in most practical heterogeneous combustion systems, the condensed phase is distributed in more than one piece and this spatial distribution can strongly affect burning and/or spread rate. From a fundamental viewpoint, the interaction between parallel surfaces offers opportunities to simplify the boundary conditions, over those for isolated surfaces, as the opposing surfaces impose a plane of symmetry between the two surfaces and if the surfaces are sufficiently large, the radiation shape factors between the surfaces and between the flame and the surfaces approach unity. This geometry also provides interesting and unique opportunities to explore the relative effects of reactant diffusion rates, radiative heat transport and conductive transport in flame spread; these transport mechanisms have been identified as critically important in microgravity.

These phenomena will be more readily studied in nonbuoyant flames as the buoyant transport will be removed, simplifying analysis and increasing the importance of conduction and radiation. In the absence of strong buoyant flows, the interaction between flames on opposing sheets extends to greater separation distances, providing greater opportunities to study the interaction via the greater spatial resolution afforded by microgravity.

These results will be significant to low gravity flame spread research in general as the parallel geometry with its plane of symmetry between the fuel surfaces is in some terms simpler than the traditional case of a single sheet in an infinite oxidizer. The results will be an extension of the existing body of knowledge concerning flame spread and burning rate over flat surfaces. The work might also have practical benefits as radiant preheating is also very significant in fire spread in building fires. Due to the significance of flame interaction in terrestrial fire safety, it is important that flame interactions be studied in low gravity to assess their importance in spacecraft fire safety.

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Progress During FY 1995:

November
Downward flame spread tests were conducted using a point diffraction interferometer to study flame interactions in 1-g. The tests were very successful, the system seems appropriate for this problem and the results were very promising.

April
Purchase of interferometer components is underway. Two approaches are currently being pursued. The first involve replication of the breadboard system borrowed in previous months and the second involves modification of a CM-1 soot volume fraction extinction system to function as an interferometer. The designer of the CM-1 system has been retained to provide the needed modifications.

May
A summer student (Eric Falk) has begun conducting 1-g and 0-g experiments in the drop tower using one of the new PIG rigs. The first tests are focusing on comparison of the results with new sample holder with previous results.

The bulk of the interferometer components have been purchased along with the components necessary for g-data video overlay for the aircraft tests.

June
Drop tower tests by Eric Falk are continuing in the drop tower. The effect of sample width and separation distance are being examined for nitrogen diluent in 1-g and 0-g. The sample holder design has been modified to allow control of the distance to the leading edge for both sheets. The results with this sample holder are repeatable. The 0-g results are now showing a more appropriate trend with separation distance: transition from extinction to a maximum value and then decreasing asymptotically to the single sheet value as separation distance is increased. Similar results are observed in 1-g but at closer separation distances. It is likely that the sample holder used in the old Pizza Oven rig masked this effect.

A proof of concept test of assembling a point diffraction interferometer by replacing one component in one of the LSP soot volume fraction system breadboards was executed successfully. The field of view and fringe visibility were acceptable. Parts are being fabricated and procured to make a better implementation of the design. A calibration system is being designed by another summer student (Kathleen Tacina). We will calibrate the interferometer against thermocouple measurements in the boundary layer over a heated vertical flat plate.

July
Drop tower tests by Eric Falk are continuing. The effect of sample width and separation distance are being examined for nitrogen diluent in 1-g. The unsteady propagation effect seen previously is being examined in detail. Improved photography reveals that the pulsations are occurring at the edges of the paper sheets. Experiments have been conducted with various frame designs and materials to change the quenching effects. No significant changes have been observed in the presence of the instability to date.

An isothermal vertical heated flat plate has been assembled for testing of the interferometer. Thermocouple measurements have been made and temporal average values agree well with the classical results reported in the literature; however, fluctuations are evident in the temperatures. Due to delayed arrival of one of the interferometer components, the flat plate is being studied using one of the branch’s rainbow schlieren systems. This has been useful for visualizing the fluctuations in the boundary layer which have been significantly reduced by controlling the boundaries more carefully and limiting the influence of ambient air flows. These problems are similar to those observed in the LSP breadboard testing: a facility in which the HVAC can be deactivated is needed.

August
Measurements were made of the temperature field of a vertical heated flat plate using the Point Diffraction Interferometer. The results compared well with thermocouple measurements at distances more than a few
millimeters from the plate. Close to the plate, the interferometer measurements were significantly higher than the thermocouple measurements. This is believed to be a result of the absence of side plates in the system which allows the boundary layer to spill over the corners of the plate, increasing the optical path length near the plate.

The first design of an adaptor to convert the LSP soot volume fraction system was not successful due to the tight alignment requirements and manufacturing tolerances. A new adjustable design is being made and the hardware will be modified to allow modification of the LSP drop tower soot volume fraction system for this purpose.

A chamber was received from the fabrication shop. It is identical to those in the general purpose combustion rigs and will be used for 1-g testing with the interferometer.

**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

*Proceedings*


Principal Investigator: Prof. Arvind Varma
University of Notre Dame

Co-Investigators:
McGinn, Dr. P.J.
University of Notre Dame

Task Objective:
This project is an analytical and experimental investigation of the mechanisms of reaction and structure formation during combustion synthesis of advanced materials from elemental reactants. Many of the processes in the stages of the synthesis are affected by gravity, and conducting the synthesis under microgravity will elucidate the reaction mechanisms to give fuller understanding of the processes and their applications.

Task Description:
Previous normal-gravity studies have shown that the reaction and structure-formation mechanisms in the "gasless combustion synthesis" process involve several stages, including melting of reactants and products, spreading of the melt, droplet coalescence, diffusion and convection, buoyancy of solid particles and bubbles in the melt, nucleation of solid products, and crystal growth. This project aims to model, isolate, control, and optimize the above stages, taking advantage of the absence of the disturbing effects of gravity.

The experimental program includes two general approaches and two reaction systems, all to be conducted under normal gravity and microgravity. The macrocombustion approach involves the combustion synthesis initiated from pellets pressed from reactant particles, with the self-sustained reaction initiated by a hot-wire coil. The first system investigated is that of nickel (Ni) particles clad with aluminum (Al), which will react to produce a mixture of nickel aluminides, Ni₃Al and NiAl. The second system is that of a pressed powder of Ni, Al, titanium (Ti), and boron (B), which will react to form a mixture of Ni₃Al and TiB₂. The reactions are conducted in a combustion chamber charged with an inert atmosphere (argon) or under vacuum. An internal holder accommodates up to three reactant (green mixture) pellets with a common igniter coil. The experimental variables include the composition and porosity of the green mixture and the initial temperature of the reaction. Diagnostics measure the combustion-front propagation velocity, temperature-time history of the process, pellet size and appearance, and the microstructure and phase composition of the products.

The microcombustion approach involves the combustion synthesis initiated from separated powders, suspended by gas flow or other means. The microcombustion approach is novel, and it greatly simplifies the processes and their interpretation for modeling. The practical implementation of the microcombustion approach is complex, however. The microcombustion tests are to use the same elemental reactants, combustion chamber, and operational conditions as the macrocombustion approach, with appropriate changes in the internal holders and tube entries.

Tests on both approaches and systems will be conducted in microgravity using the NASA Lewis Research Center free-fall drop-tower facilities. The apparatus will be adapted for use in the NASA facility. Because of the rapid progress of the processes, the reaction can be completed in the brief microgravity time available in the drop towers.

Task Significance:
The study is the first to use microgravity in this method of combustion synthesis in order to isolate the process stages and understand the reaction and structure-formation mechanisms. The combustion synthesis promises great value to the production of solid and porous materials for high-temperature aerospace and industrial applications.
Progress During FY 1995:

The first year of a planned two-year effort is complete. The macrocombustion Al-clad-Ni-reactant combustion system was investigated with ignition at the top of the vertically oriented pellets. The system was difficult to ignite at ambient temperatures, and the reaction exhibited complex modes of combustion waves, at times spinning, oscillating, interrupted, or stable. Preheating the reaction pellet (green mixture) to 200°C improved the ignition stability and increased the combustion temperature; but the combustion front was still irregular. Preheating above 200°C had little further influence. Examination of the microstructure of reacted and partially reacted products showed that the Al core melted first and reacted to form Ni₃Al. A rapid reaction occurred later in the process when the Ni shell cracked, releasing Al to accelerate the reaction.

The Al-Ni system was also investigated with the green mixture formed from pressed elemental powders rather than from clad particles. For the powder macrocombustion reactants, the combustion wave at ambient conditions was stable, but it had a spinning mode. Preheating, particularly over 300°C, had a strong effect on the powder reaction, increasing the combustion temperature and wave velocity. For both Al-Ni-reactant configurations, the combustion was complete; but the product microstructure was non-homogeneous, with voids and inclusions.

The Al-Ni-Ti-B combustion system was investigated at ambient initial temperatures only. The Ti-B strongly influenced the reaction, with combustion temperature and velocity increasing with increasing Ti-B content (increasing TiB₂ product). Green mixtures with lower Ti-B contents showed combustion waves propagating in a spin mode. With contents of 40 wgt% Ti-B or greater, the combustion wave was stable, although the wave front was not planar. Tests were also conducted with the ignition at the bottom or the side (horizontal pellet). Compared to the usual top ignition, the alternative combustion-wave directions produced an increase in combustion-wave velocity but little difference in combustion temperature or product morphology. Electron-microscope analyses of the products showed complete combustion (except for traces of Al and Ni for compositions with only 5 wgt% Ti). The microstructure for all compositions was non-homogeneous, showing "islands" of Ni₃Al embedded in a TiB₂-Ni₃Al matrix.

The normal-gravity macrocombustion tests showed a strong influence of gravity, not only on the combustion mode but also on the product grain growth and crystallization. The test chamber, power supply, diagnostics, and mounting systems were then modified for installation on a standard test frame for microgravity experiments in the NASA Lewis 2.2 Second Drop Tower. Normal-gravity bench tests were first performed with the chamber and ignition system at the Lewis Research Center. These preliminary tests showed that the modified ignition system performed adequately and the entire process from ignition to reaction could be completed in 2.2 seconds, the microgravity time limit. Certain improvements in the ignition programming, sample mounting, and vacuum connections are still in progress, based on deficiencies identified in the bench tests. A Test Request and Safety Request are under review, for approval prior to the microgravity experiments scheduled for the winter of 1995.

**STUDENTS FUNDED UNDER RESEARCH:**

| BS Students: | 3 |
| MS Students: | 0 |
| PhD Students: | 1 |

**PROJECT IDENTIFICATION:** 962-22-05-56

**NASA CONTRACT NO.:** NAG3-1644

**RESPONSIBLE CENTER:** LeRC

**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

Proceedings


Printed: 28 FEB 1996

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II. MSAD Program Tasks — Ground-based

Discipline: Combustion Science

Studies of Wind-Aided Flame Spread Over Thin Cellulosic Fuels in Microgravity

Principal Investigator: Prof. Indrek S. Wichman
Michigan State University

Co-Investigators:
DiBlasi, Prof. C.
University of Naples

Task Objective:
The objective of this research is to develop fundamental theoretical models for wind-aided flame spread over thin solid fuels in low gravity. An important aspect of this study is to develop detailed models for the combustion process in the flame anchoring region, the so-called "Triple-Flame" zone and for the fuel pyrolysis chemistry.

Task Description:
The theoretical modeling effort will explore two tracks, one fundamental, the other directly concerned with the wind-aided flame spread calculations. The fundamental part will be detailed examination of the triple flame problem near the leading edge of the spreading flame. The results of this model should provide the heat flux distribution to the supporting fuel surface. The solution techniques employed will be asymptotic and approximate methods combined with numerical methods. The second model to be examined will be wind-aided flame spread calculations. An order-of-magnitude analyses of the governing equations will be performed to identify the correct scaling of the wind-aided flame spread problem, particularly near the flame attachment point. The properly scaled equations will be solved numerically to obtain flame spread rates. The results of this study will be compared against experimental data obtained in low gravity facilities at the NASA Lewis Research Center as a part of other on-going programs.

Task Significance:
Theoretical models for the combustion process occurring at the flame anchoring point will be developed to better understand the triple flame structure. Using this basic model, numerical codes will be developed to predict wind-aided flame spread rates over thin solid fuels.

Progress During FY 1995:
Developed a simple description of the triple flame structure using a pseudo 1-D theory embedded in a 2-D calculation of the mixture fraction field, onto which the simple numerical calculation are superposed. The results are presented at the AIAA meeting.

Developed a more sophisticate modeling approach that incorporates 2-D methods. Two levels of comparison have been developed though not yet fully implemented.

Developed a detailed Navier-Stokes (laminar) numerical scheme for the splitter-plate triple flame problem. Preliminary results provide good justification for the previous simplified models.

Students Funded Under Research:

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Project Identification: 962-22-05-53
Responsible Center: LeRC

Bibliographic Citations for FY 1995:

Presentations
High Pressure Droplet Combustion Studies

Principal Investigator: Prof. Forman A. Williams  
University of California, San Diego

Co-Investigators:
Kono, Prof. M.  
University of Tokyo, Japan
Sato, Dr. J.  
IHI, Inc., Japan

Task Objective:
The focus of this international cooperative effort is on high-pressure combustion of miscible binary fuel droplets. This is a joint research program pursued by investigators at the University of Tokyo, the University of Tohoku, the University of California, San Diego, and the NASA Lewis Research Center (LeRC). It involves construction of an experimental apparatus in Tokyo and mating of the apparatus to a NASA-LeRC 2.2-Second Drop Tower in Cleveland. Experimental results are to be analyzed jointly by the Tokyo, UCSD, and NASA investigators. The project was initiated in December, 1990, and has now involved three periods of drop-tower testing by Mr. Masato Mikami (U. of Tokyo) at LeRC.

Task Description:
The research accomplished thus far concerns the combustion of individual fiber-supported droplets of mixtures of n-heptane and n-hexadecane, initially about 1 mm in diameter, under free-fall microgravity conditions. Ambient pressures ranged up to 3.0 MPa, extending above the critical pressures of both pure fuels, in room-temperature nitrogen-oxygen atmospheres having oxygen mole fractions, X, of 0.12 and 0.13.

Task Significance:
The general purpose is to study near-critical and super-critical combustion of the droplets and to see whether three-stage boiling, observed at normal gravity, persists at high pressures in microgravity.

Progress During FY 1995:
The data from the last series of drops in the 2.2 second drop tower (two droplet array of a binary fuel at high pressure in air) were analyzed to obtain the following information: 1) droplet diameter as a function of time (when the droplet was visible), 2) flame shape as a function of time, 3) droplet lifetime, and 4) flame behavior as a function of time.

Flame contraction and separation during burning was observed for the first time. This occurs when the more volatile fuel component (heptane in this case) burns initially and a merged flame between the droplets appears. As the more volatile component is depleted near the surface of the droplet, small individual flames surround the droplets. After the droplet heats and the less volatile component begins to vaporize, a merged flame again appears.

The data are not sufficient for publication, so more tests will be performed in August 1995.

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Presentations
II. MSAD Program Tasks — Ground-based

Discipline: Combustion Science

High-Pressure Combustion of Binary Fuel Sprays

Principal Investigator: Prof. Forman A. Williams
University of California, San Diego

Co-Investigators:
Kono, Prof. M. University of Tokyo, Japan
Sato, Dr. J. IHI, Inc., Japan

Task Objective:
The objective of the proposed research is to improve understanding of the combustion of sprays of multi-component fuels at elevated pressures, extending from normal atmospheric pressure to pressures above the critical pressure of the fuel. In particular, explanation of the role of previously observed three-stage droplet combustion behavior and microexplosion in spray combustion is sought. The extent to which buoyancy influences the phenomena that occur is to be determined.

Task Description:
The objective is to be achieved by theoretical analyses taking into account concentration profiles of fuel constituents within the liquid, instabilities, droplet interactions, and conditions for achieving the limit of superheat, along with drop-tower experiments employing 1.4 and 2.2-Second Drop Towers. Binary mixtures of heptane and hexadecane will be studied first, using fiber-supported droplets of diameters from 0.8 mm to 1.5 mm in air at pressures from 0.1 to 5 MPa. Later work is planned to involve free droplets and other fuels and possibly microgravity facilities that afford longer test times.

This program is a joint program with several investigators in Japan. The Science and Technology Agency of Japan is supporting research by Dr. Michikata Kono, Professor, Department of Aeronautics, The University of Tokyo, Tokyo, Japan, Dr. Takashi Niioka, Professor, Institute of Fluid Science, Tohoku University, Sendai, Japan, and Dr. Jun’chi Sato, Senior Researcher, Heat and Fluid Dynamics Department, Research Institute, Ishikawajima-Harima Heavy Industries Co., Tokyo, Japan on "Combustion of a Fuel Droplet in High-Pressure Atmospheres under Microgravity Conditions" and on "Ignition of Fuel Droplets in High Pressure, High-Temperature Environments."

Task Significance:
The proposed research will improve our understanding of the mechanisms of the combustion of high pressure fuel sprays, such as those found in diesel and gas turbine engines.

Progress During FY 1995:
The data from the last series of drops in the 2.2 Second Drop Tower (two droplet array of a binary fuel at high pressure in air) were analyzed to obtain the following information: 1) droplet diameter as a function of time (when the droplet was visible), 2) flame shape as a function of time, 3) droplet lifetime, and 4) flame behavior as a function of time.

Flame contraction and separation during burning was observed for the first time. This occurs when the more volatile fuel component (heptane in this case) burns initially and a merged flame between the droplets appears. As the more volatile component is depleted near the surface of the droplet, small individual flames surround the droplets. After the droplet heats and the less volatile component begins to vaporize, a merged flame again appears.

The data are not sufficient for publication, so more tests will be performed in August 1995.
II. MSAD Program Tasks — Ground-based

**Discipline:** Combustion Science

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Printed: 28 FEB 1996
Laser Diagnostics for Fundamental Microgravity Droplet Combustion Studies

Principal Investigator: Dr. Michael Winter United Technologies Research Center

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
This research program seeks to investigate the range of applicability of advanced diagnostics to droplet combustion under microgravity conditions while generating fundamental information relevant to other ongoing NASA programs. Using a progressive approach, advanced laser diagnostics will progress from laboratory, to flight experiments, and ultimately to drop tower facilities. In this way, these diagnostics can be developed, while providing fundamental data on droplet transport and combustion phenomena. Through consultation with NASA personnel, and other researchers in the fields of microgravity droplet transport and combustion, priorities have been established with respect to measurement parameters. The prioritized measurement parameters are:

1. Flame front position.
2. Relative gas-phase flow around droplets.
3. Droplet surface transport and internal flow.
4. Liquid-phase thermometry.

Task Description:
An integrated diagnostic unit, capable of performing these measurements has been assembled under a previous contract. The unit consisting of NASA and UTC owned equipment has been transferred or applied to the current contract. All of the major components such as lasers, detectors and data systems are currently allocated to this effort. A self-contained, miniature, nitrogen-pumped dye laser system and two-dimensional intensified CCD imaging system will form the core of the unit. Procurement of a miniature Nd:YAG laser for integration in the Laser Diagnostics Droplet Combustion facility has been initiated. The laser will be provided by United Technologies for use in these experiments.

The testing program is proceeding with ever more challenging environments proceeding from the laboratory, to flight based tests. The diagnostic approaches and instrumentation are being verified in a laboratory setting under Task II. Experiments with the intent of reducing the risks associated with high impact and droplet drift are being conducted on the Learjet, DC-9 and KC-135 aircraft, with fiber suspended, or electrodynamically levitated droplets. Initial measurements are being performed on use of PLIF measurements to describe flame front position. Gas-phase flow dynamics will be determined by velocity measurements using particle image velocimetry. Measurement of droplet surface and internal transport will also be attempted. The applicability of liquid phase thermometry in a burning droplet is being pursued at NASA LeRC and frequent consultation toward that effort is being provided by UTRC.

Task Significance:
One of the most fundamental distinctions in combustion is whether a flame is a premixed or non-premixed flame. Condensed-phase materials usually serve as reactants in practical combustion devices, either through direct injection of a condensed-phase fuel into a combustor or the initial presence of a condensed-phase fuel within the combustor. These fuels generally burn as non-premixed flames. Additionally, most hazardous flames are multi-phase diffusion flames as well. Many of these combustion systems involve burning liquid-phase fuels which are comprised of many individual droplets.
Basic understanding is best advanced by well-controlled experiments and simplified calculations. A great deal of attention has been paid to studying the combustion of individual droplets, which is the simplest example of non-premixed combustion. These single-droplet flames provide an idealized geometry for investigating the interaction of the physical and chemical processes involved. A significant means of simplifying droplet combustion is to approach the phenomena in a microgravity environment. A great deal of activity is ongoing in this area, including calculations, drop towers, the Droplet Combustion Experiment (DCE), and glove-box experiments aboard the space shuttle. Drop tower data have shown deviations from calculated results, suggesting that the experimental conditions may differ from the idealized assumptions used.

Optical diagnostics offer several advantages over physical probes because they permit nonintrusive multi-point measurements. Nonintrusive measurements are of particular importance for droplet combustion and transport in microgravity environments, where physical contact would introduce an unacceptable level of perturbations. The resolution of these diagnostics can isolate transport to length scales much smaller than the droplet diameter. These techniques can be configured to instantaneously map an entire flow field in two and three dimensions, providing either qualitative or quantitative information on the distribution of a desired scalar or vector quantity.

**Progress During FY 1995:**

**Hardware:**
An apparatus for performing laser-induced fluorescence measurements of fiber supported burning fuel droplets aboard the NASA Learjet and KC-135 and DC-9 aircraft facilities was designed and built primarily in 1993 with testing and use in 1994. The rig houses a self-contained miniature nitroge-pumped dye laser system and two-dimensional intensified CCD and unintensified CCD imaging systems. The time code generators, two VCR's, a timing system, and MacIntosh Ilci computer with image digitization hardware form the data collection/storage system. Droplet deployment, ignition and synchronization to the timing electronics is accomplished via a unique system of radio controlled model airplane controls.

**Experiments:**
A new multi-level combustion platform has been fabricated which includes a levitator. This module will be a direct replacement to the original and can be replaced in a matter of minutes between flight tests, or parabolas.

The design will rely upon electrodynamic levitation and the electrostatic levitation electrodes will be passive. While this will increase the difficulties associated with 1-g testing, it will significantly increase the optical access and preclude flame interferences at 0-g where the flame standoff distances are significantly greater. Flight safety review for the levitator module have been completed.

A new levitator has been fabricated to provide greater optical access. The maximum allowable hole area in the center of the electrode needs to be less than 51%. With this constraint, the inner ring diameter will be increased to accommodate a hole large enough to allow passage of a laser sheet large enough to illuminate a flame with a standoff distance of ~ 10 diameters over a 1 mm diameter droplet. The exact dimensions of the device are scaled accordingly. The unit is operational and is being cycled and tested.

UTC will provide a new Nd: YAG laser from Big Sky Laser of Montana. The laser specifications are for 200 mj at 532 nm and 20 Hz.

A milestone of this project is the acquisition of 2-D images of OH fluorescence from a burning droplet in microgravity. The fluorescence may be excited from radiation at several wavelengths including the 0,0 band at 308 nm and the P1(1) transition at 282 nm. For either case, the fluorescence is observed in the 308 nm band. Clearly, excitation at 282 nm is preferred because the signal can be spectrally separated from the excitation radiation. The experiment will require a few millijoules of energy at 282 nm to excite the OH molecules over a laser sheet height of 15 mm. A cost effective laser design utilizes available equipment to produce a reliable system that can operate within the constraints of the microgravity environment. Flight regulations impose some restrictions on the size and weight of the experimental hardware.
A dye laser has been built to produce 564 nm light that can be frequency doubled in a KDP crystal to produce light at 282.2 nm. It will utilize the Big Sky Nd:YAG laser for pump power. A commercial dye laser module from UTRC inventory is being modified for this application. This module is compact and contains elements that allow tuning the output wavelength over the gain region of the dye, in this case, Rhodamine 590. The cavity is a modified Littrow configuration and has no mechanism for flowing the dye solution, it uses a cuvette that will need replenishing between flight parabolas. The output will be doubled with KDP crystals. The compact Nd:YAG laser and modified dye laser module will easily fit within the hardware rack used for the flight experiments.

Results:
Ignition standoff data for the methanol indicated that the ignitor wire needed to be as close as physically possible to achieve reliable ignition. In contrast, the position of the ignitor needed to be several diameters away from the droplet surface to achieve reliable ignition in ethanol.

Successful radical chemiluminescence, PLIF and visible flame luminosity images were obtained. Initial analysis indicates that the broadband visible flame luminosity and radical chemiluminescence agree well in mapping the flame front position. Comparison between the broadband visible flame luminosity and simultaneous PLIF images of NaCl seeded burning methanol droplets indicates that the visible luminosity is not a reliable marker of the flame front position, being dominated by Na chemiluminescence for droplets seeded with NaCl.

PROJECT IDENTIFICATION: 962-22-05-44
NASA CONTRACT NO.: NAS3-27265
RESPONSIBLE CENTER: LeRC
Combustion of a Polymer (PMMA) Sphere in Microgravity

Principal Investigator: Dr. Jiann C. Yang
National Institute of Standards and Technology

Co-Investigators:
Hamins, Dr. A.
National Institute of Standards and Technology (NIST)

Task Objective:
The Objective is to determine the combustion characteristics of spherical PMMA particles in reduced gravity.

Task Description:
Experiments will be conducted to study both supported and unsupported PMMA particles burning in a controlled environment under microgravity conditions. The test conditions will be room temperature and pressure (25 °C and 1 atm). The ambient oxygen concentration will range from 19% to 70% and the particle size will be 3, 5 and 7 mm. Different ignition methods will also be tested. The diagnostic system will consist of a CCD video camera and a high speed movie camera. The video and film images will be analyzed to provide information on burning history, flame location, soot formation, burning rate, and other combustion characteristics.

Task Significance:
This experiment is the first attempt to study the combustion of a solid fuel sphere under microgravity conditions. The one-dimensional configuration will simplify the data interpretation and the test results will provide some fundamental information to solid fuel combustion science.

Progress During FY 1995:
Preliminary ignition experiments of PMMA spheres in 1g were conducted with different devices. The spheres of 5 mm in diameter were easily ignited with a small propane torch. However, ignition of the sphere (without preheating) could not be achieved when a spark ignitor (with an on-time of more than 2 secs) was used. Ignition of solid PMMA spheres was then further tested. Spark ignition of a PMMA particle coated with a thin layer of ethanol was unsuccessful. Neither was with a preheated particle. However, ignition was always achieved using a small torch or a match. The PI concluded that a micro-torch might be the appropriate means to ignite particles. Therefore, a micro-torch is being designed for ignition. The system is very similar to a cigarette ignitor using liquid butane.

The fabrication method of PMMA spheres with embedded thermocouples was devised and planned. PMMA powder will be dissolved in a solvent and an initiator (benzoyl peroxide) will then be added to the solution. The polymer solution will be cured in an oven into a spherical mold and a thermocouple inserted into the solution. Some technical problems still remain to be solved, e.g. how to avoid bubble formation in the PMMA sphere. A technique is also being tested to accurately embed a thermocouple junction at desired location inside a PMMA particle. The first step is to heat the thermocouple by passing an electric current through it. Then the heated thermocouple is lowered into a PMMA particle by using a linear positioning stage.

Some experimental equipment were consolidated. A LO-CAM (model 50) was located in-house at NIST. A scanner capable of digitizing 16 mm films was also acquired. Imaging software such as SigmaScan/Image is needed.

Dr. J. C. Yang (PI) and Dr. A. Hammins (Co-I) visited NASA Lewis Research Center and toured the microgravity facilities. They obtained first-hand information on rig construction, test procedures and other related issues through a series of discussions with people from the Space Experiment Division.

Printed: 28 FEB 1996
II. MSAD Program Tasks — Ground-based

Discipline: Combustion Science

PROJECT IDENTIFICATION: 962-22-05-63
RESPONSIBLE CENTER: LeRC

Printed: 28 FEB 1996
Study of Two-Phase Flow and Heat Transfer in Reduced Gravities

Principal Investigator: Dr. Davood Abdollahian

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
The objective is to conduct two-phase flow instability studies in vertical upflow and downflow, in both a normal gravity environment and in low gravity aboard an aircraft, to ascertain the effect of gravity on instability and boiling mechanisms.

Task Description:
The approach is to design and build a recirculating flow boiling loop which would be used aboard an aircraft to test different two-phase flow instability phenomena and measure the conditions at which critical heat flux occurs. Specifically, the instability phenomena to be examined are the following: nucleation instability, flow pattern instability, excursive instability, oscillatory instability, and density wave instability. Testing would consist of examining the flow stability and critical heat flux in normal-gravity vertical upflow and downflow as well as in low gravity aboard an aircraft.

Task Significance:
Two-phase instabilities have been responsible for a multitude of fatal and costly accidents on Earth in the oil, electric power and nuclear industries. In addition, by understanding critical heat flux better, it may be possible to improve the cooling of microcircuitry used in the electronic industry thus improving the capability of that circuitry.

Progress During FY 1995:
The flow loop and instrumentation were fully integrated into the standard Learjet racks and tested. Normal gravity testing has commenced with the test section being aligned vertically for these tests to obtain the axial symmetry found in low gravity flows. After these tests have been completed, the test section will be repositioned horizontally within the Learjet racks. The data acquisition and control software has been modified in order to provide for the fast data acquisition rate and the necessary protective measures required to avoid damaging the flow loop from both burnout and flow loop overpressurization. Hydrostatic certification of the flow loop was completed and the flow loop was charged with freon. Single phase subcooled heating tests were conducted within the test section to assess the heat loss through the insulation to ambient conditions. Testing is focusing on the parallel channel flow instability and determining the critical heat flux in both normal and reduced gravity. The parallel channel flow instability tests are to be conducted between the two phase test section and a parallel single phase line. The critical heat flux tests are to be performed with this parallel single phase line closed. Initial tests for critical heat flux have shown that even with this parallel single phase line closed, a parallel flow instability occurs between the test section and the pump bypass line at conditions not anticipated during the design of the flow loop. In order to isolate these instability effects for the critical heat flux tests, the valve in the bypass line will be closed, and the positive-displacement pump's speed will be controlled as a direct means of setting the flowrate. DC-9 testing is planned for early FY96.

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Printed: 28 FEB 1996

II-392
Colloids & Nucleation

Principal Investigator: Prof. Bruce J. Ackerson

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
Direct observations will be made of the nucleation and early growth of crystals comprised of polymethylmethacrylate (PMMA) "hard" colloidal spheres in an index matching solvent. Colloidal samples will be prepared in a particle volume fraction range which exhibits crystals of colloidal particles at equilibrium. These samples will be shear melted and the subsequent appearance of crystals monitored by direct observations/video camera recording at the first order Bragg scattering angle.

Task Description:
1. Apply classical nucleation theory in the limit applicable to hard sphere interactions to compare against data. Modern analytic (density functional) and simulation results will be compared with data as appropriate.

2. Establish the practical limiting density supersaturation that can be studied without interference due to sedimentation induced convection.

3. Conduct an exploratory investigation of the density or order parameter fluctuations leading to a critical nucleus, and if possible, characterize the size and time dependent characteristics of these fluctuations.

Task Significance:
The experimental observations of nucleation and early growth of crystals comprised of "hard" colloidal spheres will provide a critical test of classical nucleation theory.

Progress During FY 1995:
A fast algorithm was created in order to separate the crystals from the image background. This was needed since conventional structure analysis algorithms on hand could not compensate for time dependent background gradients in the images, nor did the algorithms accurately detect crystal images of widely varying contrast with the background. Since the image background gradients are not constant with time, simple initial background image subtraction would not suffice. Given that the crystal structures occupy a significant portion of the images, attempts at local mean pixel value calculations were futile if the calculations didn't take into account whether or not sampled pixel values belonged to crystals in the image. The final technique which did work was to calculate point-by-point the cumulative pixel value histogram for a region of appropriate size surrounding the pixel of interest and to evaluate where the pixel would map to if the cumulative histogram were equalized. Once this is done, a commercial particle detection algorithm is used to produce statistics on the crystals. It calculates area, perimeter, location, as well as a number of statistics for determining the morphology of the crystal images. We wrote automation code to make the processing system produce these statistics for images where the crystal population density is low enough that the images need not be modified further than the output of the crystal/background separation algorithm provides.

All of the optical equipment was mounted on a rail system. A goniometer and angle indicators were added to the arrangement to reproduce and record camera angles and sample angles with respect to the incident light sheet. Initially, a 35mm camera was used and exposures lasting thirty seconds on slide film were taken of crystal formation. An illumination system was made to transfer the slides to the computer. This is an awkward process since the slides can easily acquire dust, are easily scratched, and cannot be uniformly illuminated. The optics of the system were improved by the addition of a new CCD camera with low dark current noise capable of long term...
image exposure. This camera provides us with the means of bypassing the image background inhomogeneities induced by projecting 35 mm. slides into the image processor. Software and hardware were created to control the camera and to interface it with the existing frame grabber.

The sample preparation of the 430 nm radii particles was completed. In order to produce adequately clean cells, we were required to design and construct an acetone vapor rinsing system which provides a dust-free rinse at the final stage of cell processing. Once the cells were clean, six samples were created with estimated volume fractions of 0.495, 0.500, 0.505, 0.510, 0.515, 0.540. The samples were mixed over more than 24 hours and then set to rest. Crystals appeared earliest in the 0.540 volume fraction cell (~1 hour, 30 minutes) and latest in the 0.500 volume fraction cell (~3 days). Although the particles in these samples are on the order of two times larger than previous particles analyzed in related experiments, the crystals appear to be significantly smaller. We will try to understand the small crystal size on the basis of settling induced shear stress comparable to the crystal yield stress. Because the crystal size is smaller than expected making photographic analysis more difficult, we will proceed using our small supply of 0.22 micron radius particles which are known to produce larger crystals.

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**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Presentations**


II. MSAD Program Tasks — Ground-based

Stability Limits and Dynamics of Nonaxisymmetric Liquid Bridges

Principal Investigator: Prof. J. Iwan D. Alexander
University of Alabama, Huntsville

Co-Investigators:
Perales, Dr. J.M. Universidad Politecnica de Madrid
Meseguer, Dr. J. Universidad Politecnica de Madrid

Task Objective:
The objectives of the proposed work are:

1. To determine the stability limits of Nonaxisymmetric liquid bridges held between non-coaxially aligned disks.
2. To examine the dynamics of Nonaxisymmetric bridge configurations and Nonaxisymmetric oscillations of initially axisymmetric bridges.
3. To experimentally investigate the vibration sensitivity of liquid bridges under terrestrial and low gravity conditions.

Task Description:
The program is to have simultaneous experimental and theoretical efforts. Experimentally, normal-gravity tests using the Plateau method will be conducted to study the equilibrium shapes and stability limits of various orientations of the liquid bridge, and to study the sensitivity of liquid bridges to axial and lateral vibration.

A numerical model will be developed using Picard iterative procedure to study the dynamics of Nonaxisymmetric bridges subject to g-jitter and the vibration sensitivity of liquid bridges.

Task Significance:
Liquid bridge stability is an important factor in determining the stability of molten zones associated with floating zone crystal growth experiments. Such understanding can help better define the vibration isolation requirements for in-space processing experiments, and can greatly enhance the chances of obtaining new and better quality semi-conductor crystals.

Progress During FY 1995:
Our research involves an experimental and theoretical investigation of the statics and dynamics of nonaxisymmetric bridge configurations and nonaxisymmetric oscillations of initially axisymmetric bridges. During FY95, the effects of nonaxial acceleration on the minimum volume stability limit have been examined using a finite difference method coupled with a mapping technique to solve the equilibrium equations. The minimum volume stability limit was found to be substantially modified as the lateral component of acceleration is increased. It appears to approach, but not meet, the "zero Bond number" minimum volume limit for purely axial acceleration. This analysis has since been extended to include an investigation of maximum volume limits.

Together with coworkers at the Universidad Politecnica in Madrid we have studied the effect of vibration on the stability limits of bridges and the modification of the static stability boundaries. We have also developed two numerical methods for the description of three dimensional oscillations of liquid bridges. The first method is restricted to relative volumes of less than about 1.5. The second method is quite powerful, especially for problems involving liquid fragmentation and coalescence. The third method is based on a VOF-type approach and can handle both large and small volume bridges up to and beyond the point of breaking.

Experiments have been carried out in three areas: Lateral shearing, squeezing, and force measurements; Stability limits, symmetric and nonsymmetric breaking behavior of initially axisymmetric bridges; Vibration dynamics and
II. MSAD Program Tasks — Ground-based

Discipline: Fluid Physics

breaking behavior. The bulk of the experimental work involved force measurements using a force-deflection apparatus to measure the total force exerted by a liquid bridge on the lower disk. Measurements have been compared to theoretical predictions for various relative volumes and aspect ratios and agreement is good. We have recently installed a pressure transducer on the lower disk. This will allow us to carry out force measurements for conditions that are not accessible to the cantilever system.

For coaxial disk supports, the stability of nonaxisymmetric bridges subject to axial gravity has been investigated experimentally and theoretically. This is the first attempt that we are aware of to determine the stability limit of nonaxisymmetric bridges for ranges of relative volume and aspect ratio beyond the maximum volume margin for axisymmetric bridges.

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**TASK INITIATION:** 12/92  **EXPIRATION:** 12/95

**PROJECT IDENTIFICATION:** 962-24-05-60

**NASA CONTRACT NO.:** NAG3-1384

**RESPONSIBLE CENTER:** LeRC

**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Journals**

**Presentations**


II. MSAD Program Tasks — Ground-based

Discipline: Fluid Physics

Investigations of Multiple-Layer Convection

Principal Investigator: Prof. C. D. Andereck
Ohio State University

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
The convection patterns that develop in two-layer fluid systems are to be studied. The principal objectives are:

1. To test the predictions of theoretical investigations.
2. To study the interaction of the pattern in one layer with the pattern in the other layer.
3. To determine the role of the geometry of the test cell on the patterns that emerge.
4. To determine the impact of surfactants at the fluid interface on the interface profile and the patterns that form.

Task Description:
An experimental investigation of natural, buoyancy driven, convection in a system consisting of two superposed layers of immiscible fluids will be performed. Narrow, pseudo-one-dimensional, test cells will be constructed with insulating side walls and conducting top and bottom walls. Temperature control of the horizontal surfaces will be provided initially by circulating known temperature water next to these surfaces. Shadowgraph visualization techniques, which are sensitive to index of refraction variations resulting from temperature gradients in the fluids, will be used to determine the flow patterns. This allows for observations of the basic dynamics of competing instabilities in the two layers. A direct comparison will then be possible between existing general theories of pattern competition, and experimental results for a specific system. This will also provide a test for direct numerical simulations of two fluid layer systems. Further work to be performed includes studying the deformation of the interface as a function of control parameter and with the introduction of surfactants. Finally, the geometry of the cell will be varied. To introduce periodic boundary conditions in one dimension we will construct an annular cell. Another variation will be to construct a cell with large extent in both horizontal dimensions, thereby freeing the system to choose more complex competing patterns.

Task Significance:
Multiple-layer convection is of interest in geophysical, astrophysical and industrial settings. It is also of importance as a fundamental pattern forming nonequilibrium system. These experiments will provide a quantitative test of our theoretical understanding of this fluid dynamical system. Results on the pattern formation processes may also be directly relevant, in thin layer limits, to understanding and improving film coating processes.

Progress During FY 1995:
We have constructed and used a first test cell, and have employed Schlieren imaging to view the convection patterns. The cell consists of glass sidewalls and aluminum blocks on the top and bottom. The dimensions are such as to impose a strong alignment of convection rolls with their axes parallel to the optical path. The top and bottom blocks are cooled and heated, respectively, by water from temperature controlled baths. One of the major problems in this experiment is in fluid selection. The first fluids used were a silicone oil, Rhone-Poulenc Rhodorsil 47v10, as the upper fluid, and Fluorinert FC-70, from 3M Corporation. These fluids were chosen on the basis that they are nearly immiscible, and because their thermal and flow properties are similar enough that convection begins in both layers almost simultaneously. The Schlieren system consists of a high-intensity red LED, a large collimating lens to produce approximately parallel light through the side of the cell, a focusing lens, knife-edge and a CCD video camera with a PC-based image capture and analysis system.
The procedure followed in data collection is to first establish a desired depth ratio for the two fluids and then allow the system to equilibrate for at least 12 hours at zero temperature difference between the two aluminum blocks. The temperature is then ramped by approximately 0.1 °C, and the system is left for 3 hours to reach a steady state before data taking at that temperature difference begins. Time dependence in this system, when it occurs, has a period on the order of one hour, so a data run consists of acquiring a single video line in each layer every 30 seconds over a total period of 6 to 8 hours. The effective ramping rate therefore is of the order of 0.2 °C/day. The frames are then put together to produce space-time plots, or power spectra in space and time are computed to obtain pattern wavelengths and frequencies.

We expected that the system would be subject to oscillatory coupling of the roll patterns in the two layers, on the basis of numerous theoretical studies. But this had never been seen in experimental realizations. There are two fundamental coupling mechanisms possible. Thermal coupling is the alignment of the hot rising fluid in both layers, causing rolls that are aligned one above the other to turn with the same sense, either clockwise or counterclockwise. Mechanical coupling is the alignment of the cold falling fluid in one layer, say the lower, with the hot rising fluid in the upper layer, with the result that the rolls turn with opposite senses, in a gear-like fashion. The oscillations are predicted to be between a thermally coupled state and a mechanically coupled state over time. Weakly nonlinear analysis of the problem by another group has shown that time dependence arises as either a standing wave of coupling oscillations in a finite system, or as a traveling wave in a large system. They also predicted the type of coupling expected at onset, depending on the depth ratio of the two fluids. Computations specific to our fluids were performed by Y. Renardy, and showed similar behavior.

The principal result is that we have found experimentally oscillatory behavior similar to that predicted by theory near the onset of patterns. Specifically, we began our experiments with nearly equal layer depths, where numerical studies predicted the behavior to be oscillatory at onset. At this fraction, however, we found only mechanical coupling, up to large temperature differences. From here, we systematically decreased the Fluorinert depth until it reached ~39% of the total cell depth, where we discovered oscillations slightly above the onset of convection. Oscillations near onset continued at lower depth ratios, until the lower fluid depth was ~34% of the total cell depth, where no oscillations were observed, and the coupling was predominantly thermal. Although the onset parameters were quite different from the predictions, the frequencies and wavelengths of the oscillatory state were close to the theory.

We are now attempting to complete our systematic survey of the behavior for this pair of fluids in the rectangular cell. Since the oscillatory behavior did not occur at the onset of the patterns we are also attempting to understand this. One possibility is that the ends of the cell play a strong damping roll in the oscillatory onset. Therefore we are constructing an annular cell, which should eliminate end effects and perhaps change the character of the oscillatory states. This cell is nearly operational and should provide an important complement to the results from the rectangular cell.

**STUDENTS FUNDED UNDER RESEARCH:**

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**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Presentations**


II. MSAD Program Tasks — Ground-based

Discipline: Fluid Physics

Electrokinetic Transport of Heterogeneous Particles in Suspensions

Principal Investigator: Prof. John L. Anderson

Carnegie Mellon University

Co-Investigators:

Garoff, S.

Carnegie Mellon University

Task Objective:

There are three objectives to our research program. The first is to complete a hydrodynamic theory for the motion of slender colloidal particles in electric fields. The other two objectives involve experimental measurements. The first of these is to design and develop a microelectrophoresis apparatus and video imaging system that would enable us to follow the kinematics of colloidal doublets in electric fields. The second experimental objective is a study of the rotational rates of doublets of latex particles in solutions at different ionic strength.

Task Description:

1. The electrophoresis of nonuniformly charged slender particles, such as cylinders and prolate spheroids, is analyzed in terms of existing models for the hydrodynamics of such particles. The basic idea is to apply the Lorentz reciprocal theorem of Stokes flow with the concept of electroosmotic "slip velocity" on the surface of the particle to obtain explicit algebraic expressions or quadratures for the translational and rotational velocities of the particle of arbitrary contour shape (straight, curved, helical, etc.). This theory can be directly extended to model the electrophoresis of uniformly charged slender particles in nonuniform electric fields.

2. The microelectrophoresis apparatus must permit observation of particle motion in a constant, one dimensional electric field at field strengths ranging from 0.1 to 50 V/cm. The optical quality of the cell is important. In addition, a method for recording and analyzing position/orientation versus time for single particles and aggregates must be developed. Temperature control to better than ±1°C is desired to avoid complications of convection; however, in analyzing the rotational motion natural and forced convection resulting from the electrical field is not too much of a problem. In addition to developing this apparatus, the images must be analyzed in a way to interpret the data in terms of the existing hydrodynamic models for electrophoretic rotation.

3. Colloidal doublets formed by coagulating two latex particles of different zeta potential (i.e., different surface charge) rotate when an electric field is applied. Two theoretical models, differing only in the physical picture of the doublet, are available for the angular velocity of the doublet as a function of sphere sizes and zeta potentials. We are collecting data at different solution conditions (pH, ionic strength) for comparison with these two models.

Task Significance:

1. Many colloidal systems are not composed of spherical, uniformly charged particles. For example, clays are disks with edges that differ from the faces, and most inorganic colloids have different crystal planes exposed at the surface thereby generating a "patchiness" to the charge. Aggregates of particles also create heterogeneous supraparticles that could result in chains with a distribution of zeta potential along their length. Our theoretical work on electrophoresis of charged slender particles expands the range of understanding heterogeneous systems.

2. The now classical DLVO theory of the energetics between two colloidal particles in aqueous media predicts two states of adhesion between two spheres. The first is a "primary minimum" in the potential energy which occurs when the two spheres essentially touch. The second state of adhesion is in the "secondary minimum" where electrostatic repulsion balances attraction caused by London dispersion forces; the secondary minimum is characterized by gap distances of order 4-8 Debye screening lengths. From hydrodynamics we expect that a doublet in the primary minimum would rotate like a rigid body in an electric field, with the more positive sphere rotating toward the field direction, while in the case of a secondary minimum the apparent doublet rotation occurs with each sphere rotating at a different angular velocity to keep the hydrodynamic torque on it equal to zero. The difference in
II. MSAD Program Tasks — Ground-based Discipline: Fluid Physics

rotation rate predicted by theory for these two cases is a factor of 3. We believe that electrophoretic rotation is a tool to probe the state of adhesion of colloidal aggregates.

Progress During FY1995:

1. A theory for the behavior of uniformly charged particles in spatially varying electric fields was developed. This theory makes interesting predictions regarding alignment of rod-like and disk-like particles in converging/diverging fields, which could be used to separate such particles.

2. The first set of experiments on the electrophoretic rotation of doublets formed from dissimilar latex spheres was concluded and the paper reporting the results has been accepted for publication. These experiments indicate that the latex doublets were always in a rigid-body conformation, even when the classical DLVO theory for colloidal forces predicts the doublets were in a deep secondary minimum with a significant fluid gap (10-20 nm) between the surfaces. These results mean either that the DLVO theory is quantitatively in significant error (i.e., there was no secondary minimum), or that the surfaces of the two particles were restricted in their relative lateral (tangential) motion even though the doublet was in a secondary minimum.

3. New experiments using the electrophoretic force acting on heterogeneous doublets when they align with an applied electric field failed to show breakage into single particles even at fields 50 times greater than the colloidal restoring force predicted from DLVO theory. These electrophoretic displacement experiments with aligned doublets are continuing. The rotation and displacement experimental protocols we have developed give us a tool to probe the fundamental colloidal forces acting both perpendicular and parallel to the surfaces of the individual particles in proximity to each other.

**Students Funded Under Research:**

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**Task Initiation:** 1/93  **Expiration:** 1/96

**Project Identification:** 962-24-08-09  **NASA Contract No.:** NAG8-964  **Responsible Center:** MSFC

**Bibliographic Citations for FY 1995:**

**Journals**

**Presentations**

Anderson, J.L. "Probing the structure of colloidal doublets by electrophoretic rotation." Seminars at van't Hoff Laboratory in U. Utrecht (The Netherlands), University of Wageningen (The Netherlands), U. Sydney (Australia), U. South Australia, U. Melbourne (Australia).
II. MSAD Program Tasks — Ground-based

Discipline: Fluid Physics

Experimental Study of Liquid Jet Impingement in Microgravity: The Hydraulic Jump

Principal Investigator: Prof. C. T. Avedisian
Cornell University

Co-Investigators:

No Co-I's Assigned to this Task

Task Objective:
The primary objective of this task is the experimental study of liquid jet impingement in microgravity. The proposed experimental study is aimed at understanding the effect of gravity on a hydraulic jump formed during impingement of a liquid jet on a target plate.

Task Description:
This study is important in applications which involve cooling of a heated surface by a relatively colder liquid jet since the heat transfer rate is strongly affected by the velocity (and hence the thickness) of the liquid film spreading on the surface. The principal investigator plans to examine the effect of gravity on the location of the hydraulic jump created by an impinging water jet onto a flat surface.

Task Significance:
This experimental study will test the validity of the theoretical predictions, and provide a first step towards more detailed studies of heat transfer and phase change processes associated with jet impingement cooling of heated surfaces in microgravity environments.

Progress During FY 1995:
The progress for the project so far were: (1) four stainless steel sharp-edged orifice plates were manufactured and tested, (2) the search for suitable flow visualization methods to reveal the details of the fluid flow near the hydraulic jump, and (3) the visualization of hydraulic jump itself. Some preliminary visualization were made of hydraulic jumps under normal gravity. The primary purpose was to try various lighting arrangements for photography. The hydraulic jump profile was obtained by using a silhouette photographic method to provide a subsurface view of a jump. Experiments were also performed to examine the effect of down stream jump height on the location of hydraulic jump. The experiment will also be initiated to visualize the transition of the hydraulic jump from normal to microgravity in the drop tower.

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Task Initiation: 6/94  Expiration: 7/95
Project Identification: 962-24-05-34
NASA Contract No.: NAG3-1627
Responsible Center: LeRC

Printed: 28 FEB 1996
II. MSAD Program Tasks — Ground-based

Discipline: Fluid Physics

Studies on the Response of Emulsions to Externally-Imposed Electric and Velocity Fields:
Electrohydrodynamic Deformation and Interaction of a Pair of Drops

Principal Investigator: Prof. James C. Baygents
University of Arizona

Co-Investigators:
Stone, H.
Harvard University

Task Objective:
The objective of this study of multiphase electrohydrodynamic flows is to determine the electric field-induced microstructural response of two-phase systems, as well as to develop an improved understanding of the physico-chemical and transport process common to a variety of low-gravity flows; we examine the motion, deformation, and interaction of pairs of viscous drops owing to an applied electric field.

Task Description:
The research is a comprehensive numerical and theoretical study of several phenomena common to multiphase flows where electrical fields are used to manipulate the microstructure in the absence of any buoyancy-induced fluid motions. This class of problems appears in typical materials handling, physico-chemical processes (e.g., emulsion breaking and drop coalescence) and bioseparations processes (e.g., aqueous two-phase partitioning).

This investigation is designed to use numerical solutions to develop an improved quantitative understanding of the effect of electric fields on typical two-drop (pair) interactions that lead toward coalescence. Cases will be studied where the potential distribution is influenced by conduction processes in the limit where viscous effects dominate the hydrodynamics. Both the effects of uniform and simple nonuniform (imposed) fields will be studied. The "leaky" dielectric model and the Stokes equations will be employed to describe the constitutive behavior of the dielectric media. The numerical calculations will use established integral equation methods for solving this class of time-dependent free boundary problems.

Task Significance:
The systematic study will lead to a quantitative description and understanding of electrically-driven low-gravity fluid motion, particularly as these flows pertain to the manipulation and positioning of drops and bubbles with electric fields. Such flows are significant for several reasons. First, in the absence of the usual gravitational forces, electric fields can be used to effect phase separation processes that are controlled by drop coalescence (e.g., emulsion breaking). Second, electric fields have long been envisioned as a tool for containerless processing of materials, inasmuch as the fields can be used to manipulate fluid interfaces, such as those of a drop surface, and as a means to levitate fluid globules against weak gravitational fields.

Progress During FY 1995:
The emphasis of the FY95 work has been on three issues. First, developing analytic expressions for axisymmetric electrohydrodynamic interactions between drop pairs, ignoring deformation of the two drops. This is being done to provide an analytical benchmark and a theoretical framework with which to interpret the results of our numerical code that was developed and tested in FY94. Second, development of analytic expressions for the electrical force on spheroidal bodies in externally-applied electric fields that vary linearly with position. This work has been submitted to Chemical Engineering Science and provides a context for understanding the results of our FY94 numerical calculations on the electrohydrodynamic deformation and translation of a single drop in a nonuniform field. The last issue that we are addressing is the role of surface charge convection in drop deformation. This is being done by modifying the (boundary integral) numerical simulations done in FY93 and FY94 to account for the rearrangement of surface charge caused by motion within the drop surface.
II. MSAD Program Tasks — Ground-based

Discipline: Fluid Physics

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BS Degrees: 0  MS Degrees: 1  PhD Degrees: 0

Task Initiation: 3/93  Expiration: 3/96

Project Identification: 962-24-08-13

NASA Contract No.: NAG8-948

Responsible Center: MSFC

Bibliographic Citations for FY 1995:

Proceedings

Presentations
II. MSAD Program Tasks — Ground-based

Critical Viscosity of Xenon

Principal Investigator: Dr. Robert F. Berg

Co-Investigators:
Moldover, Dr. M.R.

Task Objective:
The objective of the experiment is to produce archival viscosity data on xenon that is closer to its liquid-vapor fluid critical point than is possible in 1-g.

Task Description:
1. Develop a low frequency, low shear rate viscometer with mK temperature control near room temperature. It will be of an electrostatically driven micro-flexure design;

2. Characterize vibration isolation sufficient to approach the critical temperature to within 600 μK while measuring viscosity to 0.2% precision;

3. Load xenon sample to within 0.3% of the critical density;

4. Choose sample geometry and do heat transfer analysis to establish expected thermal gradients and thermal equilibration times realistic for a Space Shuttle flight timeline;

5. Involve critical point dynamics theorists in data analysis before and after flight.

Task Significance:
The flight experiment will be the first direct measurement of the predicted power-law divergence of viscosity in a pure fluid. The measurement will test theories for the crossover to the critical region and for the value of the exponent associated with the divergence. Also, in combination with the results from the Critical Fluid Light Scattering Experiment (Zeno), the data will provide a stringent test of the mode-coupling theory of dynamic critical phenomena.

Progress During FY 1995:
Flight Cell Characterization:
Flight-eligible cells E, F, and G were delivered to Lewis Research Center. These cells were constructed, filled, and characterized according to a 100-item checklist. Because the nickel oscillator's anelasticity affects the transfer function at frequencies as low as 1 mHz, we also measured the oscillator's creep following a step change in the applied torque. We have shown that this creep measurement, in combination with the measurements of the oscillator's resonance frequency and Q, are sufficient to characterize the low-frequency transfer function.

Determination Of Tc:
We used observation of the liquid-vapor meniscus's appearance and disappearance within cells E, F, and G to locate their critical temperatures to a precision of better than 1 mK. All the measurements, made during a 7-month interval are referred to the temperature scale of a common thermistor, which was assumed to be stable. The critical temperatures of cells E, F, and G, which were filled from a common bottle of xenon, fell within a span of only 2 mK. The two measurements of Tc in cell E, separated by six months, are consistent to within 1 mK, indicating the stability of the thermistor with respect to Tc.
Internal Waves:
Because the xenon's density is stratifying in 1-g measurements near the critical point, the oscillator excites standing internal waves which are analogous to sloshing in fuel tanks. Although it will not be significant in microgravity, we further studied this effect to understand its influence on the 1-g measurements and to be certain of its origin. We used a coupled-oscillator technique to measure the temperature-dependent frequencies of the internal wave modes, and we found as many as five modes at each temperature. Through a collaboration with members of NIST's Computation and Applied Mathematics Laboratory, we have achieved a quantitative understanding of these results. In the simplest case, where the viscometer's symmetry allowed only two modes to couple to the oscillator, the observed modes were identified and their frequencies were compared to numerical hydrodynamic calculations. We found agreement to within 15%.

Viscosity Measurements In Cell E:
A set of viscosity measurements was made in cell E. The temperature timeline consisted of a series of temperature ramps whose rates decrease as Tc is approached. These slow ramps, including the final rate of 50 nK/s (1.6 K/year), will be needed in the flight experiment to avoid significant density gradients in the xenon.

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals


Presentations
Marangoni Effects in Boiling of Binary Fluid Mixtures Under Microgravity

Principal Investigator: Prof. Van P. Carey
University of California, Berkeley

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
Recent efforts to more fully optimize performance of power, refrigeration and thermal control systems have lead some developers to consider the use of binary working fluids. Vaporization of the working fluid is often a critical element in the performance of such systems. However, transport phenomena associated with vaporization of binary liquid mixtures is still not well understood. The main objective of this project is to separate and explore the roles of buoyancy and Marangoni effects in binary liquid mixture boiling under simulated low gravity conditions. The research will also aim to find coolant mixtures that are particularly well suited for use in a variety of applications including spacecraft thermal control.

Task Description:
This project consist of a series of experimental studies of nucleate boiling of a variety of binary mixtures. The experiments will include both 1-g and low-g data of boiling curve and critical heat flux. Testing the boiling performance of the heating surface in different orientations will establish whether the presence of Marangoni effects reduces the sensitivity of the system to the direction of the gravity vector. The least sensitive mixtures will then become the focus of tests under low gravity conditions (via NASA low-g aircraft).

Task Significance:
The experimental database for boiling of binary fluid mixtures will help to clarify the individual contributions of gravity and Marangoni effects on the heat transfer from the surface and the critical heat flux transition. It will also help to develop a capability to model these effects with reasonable accuracy to improve the designs of thermal systems.

Progress During FY 1995:
During this reporting period, the following have been accomplished:

Design, fabrication, and assembly of the binary mixture test system is complete. The test system, which will fly on the DC-9 later this year, has been leak checked and is fully operational. Boiling heat transfer data for pure water in different orientations was obtained to fully assess the instrumentation capabilities and establish a baseline to compare to the binary mixture data.

Initial laboratory experiments (in 1-g) have been conducted with water/propanol binary mixtures. These experiments span several combinations of system pressure and ambient propanol concentration. Back-to-back experiments were performed with three different heater surface orientations: (1) upward facing, (2) downward facing and (3) with the surface vertical (side facing). These results are providing valuable insight into the effect of gravity body forces on the boiling process.

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Project Identification: 962-24-05-81
NASA Contract No.: NAG3-1633
Responsible Center: LeRC
BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals
Marangoni Instability Induced Convection in Evaporating Liquid Droplets

Principal Investigator: Dr. An-Ti Chai

Co-Investigators:
Arpaci, Prof. V.R.

NASA Lewis Research Center (LeRC)
University of Michigan

Task Objective:
The objectives of the proposed effort are: (1) to study and to characterize the Marangoni instability phenomena in the near ideal configuration of an evaporating droplet in microgravity, and (2) to establish the effect of the induced convection on the droplet evaporation rate.

Task Description:
Specifically, the purpose is to study the Marangoni instability and thermocapillary convections in an evaporating liquid drop in the Fluids Experiment System (FES) developed for flight on Space Shuttle missions. When a liquid drop undergoes evaporation, its surface temperature decreases. If the droplet is free floating in a microgravity environment, the heat transfer process inside the droplet is "conduction controlled." As the process continues, a radial temperature gradient builds up at the free surface until the critical Marangoni number is exceeded. Then the onset of instability induces thermocapillary convective flows that, in turn, speed up the evaporation. The convective flow will subside when the interior of the droplet reaches a certain equilibrium temperature, and the process will return to the "diffusion controlled" mode.

Task Significance:
When the proposed experiments are successfully completed, the unique data set, which will be collected under near ideal conditions, should by itself constitute a significant contribution to our knowledge of droplet evaporation. The significance and influence of the proposed project go far beyond any unique experimental data set. With the anticipated data set, it will be possible to quantitatively evaluate the well known Maxwell's theory of droplet evaporation. Additionally, it will be possible to establish that thermocapillary instability induced convection inside a droplet can affect its evaporation process. It is our intention to use this information to pursue the development of a complete and comprehensive theory of droplet evaporation.

The value of a more comprehensive and complete theory cannot be overstated. Having a solid theoretical basis, numerical computation can be devised along with skillful scaling techniques to explore cases where direct experimentation is not feasible. The influence of the proposed work is expected to be far reaching in this regard.

Progress During FY 1995:
Experimental Effort: A proposal to conduct Glovebox experiments on "Droplet Injector and Evaporation" was submitted and a review for the final selection will be conducted in early 1996.

1. Received funding for Glovebox selection review.
2. Prototype injectors of different size have been fabricated for both bench test and Zero-G drop test to demonstrate feasibility.
3. Both manual and automated mode of operation of the injector are being studied.
4. Alternate injector designs are being considered and tested.
5. Various evaporating sample liquids are being evaluated.

Theoretical Effort: In the past two years we have tried the projection method for the instability of evaporating horizontal layers. First, we tested the method in the absence of evaporation. We then included the effect of evaporation; the results were presented at the 9th International Conference on Numerical Methods in Laminar and Turbulent Flow in Atlanta.
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Discipline: Fluid Physics

The use of the "Projection Method" for deforming droplets does not appear to be feasible. This year Professor Arpaci successfully tested the front tracking method for the dynamics of interface mechanics in general and for the evaporating droplets in particular. Preliminary results will be analyzed and reported later. Separately, we are trying to apply the chaos theory to the second transition of the thermocapillary instability of horizontal layers. However, the method is only applied for the case of no evaporation.

Special effort has been put forward by Professor Arpaci in search for the Kilmogorov microstructure of the interface dynamics. It is expected that this structure should be related to the wavelength of the growing disturbances. The following three keynote lectures on the microstructure of turbulence have been presented at international conferences: 1) 10th International Heat Transfer Conference at Brighton, England, August 14-18, 1994, 2) The International Symposium on Turbulence, Heat and Mass Transfer at Lisbon, Portugal, August 8-12, 1994, and 3) Third Latin-American Symposium on Fluid Mechanics at Caracas, Venezuela, February 5-8, 1995.

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**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Presentations**


II. MSAD Program Tasks — Ground-based

Discipline: Fluid Physics

Rewetting of Monogroove Heat Pipe in Space Station Radiators

Principal Investigator: Prof. S. H. Chan
University of Wisconsin, Milwaukee

Co-Investigators:
No Co-I’s Assigned to this Task

Task Objective:
The general objective of the program is to investigate the physics of rewetting of heated, grooved flat plates in a microgravity environment.

Specific objectives of the program are to add to the fundamental understanding of the physics of rewetting/dryout fronts; to better quantify the conditions under which rewetting occurs; and to obtain more accurate predictions of the rewetting velocity for a range of parameters.

Task Description:
The program is composed of both experimental and theoretical investigations. For the experimental investigation a grooved flat plate apparatus will be designed and constructed. Liquid will be introduced on one side of the plate to flow along the grooved surface; the other side of the plate will be electrically heated. This apparatus will be operated both on the ground and aboard a microgravity aircraft.

Data from these experiments will consist of temperature measurements in the liquid film and the heated plate. Advancing wetting front (rewetting), receding wetting front (dryout), and stationary wetting front cases will be examined.

The theoretical analysis will be made by applying experimental data on rewetting velocity and temperatures to analytical processes developed for predicting rewetting velocities on overheated nuclear fuel rods.

Task Significance:
Understanding the dryout and rewetting characteristics of grooved surfaces is important in predicting behavior of monogroove heat pipe designs proposed for the space station.

Progress During FY 1995:
When performing the ground experiments it was found that the greatest difficulty arose with the introduction of the wetting fluid to the end of the grooved plate. This was to be done in such a manner that only the capillary action was responsible for the advancement of the fluid, with other factors (i.e., gravity head, initial velocity from feed system) being minimized.

When designing the apparatus for the in flight test, this problem was at the forefront of consideration. We have come up with a design scheme in which the plate end is immersed in fluid for the duration of the microgravity run, and is out of the fluid at all other times.

Our progress in this area is as follows:
1. We have completed a drop tower with which to test the experimental apparatus. The drop time is around 0.9 seconds. With this we can evaluate the performance of the reservoir and the resilience of the overall experimental apparatus.

2. The first design iteration reservoir has been built and is ready for testing.
3. All other components for the in-flight experiment have been built and are ready to be put together as soon as the reservoir is tested.

Recently a series of rewetting experiments were run in which the entire bottom of the plate was subject to heating. In analyzing the data an interesting discovery was made. As in previous experiments, in which the plate end was heated and maintained at a constant temperature, the wetting front temperature was found to vary with several factors, the most notable being the initial temperature. This variation in wetting temperature made precise prediction and determination of the wetting front quite difficult. However, when the data (temperature vs. time) was used to generate a polynomial expression, and when the second derivative of this expression was plotted, the zero (point of inflection) of the second derivative, which indicates the time that the wetting front reach a given thermocouple location, was found to agree very well with the observed time at which the wetting front crossed the sensor. Due to its success, this technique is being implemented to determine the rewetting temperature.

We are currently running further experiments in an attempt to correlate the effects that factors such as temperature gradient and initial temperature have on the wetting temperature.

**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Journals**

**Presentations**
Marangoni and Double-Diffusive Convection in a Fluid Layer Under Microgravity

Principal Investigator: Prof. Chuan F. Chen 
University of Arizona

Co-Investigators:
Chan, C. 
University of Arizona

Task Objective:
To study the onset and the subsequent convection in a fluid layer subjected to Marangoni and double-diffusive instabilities, including the effects of cross diffusion and gravity modulation.

Task Description:
A coordinated research effort in ground-based experiments, stability analysis, numerical simulation, and a design sensitivity study is to be conducted. Experimentally, the effect of gravity modulation on the onset and subsequent convection in singly and doubly diffusive layers will be studied with improved experimental techniques. Theoretically, stability analyses of Marangoni double-diffusive instability will be conducted, and a numerical simulation using boundary element methods will be carried out to examine the interaction of finger convection with Marangoni convection, and to test the design sensitivity to optimize the flight experiment.

Task Significance:
The results of this study will enable the design of an apparatus for crystal growing and for casting in a space environment. In addition it will offer an improved understanding of materials processing, which will enhance the chances of obtaining new and better quality crystals for the semi-conductor industry and for the bio-medical industry. This study can also provide an improved understanding of the interaction of surface tension forces and double diffusive buoyancy forces which would lead to the improvement of manufacturing processes on Earth.

Progress During FY 1995:
In FY95, numerical simulations using the finite difference method were conducted to study the onset of convection in a double-diffusive layer with surface tension effects. The nonlinear evolution of perturbations was also examined using this code. The code is capable of simulating experimental results and verifying linear instability theory. Furthermore, by using the derived kinetic energy equation, sources and sinks of energy can be clearly identified, thus providing a better understanding of the mechanisms of the instabilities.

In preparation for the planned experimental investigation, numerical simulations using the finite-difference method have been applied to study the effect of sinusoidal gravity modulation on natural convection in a vertical narrow tank. In particular, we studied the transition from unicellular to multi-cellular flow. Results show that gravity modulation enhances stability for an air-filled test tank. But, for a high Prandtl number fluid, Pr=720, our results show a subcritical instability mode. It would be interesting to obtain experimental confirmation.

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II. MSAD Program Tasks — Ground-based

Transport Phenomena in Stratified Flow in the Presence and Absence of Gravity

Principal Investigator: Prof. Norman Chigier Carnegie Mellon University

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
The strategy of this study is as follows:

1. To separate the effects of molecular and turbulent diffusion from gravitational forces during the mixing of two shear layers.
2. To identify the relevant and characteristic non-dimensional parameters that govern the physical interaction between fluid layers in the range of the proposed experimental conditions.
3. To examine the interaction between buoyant plumes and stratified shear layers, and to study the rates of entertainment into buoyant plumes and stratified shear layers.
4. To study the effects of injecting solid and liquid particles into fluid streams under stable and unstable stratified shear flow conditions.
5. To identify potential instrumentation that could distinguish between molecular and turbulent diffusion in the presence and absence of gravity.

Task Description:
1. Conduct detailed measurements in turbulent mixing layer with velocity and density gradients.
2. Conduct a scoping on the interaction of buoyant heated jets and plumes interacting with stable and unstable stratified shear layers.
3. Develop scaling laws and similarity criteria for comparison with measurements made in the "main" and "reduced" scale experiments.

Task Significance:
To use the reduced gravity environment to improve the understanding of the fundamental physical processes of mixing between layers of fluids of different densities and velocities. The following are a few matters that are important to this research project:

1. To understand the process of dispersion of pollutants in a highly stable stratosphere and in outer space.
2. To understand the effect of dispersion of pollutants emitted from a space craft at high altitudes.

These results could be used to find the means of controlling or at least reducing pollutions of the atmosphere and the oceans where stratified flows prevail.

Progress During FY 1995:
During this period, Prof. Chigier has completed a series of measurements using a two-component LDV and a newly developed smoke-wire design for clear visualization of the vortex structures in the mixing layer with the first wind tunnel facility. Results provided guidance for improving the heat transfer conditions across the splitter plate. To this end, the wind tunnel has been modified to create optimized flow conditions. The newly-designed wind tunnel is in the final stages of manufacturing and assembly. The modifications are as follows:

1. Improved thermal insulation of the splitter plate by using AURA super insulation vacuum panels.

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II. MSAD Program Tasks — Ground-based
Discipline: Fluid Physics

2. Thickness of the splitter plate inside the nozzle has been increased.
3. The total length of the splitter plate has been reduced by 20'.
4. The height of the nozzle exit was doubled to increase the flow width and reduce the influence of the outer entrainment layers on the mixing layer.
5. New contraction contours were designed based on the effects of geometry on flow uniformity.

This newly-designed wind tunnel, with improved vacuum insulation for the splitter plate and redesigned contraction nozzle is expected to generate initial conditions much closer to the ideal conditions. Plans to conduct velocity and temperature measurements and to test the effectiveness of the new design are underway.

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| MS Students: 0                                   | NASA CONTRACT NO.: NAG3-1505 |
| PhD Students: 1                                  | RESPONSIBLE CENTER: LeRC |

BIBLIOGRAPHIC CITATION FOR FY 1995:

Proceedings
II. MSAD Program Tasks — Ground-based

**Discipline:** Fluid Physics

*Bubble Dynamics, Two-Phase Flow, and Boiling Heat Transfer in Microgravity*

**Principal Investigator:** Prof. Jacob N. Chung
Washington State University

**Co-Investigators:**
No Co-I's Assigned to this Task

**Task Objective:**
The main objective is to study the effects of external force fields on the nucleation, two-phase bubble dynamics and boiling transport in microgravity. The proposed research seeks to increase our understanding of bubble nucleation and growth on the heater surface, bubble removal from the heater surface by an electric field, an acoustic field or a velocity shear resulting from the relative fluid motion with respect to the surface, and bubble dynamics and heat and mass transport in boiling liquids.

**Task Description:**
In the experimental portion, visualizations with NTSC and high-speed photography and heat transfer measurements have been performed in a drop tower at Washington State University. The visualization photographs were then analyzed by a digital image analysis system. Boiling curves were developed based on measurements of heat fluxes and surface superheats for various system conditions. Special attention was focused on the critical heat flux. Analytical and numerical modeling is also included to compliment the experiments. Perturbation and asymptotic techniques will be applied for low Reynolds number bubble dynamics, linearized bubble stability analysis, and small oscillation of bubble in microgravity to account for small disturbances and the g-jitter. Because of similar heater and boiling system design, the proposed research would be easily adapted to the current Pool Boiling Apparatus (PBA) flight hardware available from NASA for future space experiments.

**Task Significance:**
The proposed research seeks to:

1. Increase our understanding of bubble nucleation and growth on the heater surface.
2. Bubble removal from the heater surface by an electric field, an acoustic field, and velocity shear resulting from the relative fluid motion with respect to the surface.

Future spacecraft power systems are likely to have greater power generation and dissipation requirements due to proposed longer space missions and more intensive space activities. Without suffering from weight penalties, two-phase boiling systems are being considered for a wide range of future space applications such as thermal control, propulsion, power generation, and thermal management. The boiling system has the potential advantage of being able to transfer a large amount of energy over a relatively narrow temperature range with a small weight requirements. For example, two-phase systems were once baselined for the space station. However, the thermo-fluid dynamics of two-phase systems in microgravity encompasses a wide range of complex phenomena that were not understood sufficiently for engineering design to proceed.

**Progress During FY 1995:**
We have made substantial progress in the following four areas:

1. **Facilities:**
The 2.1 Second Drop Tower is working well and we have completed 195 drops to date. The construction and calibration results for the drop tower design have been submitted as three journal articles. One is a general description of the drop tower and the calibration along with some initial results, the second focuses on the air bag

*Printed: 28 FEB 1996*
II. MSAD Program Tasks — Ground-based

Design and performance, and the third focuses on the residual acceleration as a function of frequency. We have also recently completed the conversion of our 0.6 Second Air bag Drop Tower into a 1.2 Second Drop Tower. We have changed from plastic to glass in our heater design with very successful results. One of these heaters has lasted for over thirty drops with heat fluxes up to 8 W/cm². Our single bubble heaters are also now sputtered on glass and can generate hundreds of individual bubbles before any noticeable degrading effects on the heater.

2. Acoustic Driven Nucleate Boiling:
The purpose of the acoustic driven boiling research is to determine if an acoustic sound field can maintain nucleate pool boiling in microgravity and measure its effects on the boiling process in microgravity and terrestrial gravity. This research is complete and has been written up as a MS thesis. The application of the acoustic sound field to the boiling process increases the heat transfer rates in Earth's gravity. In microgravity it was found that the radiation pressure can produce a large enough acoustic force to move vapor bubbles off a heater surface during microgravity. The effects of cavitation and acoustic streaming resulted in erratic bubble motion and aided in the perturbation of vapor bubbles attached to and in the vicinity of the heater and effected the heat transfer and bubble dynamics in both microgravity and terrestrial gravity. At all heat fluxes tested during microgravity, the acoustic mechanisms prevented large amounts of vapor from blanketing the heater surface.

3. Forced-Convection Nucleate Boiling:
The purpose of the forced-convection boiling research is to determine if a flow field can maintain nucleate pool boiling in microgravity and measure its effects on the boiling process in both microgravity and terrestrial gravity. We presented a paper on the work of Wang et al. 1993 at the National Convective Boiling Conference in Banff, Canada. The paper received favorable reviews and, as a result, it will be published in a new book entitled "Convective-Flow Boiling". In continuation of this work, we are presently utilizing a small flow boiling rig in the 1.2 Second Drop Tower and have completed 30-40 drops with both 1" x 1" gold film heaters and with single bubble heaters. The continued research will attempt to map the boiling regimes for various flow rates and heat fluxes in microgravity. For example, at a given heat flux, what flow rate is required in order to sustain boiling and avoid burnout of the heater surface.

4. Electrostatic Driven Nucleate Boiling:
The purpose of the electrostatic boiling research is to determine if an electric field can maintain nucleate pool boiling in microgravity and measure its effects on the boiling process in both microgravity and terrestrial gravity. We have studied how large nonuniformities in the electric field produced at the edge of two diverging-plate electrodes can produce large forces which are efficient in motion control and bubble positioning for both liquid droplets and vapor bubbles. Three electric-field experimental studies are either completed or under progress. The first study focuses on results in which a controlled electrical body force is imposed onto the boiling process for a test fluid of FC-72 which is boiled on a horizontal platinum wire heater. As this force tends to either add to or subtract from buoyancy an analogy between variable gravity boiling results and results from boiling with an electric field are presented and discussed. Second, an experiment was performed to correct some mistakes previously reported in the literature. We have submitted a paper to the J. of Applied Physics detailing this information. The third and final set of experiments uses semitransparent gold-film heaters which allow a bottom view of the boiling. We are also using a special surface that changes color with temperature and allows us to visualize how the temperature varies over the heater surface.

STUDENTS FUNDED UNDER RESEARCH:

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Task Initiation: 12/92 Expiration: 12/95

Project Identification: 962-24-05-59

NASA Contract No.: NAG3-1387

Responsible Center: LeRC

Printed: 28 FEB 1996
BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals


Proceedings

Presentations

II. MSAD Program Tasks — Ground-based

Microgravity Particle Dynamics

Principal Investigator: Dr. Ivan O. Clark

Co-Investigators:

Johnson, E.J.
Meyers, J.F.
Kjelgaard, S.O.

Discipline: Fluid Physics

Task Objective:

The objective of this research is to develop the apparatus, numerical models, and practices for enhancing the knowledge of particle transport in laminar fluid flows. Specifically, an enhanced understanding of the roles of thermophoresis (thermal gradient) and Saffman (particle crossing of velocity gradient) effects on particle transport is sought. The research also seeks to identify the critical parameters and instrumentation requirements for subsequent microgravity experiments.

Task Description:

The technical approach is a coordinated numerical and experimental investigation using geometries selected to maximize the scientific return of the research. Gravitational effects in the experimental investigation are addressed through the use of multiple orientations, relative to gravity, of the test chambers. The first step in this investigation is a parametric study to determine the order of magnitude of the competing effects for candidate test chamber geometries. These complex interacting effects include gravity, buoyancy, inertia, viscous drag, particle rotation, electrostatic charges, as well as thermal and velocity gradients. The initial parametric study will ensure a maximum scientific return from the selected geometry. Experimental studies will use the laser velocimetry (LV) and flow visualization systems developed for chemical vapor deposition reactor characterization at Langley Research Center (LaRC). Additional LV and other aerodynamic instrumentation systems are available to this research at LaRC if needed.

Task Significance:

The proposed research will result in: (1) definition of the requirements and the potential for follow-on flight experiments in transport phenomena, (2) an enhanced understanding of particle transport phenomena in thermal and velocity gradients, and (3) fluid dynamic correction factors for particle-based flow instrumentation for a range of thermophoresis and Saffman environments. The results of this research will be immediately applicable in both unigravity and microgravity for applications such as validation of particle transport theories; correction of wind tunnel research data; and design refinements for chemical vapor deposition reactors, particulate combustors, and clean rooms. In addition, experimental size constraints for microgravity experiments dictate that velocity gradients will exist in laminar flow flight experiments. Hence, Saffman effects will be present to some extent in all microgravity laminar flow experiments with particles.

Progress During FY 1995:

Steady-state numerical studies of thermophoretic flow patterns have begun. Several shortcomings of the thermophoresis algorithm used in FLUENT have been identified and an improved algorithm has been implemented. Parametric studies of velocity, length, time, thermal gradient magnitude and velocity gradient magnitude likely to be involved in gas-solid multiphase flight experiments have been assessed for 1-100 micrometer particles. The results of this parametric study indicate that flight experiment regimes are both achievable and useful. A version of FLUENT augmented to analyze particle nucleation and growth during transport has been installed and validation testing has begun.
A database has been established of particle dynamics research and particle-based instrumentation which will ensure currency of ground and flight experiments associated with this research as well as provide a valuable reference tool for other researchers in this area.

STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 11/92    EXPIRATION: 11/95
PROJECT IDENTIFICATION: 962-24-06-04
RESPONSIBLE CENTER: LaRC

Printed: 28 FEB 1996
Reactive Fluids Experiment: Chemical Vapor Deposition

Principal Investigator: Dr. Ivan O. Clark
NASA Langley Research Center (LaRC)

Co-Investigators:
Hyer, P.V.
Johnson, E.J.
Lockheed Engineering & Sciences Co.

Task Objective:
The research will develop a series of ground-based experimental investigations of the fluid dynamics of chemical vapor deposition (CVD) which will lead to an enhanced understanding of the basic sciences underlying reactive fluid interactions. It will form the basis for a proposal to perform a series of flight experiments necessary to more fully elucidate these scientific principles. This program will use past experience in chemical vapor deposition, non-isothermal flow measurements, numerical modeling of reactive fluid dynamics, and development of instrumentation to carry out the research.

Task Description:
A combined numerical and experimental approach is being used to investigate the CVD process. The experimental approach combines growth of semiconductor materials, the deposition of a model material, and the measurement of the gas flow velocities in the CVD reactor using laser velocimetry. The numerical approach models each of the experimental approaches and uses the experimental results for validation.

Task Significance:
CVD is an extremely important industrial process. It is widely used not only for the production of semiconductor and insulating materials, but also for optical coatings, wear- and corrosion-resistant coatings, paint pigments, and the production of drawing stock for optical fibers. In addition to the economic importance of these application areas for terrestrial research and manufacturing, they also represent key manufacturing capabilities for future extraterrestrial development. Each of these CVD applications takes place in reactors which have been developed through decades of empirical trial and error. Engineering design capabilities have been limited by the extreme difficulty, under Earth-gravity (1g), of separating the fluid dynamic effects of externally forced convection, buoyant thermal convection, buoyant solutal convection, and internally forced convection due to volume changes arising from both thermal and chemical effects. This research seeks to improve the ability to apply engineering design techniques to this economically important area.

Progress During FY 1995:
The numerical modeling of InP in three spatial dimensions has been refined with good results. Model/experiment agreement is best when the experimental films are thick. InP film porosity has proven to be a problem on the large-area amorphous substrates. Additional growths are being performed with GaAs and InP substrates to examine substrate effects on measured growth rates.

Laser velocimetry (LV) measurements of the flow velocities in a replica CVD reactor, which duplicates the geometry used for the InP growths, is in progress. Analysis of particle imaging velocimetry (PIV) experiments conducted in this channel are underway and are being correlated with LV and thermal imaging measurements. The transition from supply manifold to reactor inlet has proven to have a significant effect on reactor flows. Numerical modeling of this reactor geometry has been expanded to include the entry region effect on the thermal field as well as the effects of thermal gradients on the trajectories of tracer particles used for the LV measurements.
II. MSAD Program Tasks — Ground-based

**STUDENTS FUNDED UNDER RESEARCH:**

**TASK INITIATION:** 11/90  **EXPIRATION:** 11/95  
**PROJECT IDENTIFICATION:** 962-24-06-03  
**RESPONSIBLE CENTER:** LaRC

**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Presentations**
Studies of Freely Suspended Liquid Crystal Bubbles

Principal Investigator: Prof. Noel A. Clark

University of Colorado, Boulder

Co-Investigators:

No Co-I's Assigned to this Task

Task Objective:

The experimental study of smectic liquid crystal films in the form of single levitated bubbles including studies of (1) dynamic and equilibrium in-plane stress-induced shapes of levitated bubbles, (2) stress-induced quenches of the Kosterlitz-Thouless phase transition among others, and (3) studies of film permeability.

Task Description:

These experimental studies require technique developments beyond those which have been used for the study of planar films in 1g. Among the more important ones are (1) a method for inflation, levitation, positional stabilization of the bubble, (2) transmission video microscopy to monitor bubble shape and polarized reflection video microscopy to probe the local orientation, and (3) methods to change ambient gas pressure while maintaining bubble position.

Task Significance:

This study is suitable for several novel lines of experimentation of relevance to the physics of both liquid crystals and fluid thin films. These ultra thin freely suspended liquid crystal films are structures of fundamental interest in condensed matter physics. They are the thinnest known stable condensed phase preparation, making them ideal for studies of two dimensional phase transitions and of fluctuation and interface phenomena of thin fluid films. Freely suspended films have been used to provide unique experimental conditions for the study of condensed phase transitions on two dimensions.

Progress During FY 1995:

The inherent fluid-layer structure and low vapor pressure of smectic liquid crystals enable the long term stabilization of freely suspended, single component, liquid crystal films as thin as 30 Å, a single molecular layer. These structures have been intensively studied as planar films freely suspended over holes in solid substrates, experiments which have been crucial to the current understanding of liquid crystal phase behavior.

In this project the study of these smectic liquid crystal films in the form of bubbles was emphasized. This bubble geometry makes accessible a variety of important new experiments. The following experiments are of primary concern:

• Quenching the Smectic A to C Transition
  During the quick deflation of a smectic C (1/1/1/1) bubble, there is in-plane compression which forces the tilted smectic C molecules to stand up, giving transient formation of smectic A (111111). When the deflation completes the smectic C returns but the field giving the tilt direction in the layer plane is full of topological defects. These defects can be observed on the film by polarized reflection microscopy. This system is a two dimensional analog of the symmetry breaking phase transition corresponding to the appearance of matter in the early universe. A scaling model has been developed along with a computer simulation of the process which explains the time behavior.

• Permeation of Helium through Ultrathin Organic Films
  Bubble photos have shown typical 2cm diameter bubbles with thick regions (approximately 15 layers) and thin regions (approximately 5 layers). Such a bubble, if filled with helium, will completely deflate via permeation of helium through the thin region in about ten seconds. The permeation rate of helium through the film can be measured using this deflation rate. Currently, optical reflectivity is being used as a measure of film thickness.
II. MSAD Program Tasks — Ground-based

Discipline: Fluid Physics

• Annealing of a Two Dimensional Foam

In other bubble photos, the thick regions are large and contiguous. After aggressive shearing of the bubble by an air jet, the thicker region forms a foam of very small pancake-like circular islands, which slowly coalesce and coarsen and settle by gravity to the bottom. The coarsening dynamics of this novel two-dimensional system are presently being studied.

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BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals

Fluid Interface Behavior Under Low- and Reduced-Gravity Conditions

Principal Investigator: Prof. Paul Concus

University of California, Berkeley

Co-Investigators:

Finn, R.

Stanford University

Task Objective:

The general objective of this research is to gain a better mathematical understanding of the physical behavior of fluids partly filling a container or otherwise in contact with solid support surfaces, when capillary forces are predominate. Closely interrelated with the mathematical and computational studies are current and planned ground-based and in-space microgravity experiments.

Task Description:

The approach for this project is to pursue parallel theoretical, computational, and experimental studies to explore equilibrium capillary free surface interfaces in a variety of geometrical configurations. The theoretical aspects include both rigorous mathematical studies and numerical computation. In connection with the scheduled USML-2 Glovebox experiment, two configurations of particular interest, for which small changes in container shape or contact angle can give rise to large bulk reorientation of fluid, have been devised and are being studied: 1) a movable wedge and 2) canonical proboscis containers. Experimentation is carried out in collaboration with M. Weislogel at the NASA Lewis Research Center.

Task Significance:

Knowledge gained from these studies can lead toward a better understanding of surface phenomena of liquids in containers in a low-gravity environment, and in particular to obtaining new information on the significance of contact angle as an intrinsic physical property. Additionally, development of accurate, new methods for measuring contact angle and new insights for managing fluids in space can result.

Progress During FY 1995:

Work continued on studying the behavior of capillary surfaces in wedge domains, in which the contact angles on the two sides of the wedge are allowed to differ. Local and global existence criteria, previously conjectured, were proved, and precise conditions on boundary data under which surfaces are bounded at the corner were obtained. The question of when solutions must be discontinuous at the corner continues to be under study.

As part of developing a proposed glovebox/MIM experiment, a new type of discontinuous dependence on data was inferred from results of a student L. Zhou on liquid bridges between parallel plates and of another of J. McCuan's on nonexistence of tubular bridges in wedge domains. We have begun to study this unusual behavior numerically to obtain information on possible configurations and their stability.

A new form of the canonical proboscis was derived in which transitional behavior as the critical contact angle is crossed is discontinuous. This has some interest in connection with the forthcoming USML-2 Glovebox ICE experiment.

Additional mathematical work related to the capillary equation yielded new forms of the maximum principle, with quantitative estimates, and gradient bounds for the mean curvature equation.

Studies in the above topics will continue, taking into account results of the USML-2 experiment. Emphasis will be placed on obtaining further computational and mathematical results for development of the proposed glovebox/MIM experiment.
Additionally, as a first step in studying the case of contact angle that is to some extent indeterminate rather than being prescribed at a fixed value, we hope to begin looking at certain global shape estimates under these circumstances.

**STUDENTS FUNDED UNDER RESEARCH:**

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**TASK INITIATION:** 3/90  **EXPIRATION:** 3/96  
**PROJECT IDENTIFICATION:** 962-24-05-66  
**NASA CONTRACT NO.:** NCC3-329  
**RESPONSIBLE CENTER:** LeRC

**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Journals**

**Proceedings**

**Books**

**Presentations**

Finn, R., "On a proposition of Lancaster and Siegel." Preprint, Mathematics Institute, University of Leipzig, 1995.
Convection and Morphological Stability During Directional Solidification

Principal Investigator: Dr. Sam R. Coriell
National Institute of Standards and Technology

Co-Investigators:
- McFadden, G.B.
- Murray, B.T.
- Manning, J.R.

National Institute of Standards and Technology (NIST)

Task Objective:
During the directional solidification of a binary alloy, solute inhomogeneities can arise from both fluid flow and morphological instability. In microgravity buoyancy-driven fluid flow is reduced, and experiments to study the evolution of morphological patterns without the interference of fluid flow may be possible. The goal is to develop an understanding of the interaction of fluid flow with the crystal-melt interface so that solute segregation in solidifying alloys can be controlled in order to obtain materials with superior properties.

Task Description:
Included in this research are the following:

1. Calculations of cellular morphologies in the absence of fluid flow.


3. Linear stability analyses of coupled interfacial and convective instabilities.

4. Calculations of the effects of time-dependent gravitational accelerations (g-jitter) on fluid flow during directional solidification. For growth of a vicinal face at constant velocity, the effect of anisotropic interface kinetics on morphological stability has been calculated for a binary alloy. Anisotropic kinetics give rise to traveling waves along the crystal-melt interface, and can lead to a significant enhancement of morphological stability.

This ground based research will focus on providing theoretical interpretation and guidance for a series of space experiments to be carried out by J.J. Favier, R. Abbaschian, and colleagues on tin-bismuth alloys using the MEPHISTO apparatus and by K. Leonartz and colleagues on succinonitrile-acetone alloys.

Task Significance:
This comprehensive research on the directional solidification of binary alloys will determine the processing conditions required to eliminate micro segregation of impurities and produce alloys with desirable microstructures and improved properties. This research will potentially contribute to the important and challenging goal to develop and transfer to all of the consortium members (of NIST, US industry, and NASA) technology that will provide for the rapid design and prototyping of new precision cast parts, enhance product quality, and reduce rejection rates.

Progress During FY 1995:
In collaboration with A. A. Chernov, the effect of anisotropic kinetics on the morphological stability of a crystal growing at constant velocity has been treated for growth into a supersaturated solution, for growth into a supercooled melt, and for growth of a binary alloy by the Bridgman method. The dependence of the interface kinetic coefficient on crystallographic orientation is based on the motion and density of steps. Morphological instability leads to step bunching and the formation of macrosteps. Anisotropic kinetics gives rise to traveling waves along the crystal-melt interface, and can lead to a significant enhancement of morphological stability. It is well known...
that a shear flow along the crystal-melt interface generally stabilizes the interface with respect to perturbations of the interface shape along the flow direction. Thus, a shear flow introduces an anisotropy similar to that introduced by anisotropy of interface kinetics and we expect an interaction between a shear flow and anisotropic kinetics. A shear flow in the opposite direction from the step motion tends to move fluid into the steps and is somewhat equivalent to a faster translation of the perturbations in a stagnant fluid; thus, one expects such a flow to further stabilize the interface. Conversely, a shear flow in the direction of the step motion will destabilize the interface. Calculations of the interaction of a shear flow and anisotropic kinetics for melt growth of a germanium-silicon alloys have been carried out. A shear flow (linear Couette flow or asymptotic suction profile) parallel to the crystal-melt interface in the same direction as the step motion decreases interface stability in that the critical solute concentration decreases.

A shear flow counter to the step motion enhances stability for small shear rates; for larger shear rates, the neutral curve develops a bimodal structure, and the critical solute concentration slowly decreases with shear rate. The instability mechanism and the stability behavior for a wide range of processing conditions are being investigated. We plan to carry out similar calculations for the case of growth from supersaturated solution.

In collaboration with R. F. Sekerka, we have developed a simple model of the influence of natural convection on the selection of the operating state (dendrite tip velocity and tip radius) for dendritic growth of a pure material. We hypothesize that the important aspects of natural convection can be accounted for by considering the global convection that would occur in the vicinity of a sphere of radius $R$ that characterizes the size of a dendritic array that emanates from a point source. We estimate the thickness of a stagnant boundary layer $d$ surrounding this sphere by matching the value of the Nusselt number obtained from the heat transfer literature. We solve the steady-state problem of a paraboloidal dendrite at temperature $T_M$ growing toward a confocal paraboloid at temperature $T$, and at a distance $d$ from the tip. This results in a new transcendental equation, that depends on the gravitational acceleration, and transport properties of the melt, for the dimensionless supercooling in terms of the Peclet number.

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**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Journals**


**Presentations**


Interaction and Aggregation of Colloidal Biological Particles and Droplets in Electrically-Driven Flows

Principal Investigator: Prof. Robert H. Davis
University of Colorado, Boulder

Co-Investigators:
Todd, P.
Loewenberg, M.
University of Colorado, Boulder
Yale University

Task Objective:
The objective of this research is to develop a fundamental understanding of aggregation and coalescence processes during electrically-driven migration of particles (or cells) and droplets.

Task Description:
The research includes the following tasks:

• Development of a theoretical description of electrically-driven particle aggregation by computing the relative velocity between two particles in near contact with hydrodynamic, electrokinetic, van der Waals, and electrostatic double-layer interactions, and by predicting the stability conditions and the rate of pair-wise aggregation in a semi-dilute suspension;

• Formulation of a description of electrically-driven drop coalescence by developing a simplified electrokinetic description of a charged fluid interface under thin double-layer conditions, and performance of initial computations for the interactions between a pair of electrically-driven droplets; and

• Observations of electrically-driven aggregation and coalescence by conducting terrestrial experiments to test the theoretical description of electrically-driven particle aggregation and observe electrically-driven droplet coalescence, with the results leading to future design of a flight-based experiment to gather quantitative observations for testing a theoretical description of electrically-driven droplet coalescence.

Task Significance:
The fundamental study of particle aggregation in electric fields is expected to have practical application to electrically-controlled cell flocculation for cell separation and recycle in space-based bioreactors, where gravity cannot be employed as previously done. Similarly, research conducted on drop interactions and coalescence is expected to provide an understanding of electrically-driven demixing of two liquid phases, such as those encountered in biphasic aqueous extraction of biological cells and molecules under reduced gravity when buoyancy-driven demixing is weak. Finally, the theoretical descriptions of two charged, migrating particles or drops are expected to have general scientific and engineering value.

Progress During FY 1995:
A theoretical description of the near-contact interaction of two particles in an electrical field has been completed. The new results were combined with previous results for moderate and large separations in a trajectory analysis to predict pairwise aggregation rates of charged particles with differing zeta-potentials. In addition to hydrodynamic interactions, attractive van der Waals forces and repulsive electrostatic forces were included in the analysis. The key result is that particle aggregation under an electric field occurs more easily and efficiently than under a gravitational field. A study of combined electrophoretic and gravitational aggregation indicates that it may be possible to prevent aggregation by antiparallel orientation of an electric field of proper strength with the gravity vector. A theoretical description of the electrophoretic migration of drops with thin double layers due to insoluble ionic surfactants on their interfaces has been initiated.
Microvideo experiments to observe aggregation of red blood cells have been initiated, but direct quantification is difficult since even moderate concentrations (1% cells) are optically opaque, whereas very dilute suspensions have very low aggregation rates. Zone electrophoresis, in which a zone of faster-moving cells passes through a zone of slower-moving cells, are planned.

**STUDENTS FUNDED UNDER RESEARCH:**

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**TASK INITIATION:** 2/93  **EXPIRATION:** 2/96

**PROJECT IDENTIFICATION:** 962-24-08-10  
**NASA CONTRACT NO.:** NAG8-945  
**RESPONSIBLE CENTER:** MSFC

**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Journals**

II. MSAD Program Tasks — Ground-based

Microphysics of Close Approach and Film Drainage and Rupture During Drop Coalescence

Principal Investigator: Prof. Robert H. Davis
University of Colorado, Boulder

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
The overall objective of this research is to develop a comprehensive theoretical model of the relative motion, film drainage, and film rupture leading to coalescence of interacting drops dispersed in an immiscible fluid. Relative motion due to gravity, thermocapillary migration, and attractive van der Waals forces is considered. This research is concerned with the microphysics of coalescence and focuses on the near-contact interaction of a drop approaching a second drop or a surface or interface.

Task Description:
The overall goal of this microphysical research is to predict deformation, film drainage, and collision rates using fundamental theoretical analyses. The novel method used couples lubrication theory in the narrow separation gap and boundary integral theory for the drop phase. Matched asymptotic expansions are used for small times (small drop deformations) and long-times (draining film regions).

The research program is divided into three components directed at meeting the goal:

1. Near-contact relative motion for nearly spherical drops: The rate of approach and the onset of deformation and film drainage are examined for gravity-driven and thermocapillary-driven motion.

2. Evolution of drop deformation during film drainage: As the drops move closer, the natural evolution of the shape of the thin film separating them is predicted, as is the rate at which this film drains.

3. Film rupture due to van der Waals forces: When the rate-limiting coalescence step of film drainage causes an unstable film to become very thin, then attractive van der Waals forces pull the drop interfaces together and cause rupture. The rupture time and the rupture mode are determined as functions of the system parameters.

Task Significance:
Drop interactions and coalescence play key roles in a variety of natural and industrial phenomena, including liquid-liquid extraction, raindrop growth, multiphase flow, and processing of bimetallic melts within the liquid-phase miscibility gap. In liquid-liquid extraction of separation, it is advantageous to promote interactions and coalescence for efficient and faster operations, while in the processing of bimetallic materials coalescence must be controlled so that the two materials are uniformly dispersed in the final product. This research aims to provide a better understanding of the fundamental factors governing drop interactions and coalescence.

Progress During FY 1995:
The primary objective of the research project was to provide a theoretical description of the hydrodynamic interaction of two viscous droplets moving through an immiscible fluid and to use this description to predict coalescence rates. The primary focus is on the interaction of the drops when in near contact. A combination of lubrication theory for flow in the film separating two drops and boundary integral theory for flow within the drops was developed to describe the near-contact interaction. This theory was combined with prior results for the interaction of two drops at moderate and larger separations and then used in trajectory analyses and in the convection-diffusion equation to predict pairwise collision rates due to gravitational motion, Brownian motion, thermocapillary migration, and bulk fluid motion. A key result is that two spherical drops come into physical contact in a finite time in the absence of molecular effects, in contrast to the prediction for rigid spheres. When the drops become very close, however, they flatten and then dimple in the region of near contact. The film drainage rate...
then becomes very slow, and van der Waals attractive forces are required to cause film rupture and coalescence. This work is relevant to coalescence and phase separation processes in liquid-phase materials processing, flotation, raindrop formation, liquid-liquid extraction, and a variety of other natural and industrial processes. It is also of fundamental interest in providing understanding of gravitational and nongravitational mechanisms of relative drop motion and coalescence.

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**TASK INITIATION:** 5/91  **EXPIRATION:** 5/95  
**PROJECT IDENTIFICATION:** 962-24-05-35  
**NASA CONTRACT NO.:** NAG3-1277  
**RESPONSIBLE CENTER:** LeRC

**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Journals**


II. MSAD Program Tasks — Ground-based

Phase Segregation Due to Simultaneous Migration and Coalescence

Principal Investigator: Prof. Robert H. Davis
University of Colorado, Boulder

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
The objective of this research is to understand the interaction and coalescence of bubbles and drops due to thermocapillary and gravitational effects. Modeling is performed via population dynamics balances to predict the rate of phase segregation under the collective or individual action of the driving forces.

Task Description:
Significant effort is being devoted to the development and performance of ground-based experiments. The trajectories of interacting drops and the rate of phase segregation are being measured in a transparent immiscible liquid system under isothermal conditions.

Theoretical work is also being performed to predict the macroscopic phase separation and drop-size distributions due to buoyancy and thermocapillary motion and coalescence of immiscible dispersion of drops by solving the general population dynamics equations retaining both spatial and time dependencies.

Task Significance:
This research on phase segregation attempts to provide a predictive tool to determine the size distributions of drops in a heterogeneous immiscible fluid mixture. Inputs from other fundamental studies on drop interactions and coalescence are used in population dynamics models to predict the size distributions. This research will ultimately be of use in materials processing, food and beverage processing, and perhaps in the biotechnology industry.

Progress During FY 1995:
Theoretical work on spherical drops included a nonhomogeneous population dynamics model to describe the simultaneous effects of drop coalescence and gravity sedimentation on phase separation in liquid-liquid dispersions. It was predicted that the rate of phase separation initially increases with time due to coalescence forming larger drops with greater sedimentation velocities, and then decreases at later times due to the larger drops settling out of the dispersion. The predictions have been verified experimentally. Related work has been initiated with thermocapillary motion of dispersed drops in a temperature gradient.

Studies to predict collective effects of Brownian diffusion and gravitational or convective motion on drop coalescence rates were also performed. It was found that the synergistic coalescence rates are higher than the individual coalescence rates added together.

Finally, a theoretical study of the effects of drop deformation on coalescence rates has been initiated. Large deformations are described by a new, three-dimensional boundary-integral formulation. Small deformations of two drops during near contact are described by a combined lubrication/boundary-integral theory. Initial results show that small deformations reduce coalescence rates whereas large deformations increase coalescence rates.

STUDENTS FUNDED UNDER RESEARCH:

| Students/Funded | BS Students: | 0 | BS Degrees: | 0 |
| Students/BS  | MS Students: | 0 | MS Degrees: | 0 |
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TASK INITIATION: 12/92  EXPIRATION: 11/95
PROJECT IDENTIFICATION: 962-24-05-39
NASA CONTRACT NO.: NAG3-1389
RESPONSIBLE CENTER: LeRC
BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals

Presentations

Theory of Solidification

Principal Investigator: Prof. Stephen H. Davis
Northwestern University

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
This work concerns our effort to understand - on a quantitative level - how various factors affect the morphology of solidification fronts and, hence, the resulting microstructures of the solidified material.

Task Description:
In the approach, nonlinear stability theory, asymptotic, and numerical methods are used to investigate the stability of the coupled systems describing the directional solidification of binary systems from the melt.

Task Significance:
The project aims at the theoretical prediction of microstructure in crystalline materials that would allow the a priori "design" of new materials. We will endeavor to answer the following central scientific question: How and under what conditions can crystals be grown in microgravity (μg) with different and "better" properties than those grown on Earth?

Progress During FY 1995:
Progress has been made in many areas of solidification. Three areas are of particular interest.

Schulze and Davis previously showed that on the basis of linear stability theory that substantial delay of morphological instabilities can be expected if during directional solidification the crystal is given certain prescribed motions. This shear stabilization has now been shown to extend into the nonlinear range. Thus, in the absence of gravity, "perfect" crystals should be able to be grown at substantially higher speeds. It remains an open question whether this stabilization exists, as on Earth, when buoyancy-driven convection is present.

If a convective flow is imposed upon a solidifying interface, then the morphology will be substantially affected. Presently, a two-dimensional steady case is being examined in order to determine how the intensity and wave length of the convection affect the onset of cellular growth.

If one wishes to study how a melt flow reacts to say, the cellular corrugations of a solidifying interface, one requires a knowledge of the "effective" boundary conditions on long scales where the morphology has been "averaged away". Likewise, if one wishes to examine eutectic-growth instabilities and one could "average away" the eutectic scale, one could find a simple model for long-wave instabilities and the effect of fluid flow on these. Both of these efforts are in progress.

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TASK INITIATION: 10/87 EXPIRATION: 2/96

PROJECT IDENTIFICATION: 962-25-05-16
NASA CONTRACT NO.: NAG3-1737
RESPONSIBLE CENTER: LeRC

Printed: 28 FEB 1996
BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals

Presentations

Davis, S.H. "Rapid solidification." Department of Mechanical Engineering, Wayne State University, 1995.

Microgravity Foam Structure and Rheology

Principal Investigator: Prof. Douglas J. Durian
University of California, Los Angeles

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
The objective of this research is to investigate the origin of the most striking and least understood rheological properties of foam by measuring elastic and flow behavior for a sequence of aqueous foams with increasing liquid content. The microscopic structure and dynamics of the foams will simultaneously be characterized by recently developed multiple light scattering techniques.

Task Description:
Foam structure and dynamics will be measured directly and non-invasively through development and use of novel multiple light scattering techniques such as Diffusing Wave Spectroscopy (DWS). Foam rheology will be measured in a custom rheometer which allows simultaneous optical access for multiple light scattering. Microgravity conditions will ultimately be required to eliminate the increasingly rapid gravitational drainage of liquid from in between gas bubbles as the liquid:gas volume fraction is increased toward the rigidity-loss transition.

Task Significance:
This experiment will constitute the first measurement of how the surprising solid-like elastic quality of foam vanishes as the volume fraction of liquid is increased. The simultaneous measurements will also permit the first quantitative correlation of macroscopic rheological behavior with the underlying microscopic structure and dynamics, thus providing new insight into the origin of the dual solid/liquid nature of foams.

Progress During FY 1995:
In the current funding period we have made important progress both in terms of the development of multiple light scattering techniques and their application to a model foam system. As for the former, analysis of multiple light scattering data crucially assumes that photon propagation can be described by a diffusion approximation with source and boundary terms set by a phenomenological penetration depth and extrapolation length, respectively. While the accuracy of this approach can in principle be no greater than about 1%, far greater errors are introduced in practice due to inappropriate treatment of source and boundary terms. We have determined how to average over the penetration depth, and how to experimentally deduce the extrapolation length. We have nearly achieved the objective of putting the Static Transmission (ST) and Diffusing-Wave Spectroscopy (DWS) theories on firmer theoretical ground and advance the reproducibility and accuracy of their application down from the 10% level to the 1% level. This is crucial if ST and DWS are to be truly quantitative probes of foam structure and dynamics. In addition, we have introduced a new optical configuration for DWS experiments which provides for significantly better photon-counting statistics and is less susceptible to systematic errors from imperfect laser beam conditioning.

To move toward our goal of making ST and DWS truly quantitative probes, we have completed a series of measurements of the angular dependence of light diffusely transmitted through a variety of opaque samples. We have shown that such data can be analyzed in terms of the extrapolation length ratio which describes the boundary conditions to be used in diffusion theory treatments of photon propagation. This phenomenological parameter appears in the theoretical results used to analyze ST and DWS data, and must be accurately known if analyses are to quantitatively accurate. Furthermore, a two-stream theory of diffusing light spectroscopies have been developed which extends the traditional theories to thinner slabs, where diffusion theories break down.
Finally, a bubble-scale model has been developed for simulating foam rheology and its dependence on liquid content, polydispersity, and dimensionality. Varying these parameters has been a long-standing problem in the theory of foams. The key is to focus on entire bubbles rather than films or vertices. Significantly, the new model can also be used to compare with our multiple light scattering experiments.

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PROJECT IDENTIFICATION: 962-24-05-73  
NASA CONTRACT NO.: NAG3-1419  
RESPONSIBLE CENTER: LeRC

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals


Presentations
II. MSAD Program Tasks — Ground-based

The Influence of Gravity on Nucleation, Growth, Stability and Structure in Ordering Soft-Spheres

Principal Investigator: Prof. Alice P. Gast  
Stanford University

Co-Investigators:

Pine, Dr. D.J.  
Exxon Research & Engineering

Task Objective:

While previous investigations of colloidal crystals have focused primarily on the structures produced in the ordered arrays, we intend to probe the dynamics of the crystallization process, in particular, focusing on the influence of the gravitational field on ordering. In this study, we will use a novel new light scattering experiment, known as diffusing wave spectroscopy, allowing us to monitor particle motion and ordering in an otherwise turbid suspension. This approach will provide the means to study, on a particle level, a model ordering process in situ. The application of diffusing wave spectroscopy can thus be viewed as an important new noninvasive experimental probe of interparticle dynamics.

Task Description:

Among the attractive features of aqueous colloidal suspensions are long-range Coulombic repulsions diminishing the influence of hydrodynamic interactions between particles. In this system we can observe and provoke homogeneous nucleation, bcc or fcc lattice formation, defects, twinning and a crystallization growth instability analogous to those observed in molecular systems. These phenomena motivate and provide a means for us to investigate the role of interfacial tension, transport and gravity in this process. A key feature in this study is the use of density matching conditions to study the late stages of crystallite growth and ripening without the complicating factors arising from their sedimentation. In order to investigate the influence of a gravitational field on ordering in soft-sphere colloidal suspensions we propose to begin with ground-based studies of polystyrene particles of size ranging from 100 to 500 nm suspended in mixtures of hydrogenated and deuterated water. Choosing an appropriate mixture of D₂O and H₂O we can render the suspension neutrally buoyant. In this fashion we will be able to monitor nucleation rate, growth, and ripening under a variety of gravitational conditions. Future studies on more dense colloidal particles of technological importance would require actual low gravity experiments.

Task Significance:

An additional feature of this research will be an improved understanding of the transport of multiply scattered light through complex, multiphase systems. Our combination of experiments and theoretical treatments is of fundamental importance in the understanding of phase transitions and for further applications of diffusing wave spectroscopy. The interest in colloidal systems as important materials themselves and as model molecular systems is only beginning to expand to phase transition dynamics. This study will provide a definitive test of the nature of ordering dynamics, and its sensitivity to gravity. The link to molecular processes will have important implications for future studies of gas-liquid and solid-fluid transition kinetics.

Progress During FY 1995:

We began a dynamic scattering study of the influence of gravity on the structures in and growth of colloidal crystals. In order to study the opaque suspensions of colloidal particles in water, we use the multiple scattering experiment known as diffusing wave spectroscopy (DWS). We have expanded this approach by developing a new spatially resolved detection system for DWS studies of ordered colloidal suspensions. This novel scattering geometry with its variable incident beam and detector separation allows us to characterize the propagation of the diffusing light over different length scales.

In order to understand the dynamics within a single crystallite in a polycrystalline suspension, we have developed a shearing cell that produces macroscopic ordered arrays for interrogation with our spatially resolved DWS experiment.
This experiment has provided the first unambiguous evidence for the applicability of the diffusion equation for light interacting with an ordered medium, provided we observe photons scattered over distances sufficient for the light to be randomized. The autocorrelation function from the diffusive photons gives us quantitative evidence for enhanced short time particle dynamics corresponding to motion about a lattice point in a cage of nearest neighbors. For small separations between the incident laser and the detector, the measured autocorrelation function cannot be fit with the predictions of a diffusive model. These non-randomized photon scattering events show a long time "frozen-in" component in the autocorrelation function characteristic of the slow relaxation and annealing phenomena associated with crystalline systems. This dynamic scattering technique provides the opportunity to expand this project in the future to investigate additional systems with the Laser Light Scattering facility being developed at NASA Lewis Research Center.

We are also pursuing crystal growth and sedimentation studies via visualization. This promising approach will allow us to characterize the growth and dynamics of crystallites at larger length and time scales than probed in the scattering experiments. We observe the relaxation of the sharp concentration gradient between a concentrated sediment and a more dilute suspension. We control the suspending fluid density through the addition of D2O, to create conditions where the particles vary from positively to negatively buoyant. When the gravitational force and crystal growth direction are antiparallel, the expanding front is unstable. We are currently quantifying the kinetics of crystalline sediment expansion in simulated micro-gravity conditions through the use of density matched suspensions.

**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

Presentations

Gast, A.P., "Colloids and Surfaces." European Science Foundation meeting, June, 1995.


II. MSAD Program Tasks — Ground-based

**Plasma Dust Crystallization**

Principal Investigator: Prof. John A. Goree

Co-Investigators: No Co-I's Assigned to this Task

**Task Objective:**
The objective of this research is to demonstrate that charged particulates in a plasma environment form an ordered structure.

**Task Description:**
The investigator will design and test apparatus for radio-frequency (RF) plasma generation / particulate confinement, and analyze the structure of suspended particulates. This will include working with German scientists in designing microgravity experiments.

**Task Significance:**
This investigation begins a new genre of microgravity research. It will provide: (1) a macroscopic model of how atoms arrange themselves in a crystal or liquid, (2) a much-needed test of dusty plasma theories used in space physics and astronomy, and (3) an understanding of particulate contamination during plasma processing steps of microchip manufacturing.

**Progress During FY 1995:**
A series of ground-based experiments was undertaken to advance and exploit our new plasma crystal technique. The plasma crystal consists of plastic microspheres that are charged and levitated in a gas discharge plasma. They arrange themselves in ordered structures. We produced and studied crystalline lattices, fluids near the freezing point, isotropic fluids, gases, and the hexatic intermediate phase that lies between the crystalline and liquid phases of 2-dimensional media.

Analysis methods and hardware were tested in the laboratory to define concepts for future flight experiments. As collaborators for a DARA-funded German experiment that is planned as a get-away-special, we advised in the design of the flight hardware.

Theoretical simulations were carried out to characterize the inter-particle potentials for the microspheres in the plasma.

**STUDENTS FUNDED UNDER RESEARCH:**

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**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Journals**

II. MSAD Program Tasks — Ground-based

Discipline: Fluid Physics


Presentations


Fluid Mechanics of Capillary Elastic Instabilities in the Microgravity Environment

Principal Investigator: Prof. James B. Grotberg
Northwestern University

Co-Investigators:
Halpem, Prof. D.
University of Alabama

Task Objective:
The primary objective of this work is to study the fluid mechanics of pulmonary airway closure in the microgravity environment. This task is also to study the stability of liquid lined flexible tubes with surfactants in the liquid.

Task Description:
The task is to study coupled fluid-elastic instabilities of a flexible tube coated internally with a thin liquid film by both theoretical and experimental means. The principal investigator will analyze a mathematical model of post-closure filling flows and reopening phenomena using a similar approach as the closure problem. An experiment of a flexible tube containing a thin liquid layer on its wall and a liquid core of a much smaller viscosity but similar density is also proposed.

Task Significance:
The results will be of interest to most fluid dynamicists concerned with surface phenomena and dynamic fluid-solid interactions.

Progress During FY 1995:
The progress for the project so far were in theoretical modeling and experiments. For the progress of theoretical modeling, three evolution equations were derived and solved numerically using a Crank-Nicolson finite difference time matching scheme. The results for the radial oscillations show that the enclosure time is reduced when the system is oscillated with a finite frequency. These results indicate that the destabilizing effect of expiration is greater than the stabilizing effect of inhalation. For the progress of experiments, the necessary lab equipments for the experiments have been purchased and set up and run in the lab successfully. The results from these experiments indicated the increase in closure time with even small amount of surfactant in the systems. Results from these experiments using an oil film and water core in rigid tube support theoretical values derived by the PI and Co-I.

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BIBLIOGRAPHIC Citations FOR FY 1995:

Proceedings

II. MSAD Program Tasks — Ground-based

Discipline: Fluid Physics

Presentations

Effects of Convection on the Thermocapillary Motion of Deformable Drops

Principal Investigator: Prof. Hossein Haj-Hariri
University of Virginia

Co-Investigators:
Borhan, Prof. A.
Pennsylvania State University

Task Objective:
The objective of this theoretical effort is to extend the understanding of thermocapillary motion of drops by including drop deformations. The convective transport of momentum and energy as well as the effects of container walls will be accounted for by the work.

Task Description:
The focus of the initial effort is on the following tasks (a) theoretical work on the boundary-integral formulation for the axi-symmetric motion and deformation of a drop in a cylindrical container, (b) finite-volume formulation of the same problem using domain-mapping techniques, and (c) analysis of the transport of a color function to demarcate the drop boundary.

Task Significance:
This research will provide information on the motion and shapes of drops driven by interfacial tension gradients. A novel feature of this research is that to facilitate the modeling of this phenomenon using a computer, the sharp boundary between the drop and the surrounding liquid is "smeared" in the computations. The anticipated outcome of this research is the speed and shapes of the drops under various conditions and an enhanced understanding of how much the interface can be smeared before the results are intolerably degraded.

Progress During FY 1995:
Computational results have been obtained for the migration velocity of finite viscosity deformable drops, as well as bubbles, at finite values of the Reynolds and Marangoni numbers. The bubble results are in complete agreement with those in the literature. It has been demonstrated that development of accurate means of computing second derivatives on an adaptively-refined grid with a tree structure is vital for these calculations. The second derivatives were computed economically through a Taylor series-based pointwise linear-algebraic manipulation of locally calculated naive values. Extra grid refinement is required to accurately capture finite Reynolds and Marangoni number effects due to the presence of boundary layers. Analytical results have been obtained demonstrating the critical need for refinement in order to reduce interface smearing errors. The need for resolution becomes greater the further the viscosity or conductivity ratios of the drop fluid to the surrounding fluid deviate from unity. The present algorithm and code with smeared properties at the interface is capable of providing quantitatively accurate results (to within 1%) when compared to analytical results, where they exist. Previous studies of a similar nature have been qualitative in nature.

Parametric studies of the thermocapillary migration of bubbles/drops have demonstrated the appearance of shape deformations at moderate Reynolds numbers (20-50). Such deformations are found to significantly affect the migration velocity. During the past year, the code was also extended to study other problems, viz. the interaction of two bubbles or drops in thermocapillary motion, and the role of surfactant transport on moving and deformable interfaces. The latter was achieved using a new formulation wherein the surface convective-diffusion equation is solved on the refined three-dimensional cartesian grid. Predictions based on preliminary computations for the thermocapillary migration of drops in the presence of insoluble surfactants are in complete agreement with the analytical results.
II. MSAD Program Tasks — Ground-based

Discipline: Fluid Physics

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PROJECT IDENTIFICATION: 962-24-05-45
NASA CONTRACT NO.: NAG3-1396
RESPONSIBLE CENTER: LeRC

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals

Presentations
Evaporation from a Meniscus within a Capillary Tube in Microgravity

Principal Investigator: Prof. Kevin P. Hallinan
University of Dayton

Co-Investigators:
Ervin, Dr. J.
University of Dayton

Task Objective:
The primary objectives of this project are to perform (i) macroscopic studies of evaporation from a capillary meniscus, (ii) preliminary measurements of the flow field in the evaporating thin film region, and (iii) further development of the Coherent Forward Scattering Particle Image Velocimetry (CFSPIV) technique applied to the evaporating thin film region near the contact line.

Task Description:
This is a fundamental study in microgravity of evaporation from curved liquid-vapor interfaces in capillaries in the interfacial region controlled: (i) by surface tension; and by (ii) surface forces due to solid-liquid intermolecular interactions. The experiments will include simultaneous particle image velocimetry (PIV), interferometry, and wall temperature measurement in order to obtain the fluid velocities, the interface shape, and the wall temperature in the thin film region. Thermocapillary effects will be quantified by the measured wall temperature and the fluid velocity profile in the thin film region.

Task Significance:
The study of the thermocapillary effect on the interfacial instability of an evaporating extended meniscus may help explain the failure of the Capillary Pump Loop in a microgravity environment. The knowledge obtained from this study could result in radical new heat exchanger technologies that will benefit both space exploration and terrestrial applications.

Progress During FY 1995:
Work has continued and is near completion on macroscopic studies of evaporation from a heated capillary meniscus. This work has focused on: evaluating the effectiveness of heat transport from the whole of evaporating meniscus; investigating the stability of the heated and evaporating meniscus and attempting to gain an improved understanding of the mechanisms leading to the instabilities; and finally on attempting to improve the heat transfer effectiveness and interfacial stability through the use of either surface coating on the capillary wall or the addition of a non-volatile solute to the solvent. The progress has continued toward the development of the CFSPIV technique to the heated evaporating near contact line region. The configuration of an interferometric metrology setup to measure the displacement of the microscope stage to the fixed objective lens to an accuracy of 0.05 microns was completed. The production of calibration diffraction image of the light scattered by 0.2 micron polystyrene particles in methanol for particle positions ranging for -10 to 10 microns from the focal plane of the lens was accomplished. The accomplishment also included the development of a correlation technique to compare actual diffraction images individually to the calibration images to establish the position of the particles relative to the focal plane of the lens.

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Task Initiation: 12/92 Expiration: 12/95
Project Identification: 963-25-0A-21
NASA Contract No.: NAG3-1391
Responsible Center: LeRC

Printed: 28 FEB 1996
BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Presentations
II. MSAD Program Tasks — Ground-based

Interfacial Transport and Micellar Solubilization Processes

Principal Investigator: Prof. T. A. Hatton
Massachusetts Institute of Technology (MIT)

Co-Investigators:
Smith, K.A.
Massachusetts Institute of Technology (MIT)

Task Objective:
The objective of this research is to develop and implement a new diffusion cell for the fundamental investigation of the effects of surfactants on the interfacial transport of solutes between two phases. In the one case, the concern will be the retardation effect the surfactant monolayer has on solute transfer at conditions below the critical micelle concentrations. This work will assist in determining the effects of interfacial compressibility and solute and surfactant structure on the activation barriers to transport. In the other case, the rates of formation of micelles and reversed micelles at the interface will be investigated. These, too, will be affected by the prevailing solution conditions. To date, this type of study has been hindered because the techniques used for measurement of interfacial transport processes are prone to artifacts associated with ill-defined hydrodynamic conditions.

Task Description:
During this task period, we have concentrated on developing a new diffusion cell approach for the measurement of interfacial transport and solubilization rates without introducing hydrodynamic effects that can complicate data interpretation and analysis. The technique relies on the fact that many fluorophores can be bleached irreversibly when exposed to high intensity light sources for brief periods of time. The rate at which fluorescence is recovered in the bleached zone depends on the diffusion of fluorophores from outside this region. Our task has been to set up the equipment, to integrate the various components, and to establish a methodology for data analysis. A testing program to identify the important parameters and variables over which a space-based experimental study would be beneficial is to be undertaken.

Task Significance:
Interfacial transport and solubilization processes are important in many industrial, commercial and home operations, ranging from large-scale reaction and separation processes to detergency in dishwashing and laundry applications. New separation and reaction technologies based on surfactant self-assembly show promise for reducing the potential for environmental contamination in many industrial processes, or for remediation of already contaminated resources. Through the development of techniques to measure the rates at which the processes of interest occur, and relating these data to the chemical properties of the systems under study, we will be in a position to design more effectively the surfactants for specific new applications. In some cases, gravity-driven convective currents will complicate the data analysis and interpretation, and this establishes the need to conduct experiments under the low-gravity conditions of a space-based experimental program.

Progress During FY 1995:
Optimization of the FRAP system operating parameters has been carried out. To obtain acceptable levels of sensitivity, a minimum photobleaching extent of 30% is required to perturb the system sufficiently. This level of energy input into the system requires very low fluorophore concentrations, on the order of 100 nM. A video detection system of increased sensitivity has been set up to allow observation of dilute solutions. In addition, a mechanism has been devised to shield the test cell from the observation beam except at data-acquisition times to allow increases in the observation beam strength. A translation stage now permits scanning of the photobleaching beam across the sample cell parallel to the interface, allowing greater flexibility in the bleaching pattern and effectively reducing the diffusion problem to one dimension. Finally, the entire system has been automated to

Printed: 28 FEB 1996

II-449
facilitate multi-user operation. Preliminary experiments have been carried out in the water/reversed-micellar two-phase system with both fluorescein and fluorescently-labeled proteins as solutes. Experiments are also planned to investigate transport in aqueous two-phase polymer-based systems.

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**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

Proceedings
II. MSAD Program Tasks — Ground-based

Discipline: Fluid Physics

Critical Phenomena, Electrodynamics, and Geophysical Flows

Principal Investigator: Dr. John Hegseth
University of New Orleans

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
To demonstrate the feasibility of using fluid near its critical point as a compressible fluid for an electroconvection experiment.

Task Description:
Create an experimental system where a spherically symmetric density gradient is established in a compressible fluid. Such a system called Compressible Geophysical Flow Experiment (CGFE) will mimic the property of planetary fluid flows. In order to understand how such an experimental system works, knowledge of elements from fields such as critical phenomena, electrodynamics and compressible fluid dynamics have to be brought together.

Task Significance:
This experimental work will help the understanding of the Geophysical flows and their characteristics and direct consequences.

Progress During FY 1995:
The task of this ground based study is to build a working critical fluid cell that demonstrates the E field induced density distribution. This grant came into effect on 8/22/94 and we have been working on this project for nine months. Although the density gradient has not been demonstrated yet, our confidence in the success of these planned measurements is supported by some important new evidence from a recent microgravity experiment which has shown that a density gradient is induced near the critical point by an electric field in microgravity. We are in the process of manufacturing the apparatus, developing our temperature and pressure control systems, and building the mechanical driving system. We have nearly finished the first phase of the detailed procurement of the specialized parts needed for this experiment and the diagnostic equipment for demonstrating the density gradient in SF6 near the critical point.

The highly compressible critical fluid is very sensitive to the presence of gravitational body force which makes it very difficult to create a spherically symmetric density gradient on Earth. Moreover, the critical fluid is also sensitive to convection heat transfer under 1g. In addition, the high pressure of the critical cell makes it more difficult to develop the mechanical and thermal forcing mechanisms. Because of the waiting in the procurement and manufacturing processes, we have also been building and testing an incompressible fluid cell. The simplified cell will also help to predict the phenomena that will occur in space experiment. The two cells will be prepared in parallel, one critical fluid cell for low gravity testing and another incompressible fluid cell for ground based studies.

We have built and tested a rotating sphere in Dow Corning 0.65 cS silicone oil using magnetic coupling. We have also tested the Particle Velocimetry and electrical continuity using this system.

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Printed: 28 FEB 1996
Nonlinear, Resonance-Controlled Bifurcation Structure of Oscillating Bubbles

Principal Investigator: Dr. R. G. Holt
Jet Propulsion Laboratory (JPL)

Co-Investigators:
Gaitan, D.F.
Jet Propulsion Laboratory (JPL)

Task Objective:
The topic of the proposed investigation is the nonlinear dynamics of acoustically forced bubble oscillations. Specifically, the interplay between the nonlinear resonances of the system and the asymmetry imposed by 1g levitation forces required to isolate a bubble for ground-based experimentation will be investigated. The objectives of the proposed experiments are:

1) to observe the possible resonance-governed bifurcations by varying (within the limits imposed by 1g) the relevant parameters (driving pressure $P$, driving frequency $f_d$, equilibrium bubble radius $R_0$). These would be the first measurements of location and type of bifurcation ever. The expected bifurcations are saddle-node, period-doubling, Faraday (Hopf-like) and Hopf; and
2) to identify the $(P, f_d, R_0)$ regime where only mg experiments can obtain data, and to place bounds on the effect of the buoyant force on the threshold $(P, f_d)$ values for each $R_0$-dependent modal resonance bifurcation measured in 1g.

Task Description:
Single-bubble experiments in water at 1g with a sinusoidally-varying acoustic pressure are planned. Both acoustic levitation and optical levitation will be used to position the bubbles. Data acquisition will be accomplished via optical Mie scattering (for radius and frequency information) and high-speed video (for shape mode data).

$R_0$ will be varied via controlled rectified diffusion between 0.2 and 2. (normalized to the linear monopole resonant size of a bubble with fixed $f_d$). $P$ will be varied between 0.05 and 1.5 atm for the acoustic levitation experiments. $f_d$ will be varied between 0.2 and 2.5 (normalized to the linear resonance frequency of a bubble of fixed $R_0$).

The roles of surface tension, viscosity and especially surface viscosity in determining the location in parameter space where the shape bifurcations take place will be examined by varying the type and concentration of added surfactant to the host liquid, usually water. It should be possible to increase the value of $P$ where the Faraday instability sets in, perhaps "uncovering" some of the predicted saddle-node/period-doubling bifurcations for purely spherical motion even in 1g.

Task Significance:

There are two reasons for the gap in experimental knowledge. First, prior experiments, the first of their kind, were limited in scope. Second, for 1g acoustic levitation of bubbles, the same acoustic field provides levitation (positioning) and drives the oscillations. Changing $P$ changes the levitation position (which is displaced from the antinode due to gravity), making execution and calibration difficult.
II. MSAD Program Tasks — Ground-based

Discipline: Fluid Physics

There exists much theoretical work on the small amplitude, spherical problem, and this is largely borne out by experiment. Some spherical models have been pushed to large amplitudes, where a rich resonance-bifurcation structure is predicted, but has not been observed experimentally, largely due to the onset of stable shape oscillations through the Faraday instability.

Recently, some exciting new theoretical work had addressed the Faraday bifurcation by examining (for limited modes and frequencies only) the nonlinear resonant coupling (1:1 and 1:2 frequencies) between shape and volume modes. In particular, Yang, Feng and Leal [Yang, S.M., Feng, Z.C. and L.G. Leal, J. Fluid Mech. 247, 417 (1993)] show that the very nature of the coupling and relative mode stability are altered when there is some initial deformation due to an external flow; thus, for 1g experimentation, the observed onset and stability of shape oscillations will occur at lower pressures, and will likely show other anomalous features with respect to the ideal, purely isotropic case (which could be realized in microgravity).

Progress During FY 1995:
The project has been underway for a year. The techniques for data acquisition have been finalized, and a system for taking the necessary data has been assembled from scratch. The system consists of

- a levitation system capable of stably levitating sub-resonant bubbles and driving them with acoustic pressures up to 1 atmosphere. The system uses ultra-clean, gas-saturation controlled water as the fluid medium, and is capable of utilizing any desired gas or air for the bubble contents. The relative position of the levitation cell and the lasers is controlled by PC-driven stepper micropositioners, with 1 micron resolution.

- an automated and PC-controlled laser-scattering setup capable of resolving $R_0$ for a levitated bubble to 2% accuracy over the range of 10 to 120 microns via Mie angular scattering within 10 seconds, with an accuracy of ± 1 milliradian.

- an A/D system on a PC capable of gathering both the Mie angular data and Mie time-resolved data for size and frequency information, at a maximum of 2 Msamples/sec and a time-series length of 8 Msamples segmentable.

- a video-imaging system which uses a pulsed Nd-YAg (frequency-doubled) laser as a short-pulse illumination source, and a Questar telescope as the magnification element for a 700x500 pixel resolution CCD camera. The spatial resolution of the system is 1micron/pixel relative, and 1 micron absolute in both directions.

The task is poised to begin intensive data acquisition for the next fiscal year.

Students Funded Under Research: TASK INITIATION: 9/94 EXPIRATION: 9/96
PROJECT IDENTIFICATION: 962-24-04-15 RESPONSIBLE CENTER: JPL

Bibliographic Citations for FY 1995:

Journals


Proceedings
II. MSAD Program Tasks — Ground-based

Discipline: Fluid Physics

Presentations


Thermocapillary Instabilities and g-Jitter Convection

Principal Investigator: Prof. George M. Homzy

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
The objectives of this study are:

1. To study the stability characteristics and to establish the conditions under which thermocapillary flows become three-dimensional, time-dependent, and/or chaotic.

2. To analyze the g-jitter effects on surface-tension driven flow.

Task Description:
Complimentary experimental and computational studies will be conducted to characterize the transport phenomena induced by surface tension gradients or transient accelerations. Experimentally, measurements of various flow variables will be carried out to elucidate the mechanism of the instability and the dependence of instability on various parameters of the problem. The range of Marangoni numbers will be extended to study the transitions to time-dependent flows, and perhaps to turbulence. Linear stability analyses, using a combination of inverse iteration and Lanczos methods, will be conducted to help elucidate the connection between driven cavity problems and thermocapillary convection problems.

Task Significance:
The results from this study should enhance the understanding of the basic forces acting on fluids in a space-flight environment, and the accompanying fluid response. Such an understanding can contribute directly to better the fluid handling/management technologies. This can help improve planning and execution of any space experiment that has fluids involved, thus maximizing the science/technology return pursued by these space-flight experiments, which encompasses a wide range from materials processings to biomedical research.

Progress During FY 1995:
Our work has involved studies of thermocapillary convection and of g-jitter convection.

The redesign of the thermocapillary convection experimental apparatus was completed. This new design allows better temperature control, facilitates the use of LDV measurements, and allows for the variation of both the fluid depth and the cell aspect ratios. Work over the last year has been devoted to studying various transitions in flow structure in thermocapillary/buoyancy convection. We have now identified two transitions; one from a steady two dimensional flow to a steady three dimensional flow. The former is in general agreement with that we observed in an earlier set of experiments, and the latter is entirely new, never having been observed before. The newly discovered mode has been identified with so-called "meniscus convection" and work is in progress to build a simple model to explain the oscillatory convection and the mechanism and variables that determine the period of oscillation. We are in the process of preparing a manuscript on this work.

Two prototype problems in g-jitter convection have been studied. The first of these is a fully enclosed box subject to a lateral temperature gradient and vertical oscillations; the second is an open slot with wall temperatures such that both a vertical thermal stratification and a constant applied lateral temperature gradient are imposed. The results indicate that resonances are possible between the imposed jitter and either neutral gravity waves or boundary layer instability modes. These resonances are strongly dependent on fluid Prandtl number and box size. In the fully enclosed box at unit aspect ratio, parametric resonances with internal gravity waves are important, and have their...
greatest influence on streaming flows at Prandtl numbers near unity. In the modulated slot problem, streaming persists to very large levels of jitter until a stability limit is reached. The secondary time dependent two dimensional flow that ensues exhibits unexpected turbulent bursts that are interpreted through dynamical systems theory as being characteristic of two unstable states.

**STUDENTS FUNDED UNDER RESEARCH:**

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**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Journals**

**Presentations**
II. MSAD Program Tasks — Ground-based

Discipline: Fluid Physics

Turbidity of a Binary Mixture Very Close to the Critical Point

Principal Investigator: Prof. Donald T. Jacobs

The College of Wooster

Co-Investigators:

No Co-I's Assigned to this Task

Task Objective:

This experimental activity is intended to measure the light transmitted through a near critical fluid consolute point of a density matched binary mixture.

Task Description:

1. Develop a room temperature thermal control system for the sample that enables temperature control inside of ±3μK.

2. Assemble and use a phase sensitive detection system to detect the small light intensity variations expected close to the consolute point.

3. The turbidity data will be reduced according to the formalism of P. Calmettes et al. (Phys. Rev. Lett. 28, 478 (1972)) and of R.A. Ferrel (Physica A 177, 201 (1991)).

4. Undergraduate students will assist Prof. Jacobs, making his activity a strong educational experience for the students.

Task Significance:

The resulting data will be used to confirm and quantify a small exponent "h" in a denominator 

\( 1 + (q \tau_x) \)  

that describes the correlation length dependent distribution of scattered light from near critical fluids. Also, "two-scale universality" can be tested from the data set when combined with measurements of the heat capacity.

Progress During FY 1995:

The work for FY95 has been focused on the thermostat and software control. This included heater wire wrapping and thermistor installation and aging in three co-axial shells. The software work involved integrating control of the three actively temperature controlled shells. Each shell provides a factor of 10 to 100 reduction in temperature fluctuations. Short term control is now ±5 micro-K. However, room temperature swings still induce 100 micro-K swings on twelve hour time scales.

A master program that tests for stable temperature control and laser illumination levels before accepting data was worked on this summer by the supported undergraduate students.

The phase-locked-loop transmitted light detection setup has shown its sensitivity (<0.1%) by indicating a misfilled cell and temperature drifts. The light detection element of the apparatus is complete.

Three more students have received their Bachelors degrees and new students have been brought into the learning experience. One student (Anne Flewelling) was selected for an American Physical Society Summer Industrial Internship at IBM.

Printed: 28 FEB 1996
II. MSAD Program Tasks — Ground-based

**Discipline:** Fluid Physics

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**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Proceedings**

**Presentations**

Hall, P. "The turbidity of the binary fluid mixture methanol-cyclohexane near the critical point." Senior thesis 1995.
II. MSAD Program Tasks — Ground-based

Kinetic and Transport Phenomena in a Microgravity Environment

Principal Investigator: Prof. David Jasnow
University of Pittsburgh

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
Theoretical research will be carried out in several areas which involve kinetic and transport phenomena in a microgravity environment. Specifically, attention will be paid to a number of two-phase phenomena in which thermocapillarity effects play an important role. These include, kinetics of phase separation, coupled diffusing fields (e.g., temperature and concentration) and the motion of and transport through two-phase interfaces.

Task Description:
Methods proposed include modeling of conserved order parameter dynamics in two-phase situations at the coarse grained or semimicroscopic level. Optimized codes are being developed to carry out the modeling. Coupling of order parameter to hydrodynamic fields and temperature fields are included. Thermocapillary effects such as thermomigration of drops and coalescence are observed, characterized and the underlying physics analyzed. Modeling will also be performed using techniques developed in the study of dynamical systems.

Task Significance:
The modeling at the coarse grained, semimicroscopic level allows description of phenomena over length scales ranging from the correlation length on up to semi macroscopic sizes. Local thermodynamics is included using the coarse grained free energy. The interface motion is naturally tracked and surface tension dependence on temperature and other parameters is naturally included in the specification of the coarse grained free energy. Hence universal features of transport in complex situations can be extracted, and, for example, examination of phenomenological boundary conditions can be performed at a somewhat more microscopic level.

Modeling from the perspective of dynamical maps abandons the familiar partial differential equations of the subject, but preserves the essential physics, symmetries, etc. Here, if successful, a substantial improvement in computational efficiency can be achieved, at the cost of being able to compute specific material-dependent quantities.

The coarse grained modeling allows one to study phenomena from rather short length scales, i.e., on the scale of the diffuse interface thickness on up to macroscopic, the Lattice Boltzmann modeling of flows combined with parallel processing will yield efficiencies far beyond that of traditional computational fluid mechanics, and our studies of phase asymmetry indicate that a temperature gradient could be used as a control parameter in separating phases.

Progress During FY 1995:
The main effort under this theoretical project during this period has been to observe thermocapillarity effects such as thermomigration in two-phase systems, within a coarse-grained, semimicroscopic description, to characterize the phenomena and to understand the physical mechanisms for them. There are three main areas of progress: (a) The response and dynamics of a two-phase interface, in a coarse grained description, to a temperature gradient; (b) Thermomigration phenomena and phase separation kinetics in a temperature gradient, where the dominant effects are diffusion driven; and (c) A study of two-dimensional capillary driven motion of single and multiple droplets, and phase separation kinetics, including hydrodynamical effects.

Project (a) was designed to gain insight to the effect of a temperature gradient on a two-phase interface. Both using analytic approaches and numerical relaxation methods for the dynamics of a nonconserved order parameter, we have determined the motion of an interface for a nonconserved order parameter under a temperature gradient, and have analyzed the free energy landscape for an interface under different boundary conditions.
In (b) we observe, and analyze using arguments derivable from the coarse-grained free energy for the system, thermomigration phenomena in purely diffusive systems. Temperature variations of the interfacial free energy drive the motion, which occurs via diffusion. We have also considered phase separation phenomena in a temperature gradient to investigate potential anisotropy in domain growth introduced by the gradient.

In (c) we have developed a new method for forward integrating the coupled dynamical equations for a conserved order parameter (at the coarse grained, semimicroscopic level) and hydrodynamic flow fields for two-phase systems in two dimensions using a biharmonic solver. The method has been tested on single drop thermomigration in a temperature gradient, simple multiple drop configurations, coalescence, and phase separation kinetics.

Recent studies indicate that our research will continue on modeling multi-phase flows using coarse grained free energetics and the associated fluid mechanics equations as well as using lattice Boltzmann techniques. In the latter case very efficient codes for parallel processing are being developed for T3D processors and applied at the Pittsburgh Supercomputing Center. Research will also continue on phase asymmetry and on the development of interface equations to describe interface motions at the macroscopic level. In addition, an experimental group has begun careful experiments based on the ideas concerning the effect of phase symmetry.

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| BIBLIOGRAPHIC CITATIONS FOR FY 1995: |

Journals
Bhagavatula, R.S. and Jasnow, D., Phase asymmetry in thermocapillary motion. Journal of Chemical Physics, (in print 1995)

Surfactant-Based Critical Phenomena in Microgravity

Principal Investigator: Prof. Eric W. Kaler
University of Delaware

Co-Investigators:
Paulaitis, M.E.
University of Delaware

Task Objective:
Our objective is to characterize by experiment and theory the kinetics of phase separation and the metastable structures formed during phase separation in a microgravity environment. The system we are studying is a mixture of water, nonionic CIEj ethoxylated alcohol surfactants, and supercritical CO\textsubscript{2} at temperatures and pressures where the coexisting liquid phases have equal densities (isopycnic phases).

Task Description:
1. Locate and characterize ordinary and tricritical points in surfactant/water/SCF mixtures about which two coexisting phases will be matched in density.

2. Examine using scattering methods the non-equilibrium structures inside both the spinodal and binodal regions after quenching with either temperature or pressure jumps through or near a critical point.

3. Examine the kinetics of mixing of two phases "quenched" into one phase.

4. Carry out polymerization in a non-equilibrium phase.

Task Significance:
The potential ability to control the phase separation of liquid mixtures in a microgravity environment has led us to investigate novel processing strategies for materials synthesis in space. As a cost-effective first step in this research program, we have begun to examine phase separation kinetics and materials synthesis under simulated microgravity conditions in earth-based experiments. These conditions are achieved by using mixtures of surfactants and highly compressible supercritical fluids at elevated pressures to produce two co-existing fluid phases of equal density, thus eliminating the density differences that normally drive phase separation. The driving force for phase separation is obtained by rapid expansion of the supercritical fluid, which can also be polymerized to kinetically capture fluid structures in order to measure their physical, chemical, and mechanical properties. Our goal is to explore different processing strategies and the fabrication of a wide range of materials that would eventually be manufactured in space.

Progress During FY 1995:
We have continued our characterization of isopycnic phases in ternary mixtures of C\textsubscript{8}E\textsubscript{9}, CO\textsubscript{2}, and H\textsubscript{2}O. Measured densities of the water-rich and surfactant-rich phases in these mixtures verify the visual location of isopycnic phases at 30.0 C and 305 bar. The search for isopycnic phases has also been extended to different ethoxylated alcohol (CIEj) surfactants. Isopycnic phases have been found in ternary mixtures containing C\textsubscript{12}E\textsubscript{6}, CO\textsubscript{2}, and H\textsubscript{2}O, and we have visually located isopycnic phases at several different temperatures and pressures in ternary mixtures of C\textsubscript{10}E\textsubscript{6}, CO\textsubscript{2}, and H\textsubscript{2}O. Since C\textsubscript{12}E\textsubscript{6} is a stronger amphiphile than C\textsubscript{8}E\textsubscript{9}, and is available in purer form, we chose C\textsubscript{10}E\textsubscript{6} mixtures as a model system for studies of phase separation kinetics.

We have measured the binodal line on the water-rich side of the three-phase region of ternary mixtures of C\textsubscript{10}E\textsubscript{6}, CO\textsubscript{2}, and H\textsubscript{2}O at 28.2 C and 105 bar. We have found that separation is slow over the entire two-phase region, which precludes determining the critical point on this binodal from measurements of relative phase volumes. However, the observed relative critical opalescence allows us to approximately determine the critical point to be 4.5
wt% C_{10}E_6, 6.2 wt% CO_2, and 89.3 H_2O at 28.2°C and 105 bar. For a more precise determination of the critical point we plan to measure the turbidity of samples containing varying surfactant concentrations close to the binodal. The concentration closest to the critical point will have the greatest turbidity.

We are also searching for isopycnic phases in solutions containing different supercritical fluids. The motivation for changing the supercritical fluid is to replace water with D_2O to facilitate neutron scattering investigations of phase separation in a simulated microgravity environment. Two promising supercritical fluids: sulfur hexafluoride (SF_6) and trifluoromethane (R-23) have been identified based on their density, accessible critical properties, and their relative solubilities in water and surfactant.

We are measuring the binodal on the water-rich side of the three-phase region in pseudoternary mixtures of C_{10}E_6, R_{23}, H_2O, and D_2O at 25.0°C and 108 bar. The binodal for this mixture is more sharply curved than the one in CO_2 mixtures and phases separate faster than those in the CO_2-containing mixtures. These differences make it possible to more accurately approximate the critical point from the observed relative phase volumes and critical opalescence. These observations locate the critical point at 7.1 wt% C_{10}E_6 and 3.0 wt% R_{23}, with the balance H_2O/D_2O in equal weight ratios.

We are studying phase separation of the isopycnic mixtures using both small angle light scattering (SALS) to determine the rate of phase separation, and dynamic light scattering (DLS) at large angles (20-120 degrees) to characterize morphology during separation. The SALS apparatus has been calibrated by measuring the static light scattering from a dilute solution of 2.875 micron polystyrene particles. The location of the minimum in the scattered intensity agrees with that calculated from Mie scattering theory (using program written by Dr. W.J. Wiscombe at NASA Goddard Space Flight Center). The high pressure cell for the SALS experiments is under construction, and a new rectangular geometry of the bath and CCD camera setup has been shown to give much cleaner data than the circular geometry we had been using previously. Current SALS measurements on phase separating critical binary mixtures of C_{10}E_6 surfactant and water in the square cell geometry will validate the method by comparison with published literature data. In addition, we are modifying an existing high pressure cell with flat sapphire windows for use in small angle light scattering from phase separating isopycnic mixtures of surfactant-water-CO_2.

The range of length scales probed by neutron scattering compliments those examined with light scattering. We have published results showing the interactions of CO_2 at high pressures with C_{12}E_6 micelles at compositions, temperatures and pressures near the isopycnic phases described above. We will perform neutron scattering experiments on the isopycnic quaternary mixtures of C_{10}E_6, R_{23}, H_2O, and D_2O as they are phase separating in the near future. Requests for neutron beam time have been approved by NIST.

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**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Journals**

**Presentations**
Instability of Velocity and Temperature Fields in the Vicinity of a Bubble on a Heated Surface

Principal Investigator: Dr. Mohammad Kassemi

Co-Investigators:
Rashidnia, Dr. N.
NYMA, Inc., NASA Lewis Research Center (LeRC)

Task Objective:
The objective of this combined experimental/numerical work is to investigate the fluid flow and temperature fields in the vicinity of a bubble attached to a heated solid surface. The goal of the research is to investigate the interaction between thermocapillary and natural convective flows in the vicinity of the bubble and identify the parametric range for which unsteady periodic oscillations occurs on earth and in a low-gravity environment. It is hoped that the experience gained during this investigation will aid in designing and proposing the right space experiments.

Task Description:
Ground-based experiments are performed to identify the following criteria:

1. the appropriate experimental design and fluids
2. the required diagnostic tools
3. the parametric ranges for which oscillatory flow and temperature fields are obtained.

A numerical model is developed and its accuracy is tested by ground-based experimental results. The verified numerical model is used to predict the fluid flow and temperature fields in the low-g environment. The experience and knowledge base gained during this effort will be used to design an experimental system for both 1-g and low-g applications.

Task Significance:
The oscillatory thermocapillary phenomenon is not only of fundamental importance in understanding the behavior of fluid flow in space but it also has practical significance in terrestrial applications. For example, in crystal growth, the oscillatory temperature and flow fields created by unavoidable bubbles next to the solid-liquid interface may seriously affect the quality of the emerging crystal and in nucleate boiling the presence of bubbles on the solid surface may increase the heat transfer rates considerably. The results of this investigation will enhance our understanding of the role of capillary forces in fluid management, boiling processes, and materials processing for both space and ground-based applications.

Progress During FY 1995:
Although the funds for this research were initiated in late fall of 1994, significant progress has already been made. The design and construction of a test cell for the preliminary experiments has been completed. Interesting results have been obtained with this test cell using both Mach-Zehnder and Shearing interferometry and laser sheet flow visualization techniques. The 1-g experimental results have shown that both steady and periodic oscillatory motions are attainable in the vicinity of the bubble depending on the value of the Marangoni number.

As part of the theoretical/numerical study, the equations describing the temperature and flow fields around the bubble were formulated and cast into dimensions form using appropriate scaling parameters. A finite element numerical model was developed and used to simulate the temperature and flow fields around the bubble. Steady state temperature and velocity fields predicted by the finite element model are in excellent qualitative agreement with the experimental results. More rigorous quantitative comparisons are near completion and will be reported in the future. A parametric study of the interaction between Marangoni and natural convective flows including conditions pertinent to microgravity space experiments is in progress.
Based on the above preliminary simulations and experimental results, the following conclusions can be made:

1. Both experimental observations and numerical predictions indicate that the thermocapillary flow induced by bubbles on Earth is greatly influenced and affected by its inevitable interactions with buoyancy-driven convection.

2. Preliminary interferometric measurements in our laboratory demonstrate that the flow and temperature fields around the bubble can go through various oscillatory modes depending on the parametric space of experiment. The nature and origin of these modes and their dependence on the interactions between natural and thermocapillary convection are as of yet not clearly known.

3. Preliminary steady-state numerical predictions indicate that the temperature and fluid fields generated by the bubble in low-gravity and its shape are significantly different from their ground-based counterparts. There has been no direct low-gravity experimental verification or confirmation of the numerical predictions.

4. Results and conclusions obtained during ground-based experiments with regard to oscillatory convection cannot in any way be extrapolated to determine the behavior of the thermocapillary flow in space.

STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 12/94  EXPIRATION: 12/96
PROJECT IDENTIFICATION: 962-24-05-86
NASA CONTRACT NO.: NCC3-405
RESPONSIBLE CENTER: L&RC

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

NASA Tech Briefs

Presentations

**Stabilization of Thermocapillary Convection by Means of Nonplanar Flow Oscillations**

**Principal Investigator**: Prof. Robert E. Kelly
University of California, Los Angeles

**Co-Investigators**: No Co-I's Assigned to this Task

**Task Objective**: It has been demonstrated theoretically that small amplitude, nonplanar flow oscillations can stabilize Rayleigh-Benard convection. The first goal of this research is to discern if similar stabilization can be achieved for the thermocapillary (Marangoni) instability by the same means.

**Task Description**: An asymptotic expansion based on Reynolds number will be used to perform stability analysis on both Rayleigh-Benard and Marangoni-Benard convection. A numerical approach using a Fourier representation in the horizontal directions and a spectral approach in the vertical direction will be used to obtain a coupled set of ordinary differential equations in time which can be solved by use of Floquet theory.

**Task Significance**: If stabilization is possible, then the effects of finite amplitude oscillations will be determined in order to see how much stabilization is possible. And if significant stabilization is predicted, an experiment will be proposed at a later time.

**Progress During FY 1995**: A numerical analysis for finite Reynolds number was carried out to determine how much stabilization or destabilization can be achieved by means of nonplanar oscillations. One important result is that the range of stabilization (when it occurs) is limited to a certain interval of Re. For the operating conditions corresponding to Earth, the Pearson mode is most critical mode at Re - 0. As Re increases, this mode is clearly stabilized (higher Ma) up to a value of Re equal to 193. At Re = 193, the long-wave mode becomes the most unstable mode and instability occurs. The great stabilization of the Pearson mode can be achieved with proper selection of the frequency of the unsteady shear. The critical Marangoni number can be increased by a factor of 7.5 for certain cases.

The cut-off Re (at which long-wave mode destabilizes) has been found to follow Re = 1.86 sqrt(B/(Cr Pr)), where B is the Bond number, Cr is the Crispation number, and Pr is the Prandtl number. In microgravity environment, long-wave modes become dominant for high Re. It was shown that the flow can become unstable even a liquid is heated in a stable manner (from above).

**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

*Journals*
Microgravity Heat Transfer Mechanisms in the Nucleate Pool Boiling and Critical Heat Flux Regimes Using a Novel Array of Microscale Heaters

Principal Investigator: Prof. Jungho Kim
University of Denver

Co-Investigators:
Moore, C.
University of Denver

Task Objective:
The objectives are to determine the relative contribution of the various heat transfer mechanisms to the overall heat flux in a sub-cooled, nucleate pool boiling in a microgravity environment, and to obtain quantitative data measuring local heat transfer coefficients at critical heat flux conditions.

Task Description:
A two-dimensional array of microscale heaters will be used for these tests to provide control and make measurements. These heaters will be kept at a constant temperature and their power input will be measured to control the heat flux. With the ability to measure and control temperatures and heat flux over very small areas, it should be possible to adequately quantify some of the behavior during nucleate pool boiling and critical heat flux during various stages of bubble growth. Testing would be limited to normal gravity tests.

Task Significance:
The development of such a microscale heater could significantly enhance the state-of-the-art in scientific instrumentation in the area of flow boiling and two-phase flow. Understanding the boiling phenomena has tremendous impact on electronic cooling in the nuclear, oil, and electric power industries.

Progress During FY 1995:
Progress was made in the areas of heater design and construction, chip mounting, data acquisition setup, and heater calibration. A set of masks was designed and constructed for the working heaters. After four attempts, a working set of nine chips each containing an array of 148 heaters was obtained. The heaters are platinum deposited in a serpentine pattern. Numerical analysis of this heater geometry showed that the heater surface is uniform to within 0.2 degrees C. Two of these chips were mounted on a Pin Grid Array (PGA) and wire bonding between the chips and the PGA was made for the center sixteen heaters in the array. The PGA was mounted on a printed circuit board (PCB) so that connections to the heaters could be made through card edge connectors. The resistance of the heaters was found to vary from 500 to 700 Ohms. The control circuits for these heaters have been laid out on a PCB containing 16 control circuits. One PCB was made and wired with components so that testing of the circuits with the actual heaters could be performed. If this board checks out, then ten additional boards will be constructed. A circuit that controls the resistance of the heaters in the array through computer control has been constructed and tested on a protoboard. Layout of this circuit on a PCB will begin soon. A data acquisition system was also obtained during this period. The system consists of an I/O Tech Dagbook 216 capable of sampling 16 channels of data at 100 kHz. The system can be expanded to 256 channels of data. Data acquisition and reduction routines are currently being written. A board for calibrating the heaters was also designed and constructed.

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PROJECT IDENTIFICATION: 962-24-05-87
NASA CONTRACT NO.: NAG3-0386
RESPONSIBLE CENTER: LeRC

Printed: 28 FEB 1996
BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Presentations
Molecular Dynamics of Fluid-Solid Systems

Principal Investigator: Prof. Joel Koplik
City College of New York

Co-Investigators:
Banavar, J.R.
Pennsylvania State University

Task Objective:
The purpose of this theoretical research is to examine the molecular behavior of fluids in small confinements and near boundaries using molecular dynamics calculations and the statistical mechanics of classical fluids. It looks at time and spatial scales where continuum fluid mechanics provides no insight.

Examples investigated include (1) non-Newtonian fluid flows (2) phase separation of thin films of fluid mixtures (3) the breaking and coalescence of droplet interfaces driven by gravity or flows and (4) molecular scale studies of wetting and adhesion.

Task Description:
The numerical calculations are based on Molecular Dynamics, wherein one integrates the (classical) equations of motion for interacting molecules of various species, based on a prescribed interaction and specified thermodynamic operating conditions and mechanical forces. Within the framework of molecular dynamics, it is straightforward to impose a desired value of gravity or simulate the presence of g-jitter.

Task Significance:
We intend to carry out a number of calculations involving coexisting liquid, vapor and solid phases under flow conditions, with two common features. First, the systems will be studied at the molecular scale to obtain otherwise unavailable information. Second, the problems to be studied deal with fundamental issues in the physics of fluids and interfaces that are of relevance to the microgravity program.

Progress During FY 1995:
We have begun molecular studies of non-Newtonian fluid flow, using chain molecules made by tying Lennard-Jones atoms together via imposed confining potentials. The first flow considered is in a "linear Couette rheometer", a channel with sliding walls and periodic boundary conditions. We have studied steady unidirectional flows, start-up flows and time-periodic flows in order to characterize the computer fluids to be used in further studies. Two configurations of direct physical relevance are now being studied by simulation: a liquid bridge extensional flow, (relevant to forthcoming Shuttle experiments by McKinley), and flow in an expanding-contracting channel (relevant to corner flow singularities).

We have studied the "Roughening of the Liquid-vapor Interface Induced by Phase Separation". The effect occurs in a thin binary liquid film when one of the liquid phases has an affinity to the solid substrate and when the interfacial tension between the two fluids is large. We have studied this phenomenon using molecular dynamics simulations, and a phase-field model based on "Model-B" of dynamic critical phenomena. The model results are in qualitative accord with observation, and elucidates the roughening process. A paper has been submitted to Phys. Rev. Lett.

We have published a paper on "Stokes Drag at the Molecular Level" in which we have studied the translational and rotational motion of a sphere in a bulk viscous fluid using MD simulations. The results of continuum lubrication calculations are reproduced even for spheres comparable in size to the fluid molecules. However, the divergences present in lubrication theory are removed at very short separation distances due to a depletion of fluid from the region between the solids and the breakdown of no-slip boundary conditions. We have begun molecular dynamics...
simulations of the contact mechanics problem of a compressible sphere squeezed between two confining molecular plates. Interesting phenomena that can uniquely be addressed by our simulations will include the effects of a liquid layer on the rough wall and hysteretic effects associated with the cycling of the load.

We have submitted a paper "Suppression of Coalescence due to Shear and Temperature Gradients" to Phys. Fluids. The work is in collaboration with the Italian Microgravity Center in Naples. The paper contains ground and space experimental results, molecular dynamics simulations, and simple continuum hydrodynamic estimates showing how motion of an intervening fluid can suppress the coalescence of fluid bodies which would otherwise occur readily. Calculations of molecular scale interfacial dynamics are in progress. One such calculation considers the effects of contaminants and surfactant-like additives to driven interfacial rupture processes, as arise in cusp-forming experiments.

We are continuing our molecular studies of wetting processes, and MD calculations of advancing and receding contact line motion in a simulated Wilhelmy-plate configuration continue. Three types of plate surface are being considered - atomistically smooth, smooth with an isolated defect, and atomistically rough, respectively. The first shows no hysteresis as expected, the second is being analyzed to compare to earlier theoretical work, and the hysteretic aspects of the third are being pursued.

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**TASK INITIATION:** 6/90 **EXPIRATION:** 12/95

**PROJECT IDENTIFICATION:** 962-24-05-43

**NASA CONTRACT NO.:** NAG3-1167

**RESPONSIBLE CENTER:** LeRC

### BIBLIOGRAPHIC CITATIONS FOR FY 1995:

**Journals**


**Presentations**

Koplik, J., "Fluid flows at the molecular scale." Invited presentation at the APS Fluids Dynamics meeting in Atlanta, Georgia, November 20, 1994.
Fluid Dynamics and Solidification of Metallic Melts (FDSMM)

Principal Investigator: Prof. Jean N. Koster
University of Colorado, Boulder

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
The objective of this effort is to investigate surface tension-driven convective flow of low Prandtl number melts, e.g., metals, and their effect on the solid-liquid interface. (The Prandtl number is the ratio of the kinematic viscosity to the thermal diffusivity.) This will require flow visualization which will be achieved by using tracer particles and a real time X-ray system. This research will focus on fluid physics phenomena of surface-tension-driven flows in material processing. It is known that liquids for which Pr>1 have different underlying fluid physics than liquids for which Pr<<1. The research is focused on gallium melts.

Task Description:
Progress toward achieving radiographic particle image velocimetry (RAPIV) of liquid metal convection has required extensive technological development. The RAPIV will be used to obtain system flow velocity vector fields using appropriate computational analysis. RAPIV has been used successfully to detect the solid-liquid interface of solidifying gallium and to observe tungsten particles in molten gallium.

Rectangular, two-dimensional test cell geometries for the series of high temperature RAPIV experiments have been chosen. Two aspect ratios (length:height) will be used: 4:1 and 1:1 (with unit integration depth). The 4:1 ratio is the classical "Hurlie" geometry which is used often for comparative numerical studies.

The Integrated Convection Apparatus and Rotating Undercarriage Support (ICARUS) is a high-temperature (up to 1,000°C) modular furnace capable of establishing any combination of vertical and horizontal gradients in test cells of various geometries. This provides the capability to vector the gravity body force from 0 deg. (horizontal) to 90 deg. (vertical).

Task Significance:
Fluid physics is the foundation of material solidification. The primary purpose of this research is to develop a unique research capability for convective flow visualization of metallic and electronic melts during the solidification process in real time. By observing the flow pattern of a liquid metal during solidification, knowledge will be gained in understanding the solidification process. This knowledge can be used to build better processing facilities which can produce metals and electronic materials with improved properties (e.g., yield strength and toughness).

For example, titanium is used in today's aircraft, but little is known about titanium solidification. The design engineer uses a safety factor of 1.33 which causes the parts to be heavier than they need to be. If the solidification process was better understood, the titanium parts would be lighter. This would reduce the aircraft weight, thereby improving the fuel efficiency or allowing greater payload capacity.

Progress During FY 1995:
The funding for this project has been completed. The advanced research facility for liquid metal (Pr<< 1) flow visualization using an X-ray system and high temperature furnace (up to 1000°C) has been built and tested. Gallium has been selected as the test material. Visualization of the density field has been achieved with the X-ray system. A density change of 0.1% can be detected and is enhanced using false coloring. A 10°C horizontal temperature gradient was imposed. The mottle is obscuring some of the data; this problem is being addressed. Eventually, flow patterns will be observed using tracer particles. The particles must exhibit good wetting characteristics, high X-ray absorption and be neutrally buoyant.
The development of neutrally buoyant, chemically inert tracer particles with high radiographic absorptivity is essential to the RAPIV project. A substantial ongoing portion of the development effort was directed toward the design of the appropriate tracer particles: The particles are currently being developed through the support of a different task. The most promising particles are glass particles coated with nickel with a final layer of gold.

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**TASK INITIATION:** 2/91  **EXPIRATION:** 7/95

**PROJECT IDENTIFICATION:** 962-24-05-33  **NASA CONTRACT NO.:** NCC3-210  **RESPONSIBLE CENTER:** LeRC

**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Journals**


Thermocapillary Convection in Floating Zones under Simulated Reduced Gravity

Principal Investigator: Prof. Sindo Kou
University of Wisconsin, Madison

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
The main objective of the research is to enable comparison between calculated and observed patterns of thermocapillary convection in floating zones, regardless of whether or not the zones are cylindrical in shape.

Ground-based flow visualization experiments will be conducted under simulated reduced-gravity conditions, i.e., where thermocapillary convection dominates over natural convection just as in microgravity. This will include silicone-oil zones and molten zones in NaN₃ rods. Computer simulation of thermocapillary convection will be conducted and the calculated results will be compared with those observed in flow visualization.

Task Description:
A mathematical formulation will be used to quantitatively describe the optical distortions. This formulation allows the calculated flow patterns and solid/liquid interfaces to be converted into distorted ones so that they can be compared directly with those observed in flow visualization to verify the validity of computer simulation. Conversely, it can also be used to convert the observed flow patterns and solid/liquid interfaces into undistorted ones.

Task Significance:
Comparisons between calculated and observed patterns of thermocapillary convection in floating zones, though significant, have been rare (if performed at all), due to complications from optical distortions caused by the lens effect of the floating zones. With the numerical tool, thermocapillary flows in floating zone configurations can be more deeply understood. The capability of prediction can help the design of process active control, thus the quality of the material and medicine will be improved.

Progress During FY 1995:
1. Equations have been derived to relate the optical distortions caused by floating zones to their refractive indexes and free surface shapes.

2. Both computer simulation and flow visualization of thermocapillary convection have been conducted on a liquid zone of silicone oil, a molten zone in an NaN₃ rod, and floating zone crystal growth.

3. Good comparison has been achieved between calculated and observed results based on flow patterns and velocity fields.

4. An image analysis system for flow measurements was put together as planned, consisting of a PC, frame grabber, CCD camera, macro lens, TV monitor, VCR, particle tracking software, and commercial image processing software. Interaction between one software system and another was being worked out. Work on thermocapillary convection in NaN₃ molten zones was written up and submitted for publication.
II. MSAD Program Tasks — Ground-based

**Discipline:** Fluid Physics

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**PROJECT IDENTIFICATION:** 962-24-05-46

**NASA CONTRACT NO.:** NAG3-1393

**RESPONSIBLE CENTER:** LeRC

**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Journals**


Printed: 28 FEB 1996
Analysis of Phase Distribution Phenomena in Microgravity Environments

Principal Investigator: Prof. Richard T. Lahey
Rensselaer Polytechnic Institute

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
The objective of this research is to map the void distribution throughout the tube cross-sectional area in two-phase flows. The principal flow patterns to be studied are the bubbly and slug-flow regimes.

Task Description:
The research approach is to conduct normal gravity testing using equal density simulation experiments. Normal gravity experimental data, as well as available microgravity data, will be used to verify computational fluid dynamic models that are being developed. Two types of equal density simulations will be used: liquid with solid spheres, and a pair of immiscible liquids.

Task Significance:
The orientation of the phases with respect to each other and the tube wall affects the heat transfer characteristics and the work required to pump the two-phase mixture. Terrestrial applications include the nuclear, electric power and oil industries.

Progress During FY 1995:
The focus during the past year has been on the equal-density, immiscible liquid experiments using either n-butyl benzoate or silicon oil as the dispersed phase in water at 20°C. Initially, a closed loop was built whereby both phases were recovered and recycled; however, in this configuration, droplets ranging from 40 to 600 microns in diameter were generated without the ability to achieve a monodispersed droplet size. An open loop operation is being tested with a new droplet injector that will hopefully provide the capability to control the flowrate of the continuous phase, the capability to control the diameter of the dispersed phase droplets, the capability to produce the desired global void fraction, and the capability to produce a nominal droplet size and distribution.

Some single and two-phase testing has been conducted at various Reynolds numbers. It was determined that LDA did not produce an accurate measurement of the droplet size distribution. Testing is now focusing on whether the use of Fiber Phase Doppler Anemometry (FPDA) can produce a better size distribution; however, the use of receiving probes at all locations is not easily accommodated as the probes traverse the test section. Work is in progress to effect the necessary changes to utilize the FPDA system.

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STUDY INITIATION: 12/92 EXPIRATION: 11/95

PROJECT IDENTIFICATION: 962-24-05-72

NASA CONTRACT NO.: NAG3-1400

RESPONSIBLE CENTER: LeRC

Printed: 28 FEB 1996
II. MSAD Program Tasks — Ground-based

Nonlinear Drop Dynamics and Chaotic Phenomena

Principal Investigator: Dr. L. G. Leal
University of California, Santa Barbara

Co-Investigators:
Trinh, E.H.
Jet Propulsion Laboratory (JPL)

Task Objective:
The general objective of this collaborative experimental and theoretical project is to explore nonlinear phenomena associated with acoustic and/or electrostatic levitation of drops or gas bubbles in air or a viscous fluid. These include both phenomena intrinsic to large amplitude oscillations of shape (or volume, in the case of gas bubbles), and phenomena that are a consequence of the levitation process. In the former category, we include studies of newly discovered coupling between shape modes and volume oscillations for gas bubbles, and oscillations of non-spherical uncharged and charged drops in electric fields, and in combined acoustic and electric fields. When experiments aimed at understanding or exploring these fundamental, intrinsic phenomena are carried out in an acoustic field, which may be either steady or time modulated, there can be strong coupling between the bubble or drop position or shape, and the acoustic levitation force, which can lead to additional nonlinear oscillatory phenomena that can be difficult to separate from the intrinsic phenomena of interest.

Task Description:
Following earlier work based upon small deformation asymptotics, coupled with a nonlinear dynamics analysis of the weakly nonlinear amplitude equations, current theoretical work is focused upon numerical solutions for finite deformations of shape. Our most recent work has considered an inviscid drop (charged or uncharged) in a steady or time-dependent electric field. We utilize the well-known boundary integral technique for this case, but we have also developed both full finite-difference and spectral techniques to solve free boundary problems with viscous effects.

Experimental studies have utilized ground-based levitation systems located at JPL. These include both acoustic levitation for small gas bubbles, and a combination of acoustic and electrostatic levitation for viscous drops in air. The latter is particularly interesting because it allows compensation for the mean deformation associated with the levitation process, so that the mean drop shape is spherical.

Task Significance:
We have worked on nonlinear dynamical effects due to time-dependent forcing, and/or an initial deformation for both gas bubbles and viscous drops. These problems are important both from a fundamental point of view as a concrete and accessible physical system which exhibits many of the classical nonlinear phenomena, but also because the problems being studied have significant application to phenomena in multiphase flow. The work on gas bubbles has yielded a fundamentally new understanding of forced or free volume oscillations in the sense that we have shown that purely radial oscillations, without change of shape, is an exceptional phenomena. The generic behavior is that there is an exchange of energy between volume (radial) and shape modes, which becomes extremely strong near resonance. Current numerical and experimental work is aimed at understanding large amplitude effects, including conditions that may lead to sufficiently large oscillations of shape that the bubble breaks. Bubble breakup due to time-dependent fluctuations of an isotropic background pressure has not been investigated previously as a mechanism for breakup, insofar as we are aware.

The dynamics of viscous drops in an acoustic or electrostatic levitator was originally studied in the microgravity program of NASA primarily because it was viewed as the basis for containerless materials processing applications. Although this is no longer a major priority, the fundamental problem is still one of considerable interest, especially as a model system for understanding drop dynamics in electric fields for applications ranging from meteorology (raindrops) to ink-jet printers. There is also a growing interest in the possibility of using time-dependent droplet motions as a basis for measurement of material properties of the liquid or interface at extreme conditions of...
temperature. Finally, as with the bubble, the oscillating drop offers a convenient laboratory system to continue to improve our understanding of the theory of nonlinear systems.

Progress During FY 1995:
The experimental effort in FY 95 has concentrated on four separate tasks: (1) the study of large amplitude shape oscillations of ultrasonically levitated drops in a time-varying electric field; (2) the investigation of large amplitude shape oscillations of large bubbles ultrasonically trapped at reduced hydrostatic pressure; (3) the analysis of mode coupling for nonlinear oscillations of drops levitated in air and drops and bubbles levitated in a liquid medium; and (4) the investigation of large amplitude capillary waves on levitated thin liquid shells.

1. Oscillations of non-spherical uncharged and charged drops ultrasonically levitated in air and in the presence of a constant or time varying electric field have been quantitatively investigated. A soft nonlinearity has been measured for the fundamental mode of shape oscillation, sub-harmonic resonant excitation of the fundamental 1-2 and the next higher mode 1-3 has been discovered, hysteresis in the large amplitude response of the fundamental mode has been documented, and anomalous free decay frequencies for drops driven into the higher resonant modes have been observed.

2. Air bubbles up to 5 mm in diameter have been trapped in 1 g in an ultrasonic standing wave at 22 kHz and at a hydrostatic pressure reduced to about 0.3 atmosphere. Shape oscillations have been excited for various resonant modes described by spherical harmonic expansion. For the water and air combination, shape modes up to 1=5 can be excited to macroscopic amplitude. No observation of coupling to the radial mode has been possible due to the cavitation onset in the host liquid due to the high intensity acoustics required for trapping the bubbles in 1 G. An approach for a microgravity experiment to investigate this radial to shape coupling is being developed.

3. Digital video analysis of shape oscillations of drops and bubbles in a liquid has been used to investigate the resonant mode coupling of driven and freely decaying oscillations. For drops, direct coupling between the 1-6 and 1-3 axisymmetric modes has been observed for ultrasonically driven oscillations. Similar coupling between the axisymmetric 1-4 and 1-2 non-axisymmetric modes has been documented. Coupling between the first four resonant modes of freely decaying bubbles has been quantitatively measured through spherical harmonics expansion of the observed oscillations.

4. The observation of the optically scattered signal from a levitated liquid shell has been used to characterize the evolution of large amplitude acoustically-induced capillary waves. An interpretation of the power spectrum of the waves has been attempted using the interaction of symmetric and antisymmetric waves on the shell. High speed video observation has revealed a highly turbulent shell surface corresponding to a continuous frequency spectrum prior to the bursting of the shell.

The theoretical effort in FY 95 has been focused on three specific directions:

(1) Application of our numerical boundary-integral code to study the problem of an electrostatically levitated drop in a gravitational field, where both the charge Q and the electric field strength E must be nonzero. We are concerned with steady shapes in steady E fields, oscillation frequencies for perturbed initial shapes as a function of E and Q, and the response to time-dependent electric fields, among other issues. This work is currently in the final stages of publication.

(2) Re-examination of the problem of instabilities in the position of particles, bubbles or drops due to coupling with the acoustic field in levitation. We have specifically been studying the computation of acoustic pressure forces and fields when a body undergoes large (or at least not small) changes in position or shape. This has led to a new formulation of the instability of position first studied theoretically by Rudnick and Barnatz (J. Acoust. Soc. Am. 87 (1), 81 (1990)). It is also the basis for numerical computations of time-dependent position and/or shape in an acoustic pressure field.
(3) We have completed a study of the translation instability of a bubble in a time-dependent pressure field. A new asymptotic theory predicts a regime of chaotic dynamics in which a near-resonant coupling between two shape modes is modulated by direct dynamic interaction with the translational motion. Two nearby shape modes grow via interaction with a pressure-driven radial oscillation, and this produces a translational motion as described approximately by Benjamin and other earlier investigators. However, unlike earlier work, we find that the translational motion damps the shape modes and thus the process is forced to start over (ref. Feng and Leal, Phys. Fluids, 7, 1325 (1995)).

(4) We have prepared a review paper on "nonlinear bubble dynamics for the Ann. Rev. of Fluid Mechanics (to appear in 1996)."

(5) We have continued to work on numerical simulations of finite amplitude coupling of shape and volume for a gas bubble in steady and time-dependent pressure fields in support of the corresponding experimental studies.

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### TASK INITIATION: 12/91   EXPIRATION: 11/95

### PROJECT IDENTIFICATION: 963-24-07-01

### RESPONSIBLE CENTER: JPL

### BIBLIOGRAPHIC CITATIONS FOR FY 1995:

**Journals**

Oscillatory Cross-Flow Electrophoresis: Application to Production Scale Separations

Principal Investigator: Dr. David T. Leighton
University of Notre Dame

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
The task objective primarily consists of an experimental verification of the expected performance of an oscillatory cross-flow binary separation device. This device is designed to use an oscillatory electric field across the narrow gap of a specially designed electrophoresis channel to cause species with different electrophoretic mobilities to have different time/location histories within the gap. This oscillatory motion is coupled with an oscillatory fluid cross-flow to achieve rapid separation of molecules based on their electrophoretic mobilities. The system acts as a semi-permeable barrier, actively transporting molecules with mobilities either greater or lower than some adjustable value across the cell, while rejecting all others. Because the throughput is largely governed by the amplitude of the oscillatory cross-flow, protein throughputs several orders of magnitude greater than those in conventional continuous free-flow electrophoresis devices are theoretically achievable.

Task Description:
Initially, the design for the oscillatory cross-flow binary separation device will be finalized and the device itself constructed. After construction of the device, its performance will be first qualitatively tested using the dyes phenol red and naphthol green B. As the amplitude of the electric field is changed, the dyes will be selectively allowed to pass through the separation cell from one reservoir to another. Subsequently, quantitative measurements of the separation achieved by the device will be made using the proteins cytochrome-C, albumin and thyroglobulin. These readily available proteins can be analyzed using HPLC equipment available to the PI at the Center for Bioengineering and Pollution Control at the University of Notre Dame. Concurrent with the experimental studies, we will also conduct simulations of the device to investigate end effects, finite concentration effects, and the influence of more general electric fields and oscillatory cross-flows. It is hoped that these investigations will allow us to optimize the device and further improve the selectivity and throughput.

Task Significance:
Because the amplitude of the throughput is largely controlled by the amplitude of the fluid motion and only indirectly by the electrophoretic mobility, the throughput of this device is potentially several orders of magnitude greater than conventional free-flow electrophoresis devices. Experimental verification of the theoretical concepts underlying the proposed device are crucial, however. The research program is designed to 1) provide such verification and 2) to further optimize the system.

Progress During FY 1995:
The graduate student chosen for the project began 1995 by attaining theoretical background in the research group and throughout the field, designing the experimental system and selecting hardware and software. The final experimental system includes the separation cell, a bipolar power supply to drive the electric field, a specially designed syringe pump to drive the oscillatory buffer flow, and a computer and software to acquire data and accurately control and phase lock the oscillatory driving forces. Key design problems included shielding the proteins from gas generated at the electrodes in the separation cell and selecting a buffer fluid to carry the proteins through the device. To address these problems, a plexiglass cell was designed and built to investigate the resistances of the buffer with and without added membrane. Results of the experiments allowed selection of a membrane of ideal pore size and electrical resistance and a buffer solution that would both minimize Joule Heating while maintaining the protein solubility. The final design is complete and the Binary Oscillatory Crossflow Electrophoresis technique experiments are projected to begin by November 1.
II. MSAD Program Tasks — Ground-based  
Discipline: Fluid Physics

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Project Identification: 962-24-08-16
NASA Contract No.: NAG8-1080
Responsible Center: MSFC

Printed: 28 FEB 1996
Low Dimensional Models for Thermocapillary Convective Flows in Crystal Growth Processes

Principal Investigator: Prof. A. Liakopoulos
Lehigh University

Co-Investigators:
Blythe, Prof. P.A.
Lehigh University

Task Objective:
The objective of this work is to construct and validate low dimensional dynamical models of surface tension driven flows that are relevant to crystal growth processes. The models will be used to study process stability and the use of reduced-order models for control schemes.

Task Description:
A data base of 2-D, axisymmetric, and 3-D numerical solutions of the full nonlinear governing equations will be constructed using spectral element methods. These simulations are based on a variation formulation for unsteady viscous flow and an Arbitrary-Lagrangian-Eulerian description. The Karhunen-Loeve, K-L, procedure is used to determine spatial structures of the computed flow and thermal fields. Coupling of the K-L procedure with the method of weighted residuals for the full models will yield a low-dimensional representation of the flow system.

Task Significance:
Reduced-order models make feasible stability and bifurcation calculations for thermocapillary flows of practical interest. Furthermore, they can provide the basis for designing model predictive controllers for the suppression of instabilities that occur during crystal growth processes in a microgravity environment.

Progress During FY 1995:
Collection of data from direct numerical simulations of thermocapillary flows in rectangular cavities has continued. Two configurations are under investigation: a) differentially heated open cavities of aspect ratio (width/height) A = 1,2,4 and b) open shallow cavities with spatially periodic temperature distribution imposed on the free surface. In case (b) the governing equations are solved in a computational domain consisting of a single module with periodic boundary conditions on the side boundaries. All simulations are time-accurate and they incorporate the flexibility of the free surface.

We have developed computational tools for the Karhunen-Loeve decomposition (POD) of 2-D, oscillatory, convective flows and the construction of low-dimensional dynamical models. We have successfully constructed and validated low-order models for transitional, forced and free convection flows in cavities and channels and have been working on extending the methodology to problems with moving geometry and deformable meshes.

We found that it is necessary to include a minimum number of eigenmodes in the low-order model for a successful reconstruction of the full numerical data. Retaining fewer modes in the truncated series expansion either does not produce stable oscillations in time or fails to predict the correct amplitude of the oscillations. Keeping more modes however, can both reduce the accuracy and restrict the validity of the low-order models because of noise introduced by higher modes even though the energy level of the associated fluctuations is extremely low. The influence of the spatial resolution of the original data, and the number of snapshots utilized on the performance of the low-order models has been studied. In addition, the effects of scaling on the properties of the low-order models was examined.

Investigations on interfacial instabilities in two-layer Poiseuille flow are also in progress. Initially, we have neglected temperature and viscosity fluctuations. We are currently working on incorporating these effects in our analysis. Preliminary results indicate that temperature and viscosity perturbations have a pronounced effect on the
interfacial mode of instability. This is in contrast to our findings on the stability of plane, single-layer, Poiseuille flow for liquids exhibiting exponential viscosity-temperature dependence.

The Prandtl-Batchelor theorem has been successfully applied to thermally driven flows in cavities. Convective flows in cavities are characterized by the presence of single or multiple regions of closed streamlines. We have recently presented new results for the structure of closed eddies in thermally driven flows of low Prandtl numbers and high Grashof numbers. The analysis provides an extension of the classical Prandtl-Batchelor theorem. Numerical calculations given for laterally heated rectangular cavities are in excellent agreement with the extended theorem.

**STUDENTS FUNDED UNDER RESEARCH:**
- BS Students: 0
- MS Students: 0
- PhD Students: 2

**TASK INITIATION:** 6/94  **EXPIRATION:** 6/96
**PROJECT IDENTIFICATION:** 962-24-05-88
**NASA CONTRACT NO.:** NAG3-1632
**RESPONSIBLE CENTER:** LeRC

**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Journals**


**Presentations**

**Absolute and Convective Instability of a Liquid Jet at Microgravity**

**Principal Investigator:** Prof. Sung P. Lin

**Co-Investigators:**

No Co-I's Assigned to this Task

**Task Objective:**

The objective is to establish a definite role of capillary, viscous, and inertial forces in the absence of gravity by using the fluid dynamics problem of the stability of a liquid jet as a vehicle. The objective will be achieved by re-examining the known theories that can be verified completely only in microgravity. Of particular interest are the wavelengths and growth rates of instabilities in a convective Rayleigh-type jet. In addition, atomization-type Taylor-mode jets will also be studied. A wide range of Weber and Reynolds numbers will be studied, and any unexpected phenomena in microgravity that may require a new interpretation of dynamic capillary force will be examined.

**Task Description:**

The results of the proposed work will provide some benchmark knowledge in fluid dynamics. When the results obtained at microgravity are compared with the known theories for 0-g and with the known experimental results obtained at one-g, one will be able to unambiguously assess the significance of gravitational and inertial forces relative to the capillary forces over a wide range of dynamic flow parameters. The original approach was to design a rig to conduct one-g experiment during the first year (1993). It was subsequently decided to design and fabricate a rig able to conduct both low-g and one-g liquid jet experiments. Numerical modeling and preliminary rig designs were accomplished during the first two years. Ground-based low-g experiments took place during the third year in the LeRC 2.2 Second Drop Tower. Two drop campaigns were conducted. Companion one-g experiments with the same rig will be done at the PI's university. Data analysis and flight experiment definition will follow.

**Task Significance:**

From a practical viewpoint, the knowledge gained on the precise mechanisms of various modes of jet breakup will allow one to improve many existing important industrial processes. These processes include film coatings, combustion of liquid fuel, and formation of various chemical sprays. Improvements of the efficiency of these processes also bring about a drastic reduction in environmental pollution. The knowledge may also be exploited for advanced material processing.

**Progress During FY 1995:**

The theoretical aspects of the work and the rig design/build-up have now been completed. Numerous discussions between LeRC designers, electrical engineers, and technicians have taken place leading to a rig design which is giving good low-g data. The design and build-up were completed and the Clarkson personnel (I. Vihinen and A. Honohan) arrived at the drop tower on June 22nd. Some modifications to the rig were necessary and were expertly carried out by tower personnel. Extended discussions with imaging personnel on the subject of proper illumination and exposure also took place. The first drop tests occurred on June 28th. Three drop tests were done for this team on this day. Additional low-g testing occurred between July 17th to 28th and September 25th to 29th. Data on the instabilities of jets in the convective Rayleigh mode was gathered for low Reynolds numbers on the order of 10-15 using motor oil as the fluid. Additional low-g tests are planned for convective Rayleigh jets at higher Reynolds numbers.

We are now in the stage of harvesting a great deal of important scientific knowledge from the rig. We have completed approximately 40 drop tower tests. The results allow us to compare with theory the experiments of Chandrasekhar's mode for a given set of Reynolds and Weber numbers. The most stunning results are probably the
photographic evidence of absolute instability which has not been recorded before. We need and additional 120 drop tower tests for the Rayleigh and Taylor modes.

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**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Journals**

**Presentations**
Magnetorheological Fluids in Microgravity

Principal Investigator: Prof. Jing Liu  
California State University, Long Beach

Co-Investigators:  
No Co-I's Assigned to this Task

Task Objective:  
This research investigates experimentally the influence of gravity in the formation of the equilibrium structure of magnetorheological fluids.

Task Description:  
Magnetorheological (MR) fluids are colloidal suspensions whose rheological properties can be varied through the application of an external magnetic field. The key to this modification is the structure induced in the particles of the suspension. This work seeks a better understanding of the equilibrium structures and the influence of gravity to its formation, dynamics of MR fluids, and therefore, the interaction mechanism. This effort will start by identifying the fluids equilibrium structure, characterizing it, and then examining the parameters controlling its formation, including the rate of the applied magnetic field and the effect of gravitational settling. The basic experimental techniques for this work will be static laser light scattering, dynamic light scattering, and optical microscopic imaging.

Task Significance:  
This research will help us to understand the basic physics of a liquid to a solid phase transition of this so called "smart" material under the application of an external field. It will additionally provide guidelines for making technologically important materials of the magnetorheological fluids in general. For example, this material can be used in automobiles as shock absorbers, clutch controls, robotic joint controls, etc., due to the viscosity change controlled by the field. In order to do so, however, we must understand and design them better.

Progress During FY 1995:  
Dr. Martin Hagenbuechle, a postdoc from the University of Konstanz, Germany, was hired and he initiated the static (SLS) and dynamic (DLS) light scattering measurements on very dilute magnetorheological emulsions.

Using an argon ion laser, measurements were taken at a fixed scattering angle of 90 degrees. With no magnetic field applied, DLS measurements were used to check the monodispersity of the ferrofluid droplets in the emulsions. These measurements also show that without magnetic field the droplets essentially behave as hard spheres.

If a strong enough (L > 1) constant magnetic field is applied to the sample, the intensity of the scattered light is observed to increase slowly up to a saturation plateau whereas the short time self diffusion coefficient, extracted from the measured correlation functions, decreases slowly. A typical time is in the order of one hour. Both effects reflect the growth of the chains formed by the dipole-dipole interacting ferrofluid droplets and can be used to determine the chain length as a function of the time the magnetic field is applied. Turning off the field results in an immediate disintegration of the chains which is observed by the drop of the scattered intensity back to its no-field value.

The saturation intensity was also measured as a function of the field strength. Up to a field strength corresponding to L = 1 no chain formation occurs and therefore the saturation intensity is constant. For L > 1 the saturation intensity increases linearly with the field strength. This can be interpreted as a linear dependence of the chain length on the magnetic field strength.
So far the significance of the results are: 1) developing a method to monitor the chain growth, 2) chain formation is a diffusion limited process in dilute MR fluids and therefore very slow, and, 3) first step of measuring the chain fluctuation.

In order to continue and extend our studies it was important to resolve some technical problems. We built a temperature control to keep the scattering cell at a constant temperature during measurements. The Helmholtz coil we used for the previous measurements provided only field strength up to ca. 40 G. We constructed a watercooled set of coils which will allow us to proceed to higher field strengths. We modified the setup to enable us to change the scattering angle. This will allow us to measure the correlation function at different scattering vectors and, by this, probing the chain dynamics on different length scales. Finally, we replaced the classical detector optic setup by a single mode fiber which is easier to align and yields better correlation functions.

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PROJECT IDENTIFICATION: 962-24-05-90
NASA CONTRACT NO.: NAG3-1634
RESPONSIBLE CENTER: LeRC

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Presentations

Liu, J., "Field-induced structures of ferrofluid emulsions." Dept. of Physics, University of Michigan, March 6, 1995.
Cross Effects in Microgravity Flows

Principal Investigator: Prof. Sudarshan K. Loyalka  
University of Missouri, Columbia

Co-Investigators:  
Tompson, Prof. R.V.  
University of Missouri, Columbia

Task Objective:
The research objectives are to:

1. Solve the Boltzmann and the Wang Chang Uhlenbeck equations to determine the flow rates (mass and heat) and the matrix of the phenomenological coefficients, for arbitrary Knudsen number (ratio of mean free path to characteristic flow dimension), for arbitrary gas (vapor) mixtures, realistic intermolecular and gas-surface interaction potentials, and for small (linear), as well as large (non-linear), gradients.

2. Verify the results by acquiring experimental data in a diffusion cell.

3. Explore applications of the results above to simulations of flows in ampoules.

Task Description:
The experimental apparatus will be designed to test the theoretical results. The classified diffusion two-bulb setup with a connecting capillary will be used with the bulbs held at different temperatures. Results from the new theoretical and experimental understandings will then be used to study flows in specific microgravity experiments through discussions with the NASA scientists and engineers.

Task Significance:
Film growth by chemical/physical vapor deposition is a process of considerable interest in microgravity experiments. The absence of natural convection should allow better control of the growth processes, but it has been pointed out that in the highly nonisothermal ampoules, thermal slip (creep) can become a matter of significant concern even for Knudsen numbers as small as $10^{-3}$. Thus, it is important to understand and control the flows that arise from the molecular (rather than the mere continuum) nature of gases and vapors.

Progress During FY 1995:
Regarding the theoretical aspects:
• The Boltzmann Equation for a monatomic gas for rigid sphere molecules and cylindrical geometry, under noncondensing conditions was solved numerically. All phenomenological coefficients have been computed.
• Initial computations for realistic potentials (monatomic gases), as well as the velocity and the creep slip, have been completed. The creep slip is found to be dependent on the type of gas, and results confirm the accuracy of recently reported variational results.
• The variational technique also has been extended, and it has been shown that the planar flows can be computed very efficiently, for all Knudsen numbers, by use of Burnett solutions.
• The diffusion slip and the creep slip have been computed for gas mixtures. Very general expressions for the condensation and evaporation jumps for gas mixtures have also been constructed.
• For gas mixtures, an integral representation of the linearized Boltzmann operator, convenient for numerical and variational computations for all intermolecular force laws, has been obtained. The kernels are currently being used for computation of the flows.

For the measurement of the cross-flows:
• A glass two-bulb apparatus for isothermal experiments has been constructed. Experimental data on two gas
mixtures (Ar-He, N₂-He) at several pressures (1 torr to 200 torr total pressure) and mole ratios have been obtained, and are found to be in good agreement with the theoretical predictions for the diffusion slip.

- Measurements of tangential momentum accommodation coefficients of several gases and gas mixtures in the transition regime have been completed with the observation of torque on a levitated rotating sphere in controlled environments. These measurements have been extended to the slip regime. We have completed new computations for interpretations of these data and, with the help of these, we have found values for the viscosities, the slip coefficients, and the tangential momentum accommodation coefficients for several noble gases. The viscosity values are in excellent agreement with the literature data.

- A stainless steel two-bulb apparatus for both isothermal and nonisothermal experiments has been constructed and tested and has now been used for extensive nonisothermal data acquisition on several gases and gas mixtures. For simple gases, the data are in excellent agreement with the theoretical predictions over a wide range of the Knudsen number. Comparisons of the mixture data with the theory will be carried out as the theoretical computations are completed.

Regarding application of the results
- FIDAP calculations of the deposition due to cross-effects on conjunction with thermophoretic motion was explored. New computational schemes were implemented and have worked well. The slip conditions and the condensation/evaporation jump conditions are currently being implemented.

**Students Funded Under Research:**

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**Task Initiation:** 11-92  **Expiration:** 11-95

**Project Identification:** 962-24-05-64

**NASA Contract No.:** NAG3-1420

**Responsible Center:** LeRC

**Bibliographic Citations for FY 1995:**

**Journals**

II. MSAD Program Tasks — Ground-based

Controlling the Mobility of a Fluid Particle in Space by Using Remobilizing Surfactants

Principal Investigator: Prof. Charles Maldarell City University of New York

Co-Investigators:
Papageorgiou, Prof. D. New Jersey Institute of Technology

Task Objective:
This research program studies theoretically and experimentally how to use surfactants to control the mobility of the fluid interface of bubbles or drops moving through a continuous liquid phase. The interfacial mobility determines the drag exerted on the fluid particle as it is driven through the continuous phase. By controlling this mobility, the steady translational velocity of the drop can be manipulated independent of the force causing the particle to move, and this control may prove useful in material processing under microgravity which requires the management of thermocapillary driven bubbles and drops.

Task Description:
Surfactant affects the interfacial mobility by creating surface tension gradients which resist the interfacial flow: Surfactant molecules dissolved in either the continuous or drop phase kinetically adsorb from the bulk sublayer adjoining the interface onto the surface. Once adsorbed, the surfactant is convected by the surface flow to the trailing pole of the particle. Accumulation at the back end causes kinetic desorption into the bulk sublayer; this increases the sublayer concentration causing a diffusive flux of surfactant out into the bulk. At the front end, the kinetic adsorption depletes the sublayer, and forces a diffusive flux of surfactant from the bulk to the sublayer. At a steady state, diffusive, kinetic and convective fluxes balance, and a concentration gradient develops on the surface with the trailing pole larger than the leading pole. Since surfactant reduces the interfacial tension in proportion to its surface concentration, the leading pole is at a higher tension than the trailing pole. The leading pole tugs at the trailing pole, and this tangential action opposes the surface flow and hinders the interfacial mobility.

To use this retardation mechanism to manipulate the interfacial mobility, the kinetic and diffusive fluxes which maintain the concentration difference across the fluid particle must be controlled. Diffusive fluxes can be controlled by using bulk concentrations large enough to form surfactant aggregates; these aggregates act as sources of surfactant monomer thereby reducing the diffusion gradients and increasing the interfacial mobility. Kinetic fluxes are controlled by the kinetic exchange coefficient, which is determined by the surfactant structure. The overall goal is to investigate both of these mechanisms.

Task Significance:
The results can be used to control thermocapillary driven bubble motion, e.g., in a glass melt, and in the miscibility gap solidification of two phase composites. In each of these examples there is a need for controlling the thermocapillary migration velocity; in the movement of gas bubbles in a melt, it is desired to have as large a migration as possible. In the case of miscibility gap solidification, the object is to reduce the migration velocity as much as possible, so that phase separation does not occur.

Progress During FY 1995:
During this reporting period, the following have been accomplished:

1. The kinetic rate constant has been obtained for a test polyethoxylated surfactant at the air/water surface by measuring dynamic tensions during surfactant transport from the axisymmetric shape analysis of a pendant bubble. Two types of experiments were performed. First, adsorption onto an initially clean surface was observed by forming a pendant bubble quickly (< 1 sec.) by injection of gas through a needle immersed in the surfactant solution. The surfactant then adsorbed onto the freshly formed interface, reducing the tension of the bubble surface.
and elongating the bubble. The bubble elongation was captured on digitized video images. Second, desorption following compression of an equilibrium monolayer was recorded by allowing a bubble to form an equilibrium monolayer on its surface and the quickly reducing the bubble area (< 1 sec.).

2. A preliminary hydrodynamic model has been developed to describe how the terminal velocity can be controlled by the kinetic rate, for the case in which the bubble motion is buoyantly driven and no diffusion gradients exist. An inviscid incompressible bubble is assumed with a fixed spherical shape moving at a relatively low Reynolds number such that the usual Stokes equations may be used to model hydrodynamic motion. This kinetic limited model has been extended to include the effects of both diffusion of surfactant in the continuous phase. This allows for the description of the effect of the bulk concentration on the terminal velocity. The PI has shown that for fixed kinetic parameters, as the bulk concentration is increased, diffusion mass transfer can be enhanced, causing the terminal velocity to be increased.

3. The PI has used the pendant bubble technique previously utilized to measure kinetic rate constants, to measure directly the equation of state relating the surfactant concentration on the surface to the surface tension. Accurate equations of state are necessary for the theoretical modeling of the effect of surfactant on bubble motion.

**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Journals**

Pan, R., Maldarelli, C., and Green, J. Conditions for promoting kinetic control during surfactant exchange and their use in measuring kinetic parameters by the pendant bubble method. Accepted for publication to J. Colloid and Interface Science, (1995).

Pan, R., Maldarelli, C., and Green, J. Measuring the surfactant surface equation of state by using the pendant bubble as a Langmuir film balance. Accepted for publication to Langmuir, (1995).

Wang, Y., Papageorgiou, D., and Maldarelli, C. Theoretical study of diffusion limited to surfactant adsorption onto rising spherical gas bubble at low Reynolds numbers. Accepted for publication to J. Colloid and Interface Science, (1995).
Stabilization and Low Frequency Oscillations of Capillary Bridges with Modulated Acoustic Radiation Pressure

Principal Investigator: Prof. Philip L. Marston  Washington State University

Co-Investigators: No Co-I's Assigned to this Task

Task Objective:
The objectives of this investigation are to: (1) investigate the response of liquid capillary bridges to acoustic radiation pressure excitation, and (2) acoustically suppress capillary breakup of long liquid bridges.

Task Description:
Liquid bridges surrounded by another liquid are studied in a modified Plateau tank apparatus. The liquid bridge is usually a silicone-oil/tetrabromoethane mixture surrounded by water. The bridge length and location are adjusted to investigate the mode coupling of the radiation pressure of the surrounding ultrasonic field. The ultrasonic field has been mapped with a hydrophone. For dynamic studies, the spatial and temporal modulation of the radiation pressure is used to selectively excite mode resonances. After deactivating the transducers, mode frequency and rate of the free decay has been monitored optically. Static bridge shapes are measured with CCD cameras attached to digitizers and a personal computer. Conditions are being investigated experimentally and/or theoretically for acoustic stabilization of bridges surrounded by air as well as water.

Task Significance:
The proposed research furthers the understanding of the dynamics of capillary bridges which can lead to improved control of float-zone crystal growth processes. The research may also lead to an increase in dynamic gravity tolerance levels for liquid bridge related phenomena on the Shuttle.

Progress During FY 1995:
A system has been built for the deployment of neutrally buoyant bridges of a silicone-oil/tetrabromoethane mixture in an ultrasonically excited water bath. The equilibrium diameter of the bridge is fixed by the size of the support but the bridge length (and volume) may be adjusted. The amplitude of the ultrasonic field is modulated at low frequencies representative of the natural frequencies of the bridge. By optically monitoring the response of the bridge while scanning the modulation frequency and adjusting the bridge location, the desired ultrasonic excitation of bridge modes has been demonstrated. The natural frequencies as a function of bridge length were measured for weakly damped modes. Ultrasonic deformation of static bridges has also been observed and a mode excitation method using frequency modulation of the transducer drive was also demonstrated. A new static method of measuring the interfacial tension between density matched fluids was also developed. The method makes use of CCD camera images.

Modifications of the dynamical theory of liquid bridges due to the radiation pressure of a sound field were investigated. For stabilization, the sound field should be such that the radial component of the radiation pressure automatically squeezes more on the larger radius (fatter) parts of the bridge and less on the slender parts of the bridge. An analysis of the dependence of the scattering of sound on the local bridge radius suggests that this may be passively achieved for a liquid bridge in air in low gravity by appropriate design of the sound field. Another strategy is active stabilization where the bridge profile is optically sensed and the ultrasonic transducer drive is modified to bring about the desired change of radiation pressure. Both mechanisms for acoustic stabilization should cause the natural frequency of the most unstable mode to be shifted to a higher frequency when the bridge length is in the stable region. This frequency shift was experimentally detected when the active control was activated for a bridge in a Plateau tank. That observation confirms that the desired control of the ultrasonic field has been achieved and suggests that acoustic stabilization could eventually be used to suppress bridge breakup in low gravity.
II. MSAD Program Tasks — Ground-based

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PROJECT IDENTIFICATION: 962-24-05-91

NASA CONTRACT NO.: NAG3-1622

RESPONSIBLE CENTER: LeRC

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals


Study of Disturbances in Fluid-Fluid Flows in Open and Closed Systems

Principal Investigator: Prof. Mark J. McCready
University of Notre Dame

Co-Investigators:
Chang, Prof. H.-C.
Leighton, Prof. D.
University of Notre Dame

Task Objective:
The objective of this effort is to examine the different instabilities that can affect a liquid film during gas-liquid flow.

Task Description:
A generalized approach is being undertaken for gas-liquid flow in an open channel with finite length, with or without gravitational stabilization, as well as for the analytically simpler case of liquid-liquid rotating flows. Either gas-liquid low-gravity testing or comparison with existing low gravity data is planned.

Task Significance:
The presence of waves can enhance the heat transfer between phases but increase the energy required to pump the phases through a pipe network. Terrestrial applications include the nuclear, electric power and oil industries.

Progress During FY 1995:
Experimental work in the rotating Couette device has confirmed that the initial instability is super critical because the amplitude-rotation curve is exactly the same with increasing and decreasing rotation rate. The amplitude - rotation rate curve has been calculated from the complete Navier-Stokes equations using a weakly-nonlinear formulation and it agrees with experiments within the expected error. This provides the first experimental confirmation of weakly-nonlinear theory for an interfacial system. The dominant mechanism of stabilization is the transfer of energy from the unstable fundamental to the stable overtone. The mechanism of stabilization is of particular interest because it shows whether the linearly unstable waves will ever stabilize or if they grow until they roll over. In the gas-liquid channel flow experiments, the mechanism has been found to be a cubic-order self-interaction with the overtone interaction being important only in a narrow parameter range. For oil-water channel flow within the range of the experimentation, a surprising result is that the cubic self-interaction is destabilizing and that stabilization is caused by interaction between the fundamental and the mean flow. Experiments cannot confirm the mechanism, but they do show the expected saturation on linear growth at approximately the correct amplitude.

Stability calculations for oil-water channel flow have revealed the presence of two different modes that are simultaneously unstable. This has not been seen in air-liquid flows and it occurs over only a small parameter region. The experiments agree with these calculations and display the presence of a slow-moving, long-wave disturbance and a fast, short-wave disturbance simultaneously. The presence of two different unstable modes is exciting because energy transfer between long and short waves may be important in the generation of slugs in gas-liquid flows and this provides a good system to study this effect. For the flow regime transition criteria of gas-liquid flows, there appear to be conditions where long wavelength waves are unstable but no large disturbances form in both the rotating Couette device and the oil-water channel flow. This issue is critical to resolve because all of the popular models for flow regime transition (e.g. Barnea's, Hanratty's) assume that if long waves are unstable, a transition from stratified to either annular or slug flow occurs. Furthermore, comparisons of these one-dimensional models with the governing differential equations on plots of friction velocity versus liquid depth have shown significant disagreement in the location of the long wave stability curve. Because the one-dimensional models match experiments, it means that either long wave instability is not the correct criterion for say, slug formation, or that the experiments which are used to verify the theory have been done in pipes that are too short.
II. MSAD Program Tasks — Ground-based  Discipline: Fluid Physics

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TASK INITIATION: 12/92  EXPIRATION: 11/95
PROJECT IDENTIFICATION: 962-24-05-55
NASA CONTRACT NO.: NAG3-1398
RESPONSIBLE CENTER: LeRC

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals


Study of Forced Convection Nucleate Boiling in Microgravity

Principal Investigator: Prof. Herman Merte, Jr. University of Michigan
Co-Investigators: Keller, Prof. R.B. University of Michigan

Task Objective:
This task is a continuation of previous experimental work examining the effects of buoyancy on forced convection nucleate boiling. In nucleate pool boiling, bubble detachment in normal Earth gravity is usually the result of buoyant forces; in the absence of buoyancy the bubbles have a tendency to remain in the vicinity of the heating surface, eventually causing dryout at the heating surface which results in reduced heat transfer. For boiling in a flowing liquid, an additional mechanism of bubble detachment can occur: detachment due to lift and drag on the bubble, induced by the flow field. The purpose of this study is to gain an understanding of when bubble detachment will dominate, and the effectiveness of boiling in such circumstances. Also, this study is intended to clarify the relative significance of buoyancy and bulk flow inertia forces to the critical heat flux (CHF) in forced convection boiling, which will also serve to enhance the understanding of the CHF in microgravity, where buoyancy effects are absent. This information will also increase the understanding of boiling in cases where gravity is present.

Task Description:
A closed-loop flow boiling system, using R-113 as the test fluid, has been developed as part of a previous program. The temperature and pressure of the subcooled liquid R-113 are rigorously controlled at the entry to the test section. A portion of one wall of the rectangular test section is electrically heated to provide either a constant temperature or a constant heat flux. Surface temperature and heat flux measurements are made and the bubble growth/departure process is recorded visually. The relative effects of buoyancy are examined by rotating the entire flow loop. Other variables in the experiment are the flow velocity, the power input to the heater, and the amount of liquid subcooling. A long-range goal of the effort is to adapt the current test loop for testing aboard NASA's low gravity research aircraft.

The project has focused upon two general areas of study: obtaining heat transfer coefficients for nucleate boiling over a range of variables, and studying the conditions where dryout (departure from nucleate boiling) occurs. Analysis has been performed in both areas of study to develop physical explanations for the observed results.

Task Significance:
An understanding of the fundamentals of boiling in microgravity is necessary in order to predict the performance and limitations of space systems where boiling occurs, such as those for thermal control and power generation. Near absence of natural convection in microgravity also provides researchers the opportunity to study fundamental aspects of boiling which can improve performance of terrestrial power generation and process equipment.

Progress During FY 1995:
This study is intended to clarify the relative significance of buoyancy and bulk flow inertia forces to the critical heat flux (CHF) in forced convection boiling, which will also serve to enhance the understanding of the CHF in microgravity, where buoyancy effects are absent. Measurements of the CHF in subcooled forced convection boiling of R113 were made on a flat copper heater surface at varying orientations relative to gravity for low flow velocities, where buoyancy is substantial compared to flow forces. An analytical model describing the effects of the flow velocity, heater orientation and subcooling was developed, relying partly on empirical relationships derived from hot wire anemometer measurements of the void fraction and bubble frequency.
II. MSAD Program Tasks — Ground-based

The experiments show that the CHF is a strong function of the heater surface orientation at very low flow velocities, and that orientation has a considerable effect on the CHF for flow velocities below 55 cm/s. In orientations where the buoyancy and flow forces oppose each other, the CHF is generally much lower than in orientations where they act in the same direction, owing to the increase in time during which the bubbles are resident at the heater surface. The analytical model of the CHF considers the motion of larger bubbles on the heater surface, relating the inverse of the bubble residence time to the CHF. The model also includes the effects of subcooling, which act to reduce the net vapor generation rate, thereby deferring the onset of the CHF to higher levels of heat flux.

In a related study, subcooled forced convection nucleate boiling experiments with R113 were conducted with thin gold film and gold coated copper substrate flat heaters. Orientation relative to gravity, flow velocity, liquid subcooling, heat flux, and heater length aspect ratio were varied to provide a better understanding of the relative roles of various heater transfer mechanisms in the total heat flux. This work was intended to better define the boiling process and determine future work for forced convection boiling in the microgravity environment of space.

The experiments and analysis demonstrated that forced convection boiling may be described by a mechanistic approach, under which the total heat flux is divided into several contributing components. A model is presented for forced convection boiling which has an error of less than 25% for most conditions. By varying the heated length, the contributions of several of the heat transfer mechanisms were determined. Through qualitative analysis and the heat transfer model, microlayer evaporation was shown to comprise the bulk of the total heat transfer.

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BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals

Presentations
Control of Oscillatory Thermocapillary Convection in Microgravity

Principal Investigator: Prof. G. P. Neitzel
Georgia Institute of Technology

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
The objective is to experimentally investigate active control of oscillatory thermocapillary convection in planar layers. The investigative strategy is as follows:

1. Establish the appropriate basic state for a "thin" rectangular flow geometry.
2. Establish and detect the oscillatory thermocapillary convection in the form of hydrothermal waves.
3. Suppression and control of the hydrothermal waves.

Task Description:
The basic state will be investigated using available computer programs to help establish the experimental design parameters and boundary conditions. After choosing the relevant design parameters and boundary conditions, the dish which will contain the desired basic state will be constructed. The desired basic state is the return flow basic state of Smith and Davis. LDV will be used to observe and measure the basic state obtained in the experiment. The surface temperature perturbations of the hydrothermal waves will then be characterized either numerically or experimentally. Control or suppression of the hydrothermal waves will be attempted using a CO2 laser as the heat source. Locations along the free surface that appear to most effectively cancel or suppress the hydrothermal waves will be heated with a laser pulse and the duration and time of pulsing will also be compared with the predicted estimates.

Task Significance:
Crystals grown by the float-zone process can be improved by successfully suppressing undesirable fluid motion during the crystal growth process.

Progress During FY 1995:
Recent progress has been made in three key areas:

Laser-Doppler Velocimetry (LDV) measurements have been made to assess the quality of the basic state flow field. Infrared thermography measurements of the fluid layer's free surface near the critical Marangoni number have been taken to determine the true surface-temperature gradient. Most importantly, all the equipment is in place to carry out the control aspects of the project, and preliminary control experiments have been conducted.

The LDV measurements were conducted in both 1 mm and 1.5 mm layers, each at a Marangoni number that corresponds to 75% of the critical value. Velocity profiles taken in the center of the flow apparatus are in excellent agreement with theoretical predictions for an infinite layer. Measurements taken through the liquid layer's free surface from above revealed the nature of the flow near the sidewalls and endwalls of the apparatus. Typically the flow near the apparatus boundaries approached that occurring in the center of the apparatus within 1 layer depth (i.e. 1.0 mm-1.5mm). These findings have allowed us to conclude that the experimental apparatus produces a very "clean" flow, approaching that occurring in an idealized infinite layer.
Determination of the actual temperature gradient occurring on the liquid layer's free surface is important in that it allows a consistent comparison with linear stability results to be made. Infrared thermography measurements to this end have been carried out for different layer depths [0.75 mm-2.5 mm], at the respective critical Marangoni numbers. Infrared images of the free surface were digitized via a framegrabber and converted to graylevels, allowing the determination of the temperature gradient in the fully developed part of the flow away from the endwalls. The temperature gradient determined in this fashion is typically 75% of that previously estimated as the endwall temperature difference divided by the length of the fluid layer.

The control aspects of the project are in a fully functional state, and preliminary experiments have been run. For the control experiments, an infrared CO2 laser beam is focused and spread in one direction to produce a nearly uniform 1 mm thick sheet that is directed downward onto the liquid layer's free surface. The control signal comes from a thermocouple near the free surface which is amplified and passed through a phase delay before being used as input to the laser's controller. Initial experiments have shown that the liquid layer is very receptive in the infrared heating, and that a laser power of a couple of watts can suppress hydrothermal waves. Unfortunately, this much power also significantly distorts the basic flow field. Currently experiments with much less laser power (a few hundred milliwatts) are being conducted, and results should be forthcoming in the immediate future.

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**TASK INITIATION:** 2/93  **EXPIRATION:** 2/96  

**PROJECT IDENTIFICATION:** 962-24-05-61  
**NASA CONTRACT NO.:** NAG3-1454  
**RESPONSIBLE CENTER:** LeRC

**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Presentations**


Industrial Processes

Principal Investigator: Prof. Simon Ostrach
Case Western Reserve University

Co-Investigators:
Kamotani, Prof. Y.
Case Western Reserve University

Task Objective:
The objectives of this study are to gain an understanding of the role of gravity in various industrial processes and to develop new applications of microgravity.

Task Description:
The commercial processes and related basic processes which have been investigated or are being investigated, include supercritical extraction processes, coating flows, formation of bubbles in liquid flow, dynamics of liquid-gas interfaces, transport phenomena in zeolite growth, rotating electrochemical systems, and transport phenomena in crystal growth. The roles of gravity in those processes are being studied using experimental, numerical, and analytical techniques and potential benefits of microgravity are being assessed. Based on those studies microgravity experiments will be proposed.

Task Significance:
The roles of gravity in terrestrial and space-based industrial processes are being studied using experimental, numerical, and analytical techniques, and the potential benefits of microgravity applications of these processes are being assessed. Based on these studies, microgravity experiments will be proposed which will lead to an understanding of how to best utilize the environment of space for various industrial applications.

Progress During FY 1995:
1. Coating Flows
An experiment on dip coating is being conducted to study the conditions for the appearance of wavy films. The experiment covers the capillary number up to 20 and Reynolds number up to 28. When the non-dimensional film thickness exceeds a certain critical value, the film interface becomes wavy. The nature of the interfacial instability is investigated by flow visualization. According to the present experiment a coating film becomes unstable after certain Reynolds number and Stokes number are exceeded. The applicator geometry also influences the film profile and thickness. One conclusion from the present experiment is that gravity significantly influences the stability of the coating film and thus coating in microgravity is advantageous in obtaining uniform films.

2. Bubble Generation in Microgravity
The main idea in this work is to study single bubble generation with transition from the bubbly to the slug flow regime in a continuous liquid flow under microgravity. We are using the Fluid Flow Diagnostics Loop Facility at NASA/Lewis to run experiments under normal gravity conditions. Both co-flow and counter-flow systems have been designed and are now in the manufacturing process. The 1.27 cm I.D. flow conduit section is assembled and certain modification are made in the data acquisition system. After some testings with the 1.27 cm flow conduit system, pipe diameters and injection nozzle diameters will be varied to observe the changes in flow regime transition.

3. Zeolite Growth
We have found a way to grow large zeolite crystals. By adding nutrients during the growth period it is possible to
put the extra nutrients into the existing crystals without increasing the number of crystals, thereby increasing their average size. A patent has been applied for the method. The time to add the nutrients is determined by observing the shrinkage of the gel mixture. The method is considered to be very effective in growing large crystals in microgravity. However, in microgravity the gel does not shrink, but it becomes dilute gradually as the crystal growth proceeds. We found that by putting a He-Ne laser through the gel it is possible to know structural changes in the gel, which makes it possible to identify the time to add the nutrients. We are also studying theoretically the diffusion processes taking place during zeolite growth in microgravity.

4. Rotating Electrochemical Systems

The mass transfer process in shallow rotating electrochemical cells is being investigated numerically, analytically, and experimentally. Various cases relevant to rotating battery applications (Schmidt number about 3,000) have been computed numerically. When the rotational speed exceeds a certain value, secondary cells appear in the core region. We are also performing a scaling analysis to identify new dimensionless parameters and a mass transfer correlation which can classify the entire range of our interest. Based on the numerical analysis we are constructing a new electrochemical test cell. Sectioned electrodes which are specially designed to measure the local mass transfer rate have been fabricated.

5. Natural Convection in Circular Cylinders

Oscillatory natural convection in circular cylinders filled with a liquid metal is being investigated experimentally. The conditions for the onset of oscillations are being investigated under various conditions. The problem is very complex and many factors influence the oscillation phenomenon (e.g. heating rate, wall thickness).

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals


Presentations
Marangoni Effects on the Bubble Dynamics in a Pressure Driven Flow

Principal Investigator: Prof. Chang-Won Park
University of Florida

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
Perform theoretical and experimental research to investigate the effect of surface contamination on the relative motion of air bubbles in a liquid within a Hele-Shaw (HS) cell. Results of this effort will be used to enhance the understanding of the fundamental physical processes and the importance of interfacial flows that occur in a broad variety of industrial and process of applications.

Task Description:
• Focus the theoretical investigation on establishing a predictive model for the influence of surfactant on the bubble dynamics (especially on the translational bubble velocity in a pressure driven flow).

• Experimentally confirm the theoretical predictions and prove that many of the perplexing bubble shapes are the results of the surfactant influence.

Task Significance:
The results of this research will provide an understanding of the bubble motion on diffusional flux of heat and mass at the bubble-liquid interfaces. This has a close relationship to a number of applications in direct contact mass and heat transfer devices, bioengineering (e.g., blood oxygenation), and it has direct implication for the fluid systems in space-based applications.

Progress During FY 1995:
During this period, Professor Park has conducted experiments using water drops containing a predetermined amount of surfactant. The translational velocities of water drops in an oil-filled Hele-Shaw cell were investigated and compared to the prediction of Saffman and Taylor. The effects of surfactant concentration on the shape transition were also investigated. He has also carried out a theoretical analysis for a drop with a circular form to estimate the extent of retardation in the drop velocity which results from the surface concentration gradient.

Professor Park has plans to continue the project and to avoid using water drops because of unavoidable contamination problems. Instead he will conduct experiments with oil drops in an organic phase. He will also conduct a theoretical analysis to extend from a circular plan form to elliptic bubbles to account for shape effects.

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BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals
II. MSAD Program Tasks — Ground-based  

Discipline: Fluid Physics

Presentations

Nonlinear Dynamics and Nucleation Kinetics in Near-Critical Liquids

Principal Investigator: Prof. Alexander Z. Patashinski
Northwestern University

Co-Investigators:
Ratner, M.A.
Northwestern University
Pines, V.
Case Western Reserve University

Task Objective:
The objective of this theoretical research is to develop a more complete understanding of the nonlinear response of a near-critical liquid to strong perturbations of local thermodynamic equilibrium. The consideration is limited to a set of special physical situations relevant to flight experiments dealing with relaxation and nucleation in a near-critical state. It entails the hierarchical relaxation in the macroscopically homogeneous liquid following homogeneous strong, rapid temperature, and pressure perturbations of thermodynamic equilibrium, interaction of such perturbations with inhomogeneities, their influence on nucleation kinetics, and the kinetics of a sub-critical nucleus.

Task Description:
The general theoretical method is the field theory approach to near-critical systems, the scaling theory of critical phenomena, and computer simulations. The physical situations to be theoretically studied are described in the form of idealized experiments. These situations include temperature changes in a near-critical liquid at time scales shorter than the relaxation time of correlated fluctuations. The behavior of a nucleus near the phase separation line in non-stationary temperature conditions will be examined in order to find the way for direct observation of a sub-critical nucleus.

Task Significance:
The study of the response of an ensemble of large length scale fluctuations in near-critical liquid to strong and rapid perturbations of the equilibrium is expected to give an insight into phenomena accompanying such perturbations in experiments very close to the critical point, including already performed flight experiments. The relaxation of a non-equilibrium near-critical system following strong rapid perturbations is expected to yield conditions for achieving a near-critical steady state with a constant heat current and to formulate optimal conditions for a potential flight experiment.

Progress During FY 1995:
The relaxation of an ensemble of long range fluctuations following a sudden strong temperature perturbation from an equilibrium homogeneous state of a near-critical liquid was studied in a phenomenological scaling theory. The predicted phenomena include the time relaxation of the distribution function and specific "tails" in the time dependence of the temperature. The relaxation was found to follow new scaling laws. The response of the material is nonlinear, and the scaling laws are different for different signs of temperature perturbations.

By studying pressure perturbations in non-uniform, non-equilibrium near-critical liquids, we found that a temperature gradient in the initial state may result in a near-critical steady state that has unusual properties. Asymptotically, this non-equilibrium state has a vanishing temperature gradient but a finite heat current. A generalization of the fast adiabatic equilibration method is proposed as a way to archive this new state of matter. A proposal of a potential flight experiment employing this finding is submitted to NASA.

In a second area, the difficulty in observing and studying the fluctuational stage of nucleation, and the interpretation of experimental data, has motivated a computer simulation of nucleation. In order to study the early stages of nucleation, we applied the statistical theory of pattern recognition to visualize the kinetics of the local structure of a computer-generated liquid, following a deep temperature quench. It was shown that the method allows one to see and to analyze the appearance of the face centered cubic (FCC) order at the level of the first coordination shell.
II. MSAD Program Tasks — Ground-based

STUDENTS FUNDED UNDER RESEARCH:

PROJECT IDENTIFICATION: 962-24-05-07
NASA CONTRACT NO.: NAG3-1617
RESPONSIBLE CENTER: LeRC

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals

Patashinski, A.Z., A rapid temperature change near critical points. PRL, (submitted).

Presentations


II. MSAD Program Tasks — Ground-based

Discipline: Fluid Physics

Two-Phase Interfaces in Weak External Fields.

Principal Investigator: Prof. Jerome K. Percus
New York University

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
The aim of this work is to understand from first principals the behavior of two-phase interfaces in the absence of assumptions as to gravitational effects.

Task Description:
1. Analytical Statistical Mechanical formalisms will be employed. Classical Hamiltonians of the interfaces of interacting fluids will be modified or developed as needed.

2. General external fields are included.

3. Equilibrium, followed by non-equilibrium, descriptions are to be employed. It is expected that familiar and new strategies will be utilized.

Task Significance:
The above Objective is fundamental to our ability to deal with the fluid structures that abound in the real biological, chemical, and physical world.

Progress During FY 1995:
Since the state of thermal equilibrium is basic to all effective descriptions of the dynamical processes involved, its study has heavily colored our initial investigations, culminating during this year with the publication of a detailed analysis of the "mean-field hydrodynamical" limit of fluid matter, a low resolution caricature of such systems. This was joined by a more refined treatment in which the inherent interfacial thermal fluctuations, that are crucial e.g., to the timing of coalescence processes, were included. The full set of considerations was then subsumed under the aegis of the heavily studied density functional theory, but in a framework, that of expanded density functionals, in which one is able to focus at will on microscopic, mesoscopic, or macroscopic elements of the structure.

A substantial effort was mounted during this period to determine how familiar hydrodynamical concepts have to be modified and interpreted in order to make them appropriate to the multi-level structure alluded to above. This was primarily in the context of the microscopic symmetric pressure tensor which was, for the first time, expressed in the invaluable density functional format, and then used to follow the predictions of popular microscopic models on the energetics of interfacial systems. In the course of these investigations, the previously murky relation between pressure tensor and thermodynamics was completely clarified.

The process of extending thermodynamic information to interfacial dynamics was initiated along two paths. One was from the viewpoint of an inertialess lattice gas, resulting in the surprising conclusion that at this level, all transport is governed by precisely the thermodynamic free energy, albeit with a non-trivial effective particle mobility. The other aimed at understanding the fashion in which slow macroscopic motions, accounted for by a time-varying microscopic energy, generate effective thermodynamic parameters. By examining a solvable model system, it was found that all current procedures for doing so are deficient, and suitable alleviation is suggested.

We expect to devote the coming year to probing more deeply the manner in which a number of limited dynamical regimes can be analyzed with the help of our extensive equilibrium information, starting with the models of Araki...
and Munakata; Giacomin and Lebowitz; and Oxtoby. It will also be necessary to have more complete control over the complex interfacial equilibrium configurations required for intermediate stages of two phase separation, and we are developing "phase-field" techniques to this end. We hope to arrive at a stage at which the results of physical experiments can provide incisive tests of our theoretical framework.

STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 1/93  EXPIRATION: 12/95
PROJECT IDENTIFICATION: 962-24-05-76
NASA CONTRACT NO.: NAG3-1414
RESPONSIBLE CENTER: LeRC

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals

Presentations
Containerless Capillary Wave Turbulence

Principal Investigator: Dr. Seth J. Puttermann
University of California, Los Angeles

Co-Investigators:
Barmatz, M.B.
Jet Propulsion Laboratory (JPL)

Task Objective:
We are working toward the goal of studying turbulence in a broad-band spectrum of capillary waves that run around the surface of a containerlessly positioned drop of liquid. This experiment would constitute the first controlled measurement of turbulence in interacting waves.

Task Description:
The problem consists of two components. They are (a) the generation of a turbulent distribution of surface ripples and (b) the detection and measurement of this state. These issues are being approached in ground-based experiments as well as in arrangements that simulate containerless fluids in microgravity. The ground-based experiments are being carried out in a fluid which is excited with a shake table. The preflight experiments are being developed with a levitated droplet of liquid.

Task Significance:
Consequences of this experiment range from the characterization of turbulence to the determination of universal properties of nonlinear systems and signal processing. The presence of a new propagating mode (second sound) in the capillary turbulence would have important ramifications with regard to attempts to achieve controlled thermonuclear fusion.

Progress During FY 1995:
We have achieved the first controlled observations of the transition to broad turbulence in nonlinear wave interactions. These experiments studied the motion of ripples propagating on the surface of water. The key to these measurements is our ability to determine the height of the rippled off-equilibrium fluid as a function of location and time. The fluid is set in motion with a vibration exciter ("shake table"). A CCD camera mounted above the liquid and focused on the surface records the intensity of light that leaves the surface. By doping the water with "polyballs" (neutrally buoyant spheres with 1 micrometer radius) the light is forced to diffuse through the water, so that the intensity at the top surface is proportional to its local elevation. This new technique (diffusive light imaging) is our key advance. It circumvents the catastrophic problems posed by caustics in the former shadowgraph imaging technique.

Our technique can be used to measure the power spectrum or the instantaneous surface topography. Thus the first records of the full 3D profile of a localized soliton state has been obtained. Our data shows a power spectrum of surface vibration given approximately by $g^2(\omega) \sim \omega^{-4.6}$ where $\xi(x,y,t)$ is the surface height and "$\omega$" is the frequency. This compares with a theoretical dependence of $\omega^{-17.8}$. Whether we have found a mistake in the theory or if the effects of boundaries (e.g. the side walls of the dish) and a limited dynamic range has led to this discrepancy will be best answered by a flight experiment!

In a space flight the ripples will be excited on the surface of a levitated drop. For this reason the JPL group has been studying the dynamics of large amplitude ripples on a levitated drop of fluid. Excitation of large amplitude waves has been achieved with a "sting" and through modulations of the ultrasonic levitating field. Displacement spectra of the surface oscillations of levitated drops have been obtained using a modified Polytec PI interferometric...
laser vibrometer with a front-end anti-slosh optics developed by NASA Lewis (Meyer/Tin/Taylor/Mann) for UCLA/JPL (Puttermann/Barmatz/Biswa). This front end optics design was also applied to measure displacement power spectrum on a flat liquid surface with amplitudes as high as seven hundred microns (well into the turbulence regime). This new instrument is under development at NASA Lewis (funded by Code UG) and will be assigned to JPL to assist in characterizing surface capillary wave phenomena on a levitated drop.

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TASK INITIATION: 12/91  EXPIRATION: 12/94

PROJECT IDENTIFICATION: 963-24-07-03

NASA CONTRACT NO.: NAGW-2842

RESPONSIBLE CENTER: JPL

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals

Studies of Radiation-Driven and Buoyancy-Driven Fluid Flows and Transport

Principal Investigator: Prof. Paul D. Ronney

University of Southern California

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
The proposed research will consist of two sets of experiments under simulated microgravity condition. The first is a study of instabilities of radiating, initially-homogeneous gas volumes, for both optically-thin and optically-thick gases with a vertical temperature gradient between two plates. The objectives are to determine the validity of theoretical stability criteria and to determine the spectrum of the nonlinear evolution of the resulting destabilizations. The second set of experiments is a study of the stability of one-dimensional steady temperature profiles and unsteady thermal conduction waves in gases with strongly temperature-dependent radiative conductivity. The objective is to determine the stability criterion, study nonlinear evolution's of these instabilities, and develop a stability model.

Task Description:
The following are proposed problem studies:

1. Stability of a cooling optically-thin gas volume (field instability) using NH₃ gas condensible I₂ vapor or other gas with analogous properties, and measurement of the power spectrum of any resulting nonuniformities.

2. Stability of a cooling optically-thick gas volume which is opaque only at high temperatures (CMBR instability) using CO or other gas with analogous properties, and measurement of the power spectrum of any resulting nonuniformities.

3. Radiative conduction in optically-thick gases between parallel plates using SF₆.
   a. Radiatively-induced instabilities of steady planar conduction profiles at μg.
   b. Unsteady planar conduction at constant heat flux (thermal waves) at one-g and μg.
   c. Critical Rayleigh numbers at one-g at large t and ΔT.

4. Radiative conduction in optically-thick gases at μg in spherical geometry.

5. Theoretical stability analysis corresponding to items 3 and 4.

Task Significance:
The study of the coupling of internal radiation to fluid flow is important in a wide variety of practical problems including glass and semiconductor processing; oceanographic, atmospheric and astrophysical flows; plasma physics; combustion systems; solar energy collection; and heat transfer in inhabited enclosures. This work consists of a series of experiments to be carried out in the drop towers that will enhance the understanding of the physics of internal radiation to fluid flow. Results obtained from the present research could identify potential mechanisms to improve the Earth's environment and improve solar power systems.

Progress During FY 1995:
A pressure vessel to contain the experiments has been designed, built and tested. A Rayleigh-Bernard (RB) type of apparatus, i.e., heated and cooled plates, have been designed, built, and tested. A laser shearing interferometer system, suitable for use in drop tests, has been designed, built, tested, and incorporated into the above apparatus. To the PI's knowledge, this is the first interferometer system compatible with NASA-Lewis 2.2 Second Drop Tower.
experiments. The field of view of the interferometer is a circle 5 cm in diameter. The electrical system for powering and controlling the plate temperatures and interferometer system and recording thermocouple and radiometer voltages has been designed, built, tested and integrated with a NASA-supplied Digital Data Acquisition and Control System (DDACS). The above apparatus has been integrated into a NASA-furnished frame designed for use in the 2.2 Second Drop Tower.

Initial one-g tests have been performed using air and SF₆ at a chamber pressure of 1 atm. The purpose of the initial one-g tests was to verify that radiatively-active gases will have different critical Rayleigh numbers (Ra¢) for RB flow than radiatively inert gases, thereby indicating the role of radiative conductivity. As an example of this, it was found that Ra¢ was about 10 times higher for the 1 atm SF₆ mixture than that of radiatively-inert air. A comparison of the flow observed in SF₆ at values of Ra slightly above Ra¢ and at a much higher Ra shows the following. The interferometer fringes are slightly wavy at Ra > Ra¢, whereas they are severely deformed at large Ra. Also, there are closely-spaced fringes adjacent to bottom plate, indicating a thin thermal layer characteristic of radiatively-driven flow. This layer was completely stationary and was not seen in air at any Ra, thus it does not indicate a laminar-like sublayer of a turbulent thermally-induced flow. These quantitative and qualitative features indicate the importance of radiative transfer in hydrodynamic flow and instabilities, and encourage us to proceed to low-gravity tests.

A series of low-gravity experiments were performed in the NASA-Lewis 2.2 Second Drop Tower using the Rayleigh-Benard apparatus developed during the previous quarter and described. A total of 17 drop tests were conducted. The test gases were air and SF₆ at pressures from 0.2 to 2 atm. With SF₆, at the highest pressures (2 atm) and plate gaps (2 cm), a flow was observed at low-g that did not appear to be residual of the flow observed at one-g before the test. (Experiments were conducted with the hot plate on top, so that the gas between the plates is buoyantly stable, but some flow occurs above the hot plate which affects the flow between the plates to some extent.) No corresponding low-g flow was observed in air at any condition. These observations are consistent with the model postulated by the PI concerning radiation-driven flow in radiatively active gases such as SF₆.

The goal of the first drop campaign was to determine in a qualitative way when any radiation-driven flow could be observed. The conclusion of this set of tests was that such flows appear to be observable. Experiments to be conducted in the near future will focus on quantifying the characteristics of these flows and determining the conditions under which they may occur.

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BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Presentations

II. MSAD Program Tasks — Ground-based

Fluid Creep Effects on Near-Wall Solute Transport for Non-Isothermal Ampoules and Suspended Particle Transport Coefficients

Principal Investigator: Prof. Daniel E. Rosner
Yale University

Co-Investigators:
Papadopoulos, D.H.
Yale University

Task Objective:
One goal of this proposal is to initiate theoretical studies necessary to provide the basis for more realistic future ampoule-level numerical simulations for multi-component vapor transport, including supercritical vapors. A second goal is to provide predictions of particle (or macro-molecule) transport properties based on similar phenomena occurring at the particle level.

Task Description:
The following are issues to be addressed in this investigation:

1. Slip coefficients for polyatomic gases, including nondilute, disparate molecular weight mixtures.
2. Simultaneous effects of wall creep due to solute mass-transfer, including the coupling between creep associated with energy and mass transfer.
4. Appropriate "creep" conditions at "porous" solid surfaces; implications for the transport properties of suspended particles, including thermophoretic coefficients.

Task Significance:
These phenomena not only influence transport rates in microgravity crystal growth ampoules, but also the migration rates of suspended macromolecules or nanoparticles (molecular clusters and ultra fine particles).

Progress During FY 1995:
The feasibility of applying a microscopic approach based on the Boltzmann equation for low-density gases to the 'model' problem of thermal creep in a two-dimensional ampoule with strong temperature gradients established along its side-walls was investigated early on. Toward this end, a Direct Simulation Monte Carlo (DSMC) method was selected which was based on the algorithm proposed by G. Bird (1994). For simplicity, the geometry chosen is a cartesian one. The code for this purpose has been developed and thoroughly tested and validated. Statistical error has been shown to decrease with approximately the inverse first power of the sample size, as expected from the relevant kinetic theory literature. Simulations run on a Sparc 10 workstation included sample sizes up to $4 \times 10^5$ molecules.

The correlation of number density and velocity components for the test case of a dilute gas in equilibrium has been examined. It has been confirmed that the number distribution of particles within each cell follows a Poisson distribution, which essentially agrees with theoretical considerations. As a further consistency check of the code, results obtained in the non-equilibrium case agree with those found in recent computational kinetic theory literature. It was found that the sampling frequency (i.e. how often one collects data to be statistically averaged) is a crucial factor in such simulations. If correlations between successive statistical samples exist, the moments of the distribution function may be significantly affected yielding misleading results.

Upon validating methods, efforts were focused on developing a 2-D cartesian code that could handle confined geometries of interest and validating it using relevant case studies. In these simulations, a strongly non-isothermal ampoule charged with Xenon was considered. Xenon is a typical background gas used in metalorganic physical
vapor transport crystal growth applications at reduced pressures. The presence of the end walls is expected to force the creep-driven gas into a circulatory motion and thus result in the formation of a vortex rotating in the counterclockwise sense. This vortical motion has been successfully captured in microscopic level simulations.

In subsequent studies, several computational issues will be examined which arise in DSMC simulations and determinations of objective criteria to optimize the relevant simulation parameters will be made. It is hoped that eventually the microscopic approach could provide results which will guide the implementation of a computationally efficient, yet realistic, macroscopic description of the subject flows, and to economically anticipate the consequences of these phenomena in crystal growth related ampoule experiments. Finally, it is also planned to initiate an investigation of the analogous phenomenon of concentration creep using the existing well tested DSMC microscopic methods. Such creep phenomena take place when disparate molecular weight gases isothermally diffuse in the presence of concentration gradients parallel to a bounding wall.

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### TASK INITIATION: 6/94  EXPIRATION: 5/96

### PROJECT IDENTIFICATION: 962-24-05-94

### NASA CONTRACT NO.: NAG3-1654

### RESPONSIBLE CENTER: LeRC

### BIBLIOGRAPHIC CITATIONS FOR FY 1995:

**Journals**

Gas Flow from Porous Media and Microgravity Battery Spills

Principal Investigator: Dr. Robert T. Ruggeri

Boeing Company

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
This proposal is based on the hypothesis that on-orbit spacecraft battery failures can be traced to electrolyte spills resulting from gas expanding inside the capillary pores of battery electrodes. The specific objectives of this project are as follows:

1. To determine the gas capacity of three porous metals as a function of pore volume and pore diameter.
2. To determine the system temperature and pressure effect on gas capacity.
3. To determine the effect due to the external geometry of the porous material.
4. Analytically determine the effect of gravity on electrolyte spills.

Task Description:
The objectives will be accomplished by one-g experiments and analysis. The PI will identify all critical parameters that determine a porous metal's gas capacity, then demonstrate, by measurement, the effective volume of noncondensable gas contained within the pore structure of porous metal plates as a function of the identified critical parameters. Such critical parameters are expected to be pore diameter, pore volume, sample shape, temperature, pressure and surface tension. The pore volume and the effective gas capacity of three metals will be determined in volumetric gas flow experiments. The experiments will be conducted at 25°, 45°, and 60 °C in a constant temperature chamber. Zinc, silver, and nickel are the three metals that will be investigated. The effect of pressure cycles will span a range between 14.5 psia and 2.9 psia for each temperature. One to three pressure cycles will be performed for each metal specimen at each temperature. Numerical models will be developed to study the gas capacity as a function of the pore diameter, the temperature, and the ambient pressure. The effect of gravity on these experiments will be determined analytically.

Task Significance:
Spacecraft batteries have been documented to discharge electrolyte, which causes a short circuit. The electrolyte discharging mechanism is not yet fully understood. In order to prevent mission failures in the future, critical parameters which determine a porous metal's ability to hold and discharge gas should be investigated. This will lead to design of batteries that can retain electrolyte in the microgravity environment of space and thus prevent battery failure in space missions.

Progress During FY 1995:
The following have been accomplished to date:

1. The contract with Boeing Company was officially started on the first of October. A kick-off meeting was held at LeRC on November 8, 1994. Project tasks were reviewed and the approach was discussed.
2. The hardware for the ground-based 1-g experiment were acquired. Setup and checkout will take place in the next few months.
II. MSAD Program Tasks — Ground-based

Discipline: Fluid Physics

However, progress has been hindered due to the reluctance of the battery manufacturers, citing proprietary concerns, to render metal specimens for conducting the planned experiments.

Students Funded Under Research:

Task Initiation: 10/94  Expiration: 10/96
Project Identification: 962-24-05-95
NASA Contract No.: NAS3-27258
Responsible Center: LeRC
Ground Based Studies of Thermocapillary Flows in Levitated Drop

Principal Investigator: Prof. Satwindar S. Sadhal
University of Southern California

Co-Investigators:
Trinh, E.H.
Jet Propulsion Laboratory (JPL)

Task Objective:
For the measurement of the thermophysical properties of undercooled liquids, the idea of spot-heating a test sample in a levitated state is to be explored and applied to relevant materials. That is, a liquid drop levitated in an acoustic field could be heated on a small fraction of its area by a laser beam. In addition, simple filament heating will also carried out, since this can be achieved with considerable ease. The physical interference of the filament with the fluid mechanics will of course have to be taken into consideration. By carrying out the measurements of the thermocapillary effects of such heating it is possible to derive the thermal properties of the sample. However, this can only be done with the development of a successful predictive model of the system. The effort will therefore consist of both experimental and analytical work.

1.1 Analytical Part
The purpose of the analytical part of the proposed program is to develop such a model over several phases. The major thrust at present is in ground based studies with plans for a future space experiment. In the current studies therefore, the experimentation will involve significant interference of the acoustic field. Thus, for most cases for ground based studies, the drops will be deformed to a spheroidal shape. In addition, there is general asymmetry of the flow field. While it is acknowledged that many of these complexities do not arise in low gravity, there is a great deal that can be achieved by ground based studies provided the interference by the acoustic field is fully accounted for in the analysis. For model development in the direction of a zero-g space experiment, analysis will be carried out for liquid shells and compound drops.

Under the scope of the current investigation, the analytical work will consist of several tasks that will encompass the formulation of the differential equations pertaining to levitated drops, their analytical and numerical solutions and the development of results.

1.2 Experimental Part
The experimental problem of interest in this proposal is the thermal response of a spot-heated levitated drop in a convective gas flow of varying intensity. The ultimate objective of the tasks proposed is to quantitatively determine the transient and steady-state temperature distribution on the drop surface as a function of time, sample physical properties, geometry, and of the input radiant energy. Because of the coupling of thermocapillary and thermoacoustic phenomena, the interpretation of the resulting thermal state must be carried out in conjunction with the theoretical analysis of the problem. The experimental work will thus be divided into several sub-tasks, each of which must provide data that can be directly correlated with theoretical predictions. Although the final goal will be to carry out an experiment in microgravity, this proposal will limit itself to ground-based investigations using proven experimental techniques in order to correlate with and to verify the theoretical work, as well as to develop experimental methods for a potential future microgravity investigation.

Task Description:
In the usual Earth-based environment, the convective contribution arises due to buoyancy as well as to the effects of the levitation mechanism. In this particular case, the sample may be levitated in a gaseous environment by a high intensity ultrasonic field, and the convective flow field external to the specimen is caused by acoustically-driven streaming flows in addition to the normal buoyancy-driven circulation. The heat transfer problem of determining the transient and steady-state temperature distribution at the surface of the sample will also require the solution of the flow field inside the drop driven by thermocapillary effects (and perhaps also by acoustic radiation stresses).
because of the surface tension gradient introduced at the drop surface by the localized heating. Under other circumstances, the sample may be levitated in a vacuum or gaseous environment by electrostatic forces which do not generate detectable outer convective flows or droplet distortion. This approach requires, however, the permanent non-uniform charging of the drop surface; the effects of which are still unknown, but might also alter the thermally-driven capillary flows.

Under these conditions, the relevant non-dimensional parameters will thus include the Reynolds numbers of the internal thermocapillary-driven flow, of the steady outer acoustic streaming flow, and perhaps of the high frequency acoustic particle motion. The Bond number will be of relevance in order to distinguish between low gravity and Earth-based conditions. The Nusselt, Grashof and Marangoni numbers will also play a primary role. Because we shall be restricted to rather moderate temperature and to conditions far enough from the boiling point of the liquids investigated, mass transfer processes will not be taken into consideration in this case.

**Task Significance:**

The proposed research will provide fundamental understanding of the Marangoni flows associated with localized heating of drops and bubbles. For ground based studies where there is interference from the acoustic field, a sound numerical model will provide significant new information about the behavior of these complex systems. The new work on compound drops will play a fundamental role for a zero-g space experiment. Most importantly, the model development along with the experimental studies will represent fundamental groundwork for the measurement of thermophysical properties of undercooled liquids.

**Progress During FY 1995:**

2. **Analytical Part**

2.1 Spherical Particle in an Acoustic Field

The flow induced by a solid sphere oscillating in a viscous fluid when the amplitude of the oscillation is small compared with the radius of the sphere was considered by Lee & Wang [1]. They considered a sphere slightly displaced from the antinode of a standing wave. This analysis relied on the tangential velocity calculation based on an analytical algorithm. We have conducted a more detailed analysis of such a system based on Riley’s [2] method. For the flow surrounding the sphere, an expansion for small \( k a \) of the standing wave velocity was used. For a sphere displaced from the antinode, the leading order reduces to a particle in a spatially uniform flow. This was given by Riley [2]. The analysis has been extended to include the next order, which is linear in \( z \). Detailed calculations with the singular perturbation procedure have given expressions for the outer and inner field. Here, we consider the case of high frequency which means that the vorticity generated at the surface of the sphere is confined to a thin 'shear-wave' layer.

The outer and inner solutions are quite different from those obtained by Lee & Wang [1]. In particular, we notice that the inner field includes fourth order terms in Legendre polynomial derivatives. While attempts are being made to explain the discrepancies, it is likely that the conditions for the applicability of the algorithm in [1] for tangential velocity calculation may be limited. This is presently being investigated.

2.2 Internal Circulation In A Drop In An Acoustic Field

The internal flow in a drop at the antinode of a standing wave has been investigated. This was done through the application of the stress continuity condition at the liquid-gas interface. To the leading order of calculation, the internal flow field was found. It is observed that the strength of this flow exhibits an exponentially negative behavior with the frequency parameter, \( M \). For large \( M \) this is very weak. The reason for such a weak flow is mainly because of the recirculating Stokes layer. This layer is very thin and since it has opposing velocities within it, a very large shear stress is required to sustain its motion. The system cannot afford a large stress at the interface and the result is a weak internal circulation. The large drop viscosity as compared with the gas also has a role in weakening this circulation.
2.3 Relaxation of Ellipsoidal Drops

The relaxation of initially extended (ellipsoidal) drops is being examined numerically by the boundary integral method for the purpose of viscosity measurement. This work is being conducted in collaboration with Howard Stone (Harvard University). For different ratios of the drop viscosity to that of the surrounding medium has been studied for initial aspect ratios of 1.5, 2.0, 2.5 and 3.0. For Stokes flow, the evolution of the drop shape has been studied. Typically, the surface-tension forces act to restore the sphericity of the drop. The shape-time description of the relaxation process is being used to calculate the viscosity of the drop.

3. Experimental Part

3.1 Internal Circulation in Ultrasonically Levitated Drops

The internal flow within acoustically levitated droplets in 1 G has been determined to be negligible in the isothermal case and for non-rotating samples. Internal flow fields have been measured using fluorescent tracer particles and Argon-ion laser sheet illumination. For a differentially heated drop with a maximum temperature difference of about 6°C, an uncontrolled droplet rotation has prevented accurate measurement of thermocapillary flows in 1 G. Coupling between the non-isothermal environment and the ultrasonic field generates a random torque on the levitated sample.

3.2 Internal Flow Field Visualization for Electrostatically Levitated Charged Drops

Laser-heated electrostatically levitated charged droplets have been shown to be more stable under non-isothermal conditions. The internal flow fields are being currently measured using fluorescent tracer particles and ray tracing techniques for correcting the optical distortion due to the curved droplet surface.

3.3 Development of a Glovebox Flight Experiment

A breadboard and experimental procedure are being developed for potential microgravity investigations using the Middeck or Spacelab Glovebox during a future Space Shuttle mission. A scaled down version of an ultrasonic levitator coupled to a solid-state laser sheet illuminator will be implemented in the study of the stability and internal flows of droplet stabilized in microgravity in anticipation of a full scale flight experiment.

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**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Proceedings**


**Books**
Effects of Gravity and Shear on the Dynamics and Stability of Particulate and Multiphase Flows

Principal Investigator: Prof. Ashok S. Sangani
Syracuse University

Co-Investigators:
Koch, D.L. Cornell University
Louge, M. Cornell University

Task Objective:
To understand the particulate and multiphase flow behaviors and dynamics that will occur in the microgravity and Earth's gravity environments, and to investigate systems in which the inertial effects are important on the length scale of particles and bubbles.

Task Description:
Complementary theoretical, simulation and experimental approaches to achieve the aforementioned task objective. The study will consider two types of inertial suspensions that are amenable to detailed theoretical studies. The first type is a solid-gas suspension, in which the inertia of the particle and the gas viscosity are more important than the gas phase inertia. The second type is a suspension of bubbles with high Reynolds number indicating invicid flow, but with small Weber number indicating that their deformation is small. Theoretical development of sheared particulate and bubbly liquids will make use of concepts borrowed from the kinetic theory which has been successfully applied to granular flows. The kinetic theory will be complemented by a numerical simulation of the solid-gas and gas-liquid suspensions. These simulations include detailed calculation of the hydrodynamic interactions among the particles in both microgravity and Earth gravity conditions. To validate the equation of motion of the suspension systems, experimental measurements of suspension properties in simple basic flow situations are necessary. The initial Earth-based experiment will involve a sheared fluidized bed by using capacitance probes to measure the volume fraction as a function of time, the shear rate required to overcome the gravitational instability. The knowledge gained from the theoretical, simulation, and experimental work will set the necessary background to propose low gravity experiments.

Task Significance:
Shearing fluidized bed and bubbly suspensions are used in chemical processing and understanding the dynamics and flow behavior in these systems is crucial.

Progress During FY 1995:
The purpose of this project is to study, in detail, two suspensions in which inertia plays an important role at the lengthscale of particles and bubbles. The first is a gas-solid suspension at small Reynolds numbers and finite Stokes numbers, and the second are bubbly suspensions at large Reynolds numbers. With the first, the inertia of the particle phase is important, but with the second, the inertia of the continuous phase is important. Gravity has a destabilizing influence on both these suspensions and it may be possible to stabilize these suspensions by shearing them. The object of our investigation is to demonstrate this through a combination of theory, numerical simulations, and experiments.

To date we have completed an analytical investigation of sheared gas-solid suspensions in the absence of gravity. We developed kinetic theories to predict the particle-phase velocity fluctuations and stress, to cover a wide range of values of Stokes numbers, volume fraction of particles, and coefficient of restitution of particles. These theories are compared against the results of numerical simulations of gas-solid suspensions and an excellent agreement is found between the two.
We have completed dynamic simulations of shear flows of bubbly suspensions and developed a kinetic theory to explain the results of simulations. A manuscript based on this study is currently under preparation.

We are also constructing a Couette cell that will allow us to study the effects of gravity and shear on gas-solid suspensions.

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**TASK INITIATION:** 5/94  **EXPIRATION:** 5/96

**PROJECT IDENTIFICATION:** 962-24-05-96  **NASA CONTRACT NO.:** NAG3-1630  **RESPONSIBLE CENTER:** LeRC

**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Journals**


Dielectric and Electrohydrodynamic Properties of Suspensions

Principal Investigator: Dr. Dudley A. Saville
Princeton University

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
This investigation focuses on understanding those electrokinetic properties of particulate suspensions related to the so-called "electrohydrodynamic effect," specifically, the dielectric constant and electrical conductivity. These properties are of crucial importance in defining the behavior of samples in various electrokinetic separation processes. The work involves two research tasks:

1. Measurements of the electrokinetic properties of a series of suspensions with particle volume fractions between 1% and 20% by volume.

2. Development of a theory for the dielectric constant and conductivity of suspensions which encompasses the measured behavior.

Task Description:
Experimental work: We have been attempting to prepare model particles which conform to the classical theory. Previous research showed that with some particles annealing at 120°C (above the glass transition temperature) smoothed the particle surface so suspensions behaved according to the classical theory. Attempts to prepare such particles using both anionic and amphoteric latexes purchased from the Interfacial Dynamics Corporation met with limited success. The reasons for this behavior are not at all clear. Accordingly, we have been working to adsorb polymer on latex particles to increase their dipole moment. Suspensions prepared with these particles will have large dielectric constants which can be controlled by the amount and size of the adsorbed polymer.

Theoretical work: We continue to work on theoretical models of the electrohydrodynamic effect and on models of the electrokinetic behavior of dispersions. The purpose of these efforts is to provide the requisite theory to interpret our results.

Task Significance:
This research is intended to develop an understanding of one of the major obstacles to effective separation of particles by electrokinetic methods. During the past year we uncovered ways of adapting our techniques to understanding the behavior of particles in non-aqueous systems. Thus, in addition to its use in separations, our work should find applications in ceramic processing.

Progress During FY 1995:
During the past year we have focused on three activities: measuring the properties of colloidal particles with adsorbed polymer, measuring the properties of concentrated, and using electrohydrodynamic phenomena to manipulate suspensions. As the list of presentations demonstrates, these objectives have been pursued vigorously.

The work on physisorbed polyethylene oxide is almost complete and the student is writing her Ph.D. thesis.

We are currently developing an improved spectrometer to measure the properties of concentrated dispersions.

New ways of manipulating dispersions using electrohydrodynamic effects have been developed. As noted in the papers cited, we have been able to manipulate dispersions so as to form interesting structures in oils and aqueous
dispersions. In the latter case, we discovered how to form colloidal monolayers electrohydrodynamically. A paper on this topic was submitted to Science.

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**TASK INITIATION:** 6/91  **EXPIRATION:** 6/95

**PROJECT IDENTIFICATION:** 962-24-08-08

**NASA CONTRACT NO.:** NAG8-878

**RESPONSIBLE CENTER:** MSFC

**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Journals**


**Presentations**


Electrohydrodynamic Pool Boiling in Reduced Gravity

Principal Investigator: Prof. Benjamin D. Shaw  
University of California, Davis

Co-Investigators:
No Co-Is Assigned to this Task

Task Objective:
To investigate the effects of electric fields on reduced-gravity pool boiling. The electric fields are expected to significantly and controllably increase reduced-gravity nucleate boiling rates and maximum heat fluxes. The presence of an electric field will result in the production of smaller bubbles in reduced gravity and the average speed will be increased.

Task Description:
A drop apparatus will be constructed for use at the NASA Lewis 2.2 Second Drop Tower. This apparatus will consist of a pool boiling test chamber and associated instrumentation mounted on a NASA drop frame. Boiling will occur on an electrically-heated platinum wire subjected to a nonuniform external DC electric field. Boiling experiments with R-113 (trichlorotrifluoroethane, (CCl$_3$FCIF), for which $T_{>}>T_0$ or water (for which $T_{<}<T_0$) will be performed in both 1-g and $\mu$g. Data will be gathered on applied electric fields and wire heat fluxes and temperatures. High-speed motion picture photography will provide visual records of boiling phenomena.

Task Significance:
Anyone who has ever boiled water on a stove is familiar with nucleate pool boiling. Even though it is an everyday event, scientists do not understand precisely how it works, because the Earth’s gravity influences how bubbles form and grow in boiling liquids.

NASA is interested in the results from this experiment, because boiling liquids generate bubbles which are very efficient at transferring large amounts of heat. Finding new ways to dissipate heat from the space shuttle or future manned space platforms will be vital to the success of long-term missions.

The potential benefits closer to home, including more effective air conditioning and refrigeration systems, and improvements in power plants that could reduce the cost of generating electricity.

Progress During FY 1995:
The initial efforts of the past year focused upon designing an experiment to study the effects of applied electric fields on reduced-gravity pool boiling occurring with thin platinum wires heated in a host fluid. Calculations were performed to identify the experimental conditions (e.g., wire diameters, host fluids, etc.) that would be most likely to yield the best experimental data. It was decided that three fluids would be used: water, R-113, and the chlorofluoromethane refrigerant (and environmentally friendly) FC-72. A prototype boiling chamber was subsequently built and tested with water. This chamber helped us to determine the best way to support the wire and allowed us to test critical electronic components (e.g., a programmable microcontroller and programmable power supply) needed for the boiling experiments. This chamber also allowed for the evaluation of schemes to support the platinum wire. In addition, different lighting schemes for the experiments were evaluated with this prototype chamber, and a suitable lighting scheme was selected. All of the components and materials needed to construct the apparatus have now been designed and/or acquired (e.g., high-voltage power supplies, the platinum wire power supply, batteries, battery boxes, the drop frame, the boiling chamber, control electronics, boiling chamber insulation, boiling chamber heaters (cartridge and tape), a special flexible bladder to allow for volume expansion without significantly raising the chamber pressure, etc.). Construction of the apparatus is now well underway and it is expected that normal gravity experiments will be initiated within the near future. Reduced-gravity experiments (50 to 100 drops are planned) will be performed at the NASA Lewis Research Center, commencing in the fall of 1995.
II. MSAD Program Tasks — Ground-based

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PROJECT IDENTIFICATION: 962-24-05-97
RESPONSIBLE CENTER: LeRC

Printed: 28 FEB 1996
II. MSAD Program Tasks — Ground-based

Discipline: Fluid Physics

Transport Processes Research

Principal Investigator: Dr. Bhim S. Singh
NASA Lewis Research Center (LeRC)

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
The objective of this task is to promote, foster, and enhance the quality and breadth of microgravity research conducted in the discipline of fluid physics by advancing the understanding of thermal and mass transport processes when buoyancy-driven convection is reduced or eliminated.

Task Description:
The approach to achieving the task objective is to provide LeRC in-house support to assist sponsored principal investigators in the conduct of their research (particularly when that research can benefit from unique expertise or facilities at LeRC), while guiding and assisting in the definition of flight experiments.

Task Significance:
This task will assist in program planning and outreach programs in the external community, and it will conduct in-house research to advance the understanding of transport and interfacial phenomena through exploitation of the microgravity environment.

Progress During FY 1995:
Transport Process Research:

Support to Ground-Based Principal Investigators:
Support was provided for the first ever study of the absolute instability of liquid jet in microgravity using Lewis' 2.2 Second Drop Tower. Professor Lin (Clarkson University) and his students conducted approximately 40 drops using their specially designed rig. Professor Lin reported "the most stunning results are probably the photographic evidence of absolute instability which has not been recorded before". Lewis support included providing drop rig frame, consultations on design of experiment for drop tests, providing film camera and technical assistance, scheduling the drops, assistance in modifying the test set up to make it fully compatible with drop tower, and assistance in conducting the tests. Also provided support to Professor Ronney (University of Southern California) on studies of radiation-driven and buoyancy-driven fluid flow and heat transport. Professor Ronney's student conducted tests in the 2.2 Second Drop Tower. Assistance was also provided to several PIs planning to use DC-9.

Marangoni Benard Instability (MBI):
Experimental investigation on Marangoni Benard Instability (MBI) in a liquid sheet with 2 free surfaces was conducted in FY95. A 10 cSt silicone oil layer, sandwiched by an air gap from above and a thin layer of FC-43 from below, was used for the study. The onset of MBI was observed, the pattern appears to be irregular polygons. A qualitative observation of the post-onset evolution of the convection pattern indicates that the cell size increases with increasing temperature gradient across the liquid layer. Due to the lack of interfacial tension data for silicone oil-FC 75, a quantitative comparison with the theoretical results previously obtained by Duh, et. al. is not yet available. Further efforts will be devoted in FY96 to quantitatively compare the experimental data and the theories.

Surface temperature profile in Benard convection is being measured using an Inframetrics-760 infrared imager. A new apparatus was designed and built to allow better control of the cooling from top with computer-controlled random mixing of Helium gas. The use of Helium gas and an optical window made of ZnSe also minimize the
II. MSAD Program Tasks — Ground-based

Discipline: Fluid Physics

attenuation of the IR signal while maintaining good temperature control in the convection cell. Interesting mode switching was observed as a function of aspect ratio, Marangoni number, and Rayleigh number, as predicted by theoretical calculations by Narayanan et al. At the concurrent mode, pattern oscillation was also experimentally observed. This work was conducted by Duane Johnson (University of Florida), a GSRP fellow with NASA Lewis mentors Ray Skarda, and J.C. Duh.

A numerical effort to directly simulate the onset and post-onset evolution of Marangoni Benard convection started in the past year. A three-dimensional numerical code using the least-squares, finite element method (LSFEM) is currently being developed. This code will be capable of solving the unsteady, three-dimensional Navier Stokes equations, and is able to track the motion of the free surface of a liquid. We intend to use this code to explore the two different modes of onset, i.e., the pearson mode and the longwave instability mode, and to study the post-onset evolution of the convection pattern.

Thermocapillary Migration of Drops and Bubbles:

Significant progress has been made by Dr. Balasubramanian on the analysis of thermocapillary migration of a drop when convection of energy is predominant inside it. The temperature field inside and outside the drop have been determined by matched asymptotic expansions. Numerical solution of an integral equation to determine the temperature distribution inside the drop near the stagnation point and the drop migration velocity is underway.

The effect of convective transport on the temperature distribution around a slowly settling sphere in a stratified fluid is being pursued. An analysis by Merritt (1987) shows unusual logarithmic singularity in the wake region of the sphere. It has been shown that there is another solution of the problem that does not have a wake singularity.

Optical techniques for measurement of temperature distribution in a transparent liquid for use in space flight experiments have been evaluated. During IML-2 experiments, Point Diffraction Interferometry (PDI) did not perform well. Drs. Rashidnia and Balasubramaniam investigated the use of Wollaston Prism Interferometry (WPI) for this application. The results showed that with properly selected prism angle this technique can provide temperature gradient measurement for most of the range of interest for Professor Subramanian's flight experiment. Working with ESA and appropriate personnel, WPI was successfully implemented in BDPU facility.

Development of Liquid Crystal Point Diffraction Interferometry (LCPDI) was undertaken and sufficient progress was made. LCPDI was used in a Benard convection cell with 10CS silicone oil. The results of LCPDI were compared with Mach Zender Interferometry (MZI). The LCPDI proved to be much more stable and insensitive to external vibration than MZI.

Two-Phase Flow in Microgravity Environment:

Work on arranging the experiments needed to study reduced-gravity two phase flow at a pipe junction is proceeding. A series of normal-gravity tests were conducted to identify the potential problems which may arise in conducting these experiments. The instrumented test sections had been fabricated and installed in the existing Lear Jet two phase flow rig. Modifications needed to conduct the proposed experiments using that test rig are being done now. The experiments will be conducted in early FY96. The setting up of the two-phase flow diagnostics flow loop has been completed. Dr. Jayawardena (NRC) is conducting this research.

A drop tower rig for conducting two-phase gas flow tests is being currently developed to support external PIs. The rig provides liquid and gas flow capabilities and imaging as well as data acquisition and control system. A variety of test sections can be incorporated. The initial tests will be conducted using a curved circular tube.

Development of Droplet Injector:

A droplet injector capable of injecting a droplet with a minimal droplet momentum has been designed. This concept was developed by Dr. Chai. Normal-gravity tests have been completed and the device is ready for reduced-gravity testing. This type of device could have applications for several ground-based and flight investigations.
II. MSAD Program Tasks — Ground-based

Capillary Driven Flows:
Four closed form solutions concerning capillary driven flows in containers with interior corners were determined mathematically, all of which may be directly applied to the design of inspace fluid systems. Such solutions could not be determined in the past because the key to closure of the governing differential equations came from experimental observations of the flow phenomena during concurrent drop tower tests. Over 100 drops were conducted and the large data set is being compared with the quantitative theory. Two Baldwin-Wallace student interns have assisted in the collection of the data throughout the year.

An experimental study of a novel technique producing capillary driven flows which are steady was completed by M. Weislogel. The test configuration, adapted to the low-g environment, may prove ideal as a means for studying the physical phenomena associated with the moving contact line. A large data set was compiled with both drop tower and laboratory experiments which show good agreement with an over-simplified theory developed as a presentation tool for the data. The work is being prepared for publication.

A small effort was initiated by M. Weislogel and K.C. Hsieh to determine the stability of capillary surfaces in irregularly shaped containers. This study is of interest because regions of stability are probed where the surfaces exhibit discontinuous behaviors yet to be observed experimentally. Though related experimental work has been undertaken in certain flight experiments (DYLCO on IMI-2 and ICE on USML-2) the subject analysis sheds light on results which, in some cases, are indeed difficult to explain. Two high school summer shadowing students (one with SHARP) have assisted in the computer runs.

A visiting graduate student, Gerrit Woelk, from Bremen University in Germany, was mentored for four months on a problem related to surface reorientation and settling in cylinders in low gravity. The study was numerical and hinged on an analytic/empirical model for the contact line boundary condition which could be further honed by comparison to a large data set previously acquired at NASA LeRC. A functional form for the boundary condition is suggested making quantitative analysis of such system possible. The results will be included in Mr. Woelk's thesis and a joint paper is in preparation.

Others:
Gravity modulate Benard problem is under investigation using spectral methods. The case of insulated upper and lower surfaces exhibits similar behavior in (g1, Ω) space as previously published results for conductive surfaces. Multiple finger shaped regions of instability were observed in (g1, 1/W) space. For insulated surfaces, the fingers were stretched and their distorted appearance compared to the results for conductive surfaces.

On measurements of surface deformations due to thermocapillary convection several objectives were achieved. Due to the delays in fabrication of test section cells and acquisitions of a few key pieces of equipment much of the research was not able to be performed. Fortunately, by using another facility, some of the preliminary results were taken. These preliminary test runs showed quite promising results qualitatively that with the future results will be very promising. Although, measuring deformations for smaller test cell dimensions will be very tricky, it seems that the results will be accurate. Preliminary results with larger test sections showed rather good matchings with the results obtained from the rochon measuring technique which is another favorable trend. This work was performed by Dr. J. Lee NRC fellow for whom Dr. A. Chai served as research advisor.

Dr. Nengli Zhang (NRC fellow) setup temperature profiling apparatus for thin liquid layers. He refined temperature measurement accuracy to 0.01 °C and prepared for the investigation of Benard instability involving evaporation in liquid layers. Dr. A. Chai is the research adviser for Dr. Zhang.

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Printed: 28 FEB 1996

II-525
BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals

Presentations


Solute Nucleation and Growth in Supercritical Fluid Mixtures

Principal Investigator: Dr. Gregory T. Smedley
California Institute of Technology

Co-Investigators:
Wilemski, G. Lawrence Livermore National Laboratory

Task Objective:
We plan to study the nucleation and growth of naphthalene in supercritical carbon dioxide. This system is chosen due to the accessibility of relevant experimental conditions in the laboratory, and the importance of CO₂ in many technological applications. The strong dependence of solubility on pressure in supercritical fluids enables us to decouple the nucleation and growth processes so they may be studied independently.

Task Description:
1. Define experimental parameters for supercritical expansion processes. These include thermodynamic process paths, equilibrium solubilities, particle size and growth rate estimates.
2. Determine solid particle nucleation and growth rates from Mie scattering and extinction measurements.
3. Analyze experimental data, including phenomenological analytical modeling as necessary.
4. Develop and recommend a flight experiment design based on the results of the ground-based experiment.

Task Significance:
The experiments will employ rapid, but controlled, changes of the supercritical fluid pressure to vary the thermodynamic state of the fluid from a stable condition to one favoring solute nucleation and then to one fostering only solute particle growth. By using optical methods to measure the number of particles formed and their size, we can determine nucleation rates and growth rates at various thermodynamic conditions. Since the rates of nucleation of solid particles have never before been measured under these conditions, the data to be obtained will be invaluable for testing and improving nucleation theory as well as for guiding the design of a space-based experiment. One of the most important goals of the ground-based experiment will be to evaluate the effects of gravity on the measured nucleation rates. Because the density of near- and supercritical fluids varies rapidly with pressure, gravity may induce nonuniform conditions in the experimental fluid mixtures. This would make accurate ground-based measurements difficult if not impossible.

The results of this experimental/analytical work can be applied to the understanding of nucleation and growth problems found in many fields such as biotechnology (protein crystal growth), material science (sintered alloys), and analytical chemistry (supercritical fluid extraction and chromatography).

Progress During FY 1995:
The focus of the first year of this project has been to define the thermodynamic process paths likely to be of greatest interest to the scientific objectives and design an apparatus and suitable instrumentation to carry them out and measure the results. Within these tasks lies the need to trade off instrumentation sensitivity, apparatus effectiveness, and process path optimization to arrive at an acceptable compromise. The operating temperatures and pressures have been defined, and has led to a conceptual design for the apparatus. The operating process for the experiment has been conceptually set forth. A source of thorough information on the equation of state for pure CO₂ has been identified and has been used to define thermodynamic paths. These calculations have indicated that the rapid volume expansions necessary to span the supercritical operating envelope are significantly smaller than initially expected. Smaller expansions directly translate to reduced stroke and therefore reduced average velocity for the expansion piston motion; a useful design input. As a result, a high-speed servo controlled hydraulic actuator is being considered as a replacement to the pneumatic drive piston and adjustable stops. A computer and data acquisition
software have been obtained. The use of fiber optics for the scattering measurements is being considered. Pressure and temperature sensors and zero dead volume gas/optical access port concepts are being evaluated. The experimental portion of this project will be conducted at Caltech, yielding access to instruments used in aerosol science that allow classification of aerosol particles in Differential Mobility Analyzers (DMA) and measurement of number concentration using Condensation Nucleus Counters (CNC). This allows for the opportunity to calibrate the optical diagnostics used to measure number concentration and size in this research program.

Students Funded Under Research:  
Task Initiation: 8/94  Expiration: 8/97  
Project Identification: 962-24-05-69  
NASA Contract No.: C-77951-B  
Responsible Center: LeRC
Behavior of Unsteady Thermocapillary Flows

Principal Investigator: Prof. Marc K. Smith
Georgia Institute of Technology

Co-Investigators: 
No Co-I’s Assigned to this Task

Task Objective:
The objective is to investigate thermocapillary instabilities to provide an understanding of the mechanisms of flow instabilities. The study will also indicate how well the stability theory results for simple geometries apply to the behavior of thermocapillary flows in more complex geometries.

Task Description:
The research approach consists of both a theoretical and an experimental effort. Two nonlinear analytical models for study of post-critical thermocapillary flows in a bounded domain will be constructed as part of the theoretical work. The models will be used to explore the effects of Pr, interfacial heat transfer, domain size, 3-D disturbances, and interfacial deformation on system flow stability. An experimental investigation of thermocapillary instabilities of opaque, low Pr and high Pr fluids will be also be performed.

Task Significance:
This understanding will assist fluid system designers optimize system designs in which thermocapillary flows are important.

Progress During FY 1995:
We have continued our analysis of the behavior of the thermocapillary flow in a thin liquid layer by considering two more geometries: an annular ring with a free upper surface, and a thin cylindrical shell wrapped around a solid rod. Lubrication theory was used to derive the nonlinear partial differential equation describing the evolution of the free surface of the liquid layer for both geometries.

Steady-state thermocapillary flows in the annular ring geometry with heating from the inner surface were found numerically by solving the evolution equation using a pseudo-spectral method. The solution is parameterized in terms of a modified capillary number that measures the effect of surface tension and another parameter measuring the curvature of the ring. For large rings and small capillary numbers (large surface tension), the free surface of the layer is almost flat. As the capillary number increases, the deformation of the interface increases with a bulge forming near the cold end of the cavity and a depression occurring near the hot end. The thermocapillary flow has two steady-state solutions for capillary numbers less than some limit-point value. The lower-branch solutions are characterized by a smaller amount of free-surface deformation than the upper-branch solutions. As the capillary number decreases (larger surface tension), the free-surface deformation decreases on the lower branch, but increases on the upper branch. Two-dimensional stability calculations have shown that the lower branch is linearly stable and the upper branch is linear unstable. Changing the contact-line conditions from pinned contact lines to fixed contact angles greatly reduces the value of the capillary number at the limit point. For capillary numbers greater than the limit-point value, the behavior is unsteady.

As the azimuthal curvature of the ring increases, the limit-point value reduces at first and then increases. It appears to move off to infinity as the inner radius of the ring approaches zero. This indicates that a circular liquid film heated from the center would always have a stable, steady-state solution. This is in direct contrast to the two-dimensional case in which only unsteady behavior is seen for large enough capillary numbers.
Similar calculations for the thin cylindrical shell are currently underway. The results so far show that the steady shape of the interface is the reverse of what was seen before: a bulge is near the hot end of the layer and a depression is near the cold end. This behavior is the result of the capillary pressure due to the cylindrical nature of the shell geometry. Further explanations of this phenomena and the relevant stability and time-dependent calculations are being prepared.

The experimental work planned for this year is a detailed measurement of the free-surface deflection in a thin layer of molten gallium. The thickness of the liquid layer will be measured by a laser beam reflected off the free surface of the gallium. A video camera will monitor the incident and reflected positions of the beam as it passes through two diffusing screens. From these positions, it is a simple matter to calculate the layer thickness. Scans will be done to monitor the thickness in the entire layer as the end-to-end heating is increased. These measurements will be compared to the calculations for the planar liquid layer geometry done previously. We are particularly interested in the region near the hot end when the layer gets very thin and the possibility of dry-out exists.

**STUDENTS FUNDED UNDER RESEARCH:**

| BS Students | 0 |
| MS Students | 0 |
| PhD Students | 1 |

**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Presentations**


II. MSAD Program Tasks — Ground-based  

**Flow-Influenced Shape Stability: Breakup in Low Gravity**

**Principal Investigator:** Prof. Paul H. Steen  
**Cornell University**

**Co-Investigators:**  
No Co-I's Assigned to this Task

**Task Objective:**
1. Understand the influence in low gravity of flow on interface shape. For example, document and control the influence of axial flow on the Plateau-Rayleigh instability of a liquid bridge.

2. Extend the ground-based density-matching technique of low gravity simulation to situations with flow; i.e., develop Plateau chamber experiments for which flow can be controlled.

**Task Description:**
The configuration of a liquid held by surface tension under low gravity is susceptible to significant modification by liquid motion. Motion destabilizes in general but there are narrow circumstances where motion can stabilize. We identify these circumstances theoretically, by solving linear and nonlinear stability problems, and try to locate them in experiments conducted with a dynamic Plateau apparatus. Overall, the goal is an understanding of the influence of flow on shape.

**Task Significance:**
Containerless containment of liquids by surface tension has broad importance in low gravity. For space vehicles, the behavior of liquid/gas interfaces is crucial to successful liquid management systems. In microgravity science, free interfaces are exploited in various applications. Examples include float-zone crystal growth, phase separation near the critical point of liquid mixtures (spinoidal decomposition) and quenching of miscibility gap molten metal alloys. In some cases, it is desired to stabilize the capillary instability while in others it is desired to induce capillary breakup. In all cases, understanding the stability of interface shape in the presence of liquid motion is central.

**Progress During FY 1995:**
Both analytical/numerical and experimental approaches are employed.

Stability analyses include linear and nonlinear techniques. The linear stability approach has been used to analyze the shape stability of a cylindrical interface containing axial shear flows, both isothermally- and thermocapillary-driven. Computational feasibility currently limits this approach to base states that are separable flows, effectively, the axial-infinite interfaces. It is now well known that infinite cylindrical interfaces can be stabilized. For finite interfaces an alternate approach is needed. In the limit of no motion, minima of the free energy functional are obtained using the calculus of variations supplemented by numerical branch-tracing. For weak motion (creeping flow), we extend this approach using a modified functional. Near the singularity represented by the Plateau-Rayleigh limit, bifurcation theory using Liapunov-Schmidt reduction is a natural tool for the solution of the appropriate nonlinear Euler-Lagrange equation. All these analytical/numerical tools lend themselves to understanding the physics of stability in terms of simple competition mechanisms.

As for the experimental approach, a dynamic Plateau chamber has been built and is used to study liquid bridges held captive by rod-ends and embedded in an axial surrounding flow. Theory has guided the experiments to a particular window in parameter space. Such guidance is crucial since interesting stabilization effects occur over narrow parameter ranges for this problem.
II. MSAD Program Tasks — Ground-based

Discipline: Fluid Physics

Progress on theory
Disturbances may be classified by symmetry relative to the midplane of the liquid bridge. Axial gravity is antisymmetric. "Full-zone" thermocapillary effects are symmetric and "half-zone" effects, antisymmetric, for example.

Theory shows how two antisymmetric effects of opposite signs cancel with a nonlinear "bonus" that can extend stability beyond the classical limit. To prove the concept such stabilization with respect to the Rayleigh-Taylor instability has been demonstrated (by analytical solution) for two problems: 1) interface on 2D channel tilted with respect to gravity and sheared to generate a lubrication pressure; and, 2) interface in a Hele-Shaw slot with tilt and pressure induced by natural convection.

With one symmetric and one antisymmetric effect, stabilization of a different kind can occur. This kind may be relevant to stabilization of float-zones ("full-zones") in the low gravity of spacecraft, as observed in the MEPHISTO experiment.

Progress on experiment
Control of thermal disturbances has been improved. This is imperative to systematic achievement of liquid bridges beyond the Plateau-Rayleigh limit as predicted by the above nonlinear theory.

Future plans include: 1) to further refine results for symmetric/antisymmetric interactions, 2) examine float-zone data from MEPHISTO spacetlab experiment to determine the relevance of various stabilization theories, and, 3) to continue our efforts in the laboratory on bridges using axial flow and density balance as the competing disturbances.

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BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals


Printed: 28 FEB 1996
Interactions of Bubbles and Drops in a Temperature Gradient

Principal Investigator: Prof. R. S. Subramanian
Clarkson University

Co-Investigators:
Balasubramaniam, Dr. R.
NASA Lewis Research Center (LeRC)

Task Objective:
The objective of this research is to study the interactions of bubbles and drops in the presence of a temperature gradient in the continuous phase. The goal is to understand how the presence of a neighboring drop alters the motion of a test drop and whether the two drops coalesce. Both theoretical and experimental research is planned on the interactions of drops with each other or with a neighboring boundary.

Task Description:
The emphasis of the initial effort is on the design, fabrication and set up of the experimental hardware, and to check its performance. Experiments will then be performed using a suitably chosen liquid-liquid system such that the drops sink due to buoyancy. The drops will be subjected to a vertical temperature gradient; thermocapillary forces will then cause the drops to move upwards. Initially experiments will be performed with single drops and the results will be compared with existing theory. Subsequent experiments will focus on drop interactions.

Task Significance:
This research attempts to provide important information on interactions of drops, i.e., how the presence of a drop affects the motion of another, in a medium where the temperature is non-uniform. Primary emphasis is on thermocapillary flow, thereby the research is more applicable to drop interactions under reduced gravity. The nature of these pairwise interactions determines whether the drops speed up or slow down as they approach each other and whether they collide or glide past each other. Pairwise interaction data are very useful in models that track coalescence in a many drop dispersion.

Progress During FY 1995:
The apparatus for studying drop motion in a vertical temperature gradient was reassembled. Successful experiments were conducted on the migration of pairs of drops. The drops are of diethyl maleate and the continuous phase is propanediol. Because of the three-dimensional nature of the trajectories, it was necessary to obtain two orthogonal views, using two video cameras. Frame by frame analysis of the video data is currently in progress.

STUDENTS FUNDED UNDER RESEARCH:

BIBLIOGRAPHIC CITATIONS FOR FY 1995:
Presentations

Printed: 28 FEB 1996
II. MSAD Program Tasks — Ground-based

Discipline: Fluid Physics

Instability in Surface-Tension-Driven Benard Convection

Principal Investigator: Prof. Harry L. Swinney
University of Texas, Austin

Co-Investigators:
McCormick, Prof. W.D. University of Texas, Austin
Swift, Prof. J.B. University of Texas, Austin

Task Objective:
The objectives of this work are to investigate the primary and secondary instabilities in surface-tension driven convection and in double-diffusive convection in the Hele-Shaw geometry, and to characterize both the global structures and the local flow properties of Benard Convection.

Task Description:
Noninvasive optical techniques will be developed and employed to study the primary and secondary instabilities over a wide range of Marangoni numbers. Theoretically, nonlinear analyses for the long wave (k=0) instability will be conducted with surface deflection included. Direct numerical simulation of the two- and three-dimensional incompressible fluid equations will be used to investigate both weakly non-linear behavior near the primary instability and secondary instabilities in Marangoni convection.

Task Significance:
Surface tension force dominates many physical processes in space and strongly influences many important practical problems on Earth, such as the processing of commercially important materials (electronic or biological crystal growth), manufacturing (welding, coatings), as well as industrial heating, cooling, and mixing (thin film heat exchangers). This work seeks to elucidate clearly the basic mechanisms that can cause fluids to change from a simple quiescent state to a turbulent behavior. Such a transition can have a dramatic effect on the practical problems described above. Furthermore, in the course of this work, new experimental techniques such as enhanced infrared imager will be developed that may later find wider use in other space/terrestrial applications of commercial importance.

Progress During FY 1995:
— Experimental onset of hexagonal convection was determined precisely and found to agree with linear theory. Previous experiments had found instability at parameter values nearly an order of magnitude smaller than predicted.
— First measurement of hysteresis at the onset of hexagons sets a standard for comparison of competing nonlinear theories, whose current estimates of hysteresis disagree with each other and with the experiment.
— First experimental observation of a long-wavelength (non-hexagonal) instability is found at parameter values 35% smaller than predicted by linear theory. This instability, which may be the dominant primary instability for Benard convection in microgravity, leads to the formation of a large scale "dry spot".
— First experimental observations of competition between the hexagonal and long-wavelength instabilities reveal a fundamental imbalance: the presence of hexagons suppresses the long-wavelength instability, while the presence of the long-wavelength mode may induce the formation of hexagons.
— High purity (>95%) silicone oils have been refined from commercial oils, which are used in many fluids experiments. The purification method yields superior working fluids that have well-characterized physical properties and are free of deleterious evaporation and condensation effects.
— A new secondary instability leading to an ordered pattern of square convection cells has been observed. The square pattern, which bifurcates from the hexagonal state, appears at a Marangoni number of approximately 400; there is a large range of Marangoni number where both hexagons and squares coexist.

**STUDENTS FUNDED UNDER RESEARCH:**

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**TASK INITIATION:** 12/92  **EXPIRATION:** 11/95

**PROJECT IDENTIFICATION:** 962-24-05-41  **NASA CONTRACT NO.:** NAG3-1382  **RESPONSIBLE CENTER:** LeRC

**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Journals**


Schatz, M.F. and Howden, K., Purification of Silicone Oils for Fluid Experiments. Experiments in Fluids, (September 1995).


Crystal Growth and Fluid Mechanics Problems in Directional Solidification

Principal Investigator: Prof. Saleh Tanveer
Ohio State University

Co-Investigators:
Baker, Dr. G. R.  
Foster, Dr. M. R.
Ohio State University  
Ohio State University

Task Objective:
The primary objective of this task will involve efforts to build a comprehensive theory for dendritic crystal growth, with and without fluid convection. This is a difficult task as it involves a nonlinear time-dependent evolution equation that is nearly ill-posed when capillarity effects are small. Bridgman Crystal Growth situations will also be studied.

Task Description:
The effort will primarily consist of a numerical study of possible bifurcation of steady-state solutions that may be steady or oscillatory in two or three dimensions.

Task Significance:
It is expected that some interesting subclasses of problems will be found for extreme values of parameters; in these cases, it is possible that the essential phenomena can be captured by a nonlinear analysis of equations that contain only a few parameters.

Progress During FY 1995:
During the funding period of support, Jan 1994 to present, continued progress has been made in two different crystal growth problems - dendritic crystal growth and the Bridgman problem.

A. Dendritic Crystal Growth
In the small Peclet number asymptotic limit, we have carried out both numerical and asymptotic analysis with a view to understanding some controversial issues in selection theory and in the time development of dendrites.

If we recall, in studying certain classes of disturbances superposed on an initially parabolic dendrite, in the asymptotic limit of small Peclet number Pe, it was recognized that there are different regions where the equations for the temperature field and boundary conditions have different forms. In an O(1) region around the dendrite tip, the diffusive field reduces to a Laplacian. With a novel numerical scheme, we have computed highly accurate solutions in the tip region showing the evolution of an initially parabolic dendrite with nonzero surface tension. For a long time, perturbation from the initial parabola is found to be restricted to the O(1) region surrounding the tip, while the other regions play a passive role and remains steady. It was found that regardless of the initial condition, a bulbous expanding region forms near the tip in the absence of crystalline anisotropy. For long time, this bulbous region evolves in a way independent of the initial condition. In the presence of noise, represented in our formulation by certain complex singularities, this bulbous tip region will split. The calculation suggests that there can be no globally time dependent state with a locally steady tip. However, with an assumed four-fold crystalline anisotropy, with a minimal surface tension direction coinciding with the needle crystal axis, it is found that the tip region settles to a steady state where the tip radius and velocities are in accordance to microscopic solvability. This happens within the time scale where disturbances have not moved away from the O(1) region around the tip.

A family of exact solutions describing the time evolution of a zero surface tension dendrite in the O(1) tip region was reported last year. Some of these solution exist for all times and result in tip-splitting or side-branching of an
II. MSAD Program Tasks — Ground-based

Discipline: Fluid Physics

Initially nearly parabolic dendrite. Later on, a criterion involving the sign of the real part of the residues of poles was found that determined the side of the needle crystal on which disturbances advect. Some of these exact solutions result in a finite-time cusp singularity on the interface. We have addressed the relevance of some of these solutions to the actual evolution of a dendrite in the small surface tension, small Peclet number limit. For instance, through an inner region analysis, it was found that the time at which a zero surface tension solution forms a cusp is preceded by the effect of the so-called daughter singularities that result in tip fattening in the absence of crystalline anisotropy effects. The actual interface in such cases does not come close to cusp formation even for arbitrarily small surface tension. In the presence of anisotropic small surface tension, a cusp does not form either but in this case a locally steady solution results when the anisotropy axis is aligned appropriately.

The fate of various types of disturbances advecting along the sides of a needle crystal, as represented by certain types of complex singularities approaching but never hitting the real domain, is also being examined. In particular, it is seen that for an approaching complex pole, significant surface tension effects on the interface occur in a slow time scale that scales inversely with the surface tension parameter. For sufficiently small Peclet number relative to surface tension, such effects can be felt at the interface in the 0(1) region around the tip. For other relative ordering of surface tension and Peclet number, surface tension effects are only felt once the disturbance has advected to other asymptotic regions. The interaction of multiple singularities and their implication to side branching coarsening is currently being examined.

B. Bridgman Crystal Growth

In the Bridgman problem, we investigated analytically the limit of large solutal Rayleigh number in the case when the rejected solute is heavier that the rest of the alloy, i.e. in a solutally stable arrangement. Unlike the case of large thermal Rayleigh number reported last year, where to the leading order the expressions for radial segregation and crystal melt interface do not involve the heat transfer at the top edge of the insulation zone, we find that in this case the heat transfer at both the top and bottom edges of the insulation zone is involved in the explicit expressions for radial segregation and crystal melt interface shape. We also find some criteria to minimize each of these quantities.

Accurate numerical schemes are being developed to extend and verify the asymptotic scaling results found analytically. In addition, the transient problem that is free of any quasi-steady hypotheses is also being investigated numerically. The numerical results for the quasi-steady equations, so far only with the linearized equations but a more realistic no-slip side wall condition, are in accord with the asymptotic results in the large thermal Rayleigh-number limit, where a more mathematically convenient no-stress condition was used. The calculations also demonstrate clearly the efficacy of the optimal heat transfer condition suggested analytically.

STUDENTS FUNDED UNDER RESEARCH: TASK INITIATION: 1/93 EXPIRATION: 12/96
PROJECT IDENTIFICATION: 962-24-05-53
NASA CONTRACT NO.: NAS1-18605
RESPONSIBLE CENTER: LeRC

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals

Oscillatory/Chaotic Thermocapillary Flow Induced by Radiant Heating

Principal Investigator: Dr. Robert L. Thompson
NASA Lewis Research Center (LeRC)

Co-Investigators:
DeWitt, Prof. K. University of Toledo
Hsieh, Dr. K. NYMA, Inc.
Van Zandt, D. ADF

Task Objective:
The main objective of the research is to study the oscillatory and chaotic thermocapillary flows induced by radiant heating of the free surface of a high Prandtl number fluid. Both ground-based experiments and numerical analysis will be conducted to study effect of heating level (supplied by CO₂ laser), surface shape, aspect ratio, and Prandtl number on the conditions for transition from steady to oscillatory flows and then to chaotic flows. In the experiments, flow structures will be observed using a flow visualization technique and temperature distribution on the free surface will be measured using an infrared (IR) imager. Numerical results will be compared with experimental data.

Task Description:
A CO₂ laser is used to provide the heat source. The profile of the laser intensity in the radial direction can be a Heaviside function or a Gaussian function with variable beam diameter. The material of the test chamber is a copper water jacket, providing well-controlled wall temperature. The bottom wall of the test cell is insulated. An IR imager is used to measure surface temperature distribution and several thermocouples are implanted in the test chambers, including one at the bottom. Critical powers of the CO₂ laser at the onset point of oscillatory flow are measured at various aspect ratios and dynamic Bond numbers.

Task Significance:
The strategy of this study is to first compare the measured onset conditions for oscillatory flows with results from linear stability analysis. With the designed experimental conditions, surface tension and buoyancy effects are equally important. If close comparison between experimental and numerical results can be obtained, it could support the validity of numerical prediction of the onset conditions for pure Marangoni flow. This can help the design of possible space experiments. Results obtained in this study can help the design of surface tension driven convection experiments in space. Furthermore, a deeper understanding of the thermocapillary flow can be achieved, and the knowledge can be used to improve materials processing procedures.

Progress During FY 1995:
In study of post-onset oscillatory flow structures, it was found that a series of transitions occur and finally lead to chaotic motion. Power spectra of surface temperature at a fixed location were obtained from time history of the data with fast-Fourier-transformation. When laser power increases, superharmonic mode starts to appear and finally a white spectrum occurs which indicates the chaotic flow motion.

A new design of the test chamber has been made to reduce the evaporation rate of the silicone oil when the experiments are conducted under high laser power. A new stabilized CO₂ laser was also acquired for achieving more stable operating conditions. System with new test chamber and laser has been setup and tested. A series of experiments will be conducted to study the post-onset behavior of the oscillatory flow in detail.
II. MSAD Program Tasks — Ground-based

**STUDENTS FUNDED UNDER RESEARCH:**

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**TASK INITIATION:** 10/92  **EXPIRATION:** 10/95

**PROJECT IDENTIFICATION:** 962-24-05-67

**NASA CONTRACT NO.:** NAG3-1501

**RESPONSIBLE CENTER:** LeRC

Printed: 28 FEB 1996
II. MSAD Program Tasks — Ground-based

Discipline: Fluid Physics

Light Scattering Studies of Relative Motions of Solid Particles in Turbulent Flows

Principal Investigator: Prof. Penger Tong
Oklahoma State University

Co-Investigators:
Goldburg, Prof. W.I.
University of Pittsburg

Task Objective:
The primary objective of this task is to understand particle concentration fluctuations in turbulent flows, particularly how the dispersion is affected by particle inertia and the characteristics of the turbulence.

Task Description:
This proposal describes a series of experiments on the dispersion of particles in turbulent flows. The task plan is to study the particle dispersion phenomena in two well characterized turbulent flows. One is turbulent Rayleigh-Benard convection in water and the other is a turbulent grid flow in a water channel.

Task Significance:
The proposed research will add extremely important data to that presently available to further develop an understanding of the interaction of solid particles and a turbulent fluid. Such data would be valuable not only in revealing phenomenology, but also in testing model representations of turbulent suspensions.

Progress During FY 1995:
The progress for the project so far were: (1) Using dynamic light scattering, PI and his associates have measured the particle velocity decay rate as a function of the length of a thin cylindrical scattering volume (L). The results show a cut-off length (Lc) above which the decay rate is proportional to \( L^{0.6} \), and below this cut-off length the decay rate is proportional to \( L^{0.35} \). This cross over from Bodgiano-Obukhov scaling at large L to Kolmogorov scaling for smaller L is an important issue of an unresolved theoretical debate. These experiments provide a benchmark against which future theoretical models can be tested, (2) Studying sedimentation of heavy particles in a fluid at equilibrium, and (3) PI has demonstrated, for the first time, that the depletion-attraction potential is accurate to describe the colloidal interaction in polymer solutions.

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TASK INITIATION: 7/94  EXPIRATION: 6/96
PROJECT IDENTIFICATION: 962-24-05-98
NASA CONTRACT NO.: NAG3-1613
RESPONSIBLE CENTER: LeRC

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Presentations


Tong, P. "Scattering experiments in mixtures of colloid and polymers: Depletion and absorption." Physics Department Colloquium, Oklahoma State University, April 13, 1995.

Tong, P. "Depletion and absorption phenomena for colloids in polymer solutions." Symposium on Colloids/Polymer Science and Engineering, 26th Annual Meeting of the Fine Particle Society, Chicago, IL, August 22-25, 1995.
Computational Studies of Drop Collision and Coalescence

Principal Investigator: Prof. Grétar Tryggvason
University of Michigan

Co-Investigators:
Jacqmin, Dr. D.
NASA Lewis Research Center (LeRC)

Task Objective:
The objective of this research is to investigate the behavior of bubbles and drops in microgravity by full numerical solutions of the governing equations. The collision and thermal migration of drops are studied in detail to provide essential input for material processing and fluid handling in space. These problems also serve as a test bed for refinements and extensions of the numerical technique being used, thus helping to develop the capability to predict accurately the behavior of free-surface fluid systems.

Task Description:
A numerical technique, based on explicit tracking of the interface between two immiscible fluids, is used in this study. This method has now been extended to deal with both the thermal migration and the rupturing of thin films.

Task Significance:
The unique aspect of the method is that it accounts fully for both inertia and viscous effects in both fluids and allows the inclusion of surface tension. It is also well suited for complicated interface geometries and has been implemented for fully three-dimensional flows. The basic aspect of this method is described in the Journal of Computational Physics, vol. 100 (1992), p. 25.

Progress During FY 1995:
Our research is aimed at developing a basic understanding of the behavior of drops in microgravity and developing numerical tools to allow accurate predictions of their behavior. We have focused on: Collision of drops; thermal migration of both bubbles and drops; and the motion of drops in shear flows.

For collision of drops we have simulated the head-on collision of two equal size drops for a wide range of Weber and Reynolds number, which are the main controlling parameters, as well as several fully three-dimensional off-axis collisions. The simulations reproduce experimental data well, showing that the evolution of the drops depend to a large degree on the Weber number of the drops before collision, but only weakly on the Reynolds number, once it becomes sufficiently large. One of the more critical questions about drop collisions is whether the drops coalesce permanently, or separate again. Several collision modes are possible, and we have examined a number of them. For head-on collisions the drops deform into a disk-like shape as they hit each other and may separate again as surface tension pulls this disk shape back into a more spherical form, often leaving one or more drops in between the original drops. For off-axis collisions, the drops, on the other hand, may continue on their original path after the initial coalescence, and stretch apart again (usually called "grazing collision"). We have recently started to collaborate with Professor C.K. Law who sent a student to Michigan this summer to learn to use our codes. He has now simulated the evolution of several bouncing drops and reports excellent agreement with his experiments.

Our simulations of thermal migration have focused on the collective behavior of many drops as they migrate toward a hot surface. Two-dimensional simulations of a large number of drops showed a strong tendency for many (up to 25) drops to form layers across the channel, and also that the drops only deformed during the initial transient. Subsequent fully three-dimensional simulations using up to nine drops have confirmed the general behavior observed in two-dimensions, but showed that for a given Marangoni number they do not line up across the channel as rapidly as the two dimensional computations did. Since all interactions fall off much more rapidly in three dimensions as compared with two-dimensions, this is perhaps not too surprising. However, as the Marangoni number increases, the effect of the drops on the temperature field increases and the interactions become stronger.
We have examined in detail the evolution of drops in both shear flow between moving walls as well as the motion of drops in a pressure driven flow between stationary walls. Both the fluidization of a layer of drops initially near a wall as well as the long time evolution of an initially uniformly distribution of drops has been examined. For the shear flow, the results generally show a shear thickening behavior for drops when the Reynolds number is high and the Weber number is low, and the evolution is dominated by collision of the drops. For the pressure driven flows, a single drop will move to a position half way between the centerline of the channel and the wall for a fairly wide range of parameters. This effect is usually called the Segre-Silberg effect and we have found that for relatively dilute suspensions, this leads to a local concentration of drops at this location. At higher concentrations, a wall layer that is relatively free of drops is observed. Both two and three-dimensional simulations of this effect show similar behavior.

The above computations have all been done using explicit tracking of the boundaries between fluids. Although the method is robust and accurate, it is somewhat complex and for many problems it may be possible to use capturing where the interface is simply marked by as rapid change in material properties instead of full tracking. D. Jacqmin has been exploring this possibility and has now developed a code capable of doing short time simulations of relatively complex problems of both coalescence and breakup of drops. For long time simulations, the interface needs to be better preserved, and we are currently exploring ways to do that.

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### BIBLIOGRAPHIC CITATIONS FOR FY 1995:

#### Journals


#### Proceedings


#### Presentations

Nonlinear Bubble Interactions in Acoustic Pressure Fields

Principal Investigator: Prof. John Tsamopoulos
State University of New York, Buffalo

Co-Investigators:
Ashgriz, N.
State University of New York, Buffalo

Task Objective:
This project proposes both theoretical and experimental studies of the interactions between bubbles immersed in an immiscible liquid. Specific objectives include the following:

1. To investigate the conditions when linearly accelerating motion of two interacting bubbles turn into a spiral motion of one of them.

2. To study the delay and prevention of break up of accelerating bubbles.

3. To incorporate bubble velocity and fluid viscosity into a coalescence model.

4. To study the coalescence of two bubbles.

Task Description:
The analytical study will focus on bubble oscillations and interactions of moderately large amplitude and will determine conditions under which the axisymmetric flow turns into three-dimensional. For viscous liquids or large deformation either the Boundary Element method or a combination of Finite Elements with the Volume of Fluid method will be used. Bubbles will be suspended in an immiscible liquid using the acoustic levitation technique for the experimental part of this project. Bubble motions and interactions will be induced by an additional acoustic field. The bubble motions, shape oscillations, collision and breakup will be recorded with high-speed cinematography.

Task Significance:
The results from this project will be beneficial to others in the bubble dynamics studies. It is well known that the formation and collapse of cavities of gas inside a flow stream cause erosion in fluid-handling equipment. The effect of a properly tuned acoustic field may help in the correct detection of submerged objects in the production of bubble dispersions.

Progress During FY 1995:
During this reporting period, the following have been accomplished:

1. In order to distinguish the attractive Bjerknes forces from other forces, the exact pressure field has been determined in more than 300 points at 3 different levels inside the test section tank.

2. Levitation of two bubbles at some distance apart was achieved. The sizes and thus size ratios and the initial distance between them were varied. By repeatedly increasing the volume of the bubbles (injecting with a syringe needle), the Bjerknes forces between the two bubbles have been increased until they overcome the weak horizontal pressure gradient and the bubbles accelerate towards each other. Velocities and accelerations were recorded using high speed video.

3. A new mathematical description of secondary Bjerknes forces in terms of amplitude and frequency of the external pressure field; volume and size ratio of the bubbles; distance between bubbles; and material properties of the liquid...
II. MSAD Program Tasks — Ground-based

for different cases of phase shifts of bubble oscillation with respect to external forcing was developed based on a more accurate relation of the acoustic field radiated by a bubble.

4. The nonlinear interaction between two bubbles was modeled and an analytical solution was determined. Velocities and forces predicted by the new model were found to be in good agreement with the experimental results.

5. Analysis of experimental data showed that the time interval necessary for the two bubbles to overcome the separation between them can be correlated with two important dimensionless parameters: one is based on the geometrical quantities (initial distance, radii), and the other is based on the acoustic field pressure amplitude and the hydrostatic pressure at the location of the bubble.

6. Contrary to the theoretical predictions, experimental results indicated that the ratio of average accelerations approached a limiting value as the size ratio decreases. A power-law correlation was determined between the characteristic parameters of the two bubbles: initial velocities and radii.

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**Bibliographic Citations for FY 1995:**

**Journals**


Residual Accelerations in a Microgravity Environment

Principal Investigator: Prof. Jorge Viñals  
Florida State University

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
This research program aims at developing a realistic theoretical model of the high frequency components of the residual acceleration field (or g-jitter), and at studying its effect on a variety of typical fluid experiments.

Task Description:
The high-frequency components of the residual acceleration field are modeled as a stochastic or random process; that is, a succession of random values of the intensity and orientation of the acceleration. Our research is divided into two major parts, one analytic and the other numerical in character. In the first part, we formulate a hydrodynamic problem that explicitly includes a random, time-dependent gravitational acceleration which is modeled as a narrow band noise. In the second part, we develop numerical algorithms to simulate this type of random field and incorporate them into Navier-Stokes equation solvers.

Task Significance:
All space experiments performed in the manned space environment are exposed to the high frequency components of residual accelerations (g-jitter) on board the spacecraft. This effect can significantly alter experimental results. A numerical approach is taken to determine what regimes of g-jitter will effect experiments conducted in a microgravity environment. A generic class of problems is considered with emphasis on materials experiments.

Progress During FY 1995:
We have verified the stochastic model of g-jitter introduced by analyzing actual g-jitter data collected during the SL-J mission (SAMS-258). The time series of head A between MET 0017 and MET 0023 has been analyzed in detail. A scaling analysis has been performed to determine the existence of deterministic or stochastic components in the time series. We find that during this period of six hours, there appears to be a monochromatic contribution of 17 Hz with an amplitude \( \sqrt{\langle g^2 \rangle} \) = 3.56 x 10^-4 g, where g is the intensity of the gravitational field on the Earth's surface. There are two additional components that have a finite correlation time: For the component at 22 Hz we estimate \( \sqrt{\langle g^2 \rangle} \) = 3.06 x 10^-4 g and a correlation time \( T = 1.09 \) s, whereas for 44 Hz we find \( \sqrt{\langle g^2 \rangle} \) = 5.20 x 10^-4 g and \( T = 0.91 \) s. There is a white noise background of intensity \( D = 8.61 \times 10^{-6} \text{cm}^2/s^3 \).

Buoyancy driven convection induced by a fluctuating acceleration field has been studied in a two dimensional square cavity. This is a simplified model of fluid flow in a directional solidification cell subject to external accelerations, such as those encountered in a typical microgravity environment. Approximate analytic solutions to the flow field have been found in the limit of large aspect ratio. We have been able to isolate a few important characteristics of cavity flow that result entirely from the stochastic nature of the acceleration field, and that would not have been obtained under a strictly periodic gravity modulation. We have extended previous early time calculations up to the asymptotic (statistical) steady state. The flow intensity is seen to be proportional to a stochastic Rayleigh number that differs from the classical definition. This result has been validated numerically by direct solution of the Navier-Stokes equation in the Boussinesq approximation driven by a random gravitational field.

Preliminary results of experiments to measure the growth of dendrites from pure supercooled succinonitrile in microgravity were reported by Glicksman at the Microgravity Materials Science Conference held in Huntsville, Alabama, May 24-25, 1994. Subsequently, some preliminary data (based only on the analysis of telemetered binary
digital images) on dendrite growth velocity versus supercooling were published. These data indicated that dendrite growth velocities are affected by convection in Earth’s gravity for supercoolings ΔT < 1K but are also affected by convection even in microgravity for supercoolings below ΔT < 0.3K. New microgravity data for supercoolings between 0.3K and 1K agreed with “power law” extrapolation of data for larger supercoolings for a constant value of the “selection parameter” s. It occurred that a simple scaling analysis of the global convection that occurs in the melt surrounding an approximately spherical dendritic array could be used to explain these results. In collaboration with researchers at NIST, we hypothesized that the important aspects of natural convection can be accounted for by considering the global convection that would occur in the vicinity of a sphere of radius R that characterizes the size of a dendritic array that is growing from a point source and solved the steady-state problem of a paraboloidal dendrite at temperature TM growing toward a confocal paraboloid at temperature T∞ located at a distance d from the tip. Our results are in remarkably good agreement with the data of Glicksman et al. for growth of succinonitrile on Earth and in microgravity. The value of g at which this model indicates that convective effects become significant has an extremely strong dependence (~ ΔT9.5) on supercooling. Therefore, unfortunately, lowering g to 10⁵ m/s² would only lower the supercooling above which convection would have little effect on dendritic growth to about 0.1K.

**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Journals**


**Presentations**

II. MSAD Program Tasks — Ground-based

Experimental Study of the Vapor Bubble Thermosyphon

Principal Investigator: Prof. Peter C. Wayner, Jr.  
Rensselaer Polytechnic Institute

Co-Investigators:
Plawsky, Prof. J.  
Rensselaer Polytechnic Institute

Task Objective:
The objective of this effort is to better understand the physics of evaporation and condensation as they affect the heat transfer processes in a vapor bubble thermosyphon (VBT). In small systems, interfacial intermolecular forces can be used to control fluid flow and heat transfer. The VBT, one such system, consists of a small enclosed container partially filled with a liquid. When a temperature difference is applied to the ends of the VBT, evaporation occurs at the hot end and condensation at the cold end — resulting in a very effective heat transfer device.

Task Description:
A transparent VBT will be designed and developed. The microscopic intermolecular force (pressure) field, which is a function of the liquid thickness profile, will be measured using microcomputer enhanced video microscopy based on interferometry. The temperature field will be measured using the interline absorbed film thickness and small temperature sensors.

Models of the transport processes in the contact line region of a VBT which include the effects of liquid-solid and liquid-vapor intermolecular forces have already been developed. As part of this effort these models will be further refined and the transport characteristics of VBTs will be obtained by comparing the experimental data to numerical solutions of the model.

Task Significance:
By studying liquid-film thicknesses and temperatures in VBT, a better understanding of the processes can be gained that will lead to optimization of VBT designs.

Progress During FY 1995:
The experimental studies can be divided into equilibrium and non-equilibrium studies of the CVBT. We note that we have changed the name of the experimental design from VBT to CVBT (Constrained Vapor Bubble Thermosyphon) which is much more descriptive. The final results of the equilibrium studies using apolar fluids are given in the papers and presentations listed in the bibliography. We have also completed some additional equilibrium studies using the polar fluid water. The effect of pH and temperature on the wetting characteristics of the water/glass system were obtained and are being correlated. Needless to say, water is a very complex fluid to study experimentally when interfacial forces are being emphasized. However, we find that the CVBT results adds significant new information on the classical water/glass system. In addition, since most industrial metals naturally have a significant oxide coating due to corrosion and the surface characteristics of glass are close to those of a metal/oxide coating due to corrosion, the surface characteristics of glass are close to those of a metal/oxide in an engineering environment. We will present some of the results obtained using water at the 995 Annual Meeting of AIChE. We find that an apolar passive system is easier to control and use than the polar system. On the other hand, a well controlled water heat exchanger would be highly desirable for the microgravity environment.

The nonequilibrium studies are considerably more complicated than the equilibrium studies because air has to be removed from the cell and both temperature and pressure measurements are needed. During the past year, we received a customized microscope which allowed us to optimize the experimental optical measurement of the film profile. A purification system for removing the air and improving the purity of the apolar fluids was built and tested. The heat source and heat sink for the CVBT were designed and tested. Preliminary experimental and
II. MSAD Program Tasks — Ground-based

Discipline: Fluid Physics

Theoretical results using the CVBT under non equilibrium conditions were obtained. We found that we could operate the cell as a capillary controlled thermosyphon (heat pipe). The important characteristic in the Earth’s environment, where there are natural convective heat losses, is the presence of a region where the temperature gradient almost vanishes. We find that the Earth’s gravitational field restricts the design and operating conditions where this can be obtained. This demonstrates the need for a microgravity environment to remove the complications and natural convection. In addition, the relatively large dimensions of our cell require a microgravity environment for symmetry. We are rebuilding our cell to include the capability to measure the vapor pressure in the cell. This should be the final cell modification.

A numerical study of the evaporating extended meniscus in the corner of the constrained vapor bubble thermosyphon was completed for two operating conditions. We find that the model is very stiff and that the selection of the appropriate boundary conditions is not obvious. We anticipate that a complete set of experimental measurements will clarify the choice of the proper boundary conditions.

Based on the above results we can make the following conclusions:

1) The use of an image analyzing interferometer, IAI, with a constrained vapor bubble thermosyphon, CVBT, was demonstrated under equilibrium conditions at 1g.
2) Using the augmented Young-Laplace equation, good agreement between the theoretical and experimental values of the dispersion constant was obtained.
3) A gravitational field restricts the range of forces that can be studied because the curvature gradient, K’, is a function of gravity.
4) The Earth’s gravitational field also restricts the range of heat transfer conditions that can be studied because of heat losses due to natural convection.
5) Experiments using apolar fluids are easier to control than experiments using polar fluids.
6) Ground-based studies indicate that a “relatively simple” flight experiment can be designed to evaluate the augmented equilibrium Young-Laplace equation using the CVBT.
7) A flight experiment for (at least) a study of the augmented equilibrium Young-Laplace equation is warranted.
8) Ground-based non equilibrium experiments are being developed. These experiments are considerably more complicated than the equilibrium experiments. However, our preliminary results indicate that a flight experiment under non equilibrium conditions is also warranted.

The paper "Determination of the Dispersion Constant in a Constrained Vapor Bubble Thermosyphon" received the Best Paper Award at the 1995 ASME/JSME Thermal Engineering Conference. We believe that this independent evaluation is a good indication that we are both moving in the correct direction and obtaining excellent results. An expanded version of this paper was published by the AIChE Journal.

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BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals

Printed: 28 FEB 1996
**Proceedings**

**Presentations**

Study of Two Phase Flow Dynamics and Heat Transfer at Reduced Gravity

Principal Investigator: Prof. Larry Witte
University of Houston

Co-Investigators:
McQuillen, J.B.
NASA Lewis Research Center (LeRC)

Task Objective:
The objective of this study is to develop and experimentally verify theoretical models that predict gas-liquid flow regimes and their characteristics in reduced gravity.

Task Description:
Reduced-gravity experiments will be conducted in NASA aircraft to measure two-phase flow parameters for a range of tube diameters, gas and liquid flow rates, and fluid properties. The gas phase for the experiments will be air; the liquids to be employed are water, water-glycerin mixtures, and water-zonyl mixtures. A theoretical modeling effort will be integrated with the experimental efforts.

Task Significance:
The purpose of this study is to achieve a better understanding—better predictability—of two-phase (gas-liquid) flow in pipes to assist in the design of space-based power and thermal management systems and of the terrestrial-based nuclear power plants and oil and natural gas pipelines.

Progress During FY 1995:
Previous experiments included testing a 2.54 cm inner diameter tube in the reduced gravity environment aboard the NASA JSC KC-135. This work has been centered on experimental and modelling studies of the characteristics of two-phase flows in microgravity. In addition to high speed photographs of the flows, electronic measurements of void fraction, liquid film thickness, bubble and wave velocity, pressure drop and wall shear stress have been made for a wide range of liquid and gas flow rates. The effects of liquid viscosity, surface tension and tube diameter on these flows were assessed. From the data collected, maps showing the occurrence of various flow patterns as a function of gas and liquid flow rates were constructed. Normal gravity two-phase flow models were compared to the results of the microgravity experiments and in some cases modified. Models were developed to predict the transitions on the flow patterns maps. Three flow patterns, bubble, slug and annular flow, were observed in microgravity. These patterns were found to occur in distinct regions of the gas-liquid flow rate parameter space. The effect of liquid viscosity, surface tension and tube diameter on the location of the boundaries of these regions was small. Void fraction and Weber number transition criteria both produced reasonable transition models.

An additional series of low gravity tests were conducted aboard the NASA JSC KC-135 in October 1994. The focus of these tests were to obtain heat transfer coefficient measurements using a sub-cooled liquid, sensible heat transfer. These tests were conducted in a 2.54 cm. inner diameter test section using air and two liquids—water, and water-glycerin.

The test section was designed to obtain local, time-averaged heat transfer coefficients along the heated length as well as instantaneous coefficients that can be correlated with the motion of liquid waves or slugs. The test section consisted of a 56 cm nickel-plated copper tube, wrapped with etched-foil heaters. Resistance temperature elements (RTD's) were positioned between the heaters and the external tube wall to estimate the internal wall temperature. A thin-film thermocouple, capable of response times as short as 103 milliseconds, was mounted inside the tube near the exit. A film thickness probe just after the exit is used to correlate the temperature fluctuations with waves or slugs. A special mixer section is used after the heater test section to obtain the mixing-cup temperature. The heated section has been used in the UH laboratory to investigate heat transfer in falling films as a means of proving...
its effectiveness as well as collecting important additional data. Modelling of the heat transfer enhancement that occurs under interfacial waves as they pass over the surface has begun.

Additional tests were conducted in September 1995 to examine the effect of gas-liquid flows through contractions. The test section inlet diameter was 2.54 cm. and the outlet diameters were either 1.27 or 1.90 cm. Differential pressure was measured over the length of the test section, and liquid film thickness and void fraction was measured both upstream and downstream of the contraction.

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**Proceedings**


**Presentations**


Interactions Between Solidification and Compositional Convection in Alloys

Principal Investigator: Prof. M. G. Worster
Northwestern University

Co-Investigators:
Davis, Prof. S.H.
Northwestern University

Task Objective:
The project aims to quantify the effects of convection on the structure and composition of cast alloys. Particular attention is to be focused on the form and influence of convective flows through the interstices of mushy layers during solidification.

Task Description:
Combined experimental and theoretical studies will be undertaken. The laboratory experiments will involve the solidification of aqueous salt solutions, as representatives of general binary systems. The theoretical studies will employ linear and nonlinear stability theory, asymptotic and numerical methods in the development and analysis of predictive mathematical models.

Task Significance:
This study of fluid dynamics during solidification will aid the design of improved casting procedures (e.g., for the manufacture of high-performance turbine blades). Additionally it will improve our understanding of air-sea interactions in polar regions, where the formation of sea ice is a dominant contributor to the global heat budget, leading to better climate modelling and prediction.

Progress During FY 1995:
A study of the evolution of nonlinear perturbations to a growing mushy layer was completed yielding a comprehensive set of coupled amplitude equations, which were used to predict the modes of convection in a mushy layer and their stability. The conditions for absolute stability have been determined in terms of various physical parameters of the system. A new oscillatory mode of convection that we discovered has been analyzed in detail, which has elucidated important interactions between convection and solidification in mushy layers that had not previously been comprehended. Extensive laboratory experiments have been performed to study the formation and evolution of sea ice. The data from these experiments is being used to guide the development of predictive mathematical models.

Students Funded Under Research:

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BS Degrees: 0
MS Degrees: 0
PhD Degrees: 1

Task Initiation: 1/93 Expiration: 12/95

Project Identification: 962-24-05-63
NASA Contract No.: NAG3-1405
Responsible Center: LeRC

Bibliographic Citations for FY 1995:

Journals


II. MSAD Program Tasks — Ground-based

**Discipline:** Fluid Physics

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*Nucleation and Chiral Symmetry Breaking under Hydrodynamic Flows*

**Principal Investigator:** Dr. Xiao-lun Wu  
**University of Pittsburgh**

**Co-Investigators:**

No Co-I's Assigned to this Task

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**Task Objective:**

The aim of this research is to investigate nucleation and chiral symmetry breaking under shear flow. It has been proposed that the symmetry breaking is a result of a strong first order phase transition coupled to a macroscopic, velocity field. The process, often referred to as autocatalysis, is considered as one of the key steps for chemical and biological evolutions. However, its very existence remains elusive.

**Task Description:**

An individual molecule of sodium chlorate (NaClO$_3$) is symmetric, but its crystal form shows distinctive optical activity. If a solution of sodium chlorate is left undisturbed, hundreds of crystals slowly form as the solvent evaporates. If these crystal are examined under crossed polarizers, one finds that half are left-handed and the other half are right-handed. Equal percentages of the two species indicates that the energy barrier for the nucleation is the same for the different handedness. It was discovered by Kondepudi *et al.* that under gentle stirring condition the crystallization seems to proceed in one direction, favoring either all crystals being right-handed or left-handed for each individual experiment. The optical purity in each experiment is greater than 99%. However, if an ensemble of experiments is carried out under the same conditions, one again finds an equal distribution of left and right-handed species. This remarkable observation indicates that the system undergoes a transition from a totally symmetric state to a totally asymmetric state simply by the introduction of a hydrodynamic flow.

The current experiments are designed to probe two important aspects of the above observations: (1) We would like to know if the transition from the symmetric state to the asymmetric state is an abrupt transition or a smooth one, as the flow parameters are varied; and, (2) To observe the existence of autocatalysis it will be useful to find correlations between the local order parameter $O=ln_1-n_R/ln_R+n_L$, averaging over some spatial scale $l$, and hydrodynamic flow patterns in the system. Here $n_L$ and $n_R$ are the number densities of left- and right-handed crystals on the scale $l$. Low Reynolds number hydrodynamic convections, such as Taylor-Couette flow and Rayleigh-Benard convections, provide fertile testing grounds. In these hydrodynamic systems, the flow consists of many local coherent structures, rolls and eddies, and the convective mixing is most effective within these coherent structures.

**Task Significance:**

Effect of hydrodynamic convection on phase transitions is a significant problem not only for basic science but also for technology. Microgravity provides a unique environment for which some of the above theoretical ideas can be tested. Here minimization of sedimentation is crucial for both the experimental observations and for the theoretical analysis. This study will enrich our general knowledge about nucleation and growth under hydrodynamic flow and may be exploited to produce chirally pure materials.

**Progress During FY 1995:**

Over the past year we have carried out extensive measurements aimed at clarifying the physical origin of secondary nucleation in supersaturated sodium chlorate solution under controlled shear flow. Earlier experiments by Kondepudi and by ourselves clearly indicate that explosive growth of these secondary nuclei is responsible for the symmetry breaking. Our new results indicate that mechanical damage to the incipient nuclei is the dominate feature of chiral symmetry breaking, and the secondary nucleation is therefore not spontaneous, contrary to early speculations. We noted that the effect of contact interaction between nuclei and stirrer has been previously investigated; yet the severity of the effect has not been fully recognized. By varying the shear rate continuously and
using samples with different amounts of solutions, we showed that the degree of chiral symmetry breaking can be approximately tuned. Moreover, when the flow is generated by means of electric convection, rather than mechanical stirring, no chiral symmetry breaking can be observed.

The second issue of concern for this experiment is the mixing effect due to hydrodynamic convection. As noted in our previous report, hydrodynamic convection is a significant problem in a wide range of phenomena concerning transport of pollutants in geostrophic flow and heat transfer in turbulence, as well as phase transitions in fluids. In light of this we have spent a considerable effort in investigating how a passive scalar, any physical quantity (such as the distribution of a dye, temperature or nucleated crystals) that is strongly effected by the flow yet has no or negligible influence on the velocity field itself, is dispersed in a chaotic flow. To simplify the problem, the flow is confined in a plane using very thin liquid (soap) films that are freely suspended. An ingenious technique, similar to that of three-dimensional Couette cell, has been developed to drive the liquid films in a controlled fashion. Both laminar and chaotic flows can be generated using this apparatus. In the chaotic flow regime, it was observed that film thickness variations are coupled strongly to the fluctuating velocity field. A semi-quantitative calculation suggests that film thickness can be treated as a passive scalar. The measured structure function S(k), which is the Fourier transformation of two-point thick-thickness correlation function, is in remarkable agreement with Batchelor's calculation for small scale structures of a passive scalar subject to turbulence flow. The result of this work has been recently published in Phy. Rev. Lett. A more extensive work which includes statistical properties of velocity fluctuations in 2D Couette flow has just been submitted to Phy. Fluids.

The apparatus that we built not only allows us to study flow in two dimensions, but also allows us to characterize physical properties of liquid films. Among various flow parameters, the shear viscosity is perhaps one of the most important ones yet it is seldom measured. The shear viscosity measurements gave rise to a number of surprises that were not accounted for by current theory. The film viscosity work is to appear in Rev. Sci. Instr. We are confident that research using thin liquid films is rather new, and will stimulate further theoretical and experimental work in this area.

STUDENTS FUNDED UNDER RESEARCH:  
BS Students: 0  
MS Students: 0  
PhD Students: 1  

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals  


Presentations  

Oscillatory Thermocapillary Convection

Principal Investigator: Prof. Abdelfattah Zebib
Rutgers University

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
The main objective of this work is to investigate the influence of free-surface deflection on the onset of oscillatory thermocapillary convection in microgravity. The study is to investigate the flow instability of thermocapillary flow. The Hopf bifurcation will be studied using a numerical method with the consideration of free-surface deflection. In addition, the disappearance of the Hopf bifurcation will be investigated.

Task Description:
In the domain perturbation approach (valid for small capillary numbers \(Ca\rightarrow 0\)), time-dependent, two-dimensional combined buoyant thermocapillary motions in a rectangular cavity are computed using a second-order accurate finite-volume method. Two situations are investigated: The \(O(1)\) pure buoyant convection (with the Marangoni number \(Ma=0\)) is known to exhibit a Hopf bifurcation at some critical value of the Grashof number, \(Gr_{cr}\). Thus, by studying the combined thermocapillary-buoyant convection for values of

\[(Ma, \ Gr) \ near \ (0, \ Gr_{cr})\]

we seek to determine the stability boundary for onset of oscillatory motion and its nature in the limit of vanishing buoyancy, \(Gr=0\). Both positive and negative values of \(Ma\) are considered. In the second problem we consider pure thermocapillary driven convection (\(Gr=0\)). The solution to the zeroth order system is known to be stationary. Higher order effects are unknown and will be investigated.

Task Significance:
These studies can contribute to our fundamental understanding and potential control of thermocapillary instabilities. Detailed information of thermocapillary flows can be obtained through this comprehensive numerical study. Mechanisms for controlling the flows will be identified. Thus the fluid management and heat transfer processes will be better controlled in space.

Progress During FY 1995:
From the numerical study, it was found that thermocapillarity stabilizes when it acts in support of buoyancy. On the other hand, thermocapillarity destabilizes for small \(Re\) but stabilizes for moderate \(Re\) when thermocapillarity acts in opposition to buoyancy. The energy-like stability analysis shows that thermocapillarity tends to smooth out intense shear layers caused by buoyancy.

The PI has demonstrated that thermocapillary convection of small Prandtl number fluids is steady for a range of the Marangoni number, \(Ma\). It is also shown that surface deflection has a small effect on the motion as expected for small Capillary numbers.

Stability boundaries in the \(Ma\) were computed - A plane for transition to oscillatory convection in a rectangular cavity of aspect ratio \(Ar\) assuming a nondeformable interface. It was reported that the boundaries move in the direction of increasing \(Ar\) and \(Ma\) as the Prandtl number is decreased. A study based on both energy and linear stability methods to help understand these transitions is planned.
II. MSAD Program Tasks — Ground-based

Discipline: Fluid Physics

Work is in progress to determine stability boundaries for bifurcation to time-dependent convection in rectangular cavities with nondeformable free surfaces. These stability boundaries are computed in the Reynolds number - Aspect ratio (Re - Ar) plane for different Prandtl numbers. From the results we have obtained so far, we find that as Pr decreases, the critical value of Ar becomes larger. We are also conducting energy-type analysis to understand the mechanism of transition.

**STUDENTS FUNDED UNDER RESEARCH:**
- BS Students: 0
- MS Students: 0
- PhD Students: 1

**TASK INITIATION:** 2/93  **EXPIRATION:** 2/96

**PROJECT IDENTIFICATION:** 962-24-05-49

**NASA CONTRACT NO.:** NAG3-1453

**RESPONSIBLE CENTER:** LeRC

**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Journals**


Superfluid Transition of $^4$He in the Presence of a Heat Current

Principal Investigator: Prof. Guenter Ahlers  
University of California, Santa Barbara

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
The objective of this project is to study the superfluid transition in a heat current. One issue which we are addressing is whether the superfluid transition remains continuous in the presence of a heat current. A second objective is to make measurements of the effective conductivity of the system very close to but slightly above the transition temperature $T_\lambda$ as a function of the current.

Task Description:
Theoretical work by Onuki has predicted that the transition will be hysteretic. We are looking for this hysteresis in a finite current. Onuki's theory does not take the effect of gravity into consideration, and it is not clear to what extent the gravitationally induced inhomogeneity will hide the predicted effect. Recent calculations by Haussmann and Dohm have indicated that a nonlinear range of parameter space should be accessible where the conductivity will depend upon the current. This range will be exceedingly close to $T_\lambda$ where the ultra-high resolution thermometry developed previously in our laboratory will be essential, and where gravity effects will play an important role.

Task Significance:
We expect that our earth-bound measurements will yield information about possible advantages to be gained from microgravity experiments. We will have to determine whether gravity effects completely obscure the nonlinear regime, thus necessitating microgravity experiments in order to make these nonlinear effects observable, or whether useful information can be obtained in an earth-bound laboratory.

Progress During FY 1995:
Since January 1, 1995 we have continued to study the finite heat current effect on the thermal conductivity of $^4$He near the lambda transition. Here we briefly describe the problem, and then outline the progress we have made.

The superfluid transition of $^4$He has long been a testing ground for theories of critical phenomena. Particularly interesting have been the transport properties. When the transition temperature $T_\lambda$ is approached from above, the finite conductivity $\lambda$ of the fluid diverges. The precise behavior of $\lambda$ as a function of $t = 1 - T/T_\lambda$ has been studied in detail both by experiment and theoretically. As $T$ is decreased below $T_\lambda$, the effective conductivity remains infinite because the heat current $Q$ is carried by superfluid counterflow. The focus of recent work has been on the behavior as $Q$ is increased beyond the point where linear response theory applies. The properties of phase transitions under such non-equilibrium conditions are relatively unexplored, even though they are an important problem in condensed matter physics. It is well known that thermal gradients in the superfluid phase can be established by a sufficiently large $Q$ due to the generation of vortices. At even larger $Q$, it is expected that the superfluid counterflow should become (thermodynamically) unstable even if vortices are absent. A number of early attempts have been made to describe this instability theoretically, but experimental evidence for it remains scarce.

Recently, a quantitative prediction, based on the renormalization-group theory, of the line $T_\nu(Q)$ of absolute instability of the superfluid in the T-Q plane has been made by Haussmann and Dohm. This line has much in common with the spinodal line of equilibrium first-order phase transitions. When approaching it from below, one should not expect to reach it because fluctuations will induce a transition at a lower temperature, say at $T_\nu(Q)<T<T_\lambda$ (Q). However, there is no theoretical prediction of $T_\nu(Q)$ and about the nature of the state which will be reached beyond it.
With support from NASA, we obtained experimental measurements of the width and thermal resistance of an intermediate dissipative region in the range T_c<T<T_x(Q). The thermal resistance is much larger that expected from the dissipation due to vortices, but still much smaller than typical thermal resistances in the normal phase above T_x(0). Let us consider a typical example for Q ~ 40 μW/cm². Proceeding from high T, the resistivity plummets towards zero near T_x(Q) with T_x(Q) - T_c ~ 5 μK. In the new dissipative region the resistivity is over an order of magnitude smaller than it is a μK or so above T_x(Q). However, the resistivity expected from mutual friction (vortices) is several orders of magnitude smaller than the measured resistance.

We made measurements in five different cells with different spacings between parallel top and bottom plates. A consistent picture has emerged from them and from previous measurements by others. It is as follows: As the superfluid transition is approached from below in the presence of a heat current, vortices are generated above a critical temperature relatively far below T_x. Although in principle thermal gradients are associated with the vortices, these gradients are relatively small. At a temperature somewhat below T_x(Q), the superfluid counterflow which carries the heat current Q becomes thermodynamically unstable and a new phase is entered. The nature of this phase is not very well known, but we find that it has a thermal conductivity much smaller than that of the superfluid state with vortices, but an order of magnitude larger than that of the normal fluid. This dissipative state persists until the temperature T_x(Q) of absolutely unstable superflow is reached. At that point the thermal resistivity rises dramatically to values characteristic of the normal fluid.

Our measurements have a direct relationship to the NASA-sponsored microgravity experiment DYNAMX, which hopes to explore the behavior of the superfluid transition in the presence of a heat current at very small currents where gravity plays an important role. We hope that our results will be helpful in choosing the detailed parameter ranges of DYNAMX.

A paper based on our work will be submitted to Physical Review Letters in the near future.

**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Journals**


**Presentations**


Liu, F.-C. "The superfluid transition of 4He under non-equilibrium conditions." Condensed-Matter Seminar, University of California, Santa Barbara, Santa Barbara, CA, January, 1995.

Liu, F.-C. and Ahlers, G. "A vortex phase near the lambda transition in 4He." APS Meeting, San Jose, CA, March 1995.

Microgravity Test of Universality and Scaling Predictions Near the Liquid-Gas Critical Point of $^3$He

Principal Investigator: Dr. Martin B. Barmatz  
Jet Propulsion Laboratory (JPL)

Co-Investigators:
- Hahn, I.
- Israelsson, U.E.
- Rudnick, J.
   
Jet Propulsion Laboratory (JPL)
University of California, Los Angeles

Task Objective:
The objectives of this task are to test the universality and scaling laws at the liquid-gas critical point of $^3$He in a microgravity environment. The task objectives will include 1) precision measurements of the isothermal compressibility along the critical isochore to determine the critical exponent and 2) precision measurements of the constant volume specific heat along the critical isochore to determine the critical exponent $a$, and 3) sound attenuation and dispersion measurements to test dynamic scaling theories.

Task Description:
Theories describing the behavior of thermophysical properties near critical points were developed using the concept of scaling laws. These models led to the definition of universality classes where critical points of the same class are predicted to have the same critical exponents. Efforts to validate the scaling law predictions near a liquid-gas critical point in ground-based laboratories are limited due to the gravity-induced vertical density gradient associated with the divergence of the isothermal compressibility. This density gradient becomes appreciable as the critical point is approached, leading to a significant smearing of the transition. Calculations have shown that in a microgravity environment ($10^6$ g) accurate specific heat and isothermal compressibility measurements could be obtained two orders of magnitude in reduced temperature closer to the critical point. Techniques are being developed for the simultaneous measurement of both static (specific heat, sound velocity, and compressibility) and dynamic (sound attenuation and dispersion) properties. These studies will require accurate measurements of pressure ($\Delta p/p \sim 10^{11}$), density ($\Delta n/n \sim 10^8$), and temperature ($\Delta T/T \sim 10^8$). These simultaneous measurements in microgravity should provide a very stringent test of theoretical predictions.

Task Significance:
The ability to perform these simultaneous measurements in microgravity should provide a very stringent test of the universality predictions.

Progress During FY 1995:
During this last year, we designed and fabricated a heat capacity cell for the $^3$He critical point experiment. This cell has three capacitive density sensors for measuring the density of the fluid and for leveling the cell in a gravitational field. We are currently mounting the cell on the high resolution thermometer stage of the thermal control system. For thermal control, we have implemented a new fuzzy gain scheme in a proportional-integral-differential (PID) controller. Computer simulations show that the new controller settles faster with less overshoot than a regular PID controller. Testing is now in progress with the real system containing high resolution thermometers. We have also collaborated with Dr. Ulf Israelsson to evaluate a high resolution pressure gauge for compressibility measurements near the critical point that is based on silicon micromachining technology.
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**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

*Proceedings*
II. MSAD Program Tasks — Ground-based

Discipline: Low Temperature Microgravity Physics

Measurement of the Heat Capacity of Superfluid Helium in a Persistent-Current State

Principal Investigator: Dr. Talso C. Chui
Jet Propulsion Laboratory (JPL)

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
The objective of the task is to detect any changes in the heat capacity of helium as result of superfluid flow very near the superfluid transition temperature.

Task Description:
The flow in the form of persistent current will be created in a toroidal-shaped calorimeter. The heat capacity is then measured from below to above the transition, where the persistent current will decay to zero. The heat capacity will subsequently be remeasured below the transition to detect any difference. If the experiment shows that the heat capacity is different with superfluid flow, then a space experiment can be designed to map out the heat capacity curves as a function of temperature and superfluid velocity.

Task Significance:
The results will be compared to the renormalization group (RG) theory, which has recently been applied to calculate the expected behavior. Although RG theory has been remarkably successful in explaining many properties of matter near a phase transition, the effect being studied here is a unique manifestation of quantum properties of superfluid helium. The theory developed to explain this involves a way to combine RG theory and quantum mechanics through the use of the Josephson relation. The experiment will examine whether such an extension of RG theory will correctly describe nature.

Progress During FY 1995:
Recently there was new experimental evidence supporting the idea initially proposed by us that in heat flow experiments near the lambda transition, the first rise in temperature is associated with vortex creating of the type proposed by Langer and Fisher to explain the intrinsic critical velocity observed by Clew and Reppy in a superfluid gyroscope. If this interpretation is correct, it would affect the experiment design significantly. The real shift in the lambda transition temperature as predicted by the renormalization group theory (RG) cannot be easily reached.

Based on these developments, the emphasis of the experiment has shifted to charting the region of experimental interest where the RG prediction can be realized. A thermal conductivity cell has been fabricated for this purpose. The apparatus is currently in operation with high resolution thermometers capable of resolving temperature to $5 \times 10^{-11}$ K/Hz$^{1/2}$.

We have also performed theoretical studies to find ways to reach the critical velocity predicted by RG theory. We found that in a cylindrical flow the RG critical velocity can be reached. One obvious example that this is true is the case of superfluid flow near the core of a vortex where the flow velocity increases as $1/r$ until the RG critical velocity is reached, where the fluid becomes normal. This process would give a reasonable vortex core radius of about 1.6 times the correlation length. We are currently performing theoretical studies to map out the phase diagram of rotating helium in small cylinders.
II. MSAD Program Tasks — Ground-based

Discipline: Low Temperature Microgravity Physics

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BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Presentations
Nonequilibrium Phenomena Near the Lambda Transition of $^4$He

Principal Investigator: Dr. Taiso C. Chui
Jet Propulsion Laboratory (JPL)

Co-Investigators:
Israelsson, Dr. U.E.
Jet Propulsion Laboratory (JPL)

Task Objective:
The objective of this project is to obtain information on the order-parameter relaxation time in superfluid helium near the lambda transition.

Task Description:
The experiment will measure the heat capacity of superfluid helium near the lambda transition under a pressure oscillation at variable frequency. There will be a small change in the averaged heat capacity when the frequency is increased above the reciprocal of the order-parameter relaxation time, thus allowing a measurement of the relaxation time. The proposed technique is well suited for performance in space where close approach to the transition is possible without the smearing effect of gravity induced inhomogeneity. Since the relaxation time is predicted to slow down considerably near the transition, getting closer to the transition will avoid problems on earth associated with extremely fast relaxation.

Another approach to be attempted is to measure the temperature change in helium following a sudden application of pressure. This approach requires the use of a very fast bolometer.

Task Significance:
The order-parameter relaxation time is an important parameter characterizing all dynamic processes in a phase transition. Accurate measurement of this quantity will allow a stringent test of the dynamic renormalization group theory which can be applied to predict the behavior of this quantity with no adjustable parameters.

Progress During FY 1995:
Parts have been fabricated for the experimental probe. This probe is a duplicate of one that is currently in service for another project. A new post doctoral associate has joined our group. Detailed design of the experiment has begun. Some calculations were performed to understand thermodynamics of helium under a sudden pressure quench.


BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Presentations
Task Objective:
The objective of this research is to study the nucleation of quantized vortices in helium II by investigating the behavior of rotating droplets of helium II in a reduced gravity environment.

Task Description:
Two methods well-suited for levitating the helium drop in the near vacuum environment are electrostatic and/or magnetic levitation. A pure electrostatic scheme requires active feedback control, while a purely magnetic levitation requires large fields. A hybrid system is probably the best choice. Rotation can be accomplished by coupling to a charge distribution on the drop surface. We will initially use purely electrostatic levitation for studying drops. The required charging of the drops can be accomplished by forming the drops around a sharp electrode tip held at a high voltage. Film flow of helium II can be utilized to create drops at the bottom of a suitable container which can be filled by a fountain pump and situated above a pair of capacitor plates having an appropriate voltage difference between them.

Task Significance:
Nucleation phenomena, in general, are fundamental to many fields of physics and engineering. In the case of a rotating superfluid drop, it will be possible to produce a state of zero nucleation, analogous to growing a perfect defect-free crystal. It should also be possible to add a controlled impurity to cause nucleation of a quantized vortex line in the drop. At low enough temperatures, this nucleation will be a pure quantum mechanical tunnelling phenomenon. At higher temperatures it should be possible to see thermally activated nucleation taking over, for a demonstration of nucleation under more familiar classical conditions. In conventional systems it is evident that vortex lines come from some preexisting source, probably vortices trapped by pinning sites on the walls. While this kind of source of vorticity is undoubtedly important, it is not as fundamental as the "extrinsic nucleation" problem where vortex line appears when none was present before.

Progress During FY 1995:
We have investigated the placement of positive charge in superfluid helium drops and the means of levitating positively charged drops. We have succeeded this past year in suspending drops of superfluid helium, up to 1mm in diameter, in a vertical electric field between parallel plates. The drops are positively charged, as first determined by their deflection as they pass through vertical plates having prescribed potentials. The actual charges are positive helium ions produced in a controlled corona discharge using a normal point-plane geometry. The superfluid drops are simultaneously formed and charged at the positive point electrode via electron-atom collisions which occur in the forming drop. The drops are accelerated downward through a small hole in the cathode plate, which serves as the upper levitation plate. An additional plate, displaced 1 cm below the cathode is kept at a positive potential.

To make further progress, it was necessary to place a large ballast resistor in the external charging circuit to inhibit the formation of arcs and, further, to restrict the dark discharge current in the vapor to an acceptable value (below 10 nA). Drops with sufficient charge-to-mass ratio have been observed to drift laterally out of the field in the absence of additional electrodes and a feedback mechanism.
To correct the above problem, we are currently working on an electrodynamic levitator which utilizes parametric modulation of the electric field to facilitate levitation of charged drops. With this type of levitator, there is a time-averaged force which acts on the charged drop, and for certain parameter regimes levitation is achievable even in the absence of a constant electric field. The electrode surfaces are hyperbolic in cross-section and are constructed of clear epoxy, which has been coated with a thin layer of gold. The levitator we have constructed has three electrode surfaces. An upper and lower hyperbolic surface are kept at a constant potential so that the weight of the drop can be partially compensated for. The middle electrode is a hyperbolic torus to which is applied an alternating voltage of tunable frequency and amplitude. The correct values for these quantities are determined from the solution to a Mathieu equation. We have constructed an AC source with adjustable frequency and amplitude by amplifying the output of a HP signal generator using an audio amplifier and then inputting the amplified signal to a step-up transformer.

We have investigated additional means of charging that may have advantages for this project. A pulse generator, which can generate 200 ns voltage pulses of amplitudes between 4 kV and 10 kV, is connected to a fine tungsten tip which is directed vertically just above a free surface of liquid helium. We have succeeded in producing charged drops in this manner which are sprayed upward. This has the advantage of producing charged drops with small velocity near the center of the levitator. On the other hand, the amount of charge produced by this transient pulsing is less than that achieved by the DC corona and this may limit the size of drop which we can suspend in our levitator.

During the past year Greg Bauer has received his Ph.D. for work based on this project. His thesis (June, 1995) is titled "The behavior of a quantized vortex in a drop and on a sphere."

**STUDENTS FUNDED UNDER RESEARCH:**

| BS Students: | 2 | BS Degrees: | 0 |
| MS Students: | 0 | MS Degrees: | 0 |
| PhD Students: | 1 | PhD Degrees: | 1 |

**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Journals**

**Presentations**
Kinetic and Thermodynamic Studies of Melting-Freezing of Helium in Microgravity

Principal Investigator: Prof. Charles Elbaum
Brown University

Co-Investigators:
Kosterlitz, J.M.
Brown University

Task Objective:
The objective of this project is to study, experimentally and theoretically, the effects of gravity on the melting-freezing transitions, including kinetic processes and the equilibrium shape of solids. The research is carried out on helium, whose unique properties render such investigations possible on a time scale consistent with experiments in space, under microgravity conditions. Indeed, morphological changes of the solid-liquid interface (i.e., the "surface" of helium) generally occur fast enough to satisfy the time constraints mentioned above.

Task Description:
An optical system with special lighting applied to a growth cell contained in a liquid helium dewar allows viewing of the solid-liquid interface (SLI) and of crystal shapes. Rapid image capture equipment allows recording of the evolution of the SLI and of the crystal shapes in response to changes in temperature and pressure.

Task Significance:
These studies are addressing a number of fundamental questions, especially as they relate to the effects of gravity. These questions include the kinetics of first order phase transitions, the critical behavior in the evolution of crystal shapes as they approach equilibrium, faceting-roughening phenomena on various surfaces, relative and absolute values of interfacial free energy for different crystal faces, and the minimization of a system's free energy subject to various constraints. Furthermore, many applications should benefit from a deeper understanding of the above phenomena, among them crystal growth, surface configurations, sintering, and surface reactivity.

In the course of this research we discovered a new phenomenon of nucleation in a first order phase transition, whereby a nonequilibrium phase nucleates and persists in a metastable state. This nucleation occurs from overpressurized superfluid helium-4 into the body-centered cubic (BCC) solid phase, in preference to the expected equilibrium hexagonal close-packed (HCP) phase. These observations provide a new perspective on the kinetics of first order phase transitions occurring near the triple points, and in systems having two or more phases with modest free energy differences.

We are directing a part of our research effort to this newly discovered phenomenon.

Progress During FY 1995:
Nucleation and growth processes in first-order phase transitions have been the subject of extensive studies for many years. These studies are motivated by both the scientific and technological importance of such transitions. While the macroscopic, thermodynamic descriptions of such phenomena as, for example, freezing-melting and condensation-evaporation are of long standing, many of the detailed kinetic and morphological aspects still remain to be fully elucidated. In the technological domain, the success of many industrial processes depends on an understanding of first-order phase transitions. These processes cover a very wide range, which includes such diverse areas as the casting of steel, the growth of silicon crystals for semiconductor based devices, and the manufacture of pharmaceutical products. Our discovery of the nucleation of a non-equilibrium phase may have an important impact on the way many of these processes are initiated and controlled.

A first-order phase transition usually involves the nucleation and growth of a new stable phase rendered unstable by
changes in intensive variables, like temperature or pressure. We observed nucleation and phase transitions in superfluid $^4$He in which a metastable solid phase nucleates and grows from the overpressured liquid in preference to the stable phase. Ultimately, the stable phase nucleates from the superfluid independently and the metastable phase disappears. While the persistence of a phase in a metastable state is fairly common, the type of nucleation into a phase out of equilibrium described here has not been previously reported.

We account for these events in terms of interfacial free energy differences and resulting nucleation probabilities for different values of supercooling. Both classical and quantum mechanical evaluations of nucleation probabilities give similar results.

**Theoretical Effort**

The theoretical effort has been in constructing a model to explain the dramatic differences observed when a $^4$He crystal is grown from the superfluid or normal fluid. When grown from the superfluid, the crystal, either in the bcc (body-centered cubic) or hcp (hexagonal close-packed) phase, is observed to flow under gravity very much like a drop of very viscous fluid. On the other hand, when the hcp solid is grown from the normal fluid at a higher temperature and pressure, it behaves like a normal rigid crystal and does not flow under gravity. We assume that the crystal in contact with the fluid has the same properties of rigidity and that the differences are due to the different natures of the fluids.

We have constructed a model of a superfluid in contact with a solid. Since the experiments have been carried out at temperature $T > T_\text{r}$ where $T_\text{r}$ is the roughening temperature of the crystal, the crystal has a rough surface and is described by a scalar field $\phi(r)$ where $\phi = +1$ in the solid and $\phi = -1$ in the fluid. The superfluid is described by a complex field $\psi = |\psi| \exp(i\theta)$ where $|\psi|^2 = \rho_s$, the superfluid density, and the superfluid velocity $\nu_s = (S/m)\theta$. To obtain the two-fluid hydrodynamics in the liquid phase, a conserved field $q$, which is the linear combination of mass density and entropy coupling to the superfluid velocity $\nu_s$ is also included. The free energy of the system $F(\psi, q, \phi)$ is

$$F(\psi, q, \phi) = [(\partial^2 r ([1/2])|\nabla \psi|^2 + (1/2) K_\psi(|\nabla \phi|^2 + f(\psi, q, \phi))]$$

where

$$f(\psi, q, \phi) = (1/2) r_0 \nabla^2 \psi + u_0 \psi \psi + (1/2) \alpha \psi \psi + \gamma q \psi \psi + (1/4) \beta (\phi^2 - 1)^2 + (1/2) \phi \psi \psi - \phi G \nu - \Delta \phi$$

We choose the phenomenological $r_0$, $u_0$, etc., so that the coupling term $(1/2) \alpha \psi \psi \psi$ leads to an effective quadratic term $(1/2)(r_0 + \alpha \phi)\psi \psi$ so that in the liquid $r_0 + \alpha = r_0 - \alpha < 0$ and in the solid $r_0 + \alpha > 0$ so that the liquid is a superfluid ($\psi \neq 0$) and the solid is not ($\psi = 0$). The driving term $-\Delta \phi$ with $\Delta > 0$ tends to drive the system to the solid phase, as does the term $-\phi G \nu$ where $G$ is proportional to the gravitational acceleration. Since the solid is about 10% denser than the fluid, such a term will tend to drive the solid downwards, as is required physically. In equilibrium, this $F(\psi, q, \phi)$ is to be minimized and gives a reasonable description of the equilibrium phase diagram.

To describe the evolution of the system from an initial state of pure superfluid, one must supplement this with some dynamical equations which reproduce the main features of the hydrodynamics of a superfluid liquid. A set of equations which do this are

$$M \psi M r = -2 \psi - \psi L (M \psi M \psi) + \eta_\psi$$

$$M q / M t = (\lambda_\psi L (M q M)) + 2 \psi \psi \psi \psi + \eta_\psi$$

$$M \phi / M t = -\psi \psi \psi \psi + \eta_\phi$$

where $\eta_\psi$, etc. are Gaussian distributed stochastic noises obeying the fluctuation dissipation theorem.

The above set of equations should be a reasonable description of the solid/superfluid system in a gravitational field as they include a first-order solid/liquid transition with the liquid being a superfluid with $\psi \neq 0$. The aim of this work in progress is to simulate this set of equations with initial conditions $\phi = -1$ so the system is in an unstable liquid phase and will evolve into the solid by the driving term $-\Delta \phi$. We shall nucleate a drop of solid on the boundary of the system and study its evolution and we believe that we shall see the solid undergoing apparent viscous flow under gravity. Presumably this flow is related to the previously observed phenomenon of...
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Discipline: Low Temperature Microgravity Physics

melting/freezing waves under gravity. We have checked that our equations do yield this wave phenomenon. We have also constructed a model for the growth of a solid from a normal fluid which includes gravity, advection and thermocapillary effects.

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PROJECT IDENTIFICATION: 962-24-07-13

RESPONSIBLE CENTER: JPL

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Presentations
Dynamics of Superfluid Helium in Low Gravity

Principal Investigator: Mr. David J. Frank
Lockheed Martin Missiles & Space Co.

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
The objective of this project is to simultaneously record the 3-axis acceleration time history and make a video recording of the position of superfluid helium (SFHe) in a test cell while in a low-gravity environment. This data can then be used as a benchmark for validation of a 3-D CFD simulation of SFHe flow behavior.

Task Description:
The first part of the project is to build a SFHe dewar and a support equipment package to allow operation of the dewar and recording of 3-axis accelerations and video images of the fluid motion on the DC-9. The dewar will have optical windows in the side to allow viewing of the liquid helium in the inner test cell. The data recording is intended to be digital data stored on a hard disk. The float package will be self-contained except for power and supply of liquid helium.

The completed dewar float package will be taken on the DC-9 for one or more flights to observe the motion of the liquid in low gravity. The recorded accelerations will be used as an input to a LMSC-developed CFD code that incorporates the two-fluid model of SFHe. The simulation output will be compared to the actual fluid motion recorded during the DC-9 flight to verify the accuracy of the computer mode.

Task Significance:
The final product of the project will be to have a CFD code for SFHe that has been verified in low gravity. This will allow predictions of SFHe behavior on future satellites such as SIRTF, GP-B, and AXAF with increased confidence in the accuracy of the simulation.

Progress During FY 1995:
Accomplishments

The refurbishment of the SFHe dewar that will be used in the DC-9 low gravity flights has been completed. A complete new inner SFHe tank has been constructed and assembled. The tank has windows in it that allow a video camera to record the motion of the fluid. Thermometry was installed inside the tank to measure the temperature of the fluid. The dewar was built and subjected to the required proof pressure tests to qualify it’s use on the NASA Low Gravity DC-9 Aircraft. The dewar has been subjected to a number of tests at SFHe temperature to demonstrate it’s vacuum integrity and hold time. These tests were all successful. The dewar has been shipped to the Jet Propulsion Laboratory where it is being integrated with JPL's Low Gravity Aircraft Float Package. Once the integration is completed and a series of tests performed, the entire package with all the cryogenic ground support equipment will be shipped to NASA Lewis Research Center.

Peter Mason of JPL and myself have visited the Lewis facility to familiarize the DC-9 personnel with the cryogenic test apparatus and operations. This will be the first cryogenic payload that they will be flying. Due to the cryogenic aspects of this experiment, there are unique requirements for servicing the experiment. A review of these operations with them showed that, even though they are out of the ordinary, they can accommodate us. It is expected that the experiment will be conducted in January 1996.
With the completion of the hardware phase of this program, I am concentrating on the fluid modeling aspects of the project. Tests were conducted in the laboratory at 1-g to measure the SFHe slosh frequency and damping in the same dewar that will be used in the low gravity tests. The fluid models are being exercised against the data. Two models are being used. The first is a CFD (computational fluid dynamics) code by FLOWSCIENCE called FLOW3D and the second is a modified version of FLOW3D that incorporates the "two fluid model" aspects of superfluid helium.

The 1-g simulations are not yet complete, but preliminary results are showing that the slosh frequencies are being predicted correctly and that the damping coefficients calculations still need some refinements. Optimizing computational mesh sizes and convergence criteria are still being investigated. The interesting result to date has shown the fluid in 1-g behaves like a Newtonian fluid in respect to slosh frequency and damping. This is further confirmation that low gravity tests are required to get data to verify models that are to be used for predicting the behavior of SFHe in zero gravity.

Plan

It is planned to conduct the low gravity tests in January on the NASA Low Gravity DC-9 aircraft. The motion of the fluid will be recorded. Following the flight, the data will need to be reduced to get it into a format that is of use to this project. It is expected that this will take place during the month of February after the hardware has been returned to JPL. The data will be used to evaluate the CFD codes.

Due to a change in the computer resources, the CFD computer codes for this project need to transferred to another computer. Use of another computer at Lockheed will result in charges to the project that were not initially planned. Computer usage at Lockheed is now charged to the contract rather than treated as indirect costs. JPL has agreed to make their CRAY computer available. The new Lockheed computer costs and cost associated with using the JPL computer (i.e. cost of transferring to a new platform and licensing from FLOWSCIENCE) are being evaluated. In either case, it is expected that the project will be completed with the available funds. The costs associated with the build of the dewar were much lower then originally planned since JPL made use of some existing hardware. The remaining funds that were planned for construction of the dewar will be used in the computational modeling.

II. MSAD Program Tasks — Ground-based

Discipline: Low Temperature Microgravity Physics

Condensate Fraction in Superfluid Helium Droplets

Principal Investigator: Prof. J. Woods Halley
University of Minnesota

Co-Investigators:

Giese, C.
Campbell, C.
Goetz, K.
University of Minnesota
University of Minnesota
University of Minnesota

Task Objective:
The scientific goal of the proposed project is to obtain information about the condensate fraction in superfluid helium four by studying elastic scattering of helium atoms from a freely floating macroscopic sphere of the fluid.

Task Description:
During the third year we have completed experiments demonstrating the use of Cs-covered surfaces to partially suspend He for a ground based experiment. Our computational studies of various magnetic suspension methods have led to a design which is being built using neodymium iron boride magnets. We have completed preliminary application to the Florida State University National High Magnetic Field Facility for use of a magnet there for droplet suspension. Many body calculations of transmission rates in the experiment are being carried out numerically by graduate student Arun Setty.

Task Significance:
The condensate fraction of the superfluid helium wavefunction is the microscopic manifestation of Bose condensation which is universally believed to be the origin of the fluid's superfluid properties (as originally proposed by London more than 50 years ago). If successful, the experiment would be important because direct experimental study of the condensate fraction has proved extremely elusive. Only neutron scattering experiments give direct information, and interpretation of these has proved difficult.

Our basic idea is that, in a microgravity environment, it will be possible to do a tunneling experiment (analogous to a Josephson tunneling experiment in some respects) in order to study the condensate. We envision sending pulses of gaseous helium atoms at one side of a suspended sphere of superfluid helium four and detecting helium atoms emerging in coincidence from the other side of the sphere.

Progress During FY 1995:

1. Overview

The objective of this project is the completion of a ground based study of the scientific and technical feasibility of an experiment in which the presence and nature of the longrange quantum coherence (condensate) in superfluid $^4$He is detected. Pulses of gaseous helium will be fired at a suspended droplet of superfluid $^4$He and the resulting emission of helium atoms will be detected.

2. Theory

Theoretical results from the previous fiscal years are reported in references 1-4 and in last year's Progress Report. This work suggested the existence of a large effect but also clarified the need for a more complete many-body analysis of the problem. Analytical aspects of this analysis were completed in the 1994 fiscal year. This year numerical work has been undertaken to make the needed variational calculations with graduate student Arun Setty and postdoctoral visitor Sang Hoon Kim.

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In last year's progress Report we presented formal results which provided a framework for calculation of the time-dependence of the emission in the case of a pulsed incoming beam and a slab geometry, given knowledge of eigenstates of the N + 1-particle system including the superfluid and one incoming particle. To utilize these formal expressions we require approximate expressions for the most important of these eigenstates. During the last year we have been making variational Monte Carlo calculations of the wave functions describing such states. To make optimal contact with experiment at an early stage, we have elected to study solutions with boundary conditions containing an incoming, reflected and transmitted wave in a slab geometry. The systematics of the dependence of the transmission coefficient of such solutions on cross-sectional area, slab thickness, and incoming particle energy will already be useful guides to experiment, even without the full time-dependent analysis.

We are working with a variational wave function for these scattering states which depends on 5 parameters: the amplitude and phase of the transmission coefficient, the phase of the reflected wave, and two parameters characterizing the shape of the envelope of the incoming, reflected, and transmitted helium wave functions. We vary these parameters systematically to bring the result in close as possible to their known values: the energy and the values of the current at a set of points inside the slab of superfluid.

The numerical calculation of the current corresponding to this wave function has turned out to be a formidable task and has recently been completed. We are presently undertaking the optimization of the parameter set by the criteria just described. Preliminary indications are that a very large transmission coefficient will be obtained, consistent with our earlier speculations and models.

3. Suspension of Superfluid with Cesiated Surfaces

We are engaged in development of two types of ground based experiments. NASA Graduate Student Researcher M. C. Williams is carrying out the experiments at Minnesota to demonstrate the feasibility of a method for producing two parallel helium surfaces using the fact that cesium surfaces are not wet by superfluid 4He. During the past year, this experiment was successful in demonstrating the possibility of levitating a film of liquid helium in this way.

A capacitance bridge was used to measure the liquid level in a can containing a cesiated hole of 70 mm diameter in a platinum film in the bottom. We found that, consistent with theoretical predictions which we made, liquid at depths greater than about 2 mm ran through the hole at velocities consistent with critical velocities reported by others. However, a 2-mm thick film was suspended over the cesium-covered platinum hole at 1.2 K for more than two hours. We confirmed the result in a second run in which 1 mm of superfluid was suspended for a comparable time. In a second set of experiments, currently under way, the method will be used to measure the temperature dependence of the contact angle for the first time. In view of the success of this experiment, we are planning a version of the experiment for studying the condensate in a configuration in which the superfluid is a suspended slab. This, however, will require acquisition of a dilution refrigerator.

4. Magnetic Droplet Suspension

Magnetic suspension has often been suggested as a method for droplet suspension of liquid helium in a microgravity environment. Experiments at Brown University were successful this year in achieving magnetic suspension of helium droplets on Earth using superconducting magnets. As an alternative, NASA Graduate Student Researcher J. Schmidt of this group has initiated a study of the possibility of using permanent magnets for this purpose. If it can be achieved, this method would have great advantages with respect to simplicity and expense. It may even be possible to achieve suspension of helium droplets on Earth using permanent magnets. To study these possibilities, Mr. Schmidt developed computer codes for determining the optimum configuration of permanent magnets, which codes were orders of magnitude more efficient than previously existing software. A permanent magnet configuration was found which produces a field configuration which can suspend helium droplets. It consists of a pie-shaped configuration of magnets arranged so that the magnetization points inward in two dimensions toward a central point. We have shown that the resulting magnetic field configuration should be capable of suspending a small (order of 0.1 mm) superfluid helium droplet. A prototype of this magnet configuration is under construction using neodymium iron boride magnetic material and will be tested by suspending bismuth and liquid hydrogen.
Studies of suspended helium droplets at the National High Magnetic Field Facility in Tallahassee, Florida are also planned. We have made application to NHMFF for use of their facilities for two sets of experiments. In the first experiments we shall suspend superfluid helium droplets at dilution refrigerator temperatures; this has not yet been done and low temperature is required for our objectives. In the second set of experiments we shall carry out the helium atom transmission experiment.

**Students Funded Under Research:**

- BS Students: 0
- MS Students: 0
- PhD Students: 3

**Task Initiation:** 1/93  **Expiration:** 12/95

**Project Identification:** 962-24-07-14

**Responsible Center:** JPL

**Bibliographic Citations for FY 1995:**

**Journals**

**Proceedings**

**Presentations**
II. MSAD Program Tasks — Ground-based

Discipline: Low Temperature Microgravity Physics

Ultra-Precise Measurements with Trapped Atoms in a Microgravity Environment

Principal Investigator: Dr. Daniel J. Heinzen
University of Texas, Austin

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
Our task objectives for FY95 are to (i) demonstrate evaporative cooling of trapped atoms to nanokelvin temperatures, and (ii) confine these ultra-cooled atoms in an "optical box trap". Such ultracold atoms are crucially important for potential zero gravity applications because they dramatically reduce the required strength of the confining potential. In turn, employing a weaker confining potential reduces the perturbations to atomic transition frequencies introduced by the confinement and should allow for a dramatic increase in resolution and accuracy in atomic resonance experiments. These steps should bring us closer to our ultimate goal of realizing ultraprecise measurements with trapped atoms in a zero-gravity environment.

Task Description:
Evaporative cooling experiments will be carried out in a new apparatus that was recently developed in our laboratory. About $10^5$ atoms at a temperature of 10 microkelvins will be loaded into a magnetic atom trap. From there, they will be evaporatively cooled to temperatures in the nanokelvin range. The atoms will then be released from this trap into an optical box trap. In order to simulate microgravity conditions, the atoms will be levitated by an additional gradient field. The ultralow temperature of the evaporatively cooled atoms will significantly ease the requirements to generate a large-volume optical trap. This optical trap will be created by intersecting beams from single mode diode lasers, possibly using optical buildup cavities to enhance the diode laser power and to better define the laser mode. Zeeman resonance experiments on these trapped atoms may also be performed.

Task Significance:
Precision atomic resonance devices have tremendous practical and scientific importance. An important example is the atomic clock, which has widespread applications to navigation, geophysical measurement, and astrophysics. Precise tests of fundamental scientific principles such as time-reversal symmetry are also carried out with such devices. Substantial advances in the accuracy of these devices are therefore of great importance. One very promising avenue is to make use of ultracold, laser-cooled atoms. These atoms can move as much as 10,000 times more slowly than the atoms in conventional devices. This very low velocity can in principle substantially increase the accuracy of an atomic resonance device. This is because certain measurement errors are proportional to the velocity of the atoms. Unfortunately, gravity seriously limits the usefulness of laser-cooled atoms for this purpose, because it quickly accelerates the atoms back to high velocities. Thus, only in the gravity-free environment of space can the full potential of these ultracold atoms be realized. Our goal is to develop techniques that could be used to produce and store a large number of ultracold atoms in the gravity-free environment of space. If successful, atomic resonance devices based on these techniques could lead to dramatic increases in accuracy.

Progress During FY 1995:
During the past year we have built a new apparatus to evaporatively cool atoms to nanokelvin temperatures. With this new apparatus we have demonstrated loading of an optical trap with more than $10^6$ atoms at a temperature of less than 100 microkelvins. Experiments are in progress to further evaporatively cool these atoms from a magnetic trap. In a separate apparatus, we have demonstrated the generation of a TEM$_{01}$ mode laser beam in an optical buildup cavity inside a vacuum chamber. Use of such a cavity will allow us to efficiently generate a version of our "optical box" dipole force trap using only low-power diode lasers. Finally, we have carried out calculations of the "quantum control" of the cesium Zeeman sublevels using tailored RF magnetic fields. This new technique will allow us to achieve maximum sensitivity in a cesium electric dipole moment experiment.
II. MSAD Program Tasks — Ground-based

Discipline: Low Temperature Microgravity Physics

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**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Journals**


**Presentations**


II. MSAD Program Tasks — Ground-based

Discipline: Low Temperature Microgravity Physics

Dynamic Measurement Near the Lambda-Point in a Low-g Simulator on the Ground

Principal Investigator: Dr. Ulf E. Israelsson
Jet Propulsion Laboratory (JPL)

Co-Investigators:
Duncan, R.V.
Sandia National Laboratory

Task Objective:
The objective of this work is to perform dynamic measurements on a short cylindrical sample of helium very near the lambda-point in an effective gravity environment of about 0.01 g. Dynamic conditions will be created by passing a heat current through the sample. The effective low-gravity environment will be created by applying a magnetic field gradient which closely cancels hydrostatic pressure differences in the sample. Specifically, the reduced gravity conditions will enable a test of theoretical predictions of the effect of small heat currents on the nature of the lambda-transition and will allow probing of the interface region between co-existing normal and superfluid portions of the fluid. These measurements are not possible to perform in a regular lab environment on the earth due to the influence of hydrostatic pressure effects and the need to apply large heat currents to overcome hydrostatic effects, tending to perturb the fluid sufficiently to render the measurements questionable. The suppression of the lambda-transition due to heat currents will also be investigated at lower values of the heat current than possible in a one-g environment. The magnet will be procured from a magnet winding company and installed in a thermal platform under construction at JPL. Melting curve thermometers, which can operate well in strong magnetic fields, will be used for high resolution thermometry.

Task Description:
The magnet will be designed and constructed by a magnet winding company. An experimental cell will be constructed with attachment points for melting curve thermometers to enable high resolution thermometry to be performed in the high field conditions of the experimental cell. The melting curve thermometers will be constructed at Sandia under a sub-contract to JPL. A high performance thermal platform will have the experimental cell and the magnet installed into it for performing the measurements. A vibration isolated and magnetically shielded helium dewar will be used to cool the thermal platform in order to minimize noise generation and improve the fidelity of the collected data.

Task Significance:
Recent investigations of the influence of an applied heat current on the properties of helium near the superfluid transition have revealed many new phenomena. Agreement with theories based on scaled mean field calculations and dynamic renormalization group calculations is not good. The disagreement may stem from the fact that theories assume zero-gravity conditions, while experiments are performed in a one-g environment. To overcome the influence of gravity on properties near the transition in a heat current, large values of heat current are required which has detrimental effects on the very properties in need of study. It has also been predicted that imposition of a heat current will change the very nature of the lambda-transition from continuous to first order. Investigating these phenomena in a simulated low-gravity environment would enable lower heat currents to be used and would enable observation of phenomena washed out by gravity effects.

Progress During FY 1995:
During the last fiscal year, all of the components for the experiment were integrated and successfully tested at liquid helium temperatures. During this cooldown, a number of important milestones were reached. A magnetic field in excess of what is required to cancel gravity to the 99% level on the ground was successfully trapped in the magnet. All temperature sensors on the apparatus, including the melting curve thermometers, were calibrated. The operation of all sensors were verified to be within specifications even when the high field magnet was energized. In particular, the melting curve thermometers were shown to be unaffected by the extreme magnetic field environment inside the...
II. MSAD Program Tasks — Ground-based  Discipline: Low Temperature Microgravity Physics

apparatus. Also, a detailed performance study of the melting curve thermometers was completed, demonstrating that the science objectives can be achieved with the current design. We were able to demonstrate a noise level of less than 10kHz in a one Hertz bandwidth. Finally, data gathering routines were developed using Labview software and were used to take preliminary thermal conductivity measurements.

Unfortunately, two problems were identified in the initial cooldown. First, there was a small leak into the vacuum area which led to unacceptable variations in the parasitic leak into the helium cell. The leak was small enough to enable us to run the experiment as planned and develop detailed data-gathering routines which were successfully run, but the science data collected were unacceptable. Second, none of the side wall thermometers were functioning properly. Despite these problems we were able to demonstrate correct behavior of the system using applied heat currents in the 1 to 10 microWatt/cm² range. We were successfully able to ramp the top temperature of the cell in a controlled fashion at constant applied heat current such that the cell moved across the entire range of interest, i.e., from initially full of superfluid to full of normal fluid, and back down again.

Following the warm-up of the experiment, we corrected all the problems encountered during the initial cooldown in a satisfactory manner. Currently, the experiment has been successfully cooled down again, and we are in the early phases of preparing to gather useful data.

The modeling effort initiated in FY94 was also continued. The modeling was extended during the past year to study a wider range of heat currents, from 1 to 30 microWatt/cm². The model was also extended to study the effect of varying the thermal resistance between the helium in the cell and the temperature controlled top of the cell. Finally, the model was quantitatively compared to physical data taken in Prof. Ahlers’ lab. This comparison to physical data showed some discrepancies which can be attributed to the flow of heat through the cell’s side walls. During the coming year, we plan to extend the model to include the effect of the side walls.

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| PROJECT IDENTIFICATION: 962-24-04-09 | RESPONSIBLE CENTER: JPL |

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Larson, M. "Dynamic measurements near the lambda-point in a low-g simulator on the ground." International NASA Low Temperature Microgravity Physics Workshop, Pasadena, CA, March 1995.


Printed: 28 FEB 1996
II. MSAD Program Tasks — Ground-based

Discipline: Low Temperature Microgravity Physics

Atom Interferometry in a Microgravity Environment

Principal Investigator: Dr. Mark A. Kasevich
Stanford University

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
Our scientific objectives are: (1) to develop a rugged laser cooled source of atoms using DBR (distributed Bragg reflector) laser technology; (2) to use this source and these lasers to demonstrate coherent atom wavepacket manipulation techniques; and (3) to incorporate these techniques into an atom interferometer gravity gradiometer. We plan to experimentally study the performance of the prototype device to evaluate the feasibility of a space-based system.

Task Description:
In year one of the grant period we will develop the laser sources and apparatus needed for the proposed experiments. We will employ standard laser cooling and trapping techniques with atomic cesium to create the cold atomic source. Our first experiments, to be carried out near the end of year one, will focus on demonstration and characterization of the proposed coherent atom manipulation techniques. In year two we will demonstrate and characterize an interferometer accelerometer - first in a low sensitivity regime in order to study potential systematic phase shifts and subsequently in a high sensitivity configuration to explore the potential accuracy and resolution of the device. Since vibrational noise will severely hamper the performance of an Earth-bound accelerometer, we will switch to a more complicated gradiometer geometry which is far less sensitive to vibrational noise in the final stages of the proposed work.

Task Significance:
The convergence of recent advances in the field of laser manipulation of atoms with technological developments in the electronics/opto-electronics industry opens the possibility of a new class of experiments involving laser manipulated atoms in a microgravity environment. In the past five years, light-induced forces have been used to cool ensembles of atoms to temperatures below 1 μK, providing researchers with a novel source of ultra-cold atoms. These laser cooled sources have revolutionized experimental atomic physics and have led to new classes of precision time standards and inertial sensors. Application of these techniques in a microgravity environment could result in robust gravity gradiometers and gyroscopes with sensitivities exceeding current state-of-the-art devices by several orders of magnitude. Such instruments would have important applications in a number of fields. For example, satellite gradiometry studies yield important geophysical data concerning Earth and ocean dynamics. A satellite-borne accelerometer/gravimeter used in conjunction with the GPS system could be used to obtain highly accurate maps of the global Earth gravity field. In addition, techniques developed for satellite interferometer sensors might also have terrestrial applications in, for example, mineral/oil exploration and navigation. Finally, with minor modification, the techniques could be employed to develop atomic standards with unprecedented accuracy.

Progress During FY 1995:
1) We have developed, built, and tested the laser system for cooling and trapping atomic cesium. Using SDL DBR lasers at 852nm we have demonstrated that up to 108 atoms/sec can be loaded into a magneto-optic trap, and that the atoms can be cooled to temperatures below 10 microKelvin. Atoms were subsequently launched in a moving molasses configuration.

2) We have finalized the design and have nearly implemented the wavepacket manipulation laser subsystem. the system employs a high frequency acousto-optic modulator in an optical injection lock scheme to generate two high power, phase coherent, laser beams separated in frequency by 9.2 GHz. We ahve tested the system using a low frequency modulator.

Printed: 28 FEB 1996
3) We have implemented the data collection and computer controlled timing system. This system was tested during the initial laser cooling measurements.

4) We have designed a compact UHV vacuum system which will be used in the proposed gradiometry measurements.

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals

Presentations


Effect of Confinement on Transport Properties by Making use of Helium Near the Lambda Point

Principal Investigator: Prof. John A. Lipa
Stanford University

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
The objective of this project is to study the effect of confinement on the thermal conductivity of helium near the lambda point. The thermal conductivity is a transport property most readily accessible to precision measurement, allowing the effects of confinement to be quantified. The extent to which the results could be improved in a microgravity environment will also be studied.

Task Description:
Dohm and collaborators have predicted the first order departures of the thermal conductivity from the bulk behavior as on approaches the lambda point. As the transition is approached, the relevant length scale increases dramatically, allowing the effect to be measured in conventional apparatus. Higher order, nonlinear effects are also predicted, but quantitative information is not yet available. We plan to measure these effects for confinement in parallel plate geometry as a function of plate separation. Earlier measurements indicated that the first order effect was different to that predicted. This needs to be verified, and higher order contributions need to be explored.

Task Significance:
The results will be used to test the emerging theory of transport properties in confined geometries, which has application to physical and chemical processes near surfaces or in small channels.

Progress During FY 1995:
Funding for this research became available at the beginning of FY95. Progress to date included investigations of previous thermal conductivity data to look for possible finite size effects. Experiments with helium confined within gaps as small as 70 microns have been examined.

STUDENTS FUNDED UNDER RESEARCH:

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TASK INITIATION: 9/94  EXPIRATION: 8/96
PROJECT IDENTIFICATION: 962-24-04-14
 RESPONSIBLE CENTER: JPL
Theoretical Studies of the Lambda Transition of Liquid 4He

Principal Investigator: Prof. Efstratios Manousakis  
Florida State University

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
We study the critical properties of liquid helium near the superfluid transition temperature $T_x$ using recently developed numerical simulation techniques and finite-size scaling. In particular, we are interested in the scaling behavior of the superfluid density and the specific heat. We shall study different finite geometries, namely pure two-dimensional, pure three-dimensional and the crossover from two-dimensional to three-dimensional superfluidity in order to verify the validity of scaling and to determine the universal functions associated with scaling.

Task Description:
A recently developed updating technique called cluster Monte Carlo, which eliminates the long-standing problem of critical-slowing-down will allows us to approach close to the lambda point for large size lattices and, thus, extract the critical exponents and scaling properties of the physical quantities of interest. We shall study the temperature and the finite-size dependence of the superfluid density and the specific heat. From these studies we can determine the critical exponent using finite-size scaling techniques.

In addition we shall determine the superfluid/normal phase boundary $T_3(h)$ for films of thickness $h$. We shall calculate the superfluid density as a function of the film thickness and we shall examine the validity of the finite-size scaling theory. We shall also calculate the specific heat as a function of $h$ and this will be used to understand the results of the CHeX experiment.

Finally, the role of vortices and the Kosterlitz-Thouless scenario will be also examined in the course of this work. We shall calculate the renormalization group beta function for two-dimensional superfluids and we shall compare it to that predicted by the Kosterlitz-Thouless theory. In addition, we shall study with our simulation technique the intimate connection between the superfluid transition and the unbinding of vortices.

Task Significance:
The results of these studies are relevant and will be compared to the experimental measurements obtained from the lambda-point experiment (LPE) and to the confined helium experiment (CHeX).

Progress During FY 1995:
Periodic Boundary Conditions, Superfluid Density.
Using the X-Y model and a non-local updating scheme called Cluster Monte Carlo, we calculated the superfluid density $\rho_s$ of a superfluid in a film geometry, i.e. on a finite lattice of size $L \times L \times H$ (where $L >> H$). In this geometry the superfluid density shows a three- to two-dimensional crossover behavior. This means that below a certain crossover temperature the helium film behaves as if it were infinitely thick and, thus, it exhibits three-dimensional behavior. Above the crossover temperature and still below the bulk critical temperature $T_x$ the helium film shows two-dimensional behavior. Because of that the critical temperature is reduced, the superfluid phase transition occurs at temperatures $T_3(H)$ smaller than $T_x$. These reduced temperatures depend on the thickness of the helium film. In order to determine the critical temperatures $T_3$, we applied the Kosterlitz-Thouless-Nelson (KTN) theory, which was formulated for purely two-dimensional helium films, to the quantity $T/(Hr)$. Namely, by solving the KTN renormalization group equations for this quantity, we were able to obtain the values $T/(Hr)$ in the limit $L \leq 4$; thus, the dependence on the planar dimensions $L$ of the lattice was completely eliminated and
II. MSAD Program Tasks — Ground-based

Discipline: Low Temperature Microgravity Physics

T/(H\tau(H)) is a function of the film thickness only. We have been able to compute accurately the ratio T/(H\tau) for various values of the film thickness H and we found estimates for the critical temperatures \( T_c(H) \). We have also found that if we plot \( T/(H\tau(H)) \) versus \( t = T/T_c \), \( n = 0.6705 \) and is the critical exponent of the bulk correlation length, we obtained one universal curve for all film thicknesses. These results have been published in *Physical Review B*.

**Periodic Boundary Conditions. Specific Heat.**

We have studied the specific heat of the X-Y model on cubic lattices of sizes \( L \times L \times L \) and on lattices \( L \times L \times H \) with \( L \gg H \) using the Cluster Monte Carlo method. Periodic boundary conditions were applied in all directions. In the cubic case we obtained the ratio of the critical exponents \( \alpha/\eta \) from the size dependence of the energy density at the critical temperature \( T_c \). Using finite-size scaling theory, we found that, while for both geometries our results scale to universal functions, these functions differ for the different geometries. We compare our findings to experimental results and results of renormalization group calculations. These results have been published in *Physical Review B*.

**Dirichlet Boundary Conditions. Specific Heat.**

Again using the Cluster Monte Carlo method, we have studied further the specific heat of the X-Y model on lattices \( L \times L \times H \) with \( L \gg H \). In the H-direction we apply Dirichlet boundary conditions so that the order parameter in the top and bottom layers is zero. We find that our results for the specific heat of various thickness size \( H \) collapse on the same universal scaling function. The extracted scaling function of the specific heat is in good agreement with the experimentally determined universal scaling function using no free parameters. These results have been published in *Physical Review Letters*.

**Boundary effects in superfluid films. Superfluid Density.**

We have also numerically studied the superfluid density and the specific heat of the X-Y model on lattices \( L \times L \times H \) with \( L \gg H \) where in the H-direction we applied staggered boundary conditions so that the magnetization in the top and bottom layer is zero, whereas periodic boundary conditions were applied in the L-directions. We find that the system exhibits a Kosterlitz-Thouless phase transition at the H-dependent temperature \( T_c^{2D} \) below the critical temperature \( T_c \) of the bulk system. We do not observe finite-size scaling of the superfluid density with respect to \( H \). However, the data can be collapsed onto one universal curve by introducing an effective thickness \( H_{cp} > H \) into the corresponding scaling relations. Applying this idea to the data for the superfluid density of Rhee et al., we are able to scale their data reasonably well. We argue that the effective thickness depends on the type of boundary conditions. The scaling function \( f_1(x) \) of the specific heat is not very sensitive to this boundary effect within error bars and we find good agreement between \( f_1(x) \) calculated from our Monte Carlo results and the experimentally determined universal scaling function. The results will be submitted to *Physical Review B* very soon.

The calculations reported above were performed on a heterogeneous environment of workstations which include DEC Alpha, Sun, and IBM RS/6000 workstations and on the Cray-YMP supercomputer and took several months of CPU time.

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**STUDENTS FUNDED UNDER RESEARCH:**

**Task Initiation:** 1/93  
**Expiration:** 12/95

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**PROJECT IDENTIFICATION:** 962-24-07-15

**RESPONSIBLE CENTER:** JPL

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**Printed:** 28 FEB 1996
BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals


Presentations
Dynamics and Morphology of Superfluid Helium Drops in a Microgravity Environment

Principal Investigator: Prof. Humphrey J. Maris  
Brown University

Co-Investigators:  
Seidel, G.  
Brown University

Task Objective:  
The long range goal of our research is the study of the hydrodynamics of drops of superfluid liquid helium by means of microgravity experiments conducted in space. At the present time we are developing a series of earth-based experiments to levitate superfluid drops so that we can acquire data and experience that will be needed for the design of experiments in space.

Task Description:  
We are conducting a series of earth-based experiments to study the behavior of superfluid drops. We will develop a means to levitate helium drops in earth gravity, primarily by magnetic levitation. We will then investigate 1) how to inject and position drops in a microgravity chamber, 2) how to manipulate drops and to give them angular momentum, 3) how to observe accurately the vibrations and rotations of the drops, and 4) what drop sizes are best suited for the study of a variety of phenomena.

Task Significance:  
The goal is to achieve data and experience critical for the design of experiments in space.

Progress During FY 1995:  
We have completed construction of an apparatus to magnetically levitate liquid helium. The helium is levitated as a result of the force exerted on it when it is placed in a high field-gradient superconducting magnet. To balance the gravitational force, the product of the field $B$ and the field gradient $dB/dz$ must exceed 22 Tesla²/cm. A magnet meeting this specification was constructed by Oxford Instruments and has been installed in a large Janis dewar in our laboratory. The magnet is enclosed by a can filled with superfluid helium at 2 K. The experimental cell has an internal diameter of 2.5 cm and extends throughout the length of the magnet bore. The cell temperature can be varied independently of the temperature of the magnet helium bath. At the present time the cell is cooled through a heat link to a pumped $^3$He pot; in the near future we plan to add a $^4$He system to cool the cell to lower temperatures.

The cell has windows at the top and bottom. Through the use of mirrors mounted inside the cryostat and the cell, we can view levitated drops from both the side and the top. The cryostat is mounted adjacent to a large optical table on which we can mount lasers for illumination and an optical recording system. This cryostat has been designed to be suitable for a variety of experiments on helium drops, at temperatures down to 0.5 K.

For recording the motions of the drops, an EGG high speed video system has been purchased and has recently been delivered to our laboratory.

During the summer we were able to levitate helium drops with this apparatus. The magnet current that was required to achieve levitation was in accord with our expectations, and the dependence of the position of the point of stable levitation on the magnet current was in agreement with calculations. Drops could be introduced into the magnetic trap either by allowing them to drip from walls or by injecting them in with the aid of an ultrasonic transducer located below the surface of the bulk liquid lower in the cell. We were able to levitate drops indefinitely and the drop size was as large as 8 mm diameter.
In the course of these experiments we made a remarkable observation. On several occasions we introduced two drops into the trap. When these drops came together at the bottom of the trap, they did not coalesce immediately but were clearly in contact with each other. They were observed to remain in this state for more than a minute before some unknown event caused coalescence to occur. One possibility is that the drops remain separated by a thin layer of gas maintained by evaporation from each drop. We are planning experiments to test this hypothesis.

It appears that the magnetic levitation system we have developed will serve very well for ground-based exploratory studies of superfluid hydrodynamics.

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**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Journals**


**Presentations**


Weilert, M.A. "Levitation of superfluid helium." University of Massachusetts, November 9, 1995.

**Task Objective:**

Ground-based experiments will be carried out to study the density equilibration process at constant average density \(r\) in a pure fluid, \(^3\text{He}\), near its liquid-vapor critical point \((T_c, p_c)\) after a step change in temperature \(\Delta T\) of the container walls. Measurements are to be carried out for both the region above \(T_c\) (one phase) and below \(T_c\) (coexisting phases). Numerical simulations are to be performed of the density, temperature and pressure equilibration processes in \(^3\text{He}\) for the regimes above \(T_c\) and below \(T_c\) both ground-based and under reduced gravity. The measurements are to be extended to binary \((^3\text{He}-^4\text{He})\) mixtures near their liquid vapor critical point ("plait point"), and corresponding computer simulations are to be carried out.

**Task Description:**

Two flat, horizontal cells of somewhat different geometry are used in different series of experiments. The fluid is contained between two parallel flat OFHC copper plates with high thermal conductivity, kept at the same temperature that is regulated to within a few mK before and after the step \(\Delta T\). In both cells, the fluid layer height is approximately 4 mm, the diameter is 30 mm and the density is measured by two thin horizontal superposed capacitive sensors, spaced by 2 mm that record the dielectric constant. The density is then derived via the Clausius-Mossotti relation, and is recorded by both sensors, \(\rho_{\text{top}}\) and \(\rho_{\text{bottom \(t\)}}\), as a function time \(t\) after a programmed small temperature step of the cell walls. At temperature above \(T_c\), immediately after the step \(\Delta T\), the fast density change from adiabatic energy transfer ("piston effect") followed by the slow stratification change at each sensor are recorded by computer. Experiments are carried out along several near-critical isochors and along several isotherms. Below \(T_c\), the two sensors detect respectively the coexisting liquid and vapor phases. An induced temperature change in the cell walls will permit following the density evolution in both phases at the sensor locations with time.

With help of the known scaled expressions for the static and transport properties of \(^3\text{He}\) above and below \(T_c\), numerical simulations in one dimension are carried out to predict the temporal and spatial evolution of the thermodynamic parameters (density, temperature, pressure and their derivatives) and to predict the asymptotic relaxation times. This simulation is to be done at arbitrary values of the gravitational acceleration \(g\), and will assume the absence of convection - which appears justified by experimental data. The departure from equilibrium values of the properties upon temperature ramping will be investigated by simulation both above and below \(T_c\). Below \(T_c\), the simulations will also predict the motion of the interface between the liquid and vapor phases.

After completion of the \(^3\text{He}\) program, measurements and numerical simulations are to be extended to binary \((^3\text{He} (^4\text{He})\) mixtures.

**Task Significance:**

Such studies - both for pure fluids and binary mixtures - are very relevant to experiments on fluids under micro-g conditions, where investigations of static and dynamic properties near critical points are to be carried out. It is important to know how long a fluid system takes to approach closely enough thermodynamic equilibrium, and what are the basic mechanisms that control the equilibrium process. The numerical computations above \(T_c\) are to be compared with experiments. Simulations under microgravity conditions will be able to assess the permissible temperature ramping rate in experimental data taking that will enable measurements of critical properties in a quasi-equilibrium state. In the two-phase regime below \(T_c\), little is known about the equilibration dynamics and the proposed experiments are expected to substantially help in understanding these processes.
II. MSAD Program Tasks — Ground-based  Disciplines: Low Temperature Microgravity Physics

Progress During FY 1995:

During this period, there were four main projects associated with the program which consists in investigating the density equilibration of a pure fluid (³He) near the liquid-vapor critical point $T_c$. In the previous FY94, the data reduction and computer simulation had been mainly concerned with the single phase above $T_c$, which led to a detailed comparison between experiment and predictions. During FY95, we have concentrated our analysis in the region of the two coexisting phases below $T_c$. Because of the presence of these two phases, the system is much more complicated, and therefore the observed density temporal evolution is more complex — but also still more interesting than above $T_c$. Here our detection system measured separately the density in the liquid and in the vapor phase. Similarly, the computer simulation has to deal with the spatial and temporal evolution of temperature, density and pressure in the two coexisting phases. The effect of the interface, including the latent heat of phase change, is to be taken into account.

Our projects were the following:
A) Experimental data analysis along the critical isochore and along isotherms in the regime $T < T_c$, obtained in a ground-based laboratory ($g=g_0$).
B) Computer simulation of the equilibration under identical conditions of temperature and density as for the experiments, both along the critical isochore and along the isotherms, (both at $g_0$ and under microgravity conditions).
C) The drafting of a very detailed paper including both the experimental results and those from computer simulation, to be submitted by the end of October '95.
D) Complete overhaul of the apparatus, construction of a new equilibration cell with an improved geometry in preparation for a new series of measurements both in $^3$He and in binary $^3$He-$^4$He mixtures, which should help in understanding the discrepancies between predictions and our past experiments.

A) An extensive program of data analysis was carried out of the very abundant experimental results obtained below $T_c$. These consisted in the temporal density evolution $r(t)$ in $^3$He at two superposed locations after a programmed temperature step $\Delta T$ below $T_c$. Two superposed capacitive sensors measured the dielectric constant of respectively the vapor and the liquid phases, and the density could be then obtained from the Clausius-Mossotti relation. The measurements were carried out both along the critical isochore for $-8 \times 10^2 < \frac{(T-T_c)}{T_c} < -1 \times 10^3$ and along two isotherms. Of these the first one was far below $T_c$, with $\frac{(T-T_c)}{T_c} = -1 \times 10^2$, where the earth's gravity effects are negligible, and the other close to $T_c$, with $\frac{(T-T_c)}{T_c} = -3 \times 10^4$, where stratification is significant. The analysis has led to the effective relaxation times "tau".

B) A computer simulation program was developed and refined for the regime $T < T_c$. This program is the extension of the simulation described in the single phase above $T_c$ (F. Zhong and H. Meyer, Phys. Rev. E, 51, 3223 (1995). It is a computation in one-dimension (1D) that determines the spatial and temporal evolution of the temperature $T(z,t)$, density $r(z,t)$ and pressure $P(z,t)$ of the fluid in the cell after a temperature step of the wall container. Here $z$ is the vertical coordinate of the cell, and the 1D approximation assumes the radius of the cell to be much larger than the layer height. The conditions of density, $(T-T_c)$ and of $\Delta T$ in the computation are the same as in the experiments. This computer simulation was carried out below $T_c$ both in the presence of the two coexisting phases, and also in presence of only one of the phases. This latter situation arises when the interface has moved out of one of the horizontal cell walls for average densities away from the critical one and sufficiently close to $T_c$, as can be calculated from the equation of state of the fluid. This simulation for two phases proved to be much more complicated to handle than that in the case of the single phase above $T_c$, and it took correspondingly more time until all the computational problems had been satisfactorily solved. However the physics of the phenomenon proved to be all the more interesting. Here we briefly summarize the comparison of the results from experiments and from simulations:

1) On the whole, there is a reasonable agreement in the shapes and amplitudes of the density temporal evolutions between experiments and simulations, and the agreement improves as the critical point is approached and

Printed: 28 FEB 1996  II-588
stratification becomes significant. However, the differences are more pronounced than in the homogeneous phase above $T_c$. In particular, there are striking differences between experiment and simulations in the vapor phase well below $T_c$, where stratification is negligible.

2) This difference is also reflected by a discrepancy in the effective relaxation time as measured and predicted for the vapor phase. However, as $T_c$ is approached, the trend of the predicted and measured relaxation time for both vapor and liquid is similar: the relaxation time increases and then levels off. This behavior is understood as resulting from the "critical slowing down" when the thermal diffusivity $\Delta T$ along the coexistence curve tends to zero. However the divergence in the relaxation time $\tau$ is prevented by stratification that causes a vertical profile of the $\Delta T$ values, and produces the levelling off in $\tau$, just as in the single phase above $T_c$. Both experiments and simulations show the curves for $\tau$ from both regimes above and below $T_c$ to join smoothly at the critical temperature.

3) Computer simulations show also that under microgravity conditions, $\tau$ will diverge as $T_c$ is approached from both the single and the two-phase regimes.

4) The experiments and simulations along isotherms gave particularly interesting results, as they describe the change of the system when the interface is moved by changing the average density. While again there were striking differences between experiments and simulations in the behavior of the vapor along the isotherm $(T - T_c) / T_c = -1 \times 10^3$, the agreement was much better for the isotherm $(T - T_c) / T_c = -3 \times 10^4$. In both cases, it was clearly seen that the effective relaxation times are quite different for the two phases. Also, when one of the phases had disappeared from the cell, the relaxation time precipitously dropped, confirming that in the absence of the latent heat of phase transformation, equilibration is considerably accelerated.

C) A comprehensive paper has been written, and that has considerably evolved in the course of five months. In the 24 Figures both the experimental and computer simulations results are shown. A detailed discussion of the success and failure in the description of the data by the simulation is given. The title is: Density equilibration near the liquid-vapor critical point of a pure fluid: II - coexisting phases $T < T_c$, by F. Zhong and H. Meyer.

D) A new density cell with two superposed capacitive sensors has been built, and installed. This density cell has a geometry that approximates more closely to an ideal 1D configuration (flat cell) than the cell used until now. Such a redesign was mandatory, because of the discrepancies between the experiment and simulation, both above and especially below $T_c$.

The vacuum system has been overhauled, the bridge circuits for the capacitive sensors upgraded, and ground-loops eliminated. The sample handling system has also been improved and updated. This work has been carried out by Mr. Andrei Kogan, a second-year graduate student. These extensive changes have been almost completed, and it is hoped to start testing the new cryostat at low temperatures in October '95.

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II. MSAD Program Tasks — Ground-based

Discipline: Low Temperature Microgravity Physics

Precise Measurements of the Density and Thermal Expansion of 4He Near the Lambda Transition

Principal Investigator: Dr. Donald M. Strayer
Jet Propulsion Laboratory (JPL)

Co-Investigators:

Chui, Dr. T.
Jet Propulsion Laboratory (JPL)

Yeh, Prof. N.-C.
California Institute of Technology

Lysek, Dr. M.
Jet Propulsion Laboratory (JPL)

Task Objective:
The objective of this ground-based Annual NRA project is to demonstrate the value of high-precision density measurements in the study of the lambda transition of liquid helium. During the two years of Annual NRA funding, we shall demonstrate the capability to perform high-precision density measurements using superconducting cavities, applying high resolution thermometers (HRTs) for temperature control. We shall also demonstrate the ability to deconvolve nonuniformities caused by gravity from the density data.

Task Description:
We shall employ superconducting microwave cavities having Q-values near $10^{10}$, operated in modes that have standing wave patterns that are axially symmetric and whose z-dependences are well known. Upon filling the cavity with liquid helium, and adjusting the temperature near to $T_s$, measurements of the cavity resonant frequency will reflect the helium density. Very near the transition a normal-superfluid interface will form in the cavity: Moving this interface across antinodes of the standing wave pattern by high resolution temperature control will allow the density to be probed in the interfacial region. Measurements at many temperatures will provide density data for deconvolving the temperature dependence of the density from the gravity-induced effects.

Task Significance:
We expect that our earth-bound measurements will demonstrate the value of precision density measurements to exploration of the lambda transition, and study of cooperative transitions in general. The preliminary measurements to be conducted in this Annual NRA task will provide useful information about future exploration of related experiments to be conducted in the microgravity experiments. The results will lead to applications of the technique to problems that include studies at many pressures, studies of nonequilibrium effects, or studies in confined geometries.

Progress During FY 1995:
During FY95 this task has concentrated on two activities: Building the cryogenic probes to support the high-Q niobium cavity at a very stable temperature; and determining the method to process the frequency data to deduce the dependence of density on temperature very near the lambda transition.

Our analysis of the requirements for temperature stability that would demonstrate the benefits that high-resolution frequency measurements can provide for determining the behavior of the fluid near the transition showed that the platforms already existing in Prof. Yeh's laboratory were not adequate. Therefore, we set out to build a cryoprobe that will be suitable for our measurements. The building has been delayed because the graduate student we expected to work on the design was late in acquiring her Ph.D., and has been further delayed because the technician fabricating the parts for the probe has been diverted to other tasks frequently. The probe parts are all fabricated, and assembly has begun. We expect a cooldown by the end of CY95.

In the meantime, we have begun to assemble a system at JPL that will reach temperatures below 1 Kelvin so we can use the high resolution measurements to investigate the tricritical point of 3He–4He mixtures and the phase boundaries at low temperatures. A small dilution refrigerator (DR), built by Oxford Instruments, was acquired from
another group at JPL and installed in one of the labs of the Cryogenic Operations, Research, and Applications Laboratory at JPL. This apparatus was set up and put through a test cooldown, and appeared to operate normally, although the temperature resistor that was calibrated to low temperatures acted strangely, so precise knowledge of the operating temperature was not available. During the DR's original shakedown testing it reached temperatures below 0.020 K, we expect to operate at temperatures down to 0.10 K in our experiments on this apparatus. Material has been obtained for fabricating niobium cavities, and designs for cavities to operate in the TE_{01} modes are completed. Fortunately the DR has a waveguide installed that transmits frequencies in the band 12 to 18 GHz, so a stable oscillator can be set up at any mode in that range. The cavity design will allow at least the first three modes to be excited with the microwave electronics presently available.

Calculations of the frequency shift caused by the change of density near the lambda point have been performed for both the TE_{01} and TM_{01} modes being excited in a cylindrical superconducting cavity. Both these mode types yield a total shift of tens of Hertz as the superfluid-normal fluid interface passes through the cavity. The millihertz resolution of our present microwave sources and frequency measuring instruments will allow us to demonstrate the value of precision measurements. As the temperature regulating capability improves to the nanokelvin range, the frequency measurements must be improved to below microhertz resolution to fully exploit the capability of the high-resolution measurements. We have access to high stability frequency standards at JPL that can provide this stability. These improvements will be implemented if further funding is afforded this experiment. The results of our calculations of the density measurement resolution allowed with this apparatus will be reported at a meeting of the American Physical Society.

**STUDENTS FUNDED UNDER RESEARCH:**

**TASK INITIATION:** 9/94  **EXPIRATION:** 8/96

**PROJECT IDENTIFICATION:** 962-24-04-12

**RESPONSIBLE CENTER:** JPL

**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Presentations**

Analysis of Residual Acceleration Effects on Transport and Segregation During Directional Solidification of Tin-Bismuth in the MEPHISTO Furnace Facility

Principal Investigator: Prof. J. Iwan D. Alexander
University of Alabama in Huntsville

Co-Investigators:
Favier, Dr. J.-J.
Gerandet, Dr. J.-P.
Centre d'Etudes Nucleaire de Grenoble, France

Task Objective:
The objectives of this project are as follows:
1. Systematic preflight characterization of the impact of specific residual accelerations on solute transport and melt flow.
2. Analysis of instantaneous pulling rate adjustments to offset composition nonuniformity caused by residual acceleration.
3. Characterization of planned and incidental USMP-3 acceleration events and comparison and correlation with experimental results.

Task Description:
The USMP-3 MEPHISTO experiments will be fully devoted to the analysis of the plane-front solidification of a tin-bismuth alloy. The experiments focus on mass transport during solidification. Three samples will be simultaneously processed in the MEPHISTO furnace, while residual acceleration will be recorded by SAMS. In addition real-time Seebeck measurements and Peltier interface demarcation will allow for unprecedented characterization of the growth conditions, the effect of pulling velocity in global mass transport, and the sensitivity of transport and compositional uniformity to residual acceleration.

The task undertaken by the Center for Microgravity and Materials Research is to carry out numerical simulation of the impact of specific residual acceleration on the USMP-3 experiments. This includes the determination of optimum operating conditions for monitoring the response to specific types of disturbances. Thereby, the possibility of "null-responses" will be eliminated. Post-flight analysis will involve the comparison of predicted and observed experimental responses.

Task Significance:
Current understanding of residual acceleration (steady and transient g-jitter) effects on experiments conducted in low earth orbit is inadequate. Many theoretical sensitivity studies have been carried out for directional solidification experiments. However, no quantitative experimental confirmation of the predictions of these studies is available. Without such confirmation such studies are of limited use and could lead to either unnecessary design restrictions, an undesirable low gravity environment, or unsuitable experiment operating conditions. This benchmark study which correlates well-controlled and characterized experiment conditions with the predictions of realistic numerical simulation is an essential step towards optimizing use of low gravity laboratories. It is a prerequisite for:

- future design of orbital experiment facilities and choice of orbital attitude
- optimal choice of operating parameters
- assessing local vibration isolation requirements
- optimal scheduling of crew activities and quiet periods etc.

The results of this work will greatly benefit NASA's microgravity program by providing, for the first time, a definitive correlation between model predictions of g-jitter sensitivity and actual experimental results.

Printed: 28 FEB 1996
Progress During FY 1995:

The object of this work, started in March of 1995, is to approach the problem of determining the transport conditions (and effects of residual acceleration) during the plane-front directional solidification of a tin-bismuth alloy under low gravity conditions. The work involves using a combination of 2- and 3-D numerical models, scaling analyses, 1D models, and the results of ground-based and low-gravity experiments. The latter are to be conducted during the MEPHISTO experiment scheduled for USMP-3 in early 1996. The models will be used to predict the response of the transport conditions and consequent solute segregation in directionally solidifying tin-bismuth melt. Real-time Seebeck voltage variations across a Sn-Bi melt during directional solidification in MEPHISTO on USMP-1 show a distinct variation which can be correlated with thruster firings. The Seebeck voltage measurement is related to the response of the instantaneous average melt composition at the melt-solid interface. This allows a direct comparison of numerical simulations with the Seebeck signals obtained on USMP-1. The effects of such accelerations on composition for a directionally solidifying Sn-Bi alloy have been simulated numerically. USMP-1 acceleration data was used to assist in our choice of acceleration magnitude and orientation. The results show good agreement with experimental observations. We are currently focusing on the USMP-3 experiment when a more concentrated alloy will be solidified. Thus, our models must account for thermo-solutal convection.

STUDENTS FUNDED UNDER RESEARCH:  
TASK INITIATION: 5/95  EXPIRATION: 4/96  
PROJECT IDENTIFICATION:  YOF2248  
NASA CONTRACT NO.:  NAG3-1740  
RESPONSIBLE CENTER:  LeRC

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals

Proceedings

Presentations

II. MSAD Program Tasks — Ground-based

Discipline: Materials Science

Synthesis and Characterization of Single Macromolecules: Mechanistic Studies of Crystallization and Aggregation

Principal Investigator: Prof. Spiro D. Alexandratos

Co-Investigators:

Cook, K.  
Joy, D.  
Phillips, P.  
Wunderlich, B.

Task Objective:

The principal objective is to prepare single-molecule polymer amorphous particles and crystals by thermally induced precipitation/crystallization from bulk dilute solutions and by precipitation/crystallization via solvent evaporation from electrosprayed nanodroplets of dilute solutions. Polymer-polymer interactions will be controlled in order to compare the effects of interchain and intrachain entanglements on the chemical and physical properties of a polymer particle or crystal. Successful preliminary studies will be extended to studies in microgravity which will allow for a much more sensitive probing of intermolecular interactions through differences in properties of the polymers.

Task Description:

An electrospray attachment will be constructed for a conventional mass spectrometer, allowing for individual polymer chains to be isolated as they emerge from the instrument. Gravity complicates their isolation but its effect could be minimized with very dilute solutions. The morphology of the single chains will be characterized by high resolution low voltage scanning electron microscopy. Backscattered electron imaging will also be utilized, with spatial resolution of 1-3 nm. Thermal analysis will reveal details of chain motion, including details of crystallization kinetics and the effects of inter- and intrachain entanglement.

Task Significance:

The commercial and technological importance of plastics cannot be overstated. Specially designed plastics have been applied as materials in aerospace construction, medical prosthetics, and organic semiconductors. This wide range of applications stems from the wide diversity of chemical and physical properties of the macromolecules (i.e., polymers) which serve as the building blocks of the plastics we encounter. While our understanding of the origins of this diversity has advanced in recent years, it remains far from perfect. With the increasing importance of composite materials, the gap between theory and experiment may even be widening. An investigation into the role of the interactions between discrete macromolecules on the final observed macroproperties requires the availability of samples of controlled molecularity (i.e., one, two, three, ... macromolecules). It is the goal of this research to prepare and characterize samples comprised of single polymer molecules of known molecular weight, then compare the observed properties with those of comparably well-controlled systems where the number of interacting macromolecules is known. This will allow for the design of new plastics with targeted properties including strength, durability, and biodegradability.

Progress During FY 1995:

A general survey of the literature was made to identify the different methods for separation of single macromolecules. Five techniques are suitable: (1) spray methods; (2) the Langmuir method; (3) freeze drying of glassy solutions; (4) rapid precipitation and crystallization; and, (5) microemulsion polymerization. It is essential that the polymer solution used for the experiments is dilute enough to ensure the presence of single molecules without intermolecular entanglements. Methods (1) - (4) are different approaches to transfer the isolated molecules in solution to isolated solid particles. The fifth method attempts to hinder the polymerizing molecules from mixing in the first place. Methods (1) - (3) were tried in the laboratory, methods (4) and (5) are being planned.

Printed: 28 FEB 1996
II. MSAD Program Tasks — Ground-based

Discipline: Materials Science

The experimental work of the first year concentrated on electro-spray experiments (1). Polystyrene with high molecular weight and very small polydispersity was used. Small amorphous particles were obtained. The particles were relatively monodisperse, but consisted of up to ten molecules. Microscopically measured particle diameter agreed with predictions based on literature models of the spray process and may be helpful in corroborating and/or refining those models. An optimization of the procedure for better control and further decrease of the particle size is in progress. Extension of our earlier work with the Langmuir method (2) has enabled electron microscopic observations and morphological characterization of single crystals of poly(ethylene oxide). With the freeze drying method (3), small polystyrene particles were obtained and their morphology was investigated through electron micrographs. Further work will concentrate on the production of larger amounts of the single molecule particles.

STUDENTS FUNDED UNDER RESEARCH:

| BS Students | 0 |
| MS Students | 0 |
| PhD Students | 1 |

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals
A Novel Electrochemical Method for Flow Visualization

Principal Investigator: Dr. Timothy J. Anderson
University of Florida

Co-Investigators:
Narayanan, R. University of Florida
Fripp, A. NASA Langley Research Center (LaRC)

Task Objective:
The objective of this research program is to develop and demonstrate a novel electrochemical technique to visualize the dynamic states in high temperature liquid metals. This technique uses the oxygen anion-conducting ceramic electrolyte yttria stabilized zirconia in a crucible material which holds the model liquid metal tin in a Bridgman configuration. Electrochemical cells will be constructed at various positions along the side wall and bottom of the solid electrolyte. The bottom cell will operate in the electrolytic mode to establish a well defined boundary condition with respect to the titrated oxygen tracer concentration. The side wall cells will be operated in the galvanic mode to measure the dynamic oxygen concentration at the various wall locations. Well defined convective flow patterns will be established for this model geometry and the results will be compared to numerical predictions of the flow patterns. In this manner the sensitivity of the technique will be determined.

Task Description:
In order to identify the sensitivity of the technique, a cylindrical crucible with local electrochemical cells at 5 vertical and 4 azimuthal positions (20 total cells) will be constructed. A thin (1/8") alumina rod will be rotated in the melt along the central axis in an isothermal furnace to produce a well-defined dynamic state. At suitable rotation speed and aspect ratio, Taylor vortices are expected and the oxygen concentration variation along the side wall will be measured after establishing a zero concentration boundary condition at the bottom face of the melt cylinder. Next, the flow directions of dynamic states (oscillatory flow) developed in a Sn melt without the rod in a destabilizing vertical temperature gradient will be studied. In this experiment, a single cell will be electrochemically pulsed and the time response monitored at the other cells. The experiment will be repeated with different electrolytic cells and pulsing experiments will also be performed to identify the natural oscillation frequency. In a final series of experiments, the continuous response of the sensor cells will be measured to a step change in boundary condition. The results will be compared to a complete 3-D calculation of buoyancy-driven flow accounting for sidewall conduction and radial transport.

Task Significance:
In the processing of many advanced materials (e.g., the bulk crystal growth of semiconductors) the fluid flow state of the liquid determines the quality of the material produced. Although flows in the liquid are not intentionally created, natural forces produce flows. Since the melt is often at high temperature, exhibits a high vapor pressure, and is not transparent, it is extremely difficult to see the fluid flow pattern. Thus, we must rely on the results of calculations which have drawbacks. We have proposed a novel method to measure flow patterns in liquid metals by using solid state electrochemical sensors to measure the time variation of a tracer species. In a microgravity environment, it is anticipated that the magnitude of natural fluid flow will be greatly reduced and produce improved materials. In order to correlate material properties to the fluid flow pattern, we must know the pattern. This technique promises to provide such insight. Potentially, the method could be adapted for improved process control and manufacturability.

Progress During FY 1995:
A modified Bridgman ampoule, constructed from recrystallized alumina incorporated with multiple electrochemical sensors (YSZ plugs), was used to visualize the convective flow patterns in high temperature liquid metals.
Dissolved oxygen was used as the tracer species, which could be potenstiostatically injected or extracted locally at one of the sensors and the oxygen concentration changes were monitored at the other cell locations on the melt/electrolyte boundary in the galvanic mode as a function of time. Flow patterns were inferred for different aspect ratios of the melt and as a function of the imposed temperature gradient. The technique was also able to discern transcritical points in the dynamic states of the melt.

Computer-based data acquisition of the temperature and voltage readings was possible by interfacing the electrochemical cells with a PC carrying a DAS-1600 card.

We intend to test this flow visualization technique in a low gravity environment. It is essential to isolate the diffusion component from the convective component, both of which are comparable in a microgravity environment. Therefore an electrochemical cell was specifically designed to measure the molecular diffusivity of oxygen across cylindrical tin melts, prior to flow visualization studies.

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**TASK INITIATION:** 7/94 **EXPIRATION:** 7/96

**PROJECT IDENTIFICATION:** 962-21-08-26

**NASA CONTRACT NO.:** NCC8-051

**RESPONSIBLE CENTER:** MSFC
Foam Metallic Glasses

Principal Investigator: Prof. Robert E. Apfel  
Yale University

Co-Investigators:
Boa-Teh, C.  
Qiu, N.  
Yale University

Task Objective:
The scientific objective is to determine the parameter space for which foamed glasses are possible. The engineering objectives are to design apparatus to operate in this parameter space and to investigate the practicality and desirability of this process for satisfying technological needs and for producing new opportunities for the application of these materials.

Task Description:
Amorphous materials are an important class of materials because of their many unique features, such as the absence of crystal defects (e.g., grain boundaries or dislocations) and a wide compositional range over which a single-phase amorphous material can be formed. A novel processing approach and experimental design to achieve as-cast bulk amorphous materials are investigated, since a bulk form of amorphous material is required in many applications. By sudden decompression of a melt that is seeded with a volatile liquid, the dispersed "foaming" liquid vaporizes, taking its latent heat of vaporization from the melt, thereby homogeneously cooling the melt. Due to a high decompression rate, a sufficient cooling rate may be produced to yield an amorphous solid foam.

The experimental program is divided into three major sections: (1) proof of foaming principle with organic materials; (2) tests with the water-tin system, even though it is known that such systems will not form a glass; and, (3) tests of an alloy system for forming a foamed metallic glass. The first of these is to verify our expectations with regard to the foaming process and the production of a bulk foam. The second is to give us experience with a metallic system that others have worked with and which may present behaviors unique to metals and not observed with organic materials. The third step is obviously an important milestone toward foamed metallic glasses. The experimental program will be complemented by a theoretical/computational study of this highly transient "foaming" process and by comprehensive materials analysis of all product specimens.

Task Significance:
Rapid decompression of seeded melts is a novel processing approach to produce foam metallic glass, which is an open solid bulk structure that may have glass properties and low density. These foam metallic glass materials should possess few structural defects and may have many potential applications, such as lightweight and high strength structural materials, or matrix materials that can be filled with other materials to suit some special requirements.

Progress During FY 1995:
The major effort of the project has been to redesign the dynamic decompression and cooling (DDC) apparatus so that we can tackle the very difficult problem of dispersing micron-scale water drops in a melt of a low melting point alloy at a temperature slightly above the melt temperature and at a pressure slightly above the vapor pressure of water at that temperature. This fine dispersion is necessary so that the water drops do not rise out of the melt or coalesce before the dynamic decompression can be initiated. [This difficulty would be removed in a microgravity environment] In order to accomplish this task, a custom hollow ultrasonic horn extender was constructed that allows for the introduction of water drops into the melt through the horn while simultaneously producing the ultrasonic cavitation needed to cause emulsification of the water into the melt. Initial tests have shown that the elevated pressures required to prevent boiling can also suppress the cavitation that is necessary for producing a fine
dispersion. However, if the acoustic pressure amplitude of the horn can be increased sufficiently, through proper horn tuning, then the necessary cavitation may be achieved. The pressure cell that holds the melt-water mixture has been modified to take a burst disk that will provide an opportunity for a much higher decompression rate than is possible from a manual or electrically controlled valve. Efforts at theoretically modeling the DDC process continue.

**STUDENTS FUNDED UNDER RESEARCH:**

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**TASK INITIATION:** 3/93  **EXPIRATION:** 3/96

**PROJECT IDENTIFICATION:** 962-25-08-24  **NASA CONTRACT NO.:** NAG8-947  **RESPONSIBLE CENTER:** MSFC

**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Journals**

**Patents**
Apfel, R.E. "Foam Metallic Glass."
Nucleation and Cluster Formation in Levitated Droplets

Principal Investigator: Prof. Stephen Arnold
Polytechnic University, New York

Co-Investigators:
Izmailov, A.F.
Polytechnic University, New York
Myerson, A.S.
Polytechnic University, New York

Task Objective:
1. Employment and improvement of the existing experimental Electrodynamic Levitator Trap (ELT) technique in order to investigate stochastic motion of the ELT confined microdroplets of supersaturated solutions in the case where the dimensionless drag $a$ and driving $b$ parameters are much greater than one.

2. Development of an appropriate theoretical formalism to describe stochastic motion of the ELT confined microdroplets of supersaturated solutions in an atmosphere near the Standard Temperature and Pressure for the case where the dimensionless drag $a$ and driving $b$ parameters are not necessarily small (less than one).

Task Description:
Components of this research include:

1. Experimental study of stochastic motion of the confined levitated microdroplets of various supersaturated solutions. The containerless levitation allows one to considerably delay nucleation onset and, thus, provides a unique method to study the formation and evolution of subcritical solute clusters. This study includes measurement of such time-dependent microparticle characteristics as the standard deviation of its confined stochastic motion, activity of the solute dissolved, etc. Since the measured characteristics are extremely sensitive to the appearance of subcritical solute clusters (solid inclusions) inside of the studied microdroplet, the proposed experiment provides an unique opportunity to study the metastable state of matter.

2. Development of theoretical models of the microparticle confined stochastic motion for the two principally different cases: (1) when there are no solid inclusions inside (i.e., when the microdroplet solution is undersaturated); and, (2) when there are solid inclusions inside of the microparticle (i.e., when the microdroplet solution is supersaturated). These two models will be developed for the particular case where energy dissipation of the microdroplet motion in the levitator atmosphere is linear.

Task Significance:
Understanding the metastable state evolution in supersaturated solutions is of extreme importance in the problem of governed nucleation and crystal growth. Since this evolution consists of the birth-death process of subcritical solute clusters, we outline the following significance:

1. It is a challenging experimental problem to study metastability since any heterogeneity may cause instantaneous nucleation followed by crystallization. Therefore, development of the containerless (without heterogeneities due to container walls) experimental technique is of interest.

2. Analytical description of stochastic motion of the confined supersaturated solution microdroplet will allow treatment of the obtained experimental data.
Progress During FY 1995:

1. One of the most difficult things to determine in connection with nucleation and prenucleation is the diffusivity of the subcritical solute clusters (embryos). For the first time we have found a theoretical description which connects diffusivity and viscosity with water activity above the microdroplet.

2. In the process of working with our experiments we have found that nucleation very often occurs before we reach the spinodal even in containerless microdroplets. The problem has been identified as having to do with impurities. Because of this we have begun to assemble a device which will enable us to detect impurities down to a single molecule, and locate whether these impurities are in the bulk or on the surface of the droplet. The device is called an Aerosol Particle Microscope (APM). It owes its high resolution to a theoretical effort to understand the stochastic motion of a microparticle in an electrodynamic trap. This effort has led to a new principle for imaging Brownian particles in such a device, "Squeezed Imaging." A surprising result predicted by theory and confirmed by experiment is that Brownian motion of the particle's center of mass may be eliminated from long term images using a technique, which we term "Squeezed Imaging." In its current form the APM enables us to determine whether impurity molecules are on the surface of the droplet or in the bulk. For surface molecules polarization selective imaging in inelastic scattering provides the orientation of the molecule relative to the surface normal. Further studies will connect the location and concentration of impurities with nucleation.

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| PROJECT IDENTIFICATION: 962-25-08-30 | NASA CONTRACT NO.: NAG8-1060 | RESPONSIBLE CENTER: MSFC |

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals


Studies of Nucleation and Growth of Intermetallic Compounds

Principal Investigator: Prof. Robert J. Bayuzick

Co-Investigators:
Hofmeister, W.

Task Objective:
The objective of this research is to quantitatively define the nucleation behavior of selected intermetallic compounds and the relationship between degree of undercooling and the speed of growth of the solid.

Task Description:
This work focuses on the undercooling of intermetallic compounds and their pure metal constituents by containerless processing. This ground-based research involves a continuation of already established drop tube work and electromagnetic levitation (on the bench). Specifics to be addressed are maximum undercoolings, nucleation frequency, the effect of undercooling on solidification velocity in refractory metals and alloys, and microstructural development as a function of deep undercooling/solidification velocity.

Research Approach
This work will consist of a comprehensive investigation on nucleation and growth of the solid from the undercooled liquid of selected intermetallics and their pure metal constituents. In order to do this, containerless processing is essential not only to eliminate the effect of the container on nucleation but also because the metals and alloys of interest are highly reactive and become contaminated with crucible material.

Three ground-based techniques for containerless processing will be applied to the effort. These are free fall in the long drop tube at the Marshall Space Flight Center, electromagnetic levitation "on the bench" at Vanderbilt University, and electrostatic levitation "on the bench" at the Jet Propulsion Laboratory.

The intermetallic compounds chosen to be studied initially are TiAl and NiAl. In addition, studies will be done on pure Ti and pure Ni, as points of reference and because of their standalone contribution to science. The particular intermetallics are chosen because both exhibit a wide solubility range as seen in their respective phase diagrams and, within compositions ranges, exhibit a tendency to be unencumbered by competition between alternate phase selection in the deep undercooling regime.

Nucleation frequencies as a function of temperature will be obtained for each combination of specimen type and ground-based processing technique. In each case, the nucleation probability distribution and the cumulative distribution will be constructed from the nucleation frequencies. To do this, a large number of undercooling experiments on each specimen type will be conducted by each of the ground based containerless methods. From analysis of the cumulative distributions, the kinetics of nucleation of the solid from the liquid for each specimen type under each processing condition will be quantitatively defined and examined in comparison to nucleation theory.

The ordered intermetallic alloys as well as their pure constituents will also be studied in order to determine the solidification velocity as a function of melt undercooling. The velocity will be determined using ultra high speed imaging to track the solidification front as it moves through the specimen. In opaque samples this can be done by monitoring the movement of the thermal field developed by recalsscence. An instrument has been developed that consists of a 10 by 10 array of parallel tapped photodiodes and data acquisition. The system is capable of capturing
Theoretical implications of these results on solidification theory are under investigation. That at undercoolings greater than 173K the velocity remains constant. Sample weighing within the limitations of the electromagnetic levitator were processed. The surface area of the drops were changed and therefore the macroscopic heat transfer from the drops was changed. This explanation was investigated further by testing drops of varying masses. By changing the mass of the drops the surface area of the drops were changed and therefore the macroscopic heat transfer from the drops was changed. Sample weighing within the limitations of the electromagnetic levitator were processed. The experiments indicate that at undercoolings greater than 173K the velocity remains constant at ~32 m/s, independent of drop size. The theoretical implications of these results on solidification theory are under investigation.

**Task Significance:**

Intermetallic compounds and their composites are of considerable interest because of their potential use in high temperature structural applications. One strong driver for this interest is future goals for aircraft propulsion systems. Future aircraft engines will require high thrust-to-weight ratios and low specific fuel consumption, while remaining reliable, affordable, and highly maintainable. Effort involving innovative designs, advanced materials and processing, and improved design and analysis tools is required to meet Integrated High Performance Turbine Engine Technology goals for doubling propulsion system capabilities by the year 2005. Benefits of the required increase in engine cycle temperature must not be offset by increased turbine weight or cooling air. Thus, new materials and design technologies are needed for advanced high-temperature turbines.

The materials of interest in this study show potential for use in such applications because of their significantly increased high temperature strength and acceptable low temperature damage tolerance. It is therefore necessary to fully understand the nucleation and solidification behavior of these intermetallic compounds.

**Progress During FY 1995:**

The ultra high speed imaging system used for velocity determination was upgraded for improved operation. The system was enhanced by adding a programmable microprocessor such that variable frame length files (512 to 8192 frames) could be recorded. Additionally, the new processor allowed manual triggering of the data acquisition system such that low speed events and events that do not trigger data acquisition could be imaged.

The method used to determine the solidification velocity from the sets of images was significantly refined for more accurate analysis. The improvements include treating the drops as ellipsoids rather than spheres, more accurately determining the interface position on the images, more accurately relating the position of the interface on the images to the position of the interface on the drop, and more accurately determining the distance that the interface moved between images. All of the improvements to the data analysis were made in order to create a method of looking at the data that realistically related to the actual solidification event and in order to limit the number of assumptions required in the analysis. Additionally, a detailed error analysis of the velocity determination method developed was performed. The error analysis determined the magnitude of the error caused by the assumptions made in the velocity determination and it indicated that the uncertainty in the velocity measurement was ±2 m/s in the high undercooling regime.

As a benchmark for the study of the NiAl system the solidification velocity of pure nickel was measured. Samples weighing 1.0 gram were processed in a bench electromagnetic levitator. The results of the study indicate that the solidification velocity increases with increasing undercooling up to 173K undercooling (10 percent of the melting temperature (Tm)). At undercoolings of 173K or greater the solidification velocity remained constant at ~32 meters per second. This is not in accordance with present theories relating solidification velocity to the degree of undercooling. These theories predict that the solidification velocity increases continually with increasing undercooling. One possible explanation for the measurements made in this study is that there is a maximum thermal gradient reached at the interface that limits the driving force for solidification and therefore the solidification velocity.

This explanation was investigated further by testing drops of varying masses. By changing the mass of the drops the surface area of the drops were changed and therefore the macroscopic heat transfer from the drops was changed. Sample weighing within the limitations of the electromagnetic levitator were processed. The experiments indicate that at undercoolings greater than 173K the velocity remains constant at ~32 m/s, independent of drop size. The theoretical implications of these results on solidification theory are under investigation.
II. MSAD Program Tasks — Ground-based

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**TASK INITIATION:** 9/94  **EXPIRATION:** 9/96

**PROJECT IDENTIFICATION:** 962-25-08-20  
**NASA CONTRACT NO.:** NAG8-1087  
**RESPONSIBLE CENTER:** MSFC

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**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Proceedings**


**Presentations**


II. MSAD Program Tasks — Ground-based

Transport Phenomena During Equiaxed Solidification of Alloys

Principal Investigator: Prof. Christoph Beckermann
University of Iowa

Co-Investigators:
deGroh III, H.C.
NASA Lewis Research Center (LeRC)

Task Objective:
To investigate the macrosegregation and structural inhomogeneities resulting from gravity-induced thermosolutal convection and solid sedimentation during equiaxed solidification of alloys on a bulk level.

Task Description:
The melt flow and solid transport phenomena occurring during equiaxed alloy solidification are investigated in experiments using both metal alloys and transparent analogues. The drag and heat/mass transfer coefficients of single equiaxed crystals are measured by employing fabricated dendrite models and, in situ using transparent model alloys. This information is combined to develop a multi-phase/-scale simulation model that allows for the calculation of the individual solid and liquid motions during solidification and incorporates the detailed phase interactions on a microscopic scale.

Task Significance:
This investigation complements NASA-sponsored research on the influence of fluid flow in alloy casting by including the important effects of gravity-induced motion of free equiaxed crystals. This combined experimental and theoretical/numerical study will provide (1) needed fundamental understanding of how liquid convection and the movement of free equiaxed grains interrelate and produce segregation and structural zones in castings, (2) progress towards a more complete numerical simulation model of transport and solidification phenomena, and (3) a base for defining future microgravity flight experiments on equiaxed dendritic solidification.

Progress During FY 1995:
Bulk solidification experiments using both Pb-Sn alloys and ammoniumchloride-water solutions have been completed. An analysis of the compositional and structural patterns achieved in the Pb-Sn experiments is presently underway. This data is intended for comparison with predictions from the numerical simulation model.

The work on measuring and correlating the drag coefficient of single and multiple equiaxed dendritic crystals is also completed. Considerable progress has been made in measuring the mass transfer coefficient of single growing equiaxed transparent model alloy crystals settling in an undercooled melt. The ammoniumchloride crystal data have been correlated, and additional experiments are being conducted using a succinonitrile-acetone alloy. Simultaneous measurements of the dendrite tip growth velocities are also being analyzed.

A multi-scale/-phase model of equiaxed dendritic alloy solidification with melt convection and transport of free crystals has been developed. Work during the past year focused on performing numerical simulations for a variety of alloys and solidification conditions. Detailed comparisons of the model predictions with the transparent model alloy experiments (see above) have been carried out. More comparisons with the metallic alloy experiments are being planned.

The findings of this research have also been used to develop an initial concept for a future microgravity experiment involving the growth and interactions of multiple equiaxed dendrites. A proposal has been submitted in response to a recent NRA.

Printed: 28 FEB 1996
II. MSAD Program Tasks — Ground-based

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**TASK INITIATION:** 11/93  **EXPIRATION:** 1/96

**PROJECT IDENTIFICATION:** 962-25-05-23

**NASA CONTRACT NO.:** NCC3-290

**RESPONSIBLE CENTER:** LeRC

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**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Journals**


**Proceedings**


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Printed: 28 FEB 1996
Gravitational Effects on the Development of Weld-Pool and Solidification Microstructures in Metal Alloy Single Crystals

Principal Investigator: Dr. Lynn A. Boatner
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Co-Investigators:
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University of Alabama, Huntsville (UAH)

Task Objective:
The objectives of this research are to achieve an in-depth quantitative understanding of the role played by convection-driven and surface-tension-driven heat and mass transport in determining the shape and surface morphology of fusion weld pools and, thereby, to control the nature of the solidification processes that determine the microstructural and mechanical properties of welds and castings.

Task Description:
By combining ground-based results with those obtained in the low- and high-g environments provided by NASA aircraft, detailed microstructural information can be obtained through the use of single-crystal alloy specimens, and both gravitational and surface-tension-driven effects on weld-pool formation and microstructural properties can be delineated and quantified. The results of these investigations are currently yielding new information that is serving to advance our level of understanding of various phenomena which determine both weld-pool shapes, mass transport, and the morphological properties of solidification surfaces.

This research represents a new and innovative approach to the investigation of gravitational effects on melt-pool shapes, solidification phenomena, and weld microstructures. This approach is based on the application of our recently developed alloy single-crystal methods for the delineation of solidification microstructural properties and on the new quantitative analytical methods that have been developed in conjunction with the unique experimental results that can be obtained through the application of these single-crystal techniques. This approach to the study of weld and solidification microstructures begins with the growth of macroscopic single crystals of the alloy system that is to be investigated. As in the case of our previous studies, Czochralski-grown single crystals of the pure ternary alloy 70Fe-15Ni-15Cr (a compositional analog of one of the 300 series of stainless steels) are being utilized.

Task Significance:
The long-term goal of this research effort is to achieve a firm scientific basis for the development of a comprehensive scientific program in which more-complex solidification and welding experiments can be carried out on NASA aircraft, on Space Shuttle flights, and eventually on the Space Station. Since the microstructural properties of weld pools and the distribution of certain impurities within the weld are central to determining the weld mechanical strength, these experiments have practical implications regarding a wide range of construction, fabrication, and manufacturing operations both in space and on earth.

Progress During FY 1995:
Time-resolved macro-video techniques, surface profilometry, and conventional metallographic methods have been combined in carrying out detailed investigations of the surface-morphological properties of stationary melt pools formed by gas-tungsten-arc (GTA) and electron-beam heating. The investigations of the unique surface morphology represented by the formation of concentric “ring-like” “ripples” on the surface of GTA spot welds, which were initiated during FY 1994, were expanded to encompass a detailed study of the effects of various parametric variations on the melt-pool surface-morphological properties. The observed surface undulations are ascribed to fluid-dynamical effects arising from “capillary wave” oscillations of the melt pool during solidification. The analytical techniques employed in the present work have shed new light on the mechanism of surface-ripple formation during the
solidification of stationary melt pools formed on oriented single crystals of a ternary 70%Fe-15%Ni-15%Cr stainless-steel alloy. In particular, macro-video recordings of the solidification process, when analyzed on a frame-by-frame basis, indicate that liquid-phase (capillary-wave) oscillations occur, whose amplitude can be influenced by external perturbations. These oscillations in-turn produce an alternating solidification profile at the solid-liquid interface of the solidifying melt pool whose amplitude decreases as the solidification proceeds and whose frequency simultaneously increases. This phenomenon results in dimensional variations that affect the topmost dendritic solidification cells as revealed by cross-sectional metallographic analysis. The individual and combined effects of cooling rate, gravity, and external perturbations such as deliberately introduced vibrations on the formation of surface ripples were investigated. Additionally, studies of the implications of capillary-wave motion on mass transport effects in small stationary melt pools have been carried out by using our previously developed technique involving the controlled introduction of a tracer element subsequent to the formation and full development of the melt pool, followed by an elemental mapping of the tracer distribution by means of back-scattered-electron analysis. These results suggest that capillary-wave motion of the melt pool may play a significant role in the final mass-transport and distribution processes that occur during solidification.

A new laser system was assembled and utilized by G. Workman and G. Smith of the Materials Processing Laboratory of the University of Alabama in Huntsville, Alabama. This system was used to form melt pools in oriented stainless steel single crystals in both the low- and high-g conditions provided by the NASA KC-135 aircraft. In spite of some initial problems with the new laser melter, some limited data was obtained on the last available KC-135 flight, and this data is being analyzed in order to compare the results with those obtained in the course of the more-extensive FY 1995 ground-based studies. The preliminary analysis indicates that solidified surfaces are formed under both low- and high-g conditions whose properties are similar to those observed for solidification in a 1-g environment. These effects are interpreted as arising from g-jitter on the KC-135 aircraft or from instabilities in the laser power supply.

A numerical analysis and modeling of the capillary-wave mechanism was carried out by solving the Navier-Stokes equation for a set of boundary conditions and parameters appropriate to the experimental conditions. The results of these calculations were then used as the basis for a VHS video display and simulation which clearly illustrates the mechanism leading to the formation of capillary-wave-induced ripples on the surfaces of solidified stainless steel stationary melt pools.

In the course of the above-noted investigations of the formation of surface "ripples" on solidified melt pools, it was found that many of the same perturbations that led to alterations in the characteristics of the capillary-wave-induced small surface undulations concurrently led to changes in the overall surface topology. In fact, the magnitude of these overall profile alterations was up to a factor of 100 times larger than that characteristic of the surface "ripples" themselves. This finding has opened up an entirely new line of inquiry involving the role of gravitational, vibrational, atmospheric, and other effects on the gross surface morphological properties of stationary melt pools. These overall surface-profile geometries have both practical and fundamental implications that extend to the areas of welding, surface glazing, and casting.

Additional progress has been made in carrying out investigations of the microstructural properties of stationary melt or "weld" pools and has provided new information concerning the relationships between the weld-pool shape, the crystallography of the sample, and the resulting microstructural and dendritic properties of the solidified region. A detailed microstructural analysis of stationary weld pools has been made using oriented stainless-steel single crystals with a composition of 70%Fe-15%Ni-15%Cr (wt %) for the cases of stationary melt pools formed using either electron-beam or gas-tungsten-arc heating on (001), (011), and (111)-oriented planes of ternary austenitic, fcc-alloy crystals. Metallographic characterization was performed for each crystallographic plane and welding method. Since the two types of heating produce different melt-pool shapes, significant differences were observed in the microstructural characteristics of e-beam versus GTA stationary welds. These results were subsequently used in making a three-dimensional reconstruction of the solidification microstructure in each case. The results of this investigation provide an experimental basis for the future development of mathematical models for the prediction of solidification microstructures in stationary melt and weld pools.
II. MSAD Program Tasks — Ground-based

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BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Books

Presentations


Microstructure Formation During Directional Solidification of Binary Alloys Without Convection: Experiment and Computation

Principal Investigator: Prof. Robert A. Brown
Massachusetts Institute of Technology (MIT)

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
A combination of experimental and theoretical research will be aimed at developing a predictive understanding of cellular and dendritic microstructures of dilute binary alloys and lamella spacing in the growth of eutectics formed during thin-film solidification. The results will form the basis for prediction of the dependence of microstructure formation on macroscale properties of bulk solidification systems, such as the imposed temperature gradient and the sample growth rate.

Task Description:
The extensive theoretical and experimental research on microstructure formation point to the formation of cellular microstructures in binary alloys and lamella eutectics as being formed under conditions of long time-scale, spatiotemporal chaos in the pattern. These patterns involve a band of wavelengths that evolve with changes in the operating conditions, such as growth rate and temperature gradient, and include very long wavelength interactions through which the pattern communicates over length scales much larger than the characteristic cell size. The outstanding problem that will be addressed in the research is to begin to construct mean field models for solidification microstructure that are based on microscopic mechanics of individual elements in the microstructure. Experimental, theoretical and computational studies will be conducted that will lead toward this goal. These elements of the research plan are:

• Experimental studies of spatiotemporal chaos in cellular solidification and the role of externally applied forcing functions on regularization of the pattern;

• Extension of the analysis of wavelength selection to thin-film eutectic solidification;

• Experimental studies of lamellar eutectic growth in thin-film solidification; and

• The development of stochastic and mean field models for pattern formation in directional solidification.

The microstructure of metal alloys formed from directional solidification plays an important role in the mechanical and electrical properties of these materials. This investigation describes experimental and theoretical research aimed at developing a predictive understanding of cellular and dendritic microstructures of dilute binary alloys and the lamella spacing in the growth of eutectics formed during thin-film solidification. The combination of experimental and theoretical results will form the basis for prediction of the dependence of microstructure formation on macroscale properties of bulk solidification systems, such as the imposed temperature gradient and the sample growth rate. The experimental studies are carefully designed so that bulk convection, driven by density gradients, is unimportant. In this way, the results are applicable to gravity-free experiments that will be undertaken during space flight. The theoretical framework for understanding nonlinear pattern formation during solidification is so complex that there is little hope of unraveling the mechanisms for pattern formation in the presence of convection without rigorous analysis and experiment in the absence of convection. The thin-film experimental geometry offers the only mechanism for accomplishing this goal on Earth.
Task Significance:
The research has the promise of making significant progress towards the development of a theoretical framework for characterizing the formation of microstructure in alloy solidification. The experiments and microscale calculations to be conducted will lay the foundation for detailed description of the mechanisms for length scale adjustment in cellular and lamellar eutectic growth. The development of kinetic theory for describing the evolution of the microscale will give the connection between processing variables and microscale formation that is necessary for adaptation of the theory to practice in industrially important systems, and will provide a framework for the characterization of alloy systems for microstructural modeling parameters.

Progress During FY 1995:
Substantial progress in both the experiments and theory has been made in the last funding year. Experiments on the thin-film crystal growth of large collections of cells are being performed using the succinonitrile-acetone alloy. High purity succinonitrile is being produced at M.I.T. using vacuum distillation in a distillation facility that has been especially designed for this purpose. Two types of experiments are underway. First, new image processing software is being used to study the dynamics of large collections of small amplitude, two-dimensional cells. These experiments will compliment results published previously by us and will be a testbed for comparison of the theoretical simulations described below with experiments. Second, experiments in cells confined to small channels with widths less than or equal to ten cells will be performed to explore the effects of lateral dimension on nonlinear cell dynamics.

Numerical simulations of large collections of cells during solidification is particularly challenging because of the need to resolve the deforming melt-crystal interface as a function of time. As a result numerical methods that rely on fixing the mesh or grid with the interface become very involved as the deformation of the interface becomes severe. We are developing a new numerical method for simulating the dynamics of large collections of cells. The method is based on a fixed Cartesian mesh in which the moving boundary is immersed. The method is based on numerical approximation to a two-dimensional delta-function and a phase indicator function to signify the location of the interface and the melt and crystal phases. This approach follows from the work of LeVeque and Li (SIAM J. Num. Anal. 31, 1019 (1994)). Although fine finite difference or finite element grids are needed for accurate resolution of the interface, the grids are regular and are not updated with time. Moreover, semi-implicit time integration methods will make feasible the development of a highly parallelizable simulation tool. We are pursuing this approach.

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BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals


Presentations
Brown, R.A. "Large-scale numerical simulation: the link between chemical engineering science and systems engineering." Donald L. Katz Lecture, University of Michigan, April 1995.
Modeling of Convection and Crystal Growth in Directional Solidification of Semiconductor and Oxide Crystals

Principal Investigator: Prof. Robert A. Brown  Massachusetts Institute of Technology (MIT)

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
The research effort focuses on the development of a detailed understanding of the interactions of heat, mass and solute transport on the quality of crystals grown from melt by the vertical Bridgman (VB) method. Both semiconducting alloy (GeSi) and oxide (Bismuth germinate) crystals are being studied.

Task Description:
The research is aimed at developing a detailed simulation for VB crystal growth systems for semiconductor and oxide materials that begins to relate macroscopic analysis of heat, mass, and solute transport with the microscopic properties of the grown crystal. The analysis and simulation tools developed will be applied to the analysis of several crystal growth systems. The binary alloy GeSi and the pseudo-binary BGO Bismuth germinate are selected for analysis of VB growth. The research is divided into three sections:

• Development of integrated model for vertical Bridgman growth,
• Parallel processing and simulation of three-dimensional convection,
• Application to GeSi alloy crystal growth, and BGO crystal growth.

Fundamental understanding of the interactions of heat, mass, and solute transport on the quality of crystals grown from the melt is important in the design and control of systems for crystal growth in microgravity and for the interpretation of the results of experiments performed both on Earth and in space. The research program focuses on the development of the detailed analysis of these features in the vertical Bridgman (VB) crystal growth of semiconductor crystals and oxide materials used in optoelectronic applications. The analysis has two parallel goals: to develop the first integrated model and numerical analysis for the growth of these materials that accounts for the details of the design and operation of the furnace; and to link the predictions of the macro scale analysis of heat transfer and convection with the quality of the crystal as measured by the number of crystallographic defects and the compositional homogeneity of the material. The research integrates several aspects of research that are ongoing at M.I.T. to accomplish these goals: the development of numerical analysis for integrated heat transfer throughout a high temperature furnace, including internal radiation in a semitransparent material; the modeling of dislocation motion and multiplication in semiconductor materials; and applications of new robust algorithms for parallel computation.

The analysis of coupled furnace design and the prediction of material quality will be applied to two distinct crystal growth technologies that have potential application for crystal growth in microgravity: the growth of GeSi semiconductor alloys, a substrate material used for superlattices; and the growth of Bismuth Germanate or BGO, a scintillating oxide material used in high energy detector applications.

Task Significance:
The techniques for analysis and the quantitative insights developed in this research have broad application to a variety of ground-based and space flight experiments supported by NASA and its European counterpart, ESA. Many of the previous research results conducted in this research area represent theoretical collaboration with experiments supported by NASA. These interactions between detailed modeling and experiments are crucial to the design of well-controlled experiments for space, as well as optimized crystal growth on Earth. The development of a user-friendly simulator for VB growth systems will be a direct result of this program and will be made available to others in the research community.
Progress During FY 1995:

A major impediment to the application of large-scale modeling of material processing experiments in a large number of laboratories is the need for very large computer memories and disk space for the solution of the large sets of linear algebraic equations that result from finite element and finite difference discretizations of complex physico-chemical models of these processes. Iterative methods for solution of algebraic equations are the preferred method for circumventing this problem, but suffer from lack of convergence for the poorly conditioned, asymmetric equations that are typical for the complex models of interest in this project. We have addressed this problem by the development of a new set of preconditioned iterative methods for linear equation solvers. These methods use generalized Krylov iterative method with block preconditioners based on additive and multiplicative Schwarz domain decomposition methods. Here the finite element mesh is divided into domains composed of many elements. The linear equation set is decomposed on these domains into approximate linear subsets, which are each solved approximately to build a preconditioner for the iterative solution of the exact equation set. The resulting Schwarz/Krylov methods have been demonstrated by us to give much improved performance over previously available iterative decomposition methods for fluid mechanical problems. Moreover, when implemented on a single-processor computer the Schwarz/Krylov methods are competitive with the concurrent factorization and storage (CFS) methods for direct solution of linear equations. Because both the Schwarz and Krylov algorithms are highly parallel, a very high efficiency parallel algorithm is expected. We are pursuing this avenue of research.

Students Funded Under Research:

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Task Initiation: 7/93 Expiration: 7/96

Bibliographic Citations for FY 1995:

Journals

Books

Presentations
Brown, R.A. "Large-scale numerical simulation: the link between chemical engineering science and systems engineering." Donald L. Katz Lecture, University of Michigan, April 1995.
I. Evolution of Crystal and Amorphous Phase Structure During Processing of Thermoplastic Polymers

Principal Investigator: Prof. Peggy Cebe
Tufts University

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
Our objective is to study microstructure evolution during melt processing and secondary thermal treatment of high performance thermoplastic polymers which are candidate materials for advanced composite applications. We will investigate formation of structure in both the crystalline and the amorphous phases. The purpose of the study is to determine the effects of processing conditions on the resultant structure and physical properties of thermoplastics. We hope to address the manner in which self-deformation stresses affect the microstructure of the solidifying polymer melt. Processing in a microgravity environment will impose very different stress states on molten polymers during solidification, compared to ground-based processing conditions. Dependence of polymer microstructure and physical properties on processing conditions can more easily be predicted and controlled under microgravity conditions where self-deformation forces are avoided.

Task Description:
Present ground-based processing methodologies requires gravity compensation to support the processed piece during solidification. Therefore nearly all melt processing approaches (e.g., extrusion, film blowing, fiber drawing) involve very rapid quenching of the molten material to avoid imposing self deformation stresses on the melt. Rapid quenching results in inhomogeneous cross-sections, since the center of the piece cools more slowly than the exterior. In a microgravity environment, it would be possible to use very slow melt processing techniques to obtain more uniform microstructure throughout the section, minimizing the morphology gradient between the skin and the core of the section.

The proposed research is a ground-based study of slow melt processing in the gravity environment. We will process thermoplastic polymers by solidification from the melt without gravity compensation. First we will use real-time x-ray scattering at elevated temperature to study the kinetics of solidification. Then we will examine the resultant microstructure at room temperature after processing. Self deformation stresses on the melt will be studied to determine the effects of gravity on the development of crystalline structure and morphology from the (stressed) amorphous melt. We will characterize the unit cell structure of the crystal phase, the disposition of unit cells within the crystals, and the organization of crystals into larger units such as spherulites, fibrillar bundles, or shish-kebab structures. We will also study the amorphous phase structure to determine degree of chain alignment and the amount of rigid vs. mobile amorphous phase. Additionally, the location of rigid vs. mobile amorphous chains with respect to the crystals will be examined.

The principal approaches used in this study will be real-time wide and small angle x-ray scattering, which will be performed at the Brookhaven National Synchrotron Light Source and in our in-house x-ray laboratory. In addition, we will use differential scanning calorimetry, optical microscopy and birefringence, optical waveguide prism coupling, scanning electron microscopy, and dynamic mechanical spectroscopy to characterize the polymers after processing. Standard mechanical tests, such as stress-strain and impact resistance measurements, will be used to study the performance of the test pieces. One unique aspect of the proposed research is the combined study of both the crystal and the amorphous phases. We will be concerned not only with the nucleation and growth of crystals, but also with residual stresses and chain alignment in the amorphous phase.

Task Significance:
Our ultimate goal is to contribute to a fundamental understanding of microstructure evolution in thermoplastic polymers solidifying under influence of self deformation stresses, and to determine the ameliorating effects of the
microgravity environment. Once we know the effect of self deformation stress during processing, we will be better able to predict the solidification behavior of polymers in the microgravity environment. This will lead to development of unique processing applications that capitalize on the microgravity environment. Complementary experimentation under reduced gravity conditions would later be proposed to be performed on the Space Shuttle or on Space Station. Ground-based facilities, such as drop towers and KC-135, would not provide the capability for real-time studies of microstructure development under practical processing conditions.

**Progress During FY 1995:**

As part of our study of amorphous and crystalline orientation in polymers, real-time high temperature small angle x-ray scattering experiments were conducted at the Brookhaven National Synchrotron Light Source. A tensile deformation stage was used to apply uniaxial strain to polymer films 50-150 \(\mu\)m thick. A zone heater, capable of temperatures as high as 200\(^\circ\)C, heated a small portion of the films to temperatures near their glass transitions. At the same time, tensile strain was applied causing local deformation of the films. The x-ray beam was incident on the film during the deformation process, and the observed scattering pattern was typical of oriented amorphous polymers. In addition, we observed formation of voids during deformation. These experiments are combined with optical birefringence measurements of the films during and after deformation.

| STUDENTS FUNDED UNDER RESEARCH: | TASK INITIATION: 12/94 | EXPIRATION: 12/96 |
| BS Students: | 0 | |
| MS Students: | 0 | |
| PhD Students: | 1 | |

**STUDENT INTERNS:**

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Task Objective:

The objective of this research is to obtain definitive values for the normal spectral emissivity of selected high-melting-point metals by two independent techniques in order to provide a foundation for reliable radiometric temperature measurements in materials research at high temperatures, both in microgravity and on the ground.

Task Description:

The research will include accurate measurements of the normal spectral emissivity of selected metals near and at their melting points in a series of subsecond pulse-heating experiments in which the emissivity will be determined at two laser wavelengths in the range 0.5-0.9 nm by two independent techniques involving high-speed pyrometry and laser polarimetry, respectively. The simultaneous measurements by the two techniques on the same specimen will minimize a number of major experimental uncertainties, in particular those arising from specimen surface conditions and specimen purity.

Task Significance:

The results of this research will enable the establishment of reference values for normal spectral emissivity (also leading to high-temperature radiometric standards) which are critically needed for accurate temperature measurements in materials research on high-temperature liquids and melts and in the determination of their thermophysical properties at high temperatures, under either microgravity or terrestrial conditions. In addition, the proposed work will resolve the current major controversy in the scientific literature regarding the wavelength dependence of normal spectral emissivity of metals at and near their melting points.

Progress During FY 1995:

Definitive accurate measurements of normal spectral emissivity were performed on two refractory metals, molybdenum and tungsten, by two independent techniques, namely spectral radiometry and laser polarimetry. The measurements were in the wavelength range 600-650 nm and covered the temperature range 2000-2800°C for both metals. The agreement between the normal spectral emissivity values obtained by the two techniques was within 2% over the entire temperature range, demonstrating reliability of the direct measurements of normal spectral emissivity with the new laser polarimetry technique. This new technique will provide much-needed accurate data on normal spectral emissivity of selected high-temperature materials and will enable accurate measurements of true temperature from measurements of surface radiance temperature. In addition, applicability of the laser polarimetry technique to the non-contact detection of phase transformations in metals and alloys at high temperatures was shown by performing a preliminary series of experiments on nickel (magnetic transformation), iron (structural transformation), zirconium (solid-liquid transformation - melting point), and 53Nb-47Ti alloy (solid-liquid transformation - solidus point). During the coming year, definitive measurements related to the applications of the laser polarimetry technique to the detection of phase transformations will be made. Also, modifications to the laser polarimeter will be made to improve: stability of the optical system, calibration procedure, and ease of overall operation relating to optical alignment and data processing.
II. MSAD Program Tasks — Ground-based

Discipline: Materials Science

Project Identification: 962-25-08-27
NASA Contract No.: H-18067D
Responsible Center: MSFC

Bibliographic Citations for FY 1995:

Presentations

Printed: 28 FEB 1996
II. MSAD Program Tasks — Ground-based

Microgravity Chemical Vapor Deposition

Principal Investigator: Dr. Ivan O. Clark

NASA Langley Research Center (LaRC)

Co-Investigators:

Jesser, W.A.
Hyer, P.V.
Johnson, E.J.

University of Virginia
Lockheed Engineering & Sciences Co.
Lockheed Engineering & Sciences Co.

Task Objective:

This research will develop a better understanding of the scientific principles underlying chemical vapor deposition (CVD). The proposed research will determine to what extent microgravity can elucidate and separate these competing phenomena and will form the basis for a proposal to perform a series of flight experiments to more fully elucidate these scientific principles.

Task Description:

Ground-based experimental and numerical investigations will provide both basic scientific information on the heat and mass transfer effects central to the CVD process and define specific follow-on reduced-gravity investigations. This multi-pronged approach will maximize the utilization of available resources and capabilities. In the numerical modeling, both finite difference and spectral element techniques will be used and the predictions compared. In the experimental phases of the effort, a horizontal CVD reactor design will be used for the growth of a model material, such as aluminum, and a commercially important material, InP. Laser velocimetry measurements of the flow fields in the reactor will also be performed.

Task Significance:

CVD is an extremely important industrial technique with applications in the fields of semiconductors, optics, and corrosion resistance. The nature and quality of the layers formed are dependent on mass and energy transport as well as homogeneous and heterogeneous chemical reactions and nucleation. Commercial CVD processes currently employ reactors developed through decades of empirical trial and error. Scientific understanding of the CVD process is limited by the difficulty of separating the heat and mass transport due to externally forced convection and that of the internal processes of buoyant thermal convection, buoyant solutal convection, and thermal (Soret) and solutal diffusion. There is also forced convection due to volume changes arising from both reactive chemistry and thermal effects. A better understanding of these effects is essential to achieve desired improvements in perfection, uniformity, and size of grown layers and to provide an engineering design basis for these systems.

Progress During FY 1995:

The design of the test vessel is being refined using a parametric numerical study of geometric vs analytical resolution requirements for measuring the reactor effects. A candidate organometallic material has been selected and efforts are underway to develop the thermophysical properties database necessary to accurately model deposition.

Thermal imaging and particle imaging velocimetry (PIV) techniques have been applied to a model chemical vapor deposition (CVD) reactor. The thermal imaging provided much improved boundary conditions for use in the numerical modeling effort. It also demonstrated that jets were present in the reactor when hydrogen test gas was used and that the thermal image of these jets could be used as a diagnostic tool to improve the repeatability of experiments in CVD reactors. The effects of entry geometry on thermal field development have been studied with three different geometries and with three different carrier gasses.
II. MSAD Program Tasks — Ground-based

Discipline: Materials Science

Students Funded Under Research: TASK INITIATION: 11/92 EXPIRATION: 11/95
PROJECT IDENTIFICATION: 962-21-06-09
RESPONSIBLE CENTER: LaRC

Printed: 28 FEB 1996

II-619
Glass Formation and Nucleation in Microgravity: Containerless-Processed, Inviscid Silicate/Oxide Melts (Ground-Based Studies)

Principal Investigator: Dr. Reid F. Cooper  
University of Wisconsin, Madison

Co-Investigators:  
Perepezko, J.H.  
University of Wisconsin, Madison

Task Objective:

1. **Nucleation by internal oxidation or reduction of transition metal-bearing silicate glasses and melts.**

   If a change in valence state of a transition metal cation within a silicate melt is associated with a change in its structural role within the melt, one might be able to effect internal homogeneous nucleation within the melt via a change in the external environment, for example, by a redox reaction. Critical to the hypothesis is the nature of transition metal cations to make the melt into a semiconductor: Conduction electrons or electron holes are majority defect species and thus serve to decouple cation and anion diffusion fluxes that occur in an oxygen chemical potential gradient. One consequence is that oxidation or reduction reactions can occur internally (i.e., within the body of the melt) instead of solely on the surface. These reactions can result in the destabilization of the melt such that crystallization reactions occur in finely (nm-scale) dispersed regions of the melt body (e.g., the formation of Fe$^{3+}$-bearing spinel precipitates via the internal oxidation of an originally Fe$^{2+}$-bearing aluminosilicate melt). One can thus create fine-grained glass-ceramics from what would normally be non-glass-forming melts. Specific research involves control reaction experiments on silicate glasses and levitated reaction experiments (aero-acoustic and electrostatic levitation) on silicate melts.

2. **Internal nucleation of inviscid pseudobinary silicate melts via metastable liquid-phase immiscibility.**

   Binary alkaline Earth oxide-silicate melts are highly exothermic. Nevertheless, the structural variations between highly polymerized (silica-rich) and poorly polymerized (silica-poor) silicate liquids result in the creation of composition zones (on the silica-rich end of the phase diagram) where a single silicate liquid is not stable. On the silica-poor end of the diagram, this immiscibility would be metastable. As a consequence, if one can sufficiently undercool an inviscid, silica-poor melt, one could perhaps cause metastable amorphous-phase separation to occur prior to any crystallization. The phase separation could further promote the internal, fine (μm)-scale, uniform nucleation and crystallization of the material. The creation of unique glass-ceramic materials becomes a possibility. Scientifically, measurements of heat evolution rate in such droplets will address questions concerning the role of amorphous phase separation in crystalline nucleation.

Task Description:

Two research approaches are employed for the two tasks:

1. **Containerless processing for oxidation of Fe$^{2+}$-bearing alkaline Earth aluminosilicate melts via aero-acoustic levitation (AAL).** Small droplets (~3mm diameter) of ferrous iron-bearing calcium aluminosilicate glass, prepared initially by bulk melting in a controlled-oxygen activity furnace, are levitated and remelted using AAL and laser heating. The droplets thus formed are evaluated for their surface reactions, using Rutherford backscattering spectroscopy (RBS), and for their internal reactions using analytical transmission electron microscopy (AEM and TEM). The kinetics of the redox reaction are evaluated as functions of temperature, time, and oxygen activity, the latter controlled via the gas used as a levitation medium. The results of these experiments are compared to those done at low temperature on glasses of identical composition; with such a check, the study can be later extended to melts too inviscid to be glass formers. The nature of nucleation as affected by local oxygen fugacity will be explored using electron diffraction study of the internal oxidation front.
2. Drop-tube processing of pseudobinary silicate melts. Binary MgO-SiO$_2$ metasilicate compositions near the deep cristobalite (SiO$_2$)$_n$-enstatite (MgSiO$_3$) eutectic are melted in a drop tube. Initially fine, crystalline powder, the fine droplets are allowed by the degree of undercooling to experience metastable phase separation. Those droplets receiving sufficient undercooling to additionally penetrate the glass transition can be thermodynamically analyzed to explore the nature of nucleation in phase-separated amorphous materials. Primary analysis tools of the processing include secondary electron emission microscopy (scanning electron microscope), X-ray diffraction, TEM and electron microdiffraction, and differential thermal analysis and scanning calorimetry. These data should allow discrimination of the role of amorphous-amorphous interfaces on crystalline nucleation in the phase-separated amorphous droplets. The study will be extended to the binary Al$_2$O$_3$-SiO$_2$ system, the alumina-rich end producing highly inviscid melts that, if sufficiently undercooled, could produce interesting alumina/mullite glass-ceramics.

Task Significance:
Both of the specific tasks in this research are critically dependent on containerless processing: in both cases, avoiding containers eliminates the most blatant source of heterogeneity's that could promote heterogeneous nucleation. In the case of redox reactions (Task I) the containerless requirement is additionally (and particularly) important in that chemical (as opposed to structural, e.g., nucleation reactions can grossly affect the ionic-scale dynamics and structure of a transition metal-bearing melt. For example, noble-metal crucibles (e.g. platinum) that are often employed to contain refractory ionic melts will alloy with the transition metal ions incorporated in the melt: chemical diffusion different from that desired in the redox experimental results.

Microgravity comes into play when working with inviscid ceramic melts, specifically in the same two manners cited for dealing with molten metals: (1) density contrast amongst phases and (2) the need for quiescence. Both of these aspects are evident in our levitated melt drop experiments: melt viscosities of approximately 1 Pa.s (10 Poises) allow for shear-force-induced convection (droplets are far too small for thermal convection at this viscosity; processing of modestly larger melt bodies could promote thermal convection), which allows continuous exposure of new melt to the outside atmosphere thus short-circuiting the desired chemical diffusion process; in droplets avoiding convection, Ostwald ripening of internally formed ferrites allows them to sink to the bottom of the droplet, thus removing them from positions to act as internal nuclei for silicate phases.

Progress During FY 1995:
Efforts on this project concentrated in two specific areas in FY 95:

(1) Development of a physical model to describe the dynamics of oxidation in ferrous iron-bearing alkaline earth aluminosilicate glasses/melts and the application of this model to the experimental results on both natural and synthetic glasses and levitated melts and (2) initiation of a study of dynamic reduction in ferrous iron-bearing alkaline earth aluminosilicate melts.

Our experimental results on the oxidation of ferrous iron-bearing glasses and (levitated) melts provided a complex data set describing a complicated chemical diffusion process involving the motion of many ions in response to the oxidation potential. While we had known for a while since commencing this work that the one species that does not move in response to the oxidation potential is molecular or ionic oxygen, i.e., that the dynamic response is provided by cation motion that is charge-compensated by a counterflux of electron holes (polarons), the motion of which species and the thermodynamical/structural evolution of the glass or melt specimen was difficult to model. Happily, we made some intellectual breakthroughs this year, discovering that, in glasses of basaltic composition, rapid chemical diffusion of alkali ions to the oxidation front allows the oxidized glass to avoid structural change leading to nucleation of crystalline ferrites; the ion backscattering spectra and transmission electron microscopy results could thus be integrated into a single model. This model, to be published in Geochimica et Cosmochimica Acta (the premiere journal in the chemistry and physics of silicate glasses and melts) in 1996 (see bibliography), became the basis of understanding the physical process of the isothermal undercooling that occurs in the levitated melt droplets. The manuscript describing the melt dynamics should be submitted this autumn.
Experiments were initiated in dynamic reduction. Specifically, ferrous iron-bearing magnesium aluminosilicate melts (droplets suspended from F metal wire) were reduced in a carbon monoxide/carbon dioxide flowing gaseous buffer. The chemical diffusion process so involved should be (and appears to be, from our initial work) the kinetic mirror image to the oxidation process: polarons should flux to the free surface while reduced iron ions/atoms diffuse inward. The result is the internal nucleation/crystallization of metallic iron. The precipitation appears at first inspection to be periodic (Liesegang band formation). The belief is that the metal precipitates will have notably slower coarsening kinetics than ferrite precipitates in oxidized melts of the same original composition; as such, the reduction process may prove a better approach to internal nucleation of inviscid oxide and/or silicate melts than oxidation. This reduction work will be our future emphasis, and is the specific focus of the MSAD renewal proposal submitted in this fiscal year and presently awaiting decision.

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BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals


Presentations
Cooper, R.F. "Kinetic process of oxidation in Fe(superscript 2+)-bearing aluminosilicate glasses (and melts?): redox without oxygen diffusion." Petrology Seminar Series, University of California at Los Angeles, Los Angeles, CA, 1 November 1994.


Smith, D.R. and Cooper, R.F. "Homogeneous nucleation during oxidation and reduction of transition-metal-bearing silicate melts and glasses: Application to the microgravity containerless processing of inviscid ceramic melts." Wisconsin Space Grant Consortium Fifth Annual Conference, University of Wisconsin-Whitewater (Whitewater, WI), 17 August 1995.
II. MSAD Program Tasks — Ground-based

Directional Solidification in $^3$He-$^4$He Alloys

Principal Investigator: Prof. Arnold Dahm
Case Western Reserve University

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
The goal of this research is to enhance our fundamental understanding of crystal growth kinetics, liquid-solid interface morphologies, and the stability of alloys.

Task Description:
Preliminary observations of dendritic growth in two-dimensional samples of $^4$He and mixtures growth of the planar interface have been made. Systematic studies of cellular and dendritic growth in $^3$He-$^4$He alloys will be conducted to complement work that has been done in other alloys. Attempts to observe the initial instability of the planar interface will be undertaken.

Task Significance:
The study of morphologies is of intrinsic interest in testing theories of non-linear systems. Results which differ from those of classical alloys will guide both theorists and experiments in their future studies, and significant differences should result in new ideas for space-based alloy growth experiments. $^3$He-$^4$He alloys have a large advantage over other alloys in that the parameters which control the interface morphology, such as the latent heat, thermal conduction, $^3$He mean free path, and the phase diagram (temperature versus concentration), can be varied over a large range by a small change in temperature or solute concentration.

Progress During FY 1995:
The goal of this research is to observe dendritic and cellular growth of the in $^3$He - $^4$He alloy interface in regions with different values of the parameters which control the interface morphology and, in particular, in the temperature range near 1.4 K where the melting curves for different concentrations of $^3$He cross.

In our experiment solidification is driven by a pressure differential at constant temperature. Solidification occurs at pressures $\geq 25$ atm. Our initial attempts to observe cellular patterns led to some unforeseen difficulties. These include difficulty with consistency in sealing of glass windows onto the cell, $^4$He convection currents in our cell, blocking of the fill capillary with the solid phase, growth of the solid phase on the inner sides of our bellows which supply the alloy to the cell, and lack of control of location of the interface in our cell.

The difficulty in sealing windows was resolved with a new technique of epoxying indium shims between the glass windows and brass frames of our cell. This is described in the publication listed in the bibliography. Convection problems were greatly reduced and possibly eliminated by placing a sieve in front of the inlet (fill) capillary through which helium is added to increase the pressure to a value greater than the melting pressure to supercool the sample. The rest of the problems were associated with wetting of metals with the solid phase of our alloy.

Pure solid helium does not wet metals. However, we discovered that with arbitrarily small concentrations of $^3$He the solid phase would begin to grow from the metallic sides of the cell instead of advancing from the horizontal solid-liquid interface in the cell. In addition, the fill capillary would block when solid wet the walls of the capillary. We undertook a study of wetting by the solid phase of $^3$He-$^4$He alloys on different materials. We found that preferential wetting of the solid phase of alloys occurs in order of decreasing wetting on brass, epoxy, stainless steel, and brass.

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steel, and indium, and the solid phase does not wet glass. We have solved the problem of fill capillary blockage and growth on bellows by replacing our CuNi capillary with stainless steel fill lines and replacing our copper bellows with stainless steel bellows. We have designed our sieve which prevents convection currents by drilling small holes in a piece of brass shim stock and coating all parts of the sieve including the channels with indium. Our present cell has glass front and rear portions for viewing and a brass frame on the sides. The brass sides of the cell are coated with indium, the top is covered with our indium coated sieve, and the bottom from which the solid phase is grown is brass. We are presently fabricating a similar cell with thin glass replacing the indium coating the sides of the cell. Each step of improvement has increased our ability to control the location of the interface. Our last attempt to study dendrites and cellular growth indicated some difficulty with growth of the solid phase on epoxy which was squeezed into the cell during fabrication. This problem should be easily resolvable.

Our preliminary investigations have been in two directions: attempts to study the onset of instability of the planar interface, and dendritic and cellular morphologies of the interface. After initial attempts, we worked to eliminate problems such as convection currents and undesired locations of wetting of surfaces by the solid before proceeding to a more extensive study. Our work to date has been with a 100 ppm $^3$He concentration. Before some of our recent improvements, we were able to grow a planar hcp solid interface at arbitrarily small velocities. However, at some particular interface velocity dendritic growth suddenly appeared on a brass surface near the top of the cell so that the initial instability of the planar interface could not be studied. We believe that this was due to convection $^3$He currents and having brass surfaces in contact with the sample. We were unable to grow a planar interface of the bcc solid phase of the alloy. We do not know whether this is an inherent problem or a difference in wetting properties of the hcp and bcc phases.

It is straight forward to observe dendritic growth of the interface at larger interface velocities. We made preliminary measurements on two-dimensional samples of the alloy and pure $^4$He. Dendritic tips under some conditions, those which we studied, are of dimensions of 1 - 3 mm, while our cell thickness is 0.5 mm. Thus, heat flow is restricted to a plane. The boundary conditions on equations describing two-dimensional dendritic growth differ from the three-dimensional case. Some of the patterns we observed appeared to be cellular, although we are not sure of this. We believe that we have resolved the most severe problems which have interfered with a controlled study, and will begin a systematic study when our glass coated cell is sealed.

Our program is to first study the instability of the planar interface at different temperatures (different parameters). We will study the differences in planar growth of the hcp and bcc interfaces. Then dendritic growth and cellular growth will be studied. We expect to have initial phases of these investigations at a 100 ppm concentration to be finished soon. Further studies will include a more extensive study of the above and a study at different solute concentrations which will probe different parts of the phase diagram.

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**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Journals**

II. MSAD Program Tasks — Ground-based Discipline: Materials Science

Advanced Photonic Materials Produced by Containerless Processing

Principal Investigator: Dr. Delbert E. Day
University of Missouri, Rolla

Co-Investigators:
Ray, C.S.
University of Missouri, Rolla

Task Objective:
The objectives of this research were to 1) investigate non-linear optical glasses which have the potential for use as ultra-fast, all optical switches and other photonic devices for communication and advanced computer application, and 2) investigate and compare the kinetics of nucleation and crystallization for these glasses prepared by containerless melting with the crystallization kinetics for the same glasses conventionally melted in a container.

Glasses with the potential for non-linear optical (NLO) applications such as the heavy metal oxide (HMO) glasses containing PbO, Bi$_2$O$_3$, and Ga$_2$O$_3$ are, in general, highly fluid and chemically corrosive. These melts readily crystallize during cooling and develop unwanted color centers primarily due to impurities dissolved from the container. The traditional procedures used to melt these glasses yield colored and chemically inhomogeneous glasses of limited usefulness for NLO applications.

Containerless melting provides the opportunity to suppress or eliminate the undesirable heterogeneous nucleation and crystallization in such melts. Since no container is used, color centers caused by impurities dissolved from a container can be completely eliminated, even in highly corrosive melts. Containerless processing offers a viable alternative for preparing glasses with improved purity and homogeneity, and NLO properties.

Task Description:
Investigations of the NLO glasses are to be conducted with two HMO compositional systems: (1) PbO-Bi$_2$O$_3$-Ga$_2$O$_3$ and (2) compositions based on TeO$_2$. The importance of the PbO-Bi$_2$O$_3$-Ga$_2$O$_3$ glasses for NLO applications have been demonstrated by scientists at Corning Inc., but these glasses had a color ranging from orange to yellow which is believed to have come from impurities dissolved from the container. Attempts will be made to obtain colorless PbO-Bi$_2$O$_3$-Ga$_2$O$_3$ glasses by changing the melting parameters that generally affect the color of a glass such as the crucible material, melting temperature, time and atmosphere, and the starting raw materials. Containerless melting technique will also be used to process these glasses so as to eliminate the color centers that are developed from the impurities dissolved into melt from a container.

Like the PbO-Bi$_2$O$_3$-Ga$_2$O$_3$ glasses, the tellurite glasses also have a high refractive index (>1.9) along with a low Abbe number (10 to 20), or high dispersion. Calculations using the empirical equation, $n_2 = 391(n_1 - 1)/V_d^{5/4}$ which was developed by Boling et al. (N. L. Boling, A. J. Glass and A. Owyoung, IEEE J. Quantum Electronics, QE-14, 601, 1978) and widely used by others, indicate that the tellurite glasses have a non-linear refractive index, $n_2$, even larger than that of the PbO-Bi$_2$O$_3$-Ga$_2$O$_3$ glasses ($n_1$ and $V_d$ are the usual linear refractive index and Abbe number, respectively, for the glass with respect to sodium D-light). Also tellurite glasses have excellent optical transmission (>85%) over a wide range, from 400 to 3500 nm. These make the tellurite glasses promising laser hosts for a variety of rare earth ions, including Nd$^{3+}$ (primary laser wavelength 1060 nm).

The tellurite glasses for NLO applications will contain oxides of heavy metals such as Nb, Ta, Pb, and Bi to ensure high density and refractive index (linear). Generally, glasses with a high refractive index also have a non-linear refractive index (see the equation above). Minor addition of the oxides of Zn, Ga, Ca, Al and Ba in these glasses will also be considered if needed to improve glass formation. The following work will be performed for both type of HMO glasses.

(1) Measure critical cooling rate for glass formation, $R_c$ (container), using the pendant drop technique (spherical glass melt hanging from a thermocouple bead) to determine the glass forming tendency for these melts.
(2) Use containerless melting techniques to prepare the glasses if \( R_c \) (container) for these glasses appear to be very high and/or contamination of the melts with the container melts is significant.

(3) Measure and compare \( R_c \) (containerless) and \( R_c \) (container). The ratio of \( R_c \) (container) to \( R_c \) (containerless) will be used to determine the improvement in glass formation for containerless melts at 1-g. The \( R_c \) (containerless) will be measured at CRI, Inc., using their aero-acoustic levitator furnace.

(4) Measure selected properties such as the density, molar volume, linear refractive index and Abbe number, chemical durability, thermal expansion coefficient, glass transition and crystallization temperatures, and the transmission in the visible-ultraviolet and IR to determine the suitability of these glasses in practical applications.

(5) Calculate the non-linear refractive index, \( n_2 \), for each glass from the equation \( n_2 = 391(n_e - 1)/V_d^{3/4} \) to identify those compositions best suited for NLO applications.

(6) Investigate and compare the kinetics of nucleation and crystallization for these glasses prepared by traditional method and by containerless fashion.

(7) Measure XRD, IR and Raman spectra and evaluate the structure of these glasses.

**Task Significance:**

The following results are anticipated from this ground-based investigation.

(1) The effect of different cations on the NLO properties of these glasses will be understood. This result will be useful to developing glasses that have the NLO properties superior to those presently available.

(2) Comparing the properties for the glasses prepared by the traditional method with those for the glass prepared by containerless melting, the need for processing these glasses in the microgravity environment of space for achieving improved NLO properties will be known. However, the absence of gravity driven convection on the NLO properties for these glasses will remain unknown. The results from several space-borne (low gravity) glass melting experiments indicate that a glass prepared in microgravity is more resistant to crystallization and more chemically homogeneous. The optical properties including the NLO and lasing efficiency for a glass processed in microgravity are, therefore, expected to be improved compared to those for a glass at 1-g.

(3) The physical, thermal, optical, and spectroscopic properties for these HMO glasses that have the potential for NLO and laser applications will be known for the first time. The kinetics of nucleation and crystallization for these glasses along with their tendency towards glass formation will also be known.

**Progress During FY 1995:**

A. \( \text{PbO-Bi}_2\text{O}_3-\text{Ga}_2\text{O}_3 \) (PBG) Glasses:

Measurements of the nucleation and crystallization rates of heavy metal oxide glasses containing \( \text{PbO}, \text{Bi}_2\text{O}_3, \) and \( \text{Ga}_2\text{O}_3 \) were completed using differential thermal analysis (DTA). Except for the glasses containing high amounts of \( \text{PbO} \) (50-60, cat%), all the glasses investigated in the present work yielded multiple DTA crystallization peaks. These multiple peaks make it difficult to determine either the activation energy for crystallization or the relative nucleation rates at different temperatures for such glasses. A single DTA crystallization peak for a glass is ideally suitable for determining the activation energy for crystallization or the nucleation rate at different temperatures.

The activation energy for crystallization for a \( \text{60PbO.10Bi}_2\text{O}_3.30\text{Ga}_2\text{O}_3 \), cat% (PBG-12) glass, which contains a single DTA crystallization peak, was determined to be \( 470\pm15 \text{kJ/mol} \). A nucleation rate type curve was measured for this same glass using a DTA technique developed by ourselves (Ray and Day, J. Amer. Ceram. Soc. 73, 439, 1990). With this DTA technique, glass samples of constant weight and particle size are first heated isothermally in the DTA furnace for a fixed time (nucleation), and then heated at a constant rate until the glass crystallizes (crystallization). A graph of the maximum height of the DTA crystallization peak, \( (dT)_p \) as a function of nucleation temperature is a curve similar to the well known nucleation rate vs. temperature curve found in many silicate
glasses. When measured and plotted as a function of nucleation temperature, the \( (dT)_p \) for this glass is the same as that of the as-quenched glass until the glass is nucleated above \( \sim 265^\circ C \). This graph reaches a maxima at \( \sim 343^\circ C \) and then decreases to the value for the as-quenched glass at \( 375^\circ C \). In other words, the temperature for nucleating this glass ranges from \( \sim 265^\circ C \) to \( 375^\circ C \) and the temperature where the nucleation rate is a maximum, is \( 343^\circ C \).

B. Na\(_2\)O-TeO\(_2\) Glasses:

The formation, crystallization, and structure of binary Na\(_2\)O-TeO\(_2\) glasses have been studied as a function of composition using differential scanning calorimetry (DSC), X-ray diffraction (XRD) and IR spectra. The objectives of this work are to gain a better understanding of (1) glass formation and crystallization kinetics of tellurite melts and (2) the structural role of the Te-ions in these glasses. This knowledge will be valuable in controlling the composition of tellurite (TeO\(_2\)) glasses that have potential for non-linear optical (NLO) and laser applications. For DSC measurements, about 20 mg of glass powder composed of \( \sim 100 \) mm particles was sealed in an aluminum pan, which was then heated at \( 10^\circ C/\text{min.} \) in an atmosphere of flowing nitrogen until crystallization was complete. The DSC crystallization curves for five glasses, composition of Na\(_2\)O.2TeO\(_2\) (NT\(_2\)), Na\(_2\)O.3TeO\(_2\) (NT\(_3\)), Na\(_2\)O.4TeO\(_2\) (NT\(_4\)), Na\(_2\)O.5TeO\(_2\) (NT\(_5\)), and Na\(_2\)O.6TeO\(_2\) (NT\(_6\)) have been measured to date. With the exception of the NT\(_4\) glass, all the other glasses exhibit multiple crystallization peaks, indicating crystallization of two or more phases. The onset temperature for crystallization is highest, about 390 \( \pm 10^\circ C \), for the NT\(_4\) glass indicating this glass is the most stable of those measured. These sodium tellurite glasses were crystallized at their respective maximum temperatures (obtained from DSC) for 24 h and analyzed by XRD. The crystalline phase(s) found in each glass agree with the published phase diagram for the binary Na\(_2\)O-TeO\(_2\) system, with the exception that in the devitrified NT\(_4\) glass, both NT\(_4\) and NT\(_3\) crystals should be present according to the phase diagram, while only NT\(_4\) crystals were identified in this sample.

The IR spectra for these sodium tellurite glasses and their devitrified counterparts have been measured using the KBr pellet technique. Analyses of the IR spectra indicate that the structure of the NT\(_4\) glass contains primarily [TeO\(_4\)] bipyramidal groups, while the structure of all the other glasses contain both [TeO\(_3\)] pyramidal and [TeO\(_4\)] bipyramidal groups. Further analysis of the IR spectra for these glasses is continuing.

The specific heat, \( c_p \), for the Na\(_2\)O.4TeO\(_2\) (NT\(_4\)) glass was also measured as a function of heating rate, q. At q=20 C/min, the glass transition region ranges from 522 to 540 C. The specific heat below the glass transition, \( c_p(g) \), for this glass is about 380 mJ/g/deg whereas the \( c_p \) above the glass transition region, \( c_p(l) \), is about 840 mJ/g/deg. The average glass transition temperature, \( T_g \), increases with increasing heating rate and a plot of \( \ln(q) \) vs \( 1/T_g \) is a straight line. The slope of this straight line, which is the activation energy for glass transition, was about 530 kJ/mol for the NT\(_4\) glass.

C. PbO-Nb\(_2\)O\(_5\)-TeO\(_2\) (PNT) Glasses:

A new class of optical glasses in the system PbO-Nb\(_2\)O\(_5\)-TeO\(_2\) (PNT) has been developed. Their glass formation tendency, selected properties, and glass structure have been investigated with the aim of identifying glass compositions suitable for nonlinear optical (NLO) and laser applications. The properties investigated as a function of composition for these glasses include density, molar volume, thermal expansion coefficient, dissolution rate in solutions of different pH, transmission in the ultraviolet-visible (350 to 750 \( \mu \)m) and infrared (1 to 22 \( \mu \)m), and glass softening, transition and crystallization temperatures. The structure of these glasses were investigated using IR spectra from 450 to 2000 cm\(^{-1}\). The composition-property relations and IR spectra suggest that Nb\(^{5+}\) ions are located in the network and improve the glass formation tendency and chemical durability. Pb\(^{2+}\) ions primarily occupy interstitial positions in the glass network and behave as network modifying or charge compensating ions.

The refractive index of one particularly interesting PNT glass (20PbO-20Nb\(_2\)O\(_5\)-60TeO\(_2\), cat\% composition) was measured as a function of wavelength from 250 to 850 nm, from which the coefficient of nonlinear refractive index, \( n_2 \), was calculated to be about 18.4 \( \times 10^{-13} \) esu. This value of \( n_2 \) for this PNT glass is almost twice the 10.6 \( \times 10^{-13} \) esu value calculated for a PbO-Bi\(_2\)O\(_3\)-Ga\(_2\)O\(_3\) (PBG) glass which is considered to have the highest nonlinear refractive
index among all the oxide glasses up to this time. The higher $n_2$ value should give the PNT glass a faster response than the PBG glass in optical switching devices. A comparative property analysis also shows that most of the physical, thermal, and chemical properties of the PNT glasses are superior to those of the PBG glasses. For example, this PNT glass has a much lighter yellow color, a higher crystallization temperature (440°C compared to 410°C for the PBG glass), and 2 to 3 orders of magnitude higher chemical durability than the PBG glass. A clear superiority of these properties along with a higher optical nonlinearity make these PNT glasses more useful than the PBG glasses. Accordingly, a patent disclosure describing the PNT glasses has been filed with the University of Missouri subsequent to submitting a patent application.

D. Li$_2$O.2SiO$_2$ (lithium disilicate) Glass:

A knowledge of the primary crystallization mechanism, surface and/or internal (volume), in glasses is important to determining the heat treatment schedule used to fabricate glass ceramic materials like cordierite. Using a lithium disilicate (LS$_2$) glass, a DTA method has been developed that distinguishes surface from internal (or volume) crystallization. This method is fast, convenient, and requires only a small quantity of sample, about 1 g, to identify the dominant crystallization mechanism in the glass.

In this DTA method, either the maximum height of a crystallization peak, $(\Delta T)_p$ or $T_p^2/(\Delta T)_p$ is plotted as a function of particle size, where $T_p$ is the temperature at $(\Delta T)_p$ and $(\Delta T)_p$ is the width at half peak maxima (peak width). The amount of sample, about 40 mg, and the $\Delta T$ heating rate, about 15°C/min, were kept constant for all measurements with different particle sizes. Both $(\Delta T)_p$ and $T_p^2/(\Delta T)_p$ increase with increasing particle size when internal crystallization predominates and decrease when the glass crystallizes primarily by surface crystallization. This DTA method demonstrates that an as-quenched, unnucleated LS$_2$ glass crystallized primarily by surface crystallization and the same glass crystallized by internal crystallization when doped with small amounts of platinum. The surface crystallization of the LS$_2$ glass was enhanced when the glass particles were deliberately exposed to moisture prior to DTA measurements. These DTA results were confirmed by scanning electron microscopy (SEM) of crystallized bulk glasses. The validity of this DTA method is now being tested theoretically at the Washington University in St. Louis and experimentally at the University of Missouri-Rolla using NT$_n$, PNT, and PBG glasses.

**STUDENTS FUNDED UNDER RESEARCH:**

| BS Students: | 0 |
| MS Students: | 0 |
| PhD Students: | 1 |

**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Journals**


**Proceedings**

The Effect of Gravity on Natural Convection and Crystal Growth

Principal Investigator: Dr. Graham D. de Vahl Davis
University of New South Wales

Co-Investigators:
deGroh III, H.C.
NASA Lewis Research Center (LeRC)

Task Objective:
1. To experimentally measure natural convection during Bridgman growth at varying gravity-driven levels and to quantitatively determine how this convection affects parameters of critical importance to the crystal grower such as interface shape, and radial and longitudinal segregation.

2. To produce an accessible, experimentally verified numerical code capable of accurately determining levels of convection in real systems at varying gravity levels and directions, and the effects of this convection on the solidification process.

3. To supply and supplement numerical modeling efforts for the MEPHISTO II flight experiment of Dr. R. Abbaschian.

Task Description:
The major goal of many space experiments in solidification is to determine how convection influences various phenomena such as undercooling at the solid/liquid interface, redistribution of solute, and crystal quality. However, in most flight experiments, as in the MEPHISTO experiments which are being done using an opaque alloy of Bi-Sn, no measurements of convection are possible. Experiments and numerical studies are being done to estimate convection and its effects. Two codes are under development: 1. A modified version of the commercial finite element code FIDAP (due to M. Yao), and 2. A finite difference code (due to G. de Vahl Davis, E. Leonardi and students). Both may be available to investigators. In our experiments, we are using the transparent metal analog succinonitrile, which allows direct analysis of convection and interface shape.

Task Significance:
Our research will yield experimentally verified codes capable of calculating amounts of convection present in the melt at 1-g horizontal, vertical, and various intermediate angles, and in low-g with various residual gravity values, angles and frequencies. Thus it is hoped that current flight experiments, as well as future flight experiments, will benefit from this research. The models will complement the scaling, analytical modeling and parametric experimental studies of others (Coriell, Favier, Thevenard and Camel) and will be used to check the validity, extension and generalization of scaling laws.

Progress During FY 1995:
Closer collaborations have developed among MEPHISTO teams in France, the University of Florida, NASA Lewis, and the University of New South Wales. de Groh and de Vahl Davis are listed as CoI’s on R. Abbaschian’s MEPHISTO-4 relight proposal. An effort to assist the French on MEPHISTO-3 is also underway; in this effort, given the initial segregation of the starting sample, homogenization times, heat, mass and solute transport at different gravities are being modeled. Modeling research done during the summer of 1994 on MEPHISTO-1 and -2 was completed, published, and presented (see publications list).

The study of heat and mass transfer during solidification using succinonitrile and modeling of the system continued. Calibration systems for stereo image velocimetry have begun with M. Bethea and examination of PAF dyes for an additional flow velocity measuring system began with W. Lempert of Princeton University.
II. MSAD Program Tasks — Ground-based

STUDENTS FUNDED UNDER RESEARCH:

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TASK INITIATION: 11/92  EXPIRATION: 10/95

PROJECT IDENTIFICATION: 962-21-05-09

RESPONSIBLE CENTER: LeRC

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals


NASA Tech Briefs


Proceedings

Use of Synchrotron White Beam X-ray Topography for the Characterization of the Microstructural Development of Crystal - Normal Gravity Versus Microgravity

Principal Investigator: Dr. Michael Dudley
State University of New York, Stony Brook

Co-Investigators:
Larson, D.
Northrop-Grumman

Task Objective:
The objective of the research proposed here is to provide an assessment of the influence of the accelerated cooling rates, imposed by severe limitations on available flight time, on the defect microstructure of crystals grown in a microgravity environment (using, for example, modified Bridgman or Vapor Transport techniques). Results previously obtained on ground-based ZnTe samples (grown by Vapor Transport at NASA Marshall Space Flight Center) seem to indicate that if cooling rates are too high the accentuated thermal mismatch stresses can give rise to deformation processes, comprising the formation of dislocation slip bands.

The objective of the present research is to extend the preliminary work on ground-based samples, to further ground-based samples, and also to flight samples grown with different cooling rates. The microstructures of the as-grown crystals will be non-destructively characterized using the technique of Synchrotron White Beam X-ray Topography (SWBXT). Correlations between the existence of significant post-growth deformation and accelerated growth rates will be directly examined.

The influence of accelerated cooling rates on the significance of the comparison drawn between normal gravity and microgravity crystal growth can thus be determined.

Task Description:
The research proposed here will be carried out using Synchrotron White Beam X-ray Topography (SWBXT) at the Stony Brook Synchrotron Topography Facility, with is under the direction of Prof. Dudley. It will consist of SWBXT characterization of crystals grown in a microgravity environment primarily obtained from co-investigator Dr. D.J. Larson, Jr., of Grumman Corporate Research Center, as well as from other sources such as Drs. D. Gillies and C.-H. Su at NASA Marshall Space Flight Center. Crystals of CdZnTe and ZnTe will be examined, although many other systems of interest to NASA are expected to be studied, for example ZnSe, CdTe, HgCdTe and PbSnSe. Opportunities will be sought amongst the NASA crystal growth community to maximize the amount of data obtained on as many different systems as possible. Crystals grown at various cooling rates will be examined, and the influence of these rates on the resulting microstructure will be assessed.

In the approach adopted here, reflection topographs from the cylindrical outside surface of the as-grown boules will be initially examined. These will provide information on the defect structures in the region from the outside surface down to the penetration depth of the x-ray beam, which is typically 5-10 \( \mu \text{m} \). This enables an overall assessment of the distribution of defects such as twins, sub-grain and grain boundaries, and dislocations to be made. This can aid in the development of optimal wafering geometries to enable clearest visualization of the defect microstructure. Following this, both reflection and transmission topographs will be recorded from the individual wafers. Images obtained from the various wafers will be compared with those obtained from the original boule, and an overall representation of the three dimensional distribution of defects throughout the boule will thus be developed. By comparing such three dimensional representations of defects in crystals grown with various cooling rates, the influence of the cooling rate on the type and distribution of defects in the crystals can be determined.

It is anticipated that results may indicate that accelerated cooling rates, dictated by schedule limitations, have a dominant effect on the microstructure of crystals grown in a microgravity environment, thus obscuring information on the influence of the magnitude of the gravity vector. It is further anticipated that these results may lead to a
change in the criteria used to determine time allocation for individual crystal growth experiments on future flights in order to avoid effects on crystal microstructure resulting from effects unrelated to the magnitude of the gravity vector.

Task Significance:
Much effort has been, and continues to be, expended by NASA in evaluating the influence of a microgravity environment on the defect microstructures developed in crystals during crystal growth. While the influence of a microgravity environment on crystal growth is generally accepted as being beneficial, it is not clear that prior studies of this influence have been unperturbed by artifacts related to the particular choice of experimental conditions. Indeed, in order to properly compare crystal growth in a microgravity environment to that in a normal gravity environment it is important to be able to isolate the influence exerted by the difference in magnitude of the gravity vector on the resultant crystal quality. Effects associated with accelerated cooling rates, or sudden changes in temperature gradient, imposed either deliberately or inadvertently, detract from the significance of such experiments. Results already obtained from ZnTe crystals grown in a normal gravity environment seem to indicate that stresses generated by thermal mismatch effects during cooling can significantly affect the observed, as-grown defect microstructure. These stresses lead to the formation of dislocation slip bands which tend to obscure and/or break up the true growth defect microstructure. Should such effects occur in crystals grown in a microgravity environment, the true influence of microgravity on crystal quality cannot be assessed.

It is thus of paramount importance to be able to assess the effect of the accelerated cooling rates, dictated by flight schedule limitations, on resulting defect microstructures. One can gain insight into the chronological history of the development of the final defect microstructure from analysis of images recorded using Synchrotron White Beam X-ray Topography (SWBXT). This is a non-destructive imaging diffraction technique capable of revealing the detailed defect microstructure in large single crystals. Such defect microstructures in crystals grown with various cooling rates will be compared and examined for evidence of extensive, post-growth slip. Maximum usable cooling rates can thus be determined.

Determination of the influence of cooling rate on the defect microstructure of crystals is crucial for selection of experimental conditions under which the effects of the gravity vector on crystal growth quality can be usefully investigated. Once such selection has been optimized, differences in microstructure observed in microgravity grown crystals may be safely attributed to the influence of the gravity vector, and not to artifacts related to compressed growth schedules.

Should accelerated cooling rates be shown to have a detrimental effect on defect microstructures in microgravity grown crystals, it is likely that this result could modify the criteria used to determine the experimental schedules for microgravity crystal growth.

Progress During FY 1995:
In addition to continuing the previously reported characterization of CdZnTe (CZT) flight samples, the systems also studied include CZT crystals grown at MSFC by the PVT method, Ga doped Ge crystals grown at MSFC by directional solidification, and HgCdTe (MCT) flight samples grown at MSFC by the traveling heater method.

Systematically recorded topographs from wafers cut from the two CZT flight samples grown by Dr. D.J. Larson, Jr. show that defect structures in these samples are strongly influenced by cooling rates. In flight sample number 2, no occurrence of significant slip processes can be observed. However, in the flight sample number 1, which was more rapidly cooled than flight sample number 2, extensive slip bands belonging to the \(\{01\overline{1}\}\{11\overline{1}\}\) and \(\{01\overline{1}\}\{11\overline{1}\}\) (1' indicates a "1" with a line across the top) slip systems nucleated from the periphery propagate into the interior of the crystal. This observation strongly suggests that stresses induced by rapid cooling and constrained growth give rise to deformation process. Moreover, numerous subgrain boundaries generated in the area close to very rapidly cooled regions in flight sample number 1 which indicates that large thermal mismatch stresses associated with rapid cooling can be a major driving force for the formation of these subgrain boundaries. Our observations strongly suggest that modification of the cooling rate has a significant effect on the final crystalline perfection of such materials.
Defect structures in CZT ground-based samples grown by the PVT method were studied using SWBXT. Defects such as precipitates and slip bands were observed. Extensive 180° rotation twins comprising both large-volume, macroscopic twins, and thick twin lamellae were also revealed. On the other hand, irregular distributions of slip bands across twin boundaries indicate that the twinning process may have occurred earlier than the slip process and that the twin boundaries may act as barriers to dislocation motion, which agrees with previous observations in crystals of ZnTe. Subgrain boundaries of two different configurations were observed in these CZT samples. The first type is composed of dislocation cell structures with subgrains of 50-200 μm in width. The formation of dislocation cell structures may be attributed to dislocation glide and climb caused by thermal-gradient-induced stresses generated during crystal growth. On the other hand, the formation of the second type of subgrain boundary, which are long and straight, running approximately parallel to <110> directions, may arise from polygonization of slip dislocations generated during the post-growth cooling process. These observations indicate that thermal stresses induced during the crystal growth and post-growth cooling processes can play an important role in the development of defect structures in these as-grown crystals. Therefore, an optimization of thermal conditions is required to reduce the dislocation density in the as-grown crystals.

Directionally solidified Ga-doped germanium crystals subjected to Peltier pulsing were studied using SWBXT. Both the melt-back interface and marked growth interfaces were clearly visible. Our observations reveal that growth interfaces were axisymmetrically concave facing the liquid during seeding and remain concave during continued growth. The spacing between adjacent striations increases gradually indicating that the growth rates increase during crystal growth. Structural defects such as subgrain boundaries comprising dislocation cell structures and precipitates were characterized. Direct evidence of dislocations present in the seed crystal propagating into the as-grown crystal through melt-back interfaces during crystal growth was also obtained.

Structural defects in an MCT flight sample grown at MSFC using the traveling heater method and obtained from Dr. D.C. Gillies were studied by SWBXT. Defects such as subgrain boundaries inclusions, surface scratches, and a micro-crack were observed. No twins nor lattice distortion associated with inhomogeneous strain can be observed. Our preliminary results reveal that the MCT crystal exhibits a very high crystalline quality.

**STUDENTS FUNDED UNDER RESEARCH:**

| BS Students: | 0 |
| MS Students: | 0 |
| PhD Students: | 1 |

**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Journals**


Proceedings


Presentations

II. MSAD Program Tasks — Ground-based

Discipline: Materials Science

Reverse Micelle Based Synthesis of Microporous Materials in Microgravity

Principal Investigator: Prof. Prabir K. Dutta
Ohio State University

Co-Investigators:
Kresge, Dr. C.T.
Ansari, Dr. R.
Meyer, W.
Mobil Research & Development Corporation
NASA Lewis Research Center (LeRC)

Task Objective:
The objective of this research is to better understand the synthesis of microporous materials, especially those based on a network of connecting substructures. Examples of microporous materials include zeolites which are widely used as catalysts and absorbents.

Task Description:
The synthesis of zincophosphate microporous material is studied via a novel synthesis route using reverse micelles. The reverse micelles isolate small portions of reactant and allow examination of mechanism of crystal formation. The crystal growth processes are studied to determine the influence of convective flows and Stokes setting. Conditions for zincophosphate growth appropriate for an orbit experimentation are being identified.

Task Significance:
Microporous materials have extremely wide application as catalysts and absorbents. Their complex structure is derived from chemical and physical transformations which have to date eluded investigators. This research separates portions of the nucleation and growth process for examinations. It is leading to a greater understanding of how these materials form, to new synthesis routes, and eventually to new materials and thereby to changes in economics of technologies, such as petroleum processing.

Progress During FY 1995:
Three paths for zincophosphate crystal growth from reverse micellar reactants have been identified. These routes appear to follow single layer additive growth, agglomeration of subsize particles and reconstruction of an amorphous gel-like mass. The synthesis procedures are appropriate for performance on orbit. It has been shown by tests using rotating cells that the synthesis is sensitive to Stokes settling. It has also been shown that laser light scattering tracks particle growth, and provides evidence for the different dynamics of the growth processes. Detailed understanding of the growth process has come from using phosphorous NMR spectroscopy, x-ray diffraction scanning, transmission and atomic force microscopy.

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TASK INITIATION: 1/93  EXPIRATION: 12/95
PROJECT IDENTIFICATION: 963-26-05-01
NASA CONTRACT NO.: NAG3-1416
RESPONSIBLE CENTER: LeRC

Printed: 28 FEB 1996
BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals

Investigation of Local Effects on Microstructure Evolution

Principal Investigator: Dr. Donald O. Frazier
NASA Marshall Space Flight Center (MSFC)

Co-Investigators:
- Rogers, J.
- Downey, P.
- Facemire, B.
- Witherow, B.
NASA Marshall Space Flight Center (MSFC)

Task Objective:
The objective of this proposed work is to perform modeling and experimental studies on the development of microstructures and the growth of a second phase in a two-phase system. Most modeling and experimental studies focus on the average particle and the late stages of growth processes. However, experimental observations of dynamic local behaviors within the context of a particle ensemble are accessible by optical techniques, including holography. Interest extends to examining these systems in all stages of growth within the limit of holographic resolution. Additionally, holography provides and archives data to employ emerging techniques for measuring diffusion fields during growth.

Task Description:
The primary method of data collection is by the use of holography. The holographic techniques are crucial to capturing the entire cell volume. A unique capability to apply phase shifting interferometry to holography is under development by one of the co-investigators of this work. With this technique it is possible to measure the concentration gradients, hence diffusion fields, surrounding individual droplets. Such data, not attainable by any other techniques, would provide essential input to computer models. Additionally, a two-color holography technique, also under development by the same co-investigator, may provide measurement of spurious thermal gradients over the lifetime of an experiment. It should be noted that use of narrow path-length cells can introduce the effects of “mixed” dimensionality, as observed in recent analysis of an experiment performed in this laboratory. “Mixed” dimensionality refers to 3-D droplets located on a 2-D substrate. Such systems follow different scaling laws than fully 3-D systems.

The research approach includes observation of diffusional growth of the secondary phase in a transparent monotectic system. Ultimately, this work will rely on the benefits of microgravity processing to eliminate the buoyancy caused by concentration or temperature gradients. Ground work requires a transparent isopycnic system. Sensitivity to gravitational fields is tested, in the laboratory, by tethering two droplets in a test cell and comparing growth kinetics at varying temperatures. Convective flows increase with increasing conjugate phase density differences which are relatively strong functions of temperature. Quench experiments in narrow path-length cells allow observation of local effects in an ensemble of droplets. Narrow path-length is a prerequisite for establishing transparency quickly in high droplet density media.

Task Significance:
To a large extent, particle growth and distribution determine the mechanical properties of an alloy. An understanding of microstructure is essential to predicting the behavior of a material with respect to, for example, metal fatigue. The unique capabilities provided by holographic techniques enabling observations of local effects during diffusional growth of a second phase in a model transparent two-phase system allows detailed analyses of the local dynamic and resulting microstructures. Additionally, holographic studies of droplet diffusion in model systems are in agreement with Rutherford backscattering measurements of Ga and Sn “island” growth on Si. Likewise, cloud dynamics derives some of its physics from such coarsening phenomena. Diffusional studies to model the time evolution of the total surface area of particles comprising polar stratospheric clouds, for example,
suspected as catalysts for ozone decomposition, can be potentially important to the study of the rate of ozone layer depletion.

Progress During FY 1995:

Observations reveal that droplet number decays as $t^{-0.733}$ and the average radius increases as $t^{0.247}$ in the asymptotic limit. This shows good agreement with theoretical predictions for diffusional growth of spherical caps on a two-dimensional substrate which is a valid approximation for the geometry of this experiment. Unlike the global averages, closer observation of finite clusters reveal that experiment varies widely from calculations by a multipole expansion method for individual droplet evolution. The multipole model calculates time dependencies of individual droplet radii, given the droplet locations. This task records detailed microstructure evolution and provides a firm basis for refining the multipole model.

The multipole model that introduces concentration gradient asymmetries ignores non-ideal long range interactions between particles. Analogous to the Debye-Hückel theory of ionic solutions, potential sinks and sources are not uniformly distributed. Sources are more likely found near sinks, and vice versa. Overall, the behavior is statistically as expected, but near any droplet (source or sink), there is an excess of "counter" droplets ("counter" droplets being droplets bigger/smaller with respect to the droplet of critical-size within a given characteristic length). The chemical potential, therefore, of any given droplet is reduced as a result of these near-neighbor influences, or screening effects, and a droplet beyond the cluster should sense, from the screened droplet, a deviation from the ideal Gibbs-Thompson boundary condition. The unique capabilities provided by holographic techniques, enabling observations of local effects during diffusional growth of a second phase in a two-phase system, allows detailed analyses of screening effects, the local dynamic, and resulting microstructures. Non-ideal considerations in low volume fraction systems are estimable on the basis of the Debye-Hückel analogy.

This group has submitted a new proposal which will address refinements to predictive models by the incorporation of non-ideal diffusional behavior between droplet clusters.

**STUDENTS FUNDED UNDER RESEARCH:**

**TASK INITIATION:** 6/94  **EXPIRATION:** 6/96

**PROJECT IDENTIFICATION:** 962-26-08-13  **NASA CONTRACT NO.:**  NCC8-054  **RESPONSIBLE CENTER:** MSFC

**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Journals**


**Presentations**


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**Printed:** 28 FEB 1996  **II-638**
II. MSAD Program Tasks — Ground-based

**Electronic Materials**

**Principal Investigator:** Mr. Thomas K. Glasgow  
NASA Lewis Research Center (LeRC)

**Co-Investigators:**  
No Co-I's Assigned to this Task

**Task Objective:**  
The major objective of this task is to provide monitoring of a number of ground-based research projects performed in academia. The task also supports some ground-based laboratory facilities and a limited research program, particularly in the areas of mixing driven by steady and time variant acceleration, salt solidification, and physical vapor transport.

**Task Description:**  
The phenomena being studied include diffusion, coarsening, solution crystal growth, physical vapor transport, pattern formation in solidification of aggregation, solute rejection and transport, and nucleation behavior. The gravitational acceleration considered ranges from constant 1-g to low- and variable g-levels (g-jitter).

**Task Significance:**  
The important feature of this work is the coordinated approach; i.e., quantitative agreement is sought between physical and numerical experiments. Attention must therefore be given to the development of diagnostic tools as well as to advanced numerical techniques.

**Progress During FY 1995:**  
Measurements of relative solid and liquid thermal conductivity have been made in support of ground-based research. Work has continued on algorithms describing pattern formation and on mixing in variable acceleration environments.

Under this task we cover the monitoring of numerous ground-based investigations. In a notable case we provided hardware originally purchased for use at Lewis to a university investigator avoiding a duplication of a $50K expense.

Lewis personnel engaged on this task also provided services to students through presentations at elementary and high schools and through summer employment of college students in our laboratory.

A new larger diameter furnace for salt solidification has been constructed.

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**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Journals**


**Printed:** 28 FEB 1996
Combustion Synthesis of Materials in Microgravity

Principal Investigator: Prof. Irvin Glassman

Princeton University

Co-Investigators:

Brezinsky, Dr. K.
Law, Prof. C.K.

Princeton University

Task Objective:
The experimental investigation involves detailed probing and quantification of the heterogeneous flame structure, and the materials characterization of the nitride powders obtained. It builds on the experience already gained in a related NSF-supported program examining the gas phases combustion synthesis of materials through metal/halide exchange reactions. The theoretical investigation involves analyses of the propagation, structure and stability of the heterogeneous flame, and the reaction mechanisms of individual metal particles in variable density.

Task Description:
A comprehensive experimental and theoretical program has been initiated to synthesize metallic and nonmetallic nitrides (especially titanium nitride) under microgravity conditions, and to understand the underlying combustion mechanisms. The program applies the Self-propagating, High-temperature Synthesis (SHS) technique to titanium (or other metal) particle suspensions in supercritical nitrogen: the nitride particles are formed upon passage of a self-sustained flame through this suspension.

Task Significance:
Microgravity prevents particle settling, agglomeration, and liquid/metal film formations and permits the use of suspensions of particles of specified characteristics. This facilitates the production of nitride powders of high purity, uniformity, and specificity. The novel use of supercritical nitrogen to provide a supply of reactant at conditions near the critical temperature and pressure avoids bubble formation that occurs when liquid nitrogen is used and also provides a means for greatly changing reactant density with a modest change in pressure because of the very high isothermal compressibility near the critical point.

Progress During FY 1995:
The experimental results obtained in the prior fiscal year in which high yields of titanium nitride conversion were obtained from the self propagating high temperature synthesis (SHS) of undiluted titanium powder in supercritical nitrogen have been the basis for further experiments and analysis. The analysis has led to the publication of a Master's thesis and the preparation and submission of an archival publication to the Symposium (Int'l) on Combustion. Further experimental work has focused on generating a fluidized bed of titanium powder that could also undergo reaction with near supercritical nitrogen in a manner similar to the loosely packed powders.

The loosely packed powders when reacted with supercritical nitrogen at pressures near 7Mpa and at initial temperatures of approximately 132K have resulted in yields of over 70%. A systematic study of the effect of pressure on the yield revealed that pressure was not a universal correlating parameter. If filtration transport of the nitrogen to the reacting particles were controlling the yield, pressure would systematically influence the yield. Instead, it was found that the initial nitrogen density was a better correlating parameter with yields obtained both at supercritical nitrogen conditions and at those reported in the literature for conversions in high pressure nitrogen gas.

These results have suggested that synthesis experiments using fluidized beds and microgravity stabilized suspensions of titanium powders would be the next steps in the investigation of the coupling of interparticle distance with the initial density of nitrogen. In accord with both the results and the original proposal, experimental work was initiated in 1995 on the generation of the appropriate fluidized beds.
II. MSAD Program Tasks — Ground-based

Discipline: Materials Science

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PROJECT IDENTIFICATION: 962-26-05-09
NASA CONTRACT NO.: NAG3-1418
RESPONSIBLE CENTER: LeRC

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals

Thesis
II. MSAD Program Tasks — Ground-based

Evolution of Microstructural Distance Distributions in Normal Gravity and Microgravity

Principal Investigator: Prof. Arun M. Gokhale
Georgia Institute of Technology

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
1. To develop the methodology for estimation of distribution of distances between microstructural features by using digital image analysis and stereological techniques.

2. To apply the methodology to quantify the evolution of microstructural distance distributions during the materials processes such as liquid phase sintering.

3. To quantify the role of gravity in the evolution of microstructural spatial distance distributions during materials processes.

Task Description:
Digital image analysis procedures will be developed and applied to obtain the relative locations of microstructural features observed in metallurgical planes. Computer codes will be developed and utilized to obtain the two-dimensional distance distributions from the image analysis data. Computer simulations of three-dimensional microstructures and stereology will be utilized to obtain quantitative information concerning the microstructural distance distributions in three-dimensional microstructure. The methodology will be applied to quantify the evolution of microstructural distance distributions of tungsten grains during liquid phase sintering of tungsten heavy alloys in normal gravity and subsequently microgravity.

Task Significance:
An important effect of gravity on microstructural kinetics is through its effect on the spatial arrangements of features in microstructure. This research program will develop the techniques for quantitative characterization of the evolution of microstructural distance distributions during materials processes. The results should be useful to gauge the relative contributions of the intrinsic materials processes and gravity on the evolution of the spatial arrangement of features in microstructures during materials processes.

Progress During FY 1995:
Interactive software has been developed to create a "montage" of a large number of microstructural fields in the memory of the image analyzing computer. The software removes the "edge effect" problems associated with individual microstructural fields, and permits measurements of centroid coordinates and sizes of a larger number of microstructural features in a series of microstructural fields. Software codes have been developed to extract the statistical descriptors of the spatial arrangement of the microstructural descriptors of the spatial arrangement of the microstructural features, such as radial distribution function, pair correlation function, and nearest neighbor distribution function. These procedures have been applied to quantify the evolution of the spatial distribution of tungsten grains in the liquid phase sintered specimens of tungsten heavy alloys supplied by Professor German of Pennsylvania State University. Computer codes are developed to simulate the three-dimensional microstructures to study the spatial characteristics like connectivity of the microstructural features.

Students Funded Under Research:

| Students Funded under Research: | BS Students: 0 | MS Students: 1 | PhD Students: 0 |

Task Initiation: 7/94 Expiration: 7/96

Project Identification: 962-25-05-29
NASA Contract No.: NAG3-1651
Responsible Center: LeRC

Printed: 28 FEB 1996
BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals


Presentations


Evaluation of Microstructural Development in Undercooled Alloys

Principal Investigator: Dr. Richard N. Grugel
University Space Research Association (USRA)

Co-Investigators:
Flanagan, W.F.
Hua, F. Dr.

Task Objective:
The objectives of this study are to be conducted in view of experiment and pertinent theory, and will, upon completion, serve to enhance our scientific understanding of solidification processes. These include evaluating the microstructural morphology, external and internal to the sample, as functions of undercooling (ΔT), composition (C₀), and volume (V₀). Furthermore, through the use of a transparent analogue material, dynamics of the solidifying process will be directly observed and recorded for subsequent evaluation.

Task Description:
A series of cylindrical lead-tin alloy samples, some 16mm in length and 8mm in diameter, have been contained in quartz tubes and placed in a well controlled furnace. Nucleation of the solid is induced by one of the monitoring thermocouples when the desired undercooling, from 5 to 25K, is achieved. The sample is then metallographically prepared and microstructural development from the point of nucleation is followed. Parameters which have been varied are sample composition, degree of undercooling, and, to some extent, sample size. This experimental procedure has been paralleled by a modelling effort which predicts the microstructural development of a single dendrite that is initiated at the upper surface and grows down into the undercooled melt. Model verification has been complemented by direct observation of the solidification process using samples based on the transparent, metal analogue, succinonitrile-water system.

Task Significance:
The microgravity environment of space is envisioned as a novel processing arena for the solidification of metals and alloys. Contamination of high temperature and/or reactive materials is expected to be minimized as a container is not required to hold the melt and the samples are not limited in size. Greater undercoolings might also result due to elimination of heterogeneous nucleation sites with the subsequent solidification microstructure expected to consist of extremely fine constituents and/or novel phases, i.e. improved material properties. Unfortunately, processing in the microgravity environment is expensive and time consuming. Consequently, the intent of this work is to conduct a thorough, ground-based investigation which will evaluate microstructural development in bulk, undercooled alloys with the aim of ascertaining the advantage of processing in microgravity.

Progress During FY 1995:
To gain insight regarding microstructural development in previously investigated hypereutectic Pb-Sn alloys, succinonitrile - 1 wt pct water mixtures were undercooled 2, 4, 6, and 8K after which nucleation was induced at the liquid's top surface. This work utilized commercial grade succinonitrile to which 1 weight percent water was added. Repeated melting and freezing cycles determined the liquidus temperature to be ~49°C. A long rectangular tube, closed on one end, with a cross-sectional dimension of 3 x 10mm was filled to a height of 20mm with the solution. The section containing the solution was then well submerged in a large water bath, the temperature of which could be accurately controlled.

Typically, the bath temperature was raised to 65°C, held for about 10 minutes, after which it was slowly cooled until the desired undercooling, as set by the controller, was reached. Nucleation was carefully induced by a copper needle at center of the sample's top surface with the subsequent solidification event being recorded by a VCR. By measuring the dendrite tip position at selected time intervals the instantaneous growth velocity could be determined.
Upon nucleation the sample temperature rapidly increases which translates to a primary dendrite growth velocity that rapidly decreases, an effect which is magnified by increasing the initial undercooling. Agreement with the model was good for $\Delta T = 4K$, suggested when $\Delta T = 6K$, but questionable where $\Delta T = 8K$. The reason for this increasing discrepancy lies in the experimental procedure.

The model assumes that a single dendrite grows into the undercooled melt while, experimentally, a great many are nucleated. While competitive growth can quickly eliminate most of these dendrites, the latent heat generated by the initial mass does contribute to raising the sample temperature. The consequence of this is an overall slower velocity for any dendrite which escapes the pack and proceeds to make its way through the melt. By including multiple dendrites into the model considerably better agreement was achieved.

The model, however, could not account for the columnar to equiaxed dendritic transition that was observed in Pb-Sn alloys with smaller initial undercoolings. Consequently, similar experiments which utilized the above mixture were undertaken with the intent of directly observing the solidification event. Upon nucleating a succinonitrile - water mixture which was undercooled 2K a small radius of dendritic growth was seen about the needle tip. While this continued to increase dendrite fragments, originating near the point of nucleation, were seen to sink to the bottom, growing along the way. As time continues the size and number of these grains increased until their eventual pile-up impedes further movement.

The equiaxed grains arise from dendrite branches which detach near the point of induced nucleation; they do not heterogeneously nucleate in the bulk liquid. These grains continue to grow while settling at an average velocity of $0.58mm/s$. The model does predict that a SCN dendrite initiated at an undercooling of 2K would require 77s to traverse a 20mm length, an average velocity of $0.26mm/s$. The average velocity of a dendrite initiated at $\Delta T = 4K$ is 1.5mm/s and, consequently, would overgrow any settling grain; experimentally, equiaxed grains are not observed with this initial undercooling. Thus, with a mechanism to provide seed grains, it follows that equiaxed microstructures can develop when nucleation is induced at small undercoolings. Work presently ongoing is leading to the conclusion that initially formed secondary arms find themselves, after recalcence, surrounded by solvent-depleted liquid and are prone to dissolve, predominantly at the root, after which the remaining arms are free to be transported about the bulk melt.

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**BS Degrees:** 0  **MS Degrees:** 0  **PhD Degrees:** 1

**TASK INITIATION:** 12/91  **EXPIRATION:** 11/95

**PROJECT IDENTIFICATION:** 963-25-07-03  **RESPONSIBLE CENTER:** JPL

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**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Journals**


**Presentations**


II. MSAD Program Tasks — Ground-based


Influence of Free Convection in Dissolution

Principal Investigator: Prof. Prabhat K. Gupta
Ohio State University

Co-Investigators:
Korpela, S.A.
Ohio State University
Cooper, A.R.
Case Western Reserve University

Task Objective:
To study the influence of natural convection in dissolution of silica glass into a melt consisting of PbO and SiO₂.

Task Description:
The following two-part task has been undertaken. The first portion of the task consists of designing an experimental apparatus. The second portion of the task involves the design of a computer program for theoretical calculations. Each portion of the task is under way.

Task Significance:
The aim of the research is to understand the influence of free convection in dissolution dynamics of solids subjected to a corrosive environment. In terrestrial applications a better understanding of this phenomenon should lead to savings by way of improved life of silica liners in containers used in batch processing of various corrosive materials. The aim of this task is to achieve a good enough theoretical model so that the proposed ground based experiments can be faithfully simulated via computer calculations.

Progress During FY 1995:
The composition of 50 mole percent PbO - 50% SiO₂ has been selected as the melt and pure silica as the solute to examine the influence of free convection on dissolution. The density variation with mole percent of silica is found to be linear with a slope of 0.081 g/cc per mole percent of PbO. The construction of the experimental apparatus has been initiated. A computer program has been written to solve the fundamental equations describing dissolution phenomenon in presence of free convection. Results have been obtained for large Schmidt numbers by solving these equations numerically.

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TASK INITIATION: 6/94 EXPIRATION: 8/96

STUDENT IDENTIFICATION: 962-25-05-24
NASA CONTRACT NO.: NAG3-1602
RESPONSIBLE CENTER: LeRC

Printed: 28 FEB 1996
Noncontact Thermal, Physical Property Measurement of Multiphase Systems

Principal Investigator: Dr. Robert H. Hauge
Rice University

Co-Investigators:
Margrave, J.L.
Rice University

Task Objective:
The long range goal this equipment development effort is to create a prototype measurement facility for the physical and chemical properties of levitated samples. Our objectives are as follows:
1. To determine the capabilities and accuracy of newly developed techniques for measurements of surface optical properties, density, temperature and surface tension of levitated solids and liquids.
2. To test the overall performance of current concentrators in combination with controlled gas flows and axial DC magnetic fields with respect to the creation of axially symmetric RF fields and control of liquid sample rotation, vibration and translation.

Task Description:
Measurement techniques for the properties are as follows:
1. Temperature: A simultaneous measurement of sample brightness at a variety of wavelengths through the visible and near infrared is obtained along with the sample emissivity and optical constants at 632 nm.

2. Density: A triggered high-resolution video camera image of the liquid drop is obtained with backlighting of the sample by a 670-nm diode laser. Images are taken when the sample has spherical symmetry, and the density is obtained from a calculation of volume with use of an accurately determined sample profile.

3. Surface tension: Surface-tension measurements are obtained from the vibrational frequencies of the oscillating drop. The vibrational frequencies are obtained from a frequency analysis of reflected or emitted light.

4. Electromagnetic levitation coils: Current concentrators which provide both vertical and lateral electromagnetic symmetry have been developed for efficient EM coupling to the sample and improved symmetry of the positioning forces. Symmetries other than cylindrical are under investigation as a means of controlling sample rotation.

Task Significance:
Accurate methods for measuring liquid metal physical properties will permit improved modeling of liquid metal flows in direct coating of metals to near final dimensions.

Progress During FY 1995:
A fifteen channel multicolor pyrometer with wavelength coverage from 0.4 to 1.5 microns has been constructed and tested for accuracy and use with levitated liquid metal samples. It was concluded that multicolor pyrometry provides an excellent means for determining wavelength dependent changes in emissivity where the emissivity is independently measured with ellipsometry at a specific wavelength.

A final report on Contract No. 958336 with the Jet Propulsion Laboratory was submitted to the Jet Propulsion Laboratory on June 30, 1995. The title of the report is THE DEVELOPMENT OF NONCONTACT MEASUREMENT TECHNIQUES FOR HIGH TEMPERATURE LEVITATED SOLIDS AND LIQUIDS. P.I., Robert Hauge, Rice University, Co.P.I. John L. Margrave, Rice University and Houston Advanced Research Center. Leif Fredin, Houston Advanced Research Center.
II. MSAD Program Tasks — Ground-based

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TASK INITIATION: 12/91  EXPIRATION: 11/95

PROJECT IDENTIFICATION: 963-25-07-04

RESPONSIBLE CENTER: JPL

Printed: 28 FEB 1996
II. MSAD Program Tasks — Ground-based  

Discipline: Materials Science

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Microgravity Processing of Oxide Superconductors

Principal Investigator: Dr. William Hofmeister  
Vanderbilt University

Co-Investigators:
Bayuzick, R.  
Vanderbilt University
Hopkins, R.  
Westinghouse S&TC
Vlasse, M.  
NASA Marshall Space Flight Center (MSFC)

Task Objective:
Since the discovery of superconductivity in lanthanide-based perovskite systems, considerable effort has concentrated on the synthesis and characterization of these materials. Melt processing techniques have shown great promise in developing bulk crystalline materials with critical current densities necessary for practical application. The YBaCuO system has received the most intense study, as this material has shown promise for the application of both thin film and bulk materials. However, little information is available on the complete melting relations, undercooling, and solidification behavior of these materials. In general, the understanding of undercooling and solidification of high temperature oxide systems lags behind the science of these phenomena in metallic systems. Therefore, this research will investigate the fundamental melting relations, undercooling, and solidification behavior of oxide superconductors through melt processing, with an emphasis on improving ground-based synthesis of these materials. The techniques developed by this project will apply to oxide superconductors in general and will provide additional avenues for research in this field. An additional goal of this program is to develop collaboration with other groups and provide access to the unique processing strategies developed by the microgravity program.

Task Description:
The proposed research will continue experiments with YBa2Cu3O7 using drop tubes at Vanderbilt University, the 105 meter drop tube at MSFC, and aero-acoustic levitation at Containerless Research, Inc. Microstructural and compositional analyses will be performed at Vanderbilt University and the Marshall Space Flight Center. Application of acoustic levitation and microwave heating at JPL, electrostatic levitation at JPL, undercooling combined with rapid heat removal in the URQT drop tube at Vanderbilt, and addition of small amounts of Ag to the material for improving electromagnetic susceptibility, will be explored and incorporated if the techniques prove useful. Also, rare-earth substituted 1:2:3 will be processed in order to decrease the melting temperature and ease melting. In order to correctly assess thermal profiles upon melting and solidification in aero-acoustic levitation experiments, the ultra high speed thermal imager (UHSTI) at Vanderbilt University will be utilized.

Task Significance:
Since the discovery of high-Tc superconductivity in copper oxide systems, much progress has been made in the area of thin film technology for small scale applications. However, the science suffers from the lack of crystalline materials with high critical current densities that are large enough for bulk or large scale applications. Melt processing in general has proven to be the best technique for producing bulk materials with significant critical current densities (<10^4 amp cm^2 in melt texturing experiments).

The novel techniques developed in this study have brought new insight into the understanding of the phase relations in this complex system. These enhancements will help to advance the understanding of the science of high-Tc superconductivity and of the material properties of oxide superconductors. Advancements have a direct impact on the production of bulk materials for large scale applications.

Progress During FY 1995:
Investigations utilizing aero-acoustic levitation (AAL) at Containerless Research, Inc. in Chicago, Illinois were performed. This technique allows for processing samples ranging in size from 2 - 3 mm and direct visual...
II. MSAD Program Tasks — Ground-based

Discipline: Materials Science

observation is possible. In prior experiments, samples were completely molten and solidified with either a cellular structure consisting of tetragonal $\text{YBa}_2\text{Cu}_3\text{O}_7$ coupled with fine dendrites of $\text{Y}_4\text{Ba}_3\text{O}_9$ or as single phase tetragonal $\text{YBa}_2\text{Cu}_3\text{O}_7$. These samples exhibited critical current densities on the order of $10^3 \text{ A cm}^{-2}$ up to about 45 kOe applied magnetic field at $5^\circ \text{ K}$ after a post-processing anneal in flowing oxygen. These experiments were extended to include processing of Gd and Nd substituted $\text{YBa}_2\text{Cu}_3\text{O}_7$ in collaboration with R.W. McCallum at Ames Laboratory. These rare-earth compounds are known to decrease the melting temperature of the material without adversely affecting the superconducting properties. In addition, a processing atmosphere of argon was used as well as dry air. Argon-processed samples exhibit large cracks and decompose to some of the more stable compounds in this system when exposed to air. The use of ultra high speed thermal imaging (UHSTI) for accurate temperature measurement over the entire sample surface proved successful, providing valuable solidification transition information.

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Printed: 28 FEB 1996
Non-Equilibrium Phase Transformations

Principal Investigator: Dr. Kenneth A. Jackson
University of Arizona

Co-Investigators:
Zelinski, B.
University of Arizona

Task Objective:
Our computer simulations have resulted in a breakthrough in our fundamental understanding of phase changes which take place far from equilibrium. These phase changes are dominated by kinetics coupled to the thermodynamic properties of the material. The objective of this program is to explore the implications and insights provided by this breakthrough using computer simulations coupled with laboratory experimental studies.

Task Description:
The experiments involve an analysis of microsegregation that will ultimately require experimentation in a microgravity environment where the effects of convection are minimized. The program combines simulations with experimental studies, and both aspects are viewed as essential in that the simulations provide new insights for the experimental work, and the experiments verify important aspects of the simulations and keep them relevant to the real world. Experimental studies include the dopant segregation during the crystal growth of the semiconductor indium antimonide. Computer simulations predict the magnitude and orientation dependence of these effects. The simulations will be used to explore, in detail, the transition between the diffusionless regime and the diffusion-dominated regime of crystallization, and these are correlated with the transformation-induced segregation effects at various growth rates and transformation temperatures. Simulations of the early stages of the development interface instabilities using a model based on the motion of individual atoms of an alloy (rather than an analytical model which assumes that the crystal is bounded by a mathematical surface and is growing into an alloy of uniform composition) will be compared with the experiment results.

Task Significance:
The structure and properties of materials produced by transformations that occur under conditions that are far from equilibrium is one of the central topics of materials science and are important for a variety of commercial processes including rapid solidification, spray powder formation, crystal growth, condensation processes, and glass formation. Non-equilibrium phenomena and segregation effects which are observed during rapid crystallization are not predicted by the standard quasi-equilibrium thermodynamic model. Our simulations capture, for the first time, the essence of these effects which occur in non-equilibrium transformations that occur in the real world. Our Monte Carlo computer simulations predict that these phenomena depend on the undercooling, on the growth rate, and on the diffusion coefficient in the fluid phase. These effects are generally important in the regime where the phase transformation takes place far from equilibrium. Projects supported by NASA using levitation and the microgravity environment are making major contributions to our understanding of nucleation and crystallization under these conditions, in the regime where these non-equilibrium effects are very important. Our simulation results provide a new framework for the interpretation of these experiments.

Progress During FY 1995:
The analytical model which describes most of the features of the simulations has been further developed. The model includes the orientation dependence, the growth rate dependence, and the composition dependence of the segregation coefficient. The segregation coefficient in the simulations does not depend on composition for dilute alloys, but is strongly composition-dependent for alloys with greater than about 5% of alloy concentration. The analytical model predicts a novel functional form for the distribution coefficient as a function of growth rate, which provides significantly improved agreement with experiments on the segregation of bismuth in silicon. The effect of crystallographic orientation on segregation have been shown to depend on the density of kink sites at the interface.
Code has been developed to incorporate a temperature gradient into the Monte Carlo program for simulations of cellular growth. This code is capable of modeling the phenomena of fluctuating growth rates which have been observed during the laser melting of alloys, which involves an interaction between the non-equilibrium growth rate and the temperature gradient. A Bridgman apparatus has been constructed for the growth of crystals of indium antimonide, in order to measure the growth rate and orientation dependence of the incorporation of dopants during growth. The apparatus includes Peltier pulsing to determine the growth rate, and the segregation of dopant is measured by spreading resistance. A solution growth apparatus has been used to measure the singularity in the growth rate of ammonium chloride dendrites growing into an aqueous solution. These ground-based experiments are subject to uncertainties due to convection, and these experimental results will require verification in a convection free microgravity environment.

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**TASK INITIATION:** 2/93  **EXPIRATION:** 2/96

**PROJECT IDENTIFICATION:** 962-25-08-29  **NASA CONTRACT NO.:** NAG8-944  **RESPONSIBLE CENTER:** MSFC

**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Journals**

**Proceedings**


**Presentations**


II. MSAD Program Tasks — Ground-based

Combined Heat Transfer Analysis of Crystal Growth

Principal Investigator: Dr. Mohammad Kassemi
Ohio Aerospace Institute

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
The technical objective of this research effort is to provide a quantitative understanding of the role of radiation heat transfer on crystal growth in low-gravity space experiments. To accomplish this goal a generalized combined conductive-convective-radiative numerical model is developed which can accommodate various crystal growth experiments.

Task Description:
Radiation heat transfer affects crystal growth in both space and ground-based processing. This is due to the high operating temperatures of the crystal growth processes, the semi-transparency of the crucible and phase change materials and the heat transfer link between the crucible and the furnace. In ground-based experiments, radiation and convection coexist and compete for dominating the heat transfer process in the ampoule. The role of radiation, however, becomes more prominent in low-gravity environment of space where convection heat transfer is considerably minimized.

A numerical methodology was developed for radiation exchange in the generalized multi-dimensional cylindrical geometries encountered in crystal growth. The radiation scheme which is based on the Discretized Exchange Factor method (DEF) uses node-to-node exchange to calculate radiation heat transfer in an absorbing emitting scattering non gray medium. One of the advantages of this radiation model is that it can be easily incorporated into existing finite difference or finite element codes.

The radiation model was incorporated into a finite element code for fluid flow and heat transfer and a generalized radiation-convection-conduction model was developed for crystal growth in cylindrical ampoules. The solution algorithm tracks the movement of the interface during the solidification process and adjusts the finite element mesh to accommodate changes in the shape and position of the growing solid. Radiation view factors are also continuously update as the geometry is altered.

Task Significance:
Low gravity simulations performed using the combined heat transfer model, clearly indicate that radiation effects are dominant during solidification of oxide crystals, such as BSO, and may cause highly stretched and curved interfaces during low gravity solidification experiments. The convection-conduction models that do not include radiation effects rigorously cannot be used to predict the shape and movement of the solidification front correctly.

Thermal radiation governs the energy balance in many earth and space processes. It plays a crucial role in production of glasses, semi-conductors, and advanced pay-off materials such as optical crystals. This study is geared towards understanding the effects of radiation heat transfer on crystal growth in both low-g and 1-g environments and to seek possibilities of exploiting these effects for improving the quality of the growing crystal.

Progress During FY 1995:
The studies in FY94 dealt with growth of pure oxide crystals such as BSO and YAG. In their pure form, these materials are almost fully transparent to radiation in the wavelength region up to 6 microns. Therefore, oxide crystals (in their pure form) are basically subject to surface radiation loss through the crystal which acts like a light
A new banded nongray radiation model was developed to compute volumetric radiation exchange inside the crystal growth ampoule. This radiation model, designed for a generalized absorbing-emitting-scattering nongray media, is based on a variation of the Discrete Exchange Factor (DEF) method. It evaluates radiation exchange between any two points within the participating media based on node-to-node exchange.

The volumetric radiation model was tested against other existing methods for computing radiation and also experimental results based on well established benchmark cases. The results of the comparisons indicate excellent agreement with benchmark experimental results (less than 5% discrepancy). It also proves that the new algorithm has similar or better accuracy compared to the other commonly used techniques such as discrete ordinate method and P-N approximations. Therefore it can be used in situations other than crystal growth, such as combustion, where radiation calculations and models are in demand needed for the nongray gases.

The volumetric radiation model and the associated algorithm were encoded and incorporated into the finite element code FIDAP. In order to further test the numerical model, a case study based on growth of semitransparent sapphire single crystal fiber from the melt was undertaken. This study was complemented by the experiments performed by Dr. Sayir (Case Western Reserve University) and by the analytical analysis performed by Professor Korpela (Ohio State University). The predictions of the numerical model for cooling of sapphire fiber by convection, conduction, and radiation, were in excellent quantitative agreement with the experimental results. Simulations were also performed for limiting cases (within the range of validity of the analytical perturbation analysis) and numerical predictions were almost exact matches to analytical solutions.

Solidification's of BSO and YAG were also considered for both typical low-gravity and ground-based experiments. The effect of increase in absorption coefficient of the solid which can be brought about by an increase in the dopant level was examined for various microgravity and ground-based applications. The numerical predictions indicate that for both BSO and YAG an increase in absorption and emission in the solid results in a reduction of heat loss from the solidification interface. In both low-g and 1g environments volumetric radiation transfer in the solid and surface-to-surface radiation exchange between the interface and the crucible seem to have opposing effects on the curvature of the crystal interface. As the opacity of the solid increases, the extent of the interface curvature and stretching decreases. From these results it can be deduced that even small amounts of doping can have a profound effect on the interface heat balance, cooling rate of the crystal, and flow structure in the melt, for both low-g and 1g solidification experiments of oxide crystals.

Work is in progress to study the intricate effect of radiation heat transfer in growth of doped oxide materials by considering its impact on both radial and axial segregation in the melt. These effects can be substantial during growth in the microgravity environment.

**STUDENTS FUNDED UNDER RESEARCH:**

**TASK INITIATION:** 1/93 **EXPIRATION:** 1/96

**PROJECT IDENTIFICATION:** 962-21-05-10

**NASA CONTRACT NO.:** NCC3-411

**RESPONSIBLE CENTER:** LeRC

**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Journals**

II. MSAD Program Tasks — Ground-based

Discipline: Materials Science

Proceedings
Kassemi, M. and Naraghi, MHN.  "Radiation-convection interactions in solidification of semi transparent crystals."
II. MSAD Program Tasks — Ground-based  

Fundamentals of Thermomigration of Liquid Zones Through Solids

Principal Investigator: Prof. Michael J. Kaufman  

University of Florida

Co-Investigators:

Abbaschian, R.  
Gokhale, A.  

University of Florida

Task Objective:

Currently, significant resources in terms of expert manpower as well as direct costs are being expended to develop advanced materials such as the high-temperature structural intermetallics MoSi2 and NiAl, and the fast semiconductors GaAs and GaSb. Despite considerable efforts in these areas, the intrinsic properties of many of these compounds, and alloys based on them, remain poorly understood to the degree needed to enhance their development and application. For example, it is unclear if the brittle nature of many of the intermetallics of current interest is an intrinsic property of these compounds or is associated with interstitial impurities or impurity phases. Consequently, there is a need not only to produce single crystals of controlled purity, perfection and orientation, but also to understand the details of interfacial atomistics during crystal growth in order to bring about such control.

For many of the exotic compounds of technological interest currently, conventional crystal growth techniques have not proven successful in this regard because of the many problems associated with container contamination or loss of stoichiometry due to preferential vaporization of one or more of the elements with high vapor pressures (e.g., arsenic in GaAs, antimony in GaSb, silicon in MoSi2, and aluminum in NiAl). In addition, these conventional techniques are not amenable to a scientific study of correlations between thermosolutal convection and key aspects of crystal growth such as the interface (growth) temperature and morphological stability. For example, the use of electromagnetic induction to produce float zones introduces an additional electromagnetic stirring component in the liquid zone and also precludes the use of most sensors to measure interface temperatures.

Task Description:

Although this method was discovered more than 35 years ago, there have been few attempts to utilize it as a means for processing materials in spite of the fact that most materials typically can be processed (e.g., grown as single crystals, joined and sectioned) at temperatures well below their melting points. In addition, coupling of this scheme with both acoustic emission/reflection and possibly Seebeck techniques should allow the interface positions, zone length, and interface temperature to be monitored in situ in real time.

Task Significance:

The focus of this research is intended to circumvent the common problems (e.g., crucible contamination and loss of stoichiometry) associated with conventional crystal growth methods by using the relatively unique technique of TGZM to grow single crystals of materials of current technological interest. In addition, the low-growth temperatures require much lower power inputs and result in enhanced safety during both ground operations and spaceflight. Also, it is anticipated that significant, hitherto unavailable data on liquid diffusivities will be generated during these experiments. Finally, it should be emphasized that the capability of producing high quality, inexpensive crystals of some of these rather exotic compounds could greatly facilitate advancements in these fields of current technological interest.

Progress During FY 1995:

Research efforts in FY95 concentrated on characterization of thermomigration of liquid aluminum zones through <100> and <111> single crystal silicon wafers subjected to stationary temperature gradients. In addition, attempts were made to determine the rate controlling step for thermomigration: either diffusion across the molten solvent
zone or dissolution/deposition at the solid-liquid interfaces. Thermomigration of the molten aluminum zone through <111> silicon wafers produced continuous zones with planar solid-liquid interfaces. <100> type wafers, however, showed rather discontinuous zones with irregular, triangle-shaped zone segments. Determination of the rate controlling step was based on establishing a relationship between migration rate and zone thickness. Results from this portion of the study proved inconclusive resulting most likely from non-uniform wetting as a result of oxidation. Continuing work on this system is focused on overcoming the oxidation at the solid-liquid interface and also on attempts at assessing the three dimensional nature and characteristics of a planar zone propagating through single crystal silicon wafers.

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TASK INITIATION: 2/93  EXPIRATION: 2/96

PROJECT IDENTIFICATION: 962-21-08-16

NASA CONTRACT NO.: NAG8-946

RESPONSIBLE CENTER: MSFC
Compositional Dependence of Phase Formation and Stability

Principal Investigator: Prof. Kenneth F. Kelton Washington University

Co-Investigators:
Robinson, M. NASA Marshall Space Flight Center (MSFC)
Rath, T. NASA Marshall Space Flight Center (MSFC)

Task Objective:
The objectives of this research are to extend computer models developed by us to model first order phase transformations involving cases where the composition of the final phases are different from those of the initial ones. This study is important to resolve conflicts in the experimental literature, to develop theoretical methods for treating both steady state and time dependent nucleation in a partitioning system, to extend the understanding of basic nucleation phenomenon, and to aid in the eventual development of techniques for making qualitative predictions of desired phases and microstructures. This research will lead to improved methods for analyzing thermoanalytic data that will have wide applicability for phase transformations studies. In particular, it will aid in the design and data analysis of remote experiments, such as might take place in a microgravity environment. Drop-tube experiments may reveal new effects of composition on the nucleation of related complex periodic and quasiperiodic phases that will likely suggest future containerless solidification and microgravity studies.

Task Description:
The steady state and time-dependent nucleation rates as a function of temperature and SiO₂ concentration will be measured in one pseudo-binary silicate glass, Na₂O.CaO.3SiO₂. Initial work by others suggests a strong compositional dependence of the steady state nucleation rate, though the dependence of the transient behavior has not been measured. The concentration dependence of the maximum undercooling in drop-tube experiments in Ti-Mn-Si and Al-Cu-Co-Si, and if time permits, Ti-Cr-Si and Ti-Ni-Zr-Si, all metallic liquids that form quasicrystals, will be measured. These data will be used to refine a model, developed previously by us, of the magnitude and time dependence of the nucleation rate as a function of composition. That model for nucleation will then be incorporated in our numerical model for phase transformations. Initial tests of that extended model will be made by comparing calculated and measured trends of DSC data for the devitrification of Na₂O.CaO.3SiO₂ glass as a function of SiO₂ concentration.

Task Significance:
Although in most situations solid phases grow from liquids having a different composition, systematic work focusing on the development of the correct treatment of phase nucleation under these conditions is needed. We are therefore carrying out a theoretical and experimental investigation of the nucleation and crystallization kinetics in model silicate and metallic systems. The insight and new theoretical framework gained from this work will enhance our basic understanding of nucleation, provide new insights into solidification processes and lead to new methods, based on computer simulations, that will allow the analysis of crystallization data under isothermal and nonisothermal conditions. One specific application of these new methods will be the analysis of data obtained remotely, such as in a microgravity environment. This work will ultimately enhance our ability to predict and control microstructural development, which is essential to the development of new materials to meet changing technological needs.

Progress During FY 1995:
During the past year, experimental and theoretical investigations of the compositional dependence of the nucleation rate and transient time were carried out.
II. MSAD Program Tasks — Ground-based

Discipline: Materials Science

1. The ability to levitate and melt Ti-3d transition metal-Si-O samples of the desired composition has been demonstrated. To better collect undercooling data by improving the positional stability of the melted alloys within the rf coil, smaller samples of Ti-Cr-Si and Ti-Cr-Si-O and new samples Ti-Mn-Si-O and Ti-Ni-Zr, which also form phases with local icosahedral order, are being prepared.

2. The first measurements of the compositional dependence of the time-dependent nucleation rate in any system were made as a function of SiO₂ in Na₂O.CaO₃SiO₂ glasses. The diffusion coefficient and ΔG, as a function of composition were estimated from the measured growth data and liquidus temperatures respectively. Based on these estimates, the interface-limited rate equations normally assumed in the classical nucleation theory do not accurately describe the data.

3. To accurately model the measured time-dependent nucleation data, we have developed a theory of diffusion limited nucleation, which couples the stochastic nature of cluster evolution with the evolving diffusion fields near the cluster. The resulting differential equations are much more stiff than the set of coupled equations central to the classical theory. They have been solved in the limits of small times; these solutions will be extended to longer times in the coming year.

Student Funding: 0 BS Students, 0 MS Students, 1 PhD Student

Project Identification: 962-26-08-14
NASA Contract No.: NCC8-049
Responsible Center: MSFC

Bibliographic Citations for FY 1995:

Journals


Task Significance:
Polymeric membranes are used for a variety of purposes ranging from water purification to environmental cleanup of effluent streams. Membranes remove impurities because of submicroscopic pores. One problem in creating submicroscopic pores in membranes is the propensity to form large pores or macrovoids which "short-circuit" the membrane's ability to separate dissolved impurities. Optimal means for eliminating macrovoids have yet to be developed owing to our lack of knowledge of what causes them. This research exploits the unique low-g environment to discriminate between two hypotheses advanced to explain macrovoid formation. The results of this research should enhance our ability to eliminate these undesirable defects during membrane manufacture.

Progress During FY 1995:
The following progress has been made in addressing the tasks listed above:

1. Specification of membrane-casting solutions: The composition range of the casting solutions (cellulose acetate, acetone, water) has been determined to satisfy the criteria of low-gravity experimentation: macrovoid formation...
These results are consistent with our original suggestion that solutocapillary flow, and not diffusion, is the cause of macrovoids; membranes cast in level flight were the same as on the ground completely perforated the membrane; membranes cast in 2-g were, according to our observations, lacking in level flight. The results were clear: membranes cast in low-g were saturated with macrovoids, some of which

guidance from Dr. Owen and from the BioServe Space Technologies Center staff, he assembled supporting flight

bifurcated fiber optic and measuring reflected

depths ranging from 80 to 259 mm. Mass transfer of evaporating acetone is extremely rapid, being driven down a steep gradient by adsorption to a 100-fold excess of activated carbon. The exact transfer rate is being determined from a modified comprehensive model (see below). The reaction is initiated by pulling a trigger and allowing a sliding block to spread the casting solution during a period of several milliseconds. Phase inversion occurs in less than 20 seconds, and the membranes are subsequently dried. This process is tracked by optical monitoring using a bifurcated fiber optic and measuring reflected light from the surface of the casting cell. The fiber-optic data are accumulated simultaneously with g-sensor and parabola-count data on a personal computer. This real-time measurement capability for studying membrane formation in low-g is novel and represents an added feature which we did not anticipate in our original proposal. It has added significantly to the value of the data.

3. Perform ground-based control experiments: Numerous membranes have been cast in the laboratory using the MCA's, and the conditions for forming macrovoids, as evaluated by scanning electron microscopy (SEM), are reproducible.

4. Carry out low-g experiments in KC-135 flights: Mr. Konagurthu completed all of the pre-flight physiological and medical procedures and is qualified to perform experiments on NASA's KC-135 low-g aircraft. Subsequent to guidance from Dr. Owen and from the BioServe Space Technologies Center staff, he assembled supporting flight hardware and performed low-g experiments for three days. About 50 membranes were cast during low-g, 2-g and level flight. The results were clear: membranes cast in low-g were saturated with macrovoids, some of which completely perforated the membrane; membranes cast in 2-g were, according to our observations, lacking in macrovoids: membranes cast in level flight were the same as on the ground -- a moderate incidence of macrovoids. These results are consistent with our original suggestion that solutocapillary flow, and not diffusion, is the cause of macrovoid formation.

5. Carry out structural characterization of membranes: SEM has been used to quantitate the incidence of macrovoids and to seek any other unusual characteristics. The only usual characteristic observed so far is "tunneling macrovoids", those that completely penetrate the membrane resulting in a straight hole from one side to the other.

6. Model development: A coupled heat- and mass-transfer model is under continuing development in parallel with experimental research. It consists of a combined FORTRAN code by Shojaie and SPRINT subroutines by Berzins. It has been partially adapted to account for heat and mass transfer in low-g.

**STUDENTS FUNDED UNDER RESEARCH:**

| BS Students: | 0 |
| MS Students: | 0 |
| PhD Students: | 1 |

**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Proceedings**
Konagurthu, S., Krantz, W.B., and Todd, P. "Use of low-g to test alternative hypotheses for macrovoid growth in polymeric membranes." Euromembranes 95 (1995).

**Presentations**
Konagurthu, S., Krantz, W.B., and Todd, P. "Experimental studies of hypotheses for macrovoid formation in polymeric membranes." Regional Meeting of the Southwestern and Rocky Mountain Division of AAAS, Norman, Oklahoma, May 1995.

Containerless Property Measurement of High-Temperature Liquids

Principal Investigator: Dr. Shankar Krishnan

Co-Investigators:
Nordine, P.C.

Task Objective:
The task objective is to measure the optical properties of high-purity liquid metals and alloys (Al, Zr, Ni, and Ni-Zr alloys) over wide wavelength (220–1100 nm), temperature (Tm + 300 K), and composition ranges under containerless conditions. Spectroscopic, pulsed dye-laser ellipsometry is used to obtain the complex dielectric functions and spectral emissivity data at high temperatures on clean liquids using electromagnetic levitation. These data are needed for accurate noncontact temperature measurement, and for measurements of the total hemispherical emittance by integration of emissivity over the wavelength range of thermal emission.

Another major outcome of this research will be the determination of the optical properties of liquids over a wide wavelength range. These measurements are of fundamental importance to advancing the theory of liquid metals. For example the presence or absence of important interband transitions provides information on the valence, bonding, joint density-of-states, and the extent to which nearly-free electron behavior is exhibited by liquid metals and alloys.

Task Description:
The research approach is to levitate liquid metals and alloys (Al, Ni, Zr, and Ni-Zr alloys) electromagnetically and use a spectroscopic pulsed dye-laser ellipsometer to measure the complex dielectric function and the spectral emissivity in the wavelength range 220-1100 nm. Pulsed radiation is generated by a Molectron dye-laser, and the wavelength is automatically set and adjusted by a laboratory computer. Light reflected by the specimen is collected and analyzed by a unique rotating analyzer ellipsometer.

The rotating components of the ellipsometer are motorized and controlled by the computer. The signals are detected by a pair of photodiodes and an EG&G boxcar averager is used to extract the mean value of the light intensities at the two photodiodes which receive the orthogonally polarized components of the reflected light. The signals are automatically measured by the computer.

A Molectron dye laser is used to generate pulsed radiation in the 220–1100 nm wavelength range. The light is steered through several mirrors, and transmitted through a pair of Glan Thomson linear polarizers. The second polarizer is fixed in azimuth, and rotation of the first polarizer allows light levels to be adjusted to the optimum values. The light is incident on the liquid specimens at a fixed incident polarization, and the reflected polarization is analyzed for its new azimuth and ellipticity.

A modified rotating analyzer ellipsometer is used to measure the outgoing polarization at 6 azimuths of the analyzer. The analyzer is of the beamsplitting type such that the two orthogonal components of the beam are simultaneously obtained. Three intensity ratios are measured at three independent pairs of azimuths. The complex dielectric constant, indices of refraction, and spectral emissivities are derived from standard equations. The light intensities are detected by a pair of high speed, UV-enhanced silicon photodiodes and measured by a pair of gated integrators.

The emphasis of measurements on liquid metals and alloys is to determine the temperature and (for alloys) composition dependence of all optical properties over the accessible wavelength range. Measurements are possible from approximately 0.8 of the melting temperature, (Tm) in undercooled liquids to at least 300K above Tm. The observed effects of temperature and composition on optical properties and the wavelength dependence of these properties are interpreted in terms of liquid structure and bonding.
The liquid nickel, zirconium, and their alloys have been chosen to be investigated in this research because they display unique glass forming behavior, typify early and late transition metals, and because they are also of interest to other NASA investigators. Measurements at 633 nm on these metals is being conducted in collaboration with Professors Bayuzick of Vanderbilt University and Johnson of the California Institute of Technology.

Task Significance:
The relevance and significance to the microgravity program are twofold. First, the spectral emissivity and total hemispherical emissivity form the basis for new noncontact thermophysical property measurements in microgravity experimentation. For example, knowledge of total hemispherical emittance allows heat capacity and thermal diffusivity to be accurately determined from free-cooling and pulse-heating experiments. Spectral emissivity data allow true specimen temperatures to be determined using optical pyrometry. Second, spectral emissivity measurements on specific materials are needed by other NASA investigators in ground-based and microgravity experimentation.

Progress During FY 1995:
During the past year, work has continued towards optical property measurements on liquid metals and alloys. Materials investigated include liquid Ni, Fe, Ni-75%Zr, and some of the materials investigated by other PI's on the TEMPUS experiments. The optical properties including the complex dielectric function and the spectral emissivities for these liquid materials have been measured in the wavelength range of 360-1000 nm (1-4 eV) and at selected wavelengths as functions of temperature. Based on these results, some modelling has also been conducted to extract approximate values of the total hemispherical emissivity needed in the analysis of heat transfer. A few papers and presentations summarizing the results have also been made. During this last year of performance, two NRA proposals have been submitted for continuing work in the area of optical and thermophysical property measurements. Work has also continued to disseminate results obtained in our laboratory over the past year, in the form of three new publications.

STUDENTS FUNDED UNDER RESEARCH:

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals


Presentations
Noise and Dynamical Pattern Selection in Solidification

Principal Investigator: Prof. Douglas A. Kurtze
North Dakota State University

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
To understand the fundamental mechanisms by which a pattern-forming system can change the spacing of its pattern, and to devise a means of calculating the preferred spacing to which external noise will drive the pattern.

Task Description:
Numerical computation of the time evolution of mathematical models of solidification fronts, and analytical arguments to extract the preferred spacing from the model equations are both aspects of this task.

Task Significance:
Properties of materials grown from the melt are largely determined by their microstructure, which in turn is strongly affected by the front shape during solidification. This research will establish whether processing in a noisy environment (such as the Space Shuttle) can lead to more reproducible, and hence better controlled, front shapes than processing in a quieter environment. This study will definitively enhance our understanding of the fundamentals of solidification. A more complete study (such as this) is required for the design of materials with specific materials properties; a difficult task, but an important practical goal.

Progress During FY 1995:
Using an HP workstation acquired through this grant, we have been investigating the unstable "saddle-point" steady states of the Swift-Hohenberg equation. The Swift-Hohenberg equation can be derived from a free energy functional, and the periodic steady state with the lowest value of this functional is the preferred state which is selected by the action of random noise. The saddle-point states are saddles on the free energy surface which separate the local minima corresponding to periodic steady states, and so they are the most likely fluctuations which change the wavelength of the pattern. We have calculated the free energies of the periodic states for small values of the instability parameter, and are working to calculate the free energies of the saddles. This will allow us to determine the rate at which noise drives the system to its preferred wavelength. This work is being done in collaboration with undergraduate research assistant Jed Overmann, who has been funded by the Science Bound program run ASEND, which administers the NSF EPSCoR grant to North Dakota. I expect that he will return to work on this project in the fall (his sophomore year) and will be supported by this grant. I am working on analytical arguments to identify whether a given steady state is the preferred one or not.

STUDENTS FUNDED UNDER RESEARCH:

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TASK INITIATION: 10/94  EXPIRATION: 10/96

PROJECT IDENTIFICATION: 962-25-05-30

NASA CONTRACT NO.: NAG3-1603

RESPONSIBLE CENTER: LeRC

Printed: 28 FEB 1996
Microstructural Development during Directional Solidification of Peritectic Alloys

Principal Investigator: Dr. Thomas A. Lograsso
Iowa State University

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
The mechanisms of and conditions required for the formation of peritectic solid dendrites, cells, or planar growth between the primary solid dendrites or cells and the transitions between these structures will be investigated. In addition, alternating bands of solid phases perpendicular to the growth direction have been observed in peritectic systems. However, the precise conditions under which these bands form and the mechanisms by which these bands form are not clearly understood. It has been postulated that the development of these different structures is related to changes in the composition of the liquid. A model has been developed that predicts the compositional variations that occur in the liquid under the assumption of no convection. This model will be compared to experimental results to evaluate the importance of convective effects on the formation of various structural characteristics.

Task Description:
This research will focus on:

1. Establishing the growth conditions in terms of temperature gradient and growth rate for the formation of planar, cellular, and dendritic growth of the peritectic solid in a two-phase peritectic under convection-free conditions, with an emphasis on the morphological transition from cellular front primary and planar front peritectic solid to single phase planar front solid; and

2. Determining the effect of convection-free conditions on the formation of a banded structure during directional solidification of a two-phase peritectic alloy.

The research approach is to initially focus on two alloy systems, In-Sn and Sn-Cd. In-Sn has a very low peritectic temperature, which allows for the possibility of direct observation during solidification. In addition, In and Sn have essentially equal densities in the liquid state in the temperature range of interest, which should minimize density-driven convection. In the Sn-Cd system, the solute element (Cd) is more dense in the liquid state, which should minimize density-driven convection when solidification occurs upward. This system has been well characterized and is more tractable in terms of sample preparation than In-Sn. These results can be compared to previous work on the Pb-Bi system, in which the solute element (Bi) is less dense and thus provides a driving force for mixing in the liquid through convection.

Task Significance:
This research effort will establish the ground-based data required for the identification of the appropriate alloy system, composition, and growth conditions for microgravity processing. Studying solidification for these types of systems is important, because a number of industrially significant materials solidify in a similar manner such as brass, bronze, some steels and high temperature superconductors. The utilization of a microgravity environment greatly facilitates the analysis and modeling of data, since gravity-induced mixing in the liquid can strongly affect the solidification process.

Progress During FY 1995:
The research effort concentrated on experiments in the Sn-Cd system using solidification conditions which produced banded microstructures. Initial experiments with different compositions allowed the selection of the alloy with the most regular and repeating bands. Focusing on this particular composition, band lengths and band compositions
were measured as a function of growth velocity. These lengths and compositions did not vary significantly with solidification distance, which suggested that long range convection did not occur in the liquid. However, the dependence of band length on growth velocity was different than that predicted by the model, indicating that other factors, such as nucleation behavior or localized mixing in the liquid near the solid-liquid interface, need to be evaluated. The band compositions showed no variation with growth velocity, which is consistent with the model. However, the specific compositions determined for the phases raise questions about the accuracy of the published phase diagram near the peritectic isotherm; a quantitative comparison of the model with the experimental results requires a verification of the phase compositions at the peritectic temperature.

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals
Numerical Investigation of Thermal Creep and Thermal Stress Effects in Microgravity Physical Vapor Transport

Principal Investigator: Dr. Daniel W. Mackowski  
Auburn University

Co-Investigators:  
Knight, R.  
Auburn University

Task Objective:  
The objective of this research is to quantitatively predict the roles of thermal creep and thermal stress on microgravity physical vapor transport through detailed numerical calculations.

Task Description:  
The research will formulate thermal creep and stress effects into a variable property, two-dimensional representation of heat and mass transfer in cylindrical ampoules under typical operating parameters. Physical conditions at which thermal creep and stress significantly alter vapor transport from a pure diffusion-limited mode, as well as the resulting effects upon mass transfer rates and mass flux uniformity at the crystal surface, will be determined. The research will benefit the prediction and development of microgravity crystal growth technology by providing, for the first time, an accurate accounting of all relevant physical vapor transport mechanisms.

The potential for growing high-quality crystals in closed, cylindrical ampoules using chemical vapor transport and physical vapor transport processes under microgravity conditions has been demonstrated in a number of experiments. The development of this technology will require an accurate modeling of the relevant vapor transport mechanisms occurring in buoyancy-free environments. Previous numerical efforts to predict microgravity crystal growth rates have employed a diffusion-limited formulation of vapor transport. However, theoretical arguments strongly indicate that the non-isothermal conditions encountered in ampoules can lead to an appreciable motion of the bulk fluid as a result of previously unrecognized phenomena. Temperature gradients tangential to the ampoule side walls can, through the mechanism of thermal creep, drive a slip flow of gas over the surface. In addition, temperature gradients within the gas itself can induce a fluid stress, and resulting fluid motion, through the mechanism of thermal stress. Order-of-magnitude estimates suggest that, under typical ampoule conditions, the fluid velocities resulting from thermal creep and thermal stress can be comparable to vapor diffusion velocities.

Specific tasks include:

• Develop code for numerically solving governing differential equations. Assemble thermodynamic and transport property database of commonly used PVT samples and carrier gases.

• Perform numerical calculations of diffusion-buoyant convection vapor transport in the absence of thermal creep and thermal stress. Check numerical results with previously published results.

• Incorporate thermal creep boundary conditions and thermal stress constitutive law into numerical code. Determine model parameter values at which thermal effects significantly alter vapor transport from diffusion and buoyant convection predictions.

• Determine the individual effects of thermal creep and thermal stress on vapor mass transport rates and the uniformity of mass flux at the crystal surface. Based on numerical results, develop rational expressions for estimating the relative contributions of the thermal vapor transport mechanisms to mass flux as a function of ampoule, sample, and carrier parameters.

Printed: 28 FEB 1996  
II-670
Task Significance:

Under earthbound conditions, heat and mass transfer within nonisothermal physical vapor transport (PVT) crystal growth ampoules will often be dominated by buoyant convection. The rationale for performing PVT experiments in microgravity conditions is that the buoyant convective mechanisms will be suppressed, and crystal growth will occur in a diffusion-limited mode. However, qualitative arguments indicate that the convective mechanisms of thermal creep and thermal stress, which are usually negligible in comparison to buoyant convection in earthbound environments, could significantly affect mass transfer rates in microgravity PVT systems. A quantitative understanding of the role of these mechanisms in microgravity PVT crystal growth processes requires detailed numerical modeling.

Progress During FY 1995:

Our efforts during this period focused on: 1) formulating and examining the effects of thermal stress convection in nonisothermal ampoules; and, 2) examining the effects of conduction heat transfer through the solid crystal on the temperature and mass flux uniformity at the crystal interface.

Thermal stress is predicted from the Burnett contributions to the fluid stress tensor — which constitute the second-order approximation to the Boltzmann equation for small Knudsen flows (the Navier-Stokes equations being the first order approximation). In an effort to account for all sources of convection in μg PVT, we have included the thermal stress terms in our 2-D and transient numerical model of binary PVT. Our predictions indicate that: 1) thermal stress convection is only significant for extremely high temperature gradient conditions (i.e., ≈ 100 K/cm); and, 2) the thermal creep flows generated from these temperature gradients invariably overwhelm the thermal stress flows. Consequently, in regard to crystal growth, we do not believe that conditions exist in which thermal stress, by itself, would have an appreciable effect on the growth rates and morphology of the crystal. However, the thermal stress flows that we have predicted are intriguing in themselves. Under earthbound conditions thermal stress convection in nonisothermal gases will be insignificant compared to buoyant convection. The μg environment, on the other hand, offers conditions in which thermal stress convection could become a significant if not dominant mode of convection in nonisothermal gases.

Previous PVT numerical modeling efforts have taken the crystal interface to be planar and isothermal. In reality, conduction heat transfer through the solid, coupled with the latent heat release of the depositing crystal, can lead to nonuniformities in the crystal surface temperature. This, in turn, will affect the saturation partial pressure of the nutrient above the crystal and thus alter the arriving flux. To examine the effects of solid phase heat transfer, we have: 1) developed an analytical model (based on steady state and 1-D convection/diffusion) to predict the critical system parameters at which crystal growth is affected by solid phase heat conduction; and, 2) extended our numerical model to include solid phase conduction through the crystal/end wall solid. Our conclusions are: 1) the 2-D numerical simulations of crystal growth, including solid phase heat transfer, are in very good agreement with the 1-D analytical model; and, 2) conduction through the solid acts to even out the distribution of mass flux at the interface. This effect is the result of the thicker regions of the crystal having a relatively higher temperature than the thinner regions, which consequently slows down the growth rate of the thicker regions.

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Printed: 28 FEB 1996
Polymerizations in Microgravity: Traveling Fronts, Dispersions, Diffusion and Copolymerizations

Principal Investigator: Prof. Lon J. Mathias
University of Southern Mississippi

Co-Investigators:
Lochhead, R.
University of Southern Mississippi
Pojman, J.
University of Southern Mississippi

Task Objective:
Polymerization studies of bulk-phase diffusion and polymerization in monomer and co-monomer mixtures or in monomer droplets in a non-solvent are being performed to study density effects on polymerization.

Task Description:
These experiments will allow determination of fundamental monomer and polymer properties. The diffusion rate constants to be measured in the absence of convective mixing have not been accurately measurable on earth. This data will provide new insights into the fundamental behavior of polymers in a variety of situations important to their formation and processing. For example, molecular weights and distributions are directly dependent on the relative diffusion behavior of monomers and polymers. Processing of polymers, either in solution or from the melt, involves molecular reorganization and motion. The combination of convective mixing with diffusion determines the ability of polymers to effectively orient into crystalline domains. A further example is provided by copolymerizations at high conversion. In this regime, which is industrially important on Earth, the diffusion constants of monomers and polymers become extremely important. The co-polymer composition throughout the reaction must be amenable to calculation and prediction. This is currently not possible without accurate knowledge of polymer and monomer diffusion constants.

Three separate but closely related research investigations are being conducted that all deal with developing a fundamental understanding of monomer and polymer diffusion during polymerization or processing under widely different conditions, and with applying this knowledge to the formation of unique materials. The three projects all involve bulk-phase diffusion and polymerization in neat monomer or co-monomer mixtures, or in monomer droplets dispersed in nonsolvent. All three deal with diffusion effects that should uniquely manifest themselves under microgravity where analysis by optical, interference and light scattering will give complete understanding of macroscopic down to molecular level behavior.

Task Significance:
Microgravity offers a unique environment for studying polymer diffusion and polymer polymerization reactions. The absence of convection currents, which are the major mode of mixing at the molecular level on earth, are eliminated or reduced in the microgravity environment. More importantly, the prediction of unique co-polymer composition development in microgravity will allow controlled formation of new compositions of matter. The absence of mixing at the molecular level should produce unique short-block co-polymers available for the first time for co-monomer compositions which normally lead to random or long-block co-polymers under good mixing.

Progress During FY 1995:
Significant progress was made during the year on both ground-based and flight experiments. Despite the fact that our project is just over two years into its first three year cycle, we have not only developed sufficient ground-based data to design experiments for microgravity, but we have actually carried out a number of flight experiments and have developed hardware and schedules for additional flight experiments during the coming year. Flight experiments include:
II. MSAD Program Tasks — Ground-based

Discipline: Materials Science

- KC-135 homopolymerizations of methyl methacrylate which showed significantly higher molecular weights in microgravity than on earth. A repeat is planned in the near future along with model development and correlation of results with light intensity and gravity.

- A GAS can experiment returned in September 1995 from the Space Shuttle in which several types of polymerizations were evaluated. Preliminary results confirm successful reaction although detailed analysis must still be done.

- Two weeks of KC-135 experiments were prepared for October 17-27, 1995, involving homo- and co-polymerizations, including experiments designed to test spinodal decomposition and phase separation dynamics using twenty second intervals in a photo reactor.

- A sounding rocket experiment involving traveling fronts began development. Reactor design was modified per NASA instruction in preparation for final testing the second week in October for a January-February 1996 flight.

- Several industrial sponsors were lined up for Code C sponsorship of the 3M Gosmar rapid mixing reactor. Experiments will involve polyanaline single crystal formation (IBM collaboration) and several experiments with sterically stabilized polymer dispersions (GE and Calgon main sponsors). This will be designated for Shuttle flight in the next twelve - eighteen months.

Ground-based work has been as extensive as our flight efforts. Specifically, we have accomplished much in all three components of the project:

- Developed a series of new monomers for photopolymerization which are much faster than other methacrylates and that will allow detailed evaluation of homo- and co-polymerization studies in microgravity using both thermal and photoinitiation.

- Evaluated the role of polymerization fronts in developing new compositions of matter such as composites, filled and reinforced thermosets, and thermally generated two-component interpenetrating polymer networks.

- Carried out detailed evaluation of the absorption of hydrophobically-modified polymers at oil-water interphases during extremely slow (15 hour) quiescent conditions as well in rapid experiments in a turbulent flow regime. It was confirmed that a critical Reynold's number must be attained to allow absorption of the polymers to confer emulsion stability. Experiments were carried out under isopycnic conditions to mimic microgravity.

The extensive flight and ground-based effort for this year is substantiated by the numerous publications, preprints, and presentations given. This has been an extremely productive year. In fact, to the best of our knowledge, we have accomplished more during the first two years of this project than any PI or group of PIs in the history of NASA-funded materials research. And, we have done it with less: $200,000 to date, and $300,000 over three years.

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**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Journals**

II. MSAD Program Tasks — Ground-based

**Discipline: Materials Science**


**Proceedings**


**Presentations**


Lochhead, R.Y., and Rulison, C.J. "The kinetics of polymer diffusion to liquid/liquid interfaces by drop volume techniques."
Lochhead, R.Y., and Rulison, C.J. "An investigation of the ability of poly(acrylic acid) microgels to stabilize oil-in-water emulsion systems under shear flow conditions."


II. MSAD Program Tasks — Ground-based

Quantitative Analysis of Crystal Defects by Triple Crystal X-Ray Diffraction

Principal Investigator: Dr. Richard J. Matyi
University of Wisconsin, Madison

Co-Investigators:
Gillies, D.
NASA Marshall Space Flight Center (MSFC)

Task Objective:
The objective of this task involves the development of various x-ray diffraction methods for the analysis of semiconductor materials, particularly ZnSe and its ternary alloys. The specific goals are: (1) the development of high resolution triple crystal x-ray diffraction (HRTXD) methods for the quantitative analysis of structural defects in semiconductor crystals; (2) the application of these analytical methods to compound semiconductor crystals (grown both in ground-based experiments and, eventually, in microgravity) to achieve a better understanding of the effects of the crystal growth environment on the generation of defects; and, (3) the transfer of this analysis technology to the Space Sciences Laboratory at the Marshall Space Flight Center (MSFC).

Task Description:
The overall investigative approach of the research is the coupling of a new materials characterization tool (high resolution triple crystal x-ray diffraction) to the development of a new crystal growth system (ZnSe and its ternary alloys grown in microgravity). ZnSe and various alloy samples are being supplied by MSFC and are currently being analyzed by HRTXD to articulate the mosaic structure and the density of statistically distributed dislocations. Diffuse scattering is also being used to monitor the surface preparation. Plans are being made to investigate the use of anomalous transmission as an alternate means of characterizing the defect structure, and to measure the volume fraction of twinned material using x-ray methods. The information obtained in these analyses is being forwarded to MSFC for corroboration by other analytical methods.

Task Significance:
The Microgravity Science and Applications Division at the Marshall Space Flight Center is engaged in a program under an approved flight experiment for the growth of the important semiconductor ZnSe and its alloys in space. Of prime importance in any crystal growth process is the reduction of the density of grown-in structural defects and the understanding of the relation between crystal growth and defect generation. Recent research at the University of Wisconsin, Madison has demonstrated HRTXD is capable of clearly differentiating the defect-free, "perfect crystal" regions of a sample from regions that are structurally defective. In this research program, we will use HRTXD and related methods to examine the evolution of crystal growth defects as a function of crystal growth and relate the nature and quantity of these defects to the growth process. This differentiation and measurement of the effect of defects will be used to give a clearer insight into the effects of microgravity on the growth of technologically-important crystals.

Progress During FY 1995:
Selected samples of crystals grown both on earth and in microgravity have been studied using our HRTXD capability. In one of our first analyses we studied the diffracted intensity about the 333 reciprocal lattice point from a sample of CdZnTe grown on USML-1. The data showed a well-defined crystal truncation rod, or surface streak, extending perpendicular to the surface. The presence of a well-defined surface streak is indicative of two qualities of the diffracting crystal: (1) the bulk crystal has sufficient structural perfection in order to diffract dynamically, and, (2) the crystal surface is of high enough quality to produce the surface streak at all. It should be noted, however, that the surface streak in this sample was less intense than is typically seen in samples of highly perfect Ge or GaAs; this degradation is likely to be due to the grown-in dislocation density in this sample. This interpretation is supported by the diffuse intensity located off the surface streak in the vicinity of the 333 reciprocal lattice point. A roughly symmetric distribution of diffuse intensity about the 333 point was observed; this is indicative of a...
relatively isotropic distribution of dislocations in the diffracting volume. In contrast to the relatively perfect CdZnTe, a 111 reciprocal space map of a ZnTe sample grown at the MSFC revealed multiple surface streaks; this behavior is characteristic of a sample with large mosaic blocks that are relatively perfect (low in dislocation density) but with a high degree of misorientation with respect to each other. This interpretation was in agreement with the topographic observations of the same material described above.

Reciprocal space maps have also been obtained from ZnSe samples grown at MSFC. A ZnSe crystal with a cleaved [110] surface showed both a well-defined surface streak and a relatively large amount of diffuse scatter distributed perpendicular to the [110] surface normal direction. The diffuse scatter indicates a high dislocation density; however, the uniformity of this intensity suggested that the dislocations were randomly distributed throughout the crystal and not concentrated into subgrain boundaries. A reciprocal lattice map from a ZnSe sample with a polished surface showed the complete absence of any surface streak, and both the intensity and the angular extent of the diffuse intensity indicated a highly defective surface region in this sample. This interpretation agrees with observations from x-ray synchrotron topography of the same sample, thus supporting our belief in the complementary nature of the two characterization techniques.

In order to better understand the results of our HRTXD analyses, work is currently underway to model the observed intensity distribution. This modeling effort involves the simulation of the triple crystal diffraction scans with the inclusion of the effects of the strain fields of an assumed dislocation population. While still in its early stages, this modeling effort appears to show the correct trend in the dependence of the diffuse scattering as a function of deviation from the exact Bragg condition for a sample containing $10^6$ edge dislocations/cm$^2$ in CdTe; work on refining and extending this modeling effort is currently in progress.

**STUDENTS FUNDED UNDER RESEARCH:**

**TASK INITIATION:** 7/94  **EXPIRATION:** 7/96

**PROJECT IDENTIFICATION:** 962-21-08-28

**NASA CONTRACT NO.:** NCC8-052

**RESPONSIBLE CENTER:** MSFC
The Interactive Dynamics of Convection, Flow and Directional Solidification

Principal Investigator: Prof. T. Maxworthy
University of Southern California

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
In order to assess and control the effects of residual spacecraft accelerations on the growth of high quality crystals, full numerical simulations will be conducted. It is anticipated that this approach will lead not only to a minimization of the adverse effects of the flow on the crystal growth process, but also to the development of strategies for employing externally imposed flow fields to gain some degree of control over the solidification dynamics. Questions of interest concern the selection of length scales, the persistence of traveling waves, and of oscillatory instabilities in the nonlinear regime. Time dependent direct numerical simulations performed will allow investigation of these mechanisms in isolation, as well as with their combined effects.

Task Description:
1. Study the types of morphological instability that are to be found during the directional solidification of binary mixtures;

2. Attempt the control of such instabilities using forced flow over the solidifying interface and assess the usefulness of reduced gravity in modifying the process, thereby improving the quality and reducing the defect in manufactured products.

Task Significance:
The basic mechanisms involved in controlling such nonlinear dynamical phenomena are of value to other fields of science and engineering, and the space environment may provide a suitable test bed for examining such ideas. This could be beneficial to control the solidification process by employing externally imposed flow fields to improve the quality and reduce defects in manufactured alloy products.

Progress During FY 1995:
As of January 1996 the design and construction of the basic apparatus has been completed. This includes a cell-traversing subsystem, that was not part of the original proposal, but that was found necessary to incorporate the test-cell with the Leitz Interferometric Microscope made available by the Materials Research Section of NASA LeRC. Also, a visit by Dr. Eli Raz was arranged in order to become familiar with the operation and idiosyncrasies of the microscope as quickly as possible. This visit proved to be particularly effective in pointing out a number of problem areas that were then addressed in a subsequent redesign. Also, during this period the acquisition of a supply of 99.999% pure SCN became a source of difficulty. The only known source, Professor M. Glicksman, has a huge backlog of orders. Because of this strong demand and the long time-scale of the production method, we believe that this source cannot be relied upon to supply the needed material in a consistent and timely fashion. Two solutions have been proposed. The first is to build our own zone-refining system based on a commercially available device. However, presently funds are not available to do this. The second solution is to use commercially available SCN (99.9% pure) and use this as the base, "pure" material against which the effect of adding the second component, acetone, can be compared. Until our new proposal, submitted to the NASA Microgravity Program in August 1995 is funded, we will take the second approach. Experimentation on the techniques needed to fill the cell and to operate the heating and cooling, and especially, the flow-control systems was begun in early January 1996 with full operation expected by late February of this year.
We have been fortunate in securing independent, one year funding for a student to work on this project. His experience in this area of research has proved invaluable in modifying the original design to include a number of refinements that will improve the overall operation of the system. However, his continued employment by the project depends critically on the fate of the above mentioned proposal.

Independently, we have also begun work on an interesting fluid dynamical analog to the problem of directional solidification. This steady state version of the so-called “Printer’s Instability” is easier to work with experimentally and it is possible to test many of the features of the prototype problem with relative ease. In particular, the problem of the dependence of the instability wave length on the operating parameters, especially the cell gap-width, can be studied in depth and used to interpret the solidification experiments.

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PROJECT IDENTIFICATION: 962-25-05-26
RESPONSIBLE CENTER: LERC
Y$_2$BaCuO$_5$ Segregation in YBa$_2$Cu$_3$O$_{7-x}$ During Melt Texturing

Principal Investigator: Dr. Paul J. McGinn

University of Notre Dame

Co-Investigators:

No Co-I's Assigned to this Task

Task Objective:

The objective of this ground-based investigation is to understand the segregation of fine second phase Y$_2$BaCuO$_5$ (211) precipitates during so-called "melt texture growth" of the superconductor YBa$_2$Cu$_3$O$_x$ (123).

Task Description:

In melt texturing of 123, one of the goals is to refine the 211 that is present in the textured microstructure as much as possible to enhance flux pinning. However, segregation of fine 211 will affect the ability of the 123 to pin magnetic flux. It is unclear if segregation is an unavoidable consequence of precipitates being pushed by the solidification growth front, and what role is played by gravitational effects. The goal of this project is to understand the causes of 211 segregation. The effect of a number of factors on texturing will be examined to determine what is causing the observed segregation.

Through a series of melt texture growth experiments the effect of Pt additions on 211 size, and more importantly the 211 size dependence of segregation, will be determined. The effect of 123 growth morphology on 211 segregation will also be examined. This will allow for determination of whether the observed segregation is specific to only one morphology of 123 growth, or is characteristic of all 123 growth. The growth mode will be altered by using different techniques to achieve 211 refinement. It is anticipated that containerless processing experiments will be included in this part of the study as they will provide the best opportunity to observe impurity-free growth.

After the effect of the above factors is understood experiments will be performed to attempt melt texturing of 123 in a microgravity environment.

Task Significance:

It is expected that this research will identify important factors affecting the segregation of fine 211 particles during the melt texturing of the 123-type high temperature superconductors. It will enhance the understanding of the crystal growth process and the development of the microstructure. It is anticipated that knowledge gained from working with this technologically important system will be applicable to other systems exhibiting similar crystal growth features.

Progress During FY 1995:

Using a solid modeling program, a three-dimensional structure model of the 211 particle segregation in 123 domains was created. The model allows for visualization of the three-dimensional 211 segregation patterns in the 123 domains at various viewing angles. The model can be rotated and tilted along any axis to get a two-dimensional projection, allowing one to see what a pattern should look like in the two-dimensional plane of polish of a sample. A model of 211 particle segregation on three sets of mutually intersecting (110) type of planes in 123 domains has been proposed which can be used to generate 211 particle tracks similar to those observed in polished microstructures. Use of the model allows one to produce segregation patterns duplicating those seen in sample cross sections. Either of two proposed Y123 growth mechanisms can lead to the description of 123 domains consisting of mutually intersecting planes containing 211 particles, and such a structure explains all the track patterns that are observed in samples.
II. MSAD Program Tasks — Ground-based

STUDENTS FUNDED UNDER RESEARCH:

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TASK INITIATION: 6/94  EXPIRATION: 6/95

PROJECT IDENTIFICATION: 962-21-08-29
NASA CONTRACT NO.: NAG8-1064
RESPONSIBLE CENTER: MSFC

BIBLIOGRAPHIC Citations for FY 1995:

Presentations
Interaction of Hele-Shaw Flows with Directional Solidification: Numerical Investigation of the Nonlinear Dynamical Interplay and Control Strategies

Principal Investigator: Prof. Eckart H. Meiburg
University of Southern California

Co-Investigators:
Maxworthy, T.
University of Southern California

Task Objective:
In order to assess and control the effects of residual spacecraft accelerations on the growth of high quality crystals, full numerical simulations will be conducted. It is anticipated that this approach will lead not only to a minimization of the adverse effects of the flow on the crystal growth process, but also to the development of strategies for employing externally imposed flow fields to gain some degree of control over the solidification dynamics. Questions of interest concern the selection of length scales, the persistence of traveling waves, and of oscillatory instabilities in the nonlinear regime. Time dependent direct numerical simulations performed will allow investigation of these mechanisms in isolation, as well as with their combined effects.

Task Description:
1. Study the types of morphological instability that are to be found during the directional solidification of binary mixtures;
2. Attempt the control of such instabilities using forced flow over the solidifying interface and assess the usefulness of reduced gravity in modifying the process, thereby improving the quality and reducing the defect in manufactured products.

Task Significance:
The basic mechanisms involved in controlling such nonlinear dynamical phenomena are of value to other fields of science and engineering, and the space environment may provide a suitable test bed for examining such ideas. This could be beneficial to control the solidification process by employing externally imposed flow fields to improve the quality and reduce defects in manufactured alloy products.

Progress During FY 1995:
Over the last year, our efforts have focused on the development and implementation of a highly accurate computational approach. Since the computational domain has a curved boundary at the solidification front, we employ a conformal mapping approach to obtain a rectangular domain. By assuming periodicity in the spanwise direction, we can perform this mapping analytically, which has the advantage of providing us with analytical relationships between the original and the transformed variables. We have derived the relevant equations, boundary conditions and transformation terms in the computational domain. Since the problem is nonperiodic in the direction of solidification, we employ a compact finite difference method in this direction, while a spectral representation has been chosen for the spanwise direction. For the case without flow, we have numerically computed the growth rates of small wavy perturbations to the interface and made preliminary comparisons with the classical linear stability theory of Mullins and Sekerka. The comparisons indicate excellent agreement. We have furthermore implemented the effect of surface energy into the computational scheme as well. Again, comparison with linear stability results confirms the high accuracy of our computational approach. Currently we are working on implementing the effects of flow in the melt. As soon as this step is finished, we can carry out fully nonlinear simulations of a variety of interesting cases, in order to investigate various possibilities of flow control.
II. MSAD Program Tasks — Ground-based

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| PROJECT IDENTIFICATION: 962-25-05-27 |
| NASA CONTRACT NO.: NAG3-1619          |
| RESPONSIBLE CENTER: LeRC              |

Printed: 28 FEB 1996
The Synergistic Effect of Ceramic Materials Synthesis Using Vapor-Enhanced Reactive Sintering Under Microgravity Conditions

Principal Investigator: Prof. John J. Moore  
Colorado School of Mines

Co-Investigators:  
Ready, Prof. D.  
Colorado School of Mines

Task Objective:  
The primary objective of this research is to develop a technique for processing advanced ceramics in the form of fine powders, whiskers or platelets. The secondary objective is to determine the extent of vapor phase reactions, including convection, in combination with gravity driven flow, in the process. Important concerns include product particle size and purity. Products should have particle sizes allowing efficient handling and subsequent processing and/or function. Control of the chemical reactions and particle sized is necessary for the achievement of this objective.

Task Description:  
Titanium diboride is synthesized according to the reaction Ti+2B=TiB₂ and titanium diboride + alumina (Al₂O₃) composites are synthesized by the reaction 3TiO₂+3B₂O₃+10Al=3TiB₂+5Al₂O₃. The first reaction was chosen for its relatively simple solid-solid reaction, allowing the fundamentals of the reaction to be accurately evaluated. The TiB₂ is formed via a combustion synthesis reaction in the propagating mode, which has high product purity and rapid reaction rates as inherent advantages of this process. The second reaction also uses the propagating mode, but is complicated by the fact that the reacting components become both molten and gaseous during the ignition process. Convection, therefore, plays a much greater role than in the first reaction. Both reactions will be carried out in both inert, i.e., Ar gas, and reactive, i.e., HCl gas, environments in order to evaluate the effects of vapor transport on the combustion synthesis reaction. A complete examination of the process variables, including micro and increased gravity, will be completed to examine the effect on the reaction.

The project has taken on two new areas of interest during the FY 1995. The first area is the effect of varying amounts of liquid of varying specific gravity at the reaction front. Reactions will be investigated to achieve liquids with differing specific gravity and quantity at the reaction front. The effect of gravity on density-driven fluid flow will be an important variable in investigating the role of liquid at the reaction front. The first set of experiments will have a range of compositions between TiB and TiB₂ to obtain an increasing amount of liquid at the reaction front.

The second area of interest is the development of a numerical model of the heat transfer processes that take place in a Ti + 2 B = TiB₂ combustion synthesis reaction. This model should predict the effects of microgravity on the heat transfer process, especially convection.

Task Significance:  
Fine powders may be used as raw materials for subsequent processing techniques, while platelets and whiskers may be used as reinforcement materials in composite structures. Evaluation of reactions in microgravity enhance basic understanding of ground-based reaction mechanisms.

Progress During FY 1995:  
Effects of gas pressure on the combustion synthesis of TiB₂ in both argon and HCl have been investigated. In both cases as the pressure increases the ignition temperature increases and the combustion temperature decreases. It is also noted that as the combustion temperature increases the grain size of the product increases. Experiments in microgravity have been completed, and all reactions show a large increase in combustion temperature over
II. MSAD Program Tasks — Ground-based

ground-based results, as well as a more uniform grain size. Microgravity environments decrease the effect of convection, but the reaction mechanisms that depend on convection have yet to be fully clarified.

The alumina-titanium diboride system has also been investigated using reactive and inert gas pressure and microgravity. In this system, a vapor-liquid-solid reaction occurs at the reaction front, producing different results. Under normal conditions, gas pressure increases, ignition temperature decreases, and the combustion temperature increases, producing varied microstructures. In addition, the reactive gas seems to have little effect on the combustion temperature. Microgravity results show large increases in combustion temperatures. However, microstructures still show large variations even within a given sample.

The data acquisition equipment is being computerized. The thermocouple and pyrometer signal are monitored using a computer. These signals are processed and converted to engineering units. This system is being tested and used in current reactions.

An experimental system has been designed and is being built to study in detail the heat transfer processes. The system will provide a two dimensional surface temperature profile of the pellet throughout the reaction process. The temperature profile will be taken by a thermal imaging camera, and captured by a computer. The image will then be calibrated to provide the temperature at every point on the surface. The system should be completed in the 1996 fiscal year.

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BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals


Proceedings

Books
Diffusion, Viscosity, and Crystal Growth in Microgravity

Principal Investigator: Prof. Allan S. Myerson
Polytechnic University, New York

Co-Investigators:
Izmailov, A.
Polytechnic University, New York

Task Objective:
The results of this research should improve the operation and data analysis of existing microgravity crystal growth experiments and aid in the design of new hardware for such experiments.

Task Description:
Components of this research include:

• Experimental studies of the diffusion coefficients and viscosity of triglycine sulfate (TGS), KDP, and other compounds of interest to microgravity crystal growth in supersaturated solutions as a function of solution concentrations, solution age, and solution history;

• Development of a theoretical model of diffusion and viscosity in the metastable state;

• Development of a model of crystal growth from solution including nonlinear time-dependent diffusion and viscosity effects;

• Employment of the model with and without buoyancy-driven convective flows to predict results of Earth and microgravity crystal growth experiments and to compare these results with experimental results; and

• Development of a computer simulation of the crystal growth process that will allow simulation of microgravity crystal growth, including the effects mentioned above.

Plans are to develop the adequate physical description of the crystal growth process in the supersaturated solutions. To achieve this goal, it will be necessary to take into account the nontrivial dependencies of such transport coefficients as diffusivity and viscosity on the solute concentration and solution temperature and age. The research program's approach will include obtaining the characteristic time for the crystal growth process in the supersaturated solutions, i.e., to obtain the duration of the metastable state relaxation that leads the system "crystal + supersaturated solution" to the equilibrium state. This equilibrium is the equilibrium between the crystal surface and the entire remaining volume of the surrounding saturated solution.

Another facet of the program will theoretically determine from the proposed model the difference in the concentration, temperature, and convective flow fields between an Earth-grown crystal and one grown in microgravity. This will also allow a comparison of the gains in face size and stability that might accompany microgravity crystal growth.

Comparison of theoretical predictions to the real experimental data will verify the theoretical model and determine numerical values for model parameters that have not been measured.

Task Significance:
Since crystallization from solution occurs in supersaturated solutions, the properties of these solutions and their role in nucleation and crystal growth are of interest. Recognition of this has led to a number of studies of supersaturated solutions and their properties.
II. MSAD Program Tasks — Ground-based

Discipline: Materials Science

Progress During FY 1995:
Diffusion coefficient of TGS has been measured at several temperatures in the concentrated supersaturated regions.

A method to calculate the degree of association and the number average cluster size of ADP, KDP, and TGS from activity data in the supersaturation region was developed and is based on the assumption of the clusters being in the form of electrically neutral Bjerrum pairs and their aggregates.

Gradient column experiments to examine clustering in TGC, KDP, and ADP were conducted.

Analysis of concentration profiles around growing crystals accounting for the effect of concentration dependent diffusion coefficient and viscosities was continued.

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BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals


Presentations


II. MSAD Program Tasks — Ground-based

An Electrochemical Method to Measure Diffusivity in Liquid Metals

Principal Investigator: Prof. Ranga Narayanan
University of Florida

Co-Investigators:
Anderson, T.
University of Florida
Fripp, A.
NASA Langley Research Center (LaRC)

Task Objective:
A research program will be conducted that uses coulometric titration to measure oxygen diffusivity in liquid metals. Important aspects of the program are to estimate the effect of low gravity on the diffusivity measurements and to design experiments where the use of a low gravity environment significantly reduces the adverse effects of convection in the melt. The availability of these "benchmark" values of diffusivity will be useful for assessing the reliability of different experimental designs and operational procedures for measuring diffusivity on earth.

Another science objective of this study is to establish a clearer picture of the constitutive behavior of "Fickian diffusion" of oxygen in liquid metals. In particular, we will accurately measure the temperature dependence of the oxygen diffusion coefficient.

Task Description:
In this study an existing experimental "cell" that has been modified by these investigators is used. An example of a system that is studied is the diffusion of oxygen in tin. The experimental arrangement consists of two electrochemical cells sharing a common working electrode, viz: tin. A representation of the cell is given by:

\[
\text{Cu,CuO II YSZ II O in Sn II YSZ II Cu,CuO}
\]

Here yttria stabilized zirconia (YSZ) is the solid state electrolyte through which oxygen ions are transported into or out of the liquid tin. In these experiments, one cell is used to establish a known boundary condition, usually a negligibly small concentration, while the second cell is operated in an open circuit mode to measure the concentration at the opposite boundary. The solid state electrolytes and tin are physically arranged so that the gravity vector is co-linear with the gradient of concentration.

One of the YSZ plates is at the top and has a small overflow port to accommodate the expansion of tin, upon heating. Care is taken to avoid leakage of oxygen from this or other sources. Temperature gradients, in this so-called isothermal experiment, are reduced in order to avoid natural convection under earth's gravity. Under earth's gravity, it is also important to ensure that oxygen concentration gradients are parallel to the gravity vector so that the melt is not "top heavy." This means that the oxygen concentration should be low at the "bottom" of a vertically oriented cell. This in turn implies that oxygen depletion takes place from the bottom or oxygen addition takes place from the top.

Task Significance:
The availability of a method that gives benchmark values of diffusivity in liquid metals has many uses. These include the assessing of the reliability of different experimental designs and operational procedures for measuring diffusivities under Earth's gravity, as well as defining the constitutive relationships in liquid metals. This latter use is of fundamental importance in understanding the behavior of liquid metals and alloys.

Progress During FY 1995:
A leading post-doctoral researcher with solid state electrochemical background joined the project last December. The results obtained during this period are as follows:
The current operational parameters involved in measuring the molecular diffusivity of oxygen in liquid tin included a square base geometry for the fluid cell in addition to the circular geometry with various cell configurations and measurements on liquid lead with a circular geometry. The reliability of the previously measured values at NASA with liquid tin and with circular cell geometry had been confirmed by repeating similar experiments for different aspect ratios. In the first mode of operation, the concentration gradient was such that the fluid was heavy at the bottom and lighter at the top. The reverse was true for the second mode. In the second mode, the fluid was unstable and gave rise to convective flows. The measurements on liquid tin with square base geometry indicated that the fluid flow was less intense than with the circular geometry. Spontaneous convection was observed in the second mode of operation.

Transient measurements were conducted to measure the diffusivity of oxygen in liquid lead and it was inferred that liquid lead is more prone to convective flows than liquid tin.

All of these experiments confirm our conjecture that convection interferes with accurate diffusivity measurements and that a low gravity environment will be essential to obtain the important physical property of oxygen diffusivity in liquid metals.

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**TASK INITIATION:** 2/93  **EXPIRATION:** 2/96  **PROJECT IDENTIFICATION:** 962-25-08-26  **NASA CONTRACT NO.:** NCC8-20  **RESPONSIBLE CENTER:** MSFC

**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Proceedings**


**Presentations**

Crystal Growth and Segregation Using the Submerged Heater Method

Principal Investigator: Prof. A. G. Ostrogorsky
Rensselaer Polytechnic Institute

Co-Investigators:
Müller, G. Universität Erlanger-Nürnberg
Monberg, E. AT&T Bell Labs

Task Objective:
Fundamental studies of solute segregation at low levels of melt convection will be conducted using a programmable multi-zone furnace modified for growth by the Submerged Heater Method. The specific objectives of these studies are to:

1. Evaluate the suitability of the multi-zone furnace with the submerged heater to serve as a ground-based solidification facility, useful for pre- and post-flight studies.

2. Seek explanations for previous space experiments not demonstrating the diffusion controlled segregation.

3. Determine the criteria that will allow the diffusion-controlled segregation.

4. Evaluate the benefits of using the SHM within a PMZF for future space experiments.

Task Description:
The research plan is to use the Submerged Heater Method in ground-based experiments for studies of mass transport in the melt at low Ra numbers. The effect of the equilibrium coefficient \( k \) will be particularly investigated. The studies will focus on electronics materials previously studied in space (e.g., Ge, InSb) doped with elements having different equilibrium distribution coefficients, ranging from \( k \sim 0.5 \) to \( k \sim 10^{-3} \). Directional Solidification and zone melting procedures will be used during growth. The studies will be conducted using a programmable multi-zone Mellen furnace modified for the SHM. The ground-based experimental and theoretical work will be accompanied by numerical simulations. Numerical simulations will be used to model the transfer processes in the melt and to optimize the growth conditions. Future space experiments designed for the SHM will also be modeled.

Task Significance:
We hope to demonstrate that the multi-zone furnace with the submerged heater is useful for pre- and post-flight studies. The pre-flight studies may help to optimize the future space experiments. Our current "post-flight studies" focus on solvent-solute systems previously used in space (doped Ge, InSb and GaSb). We hope to explain why some space experiments did not result in diffusion controlled segregation.

Progress During FY 1995:
Fourteen doped Ge single crystals were grown in an 18-zone "Mellen" furnace using the submerged heater method (SHM). The influence of the level of convection on macro- and micro-segregation in Ga-doped Ge crystals was investigated. When used without rotation, the submerged heater drastically reduces convection at the solid liquid interface. In several experiments, the submerged heater was set into rotation or oscillation to produce vigorous mixing (yielding close-to perfectly stirred melt). Exceptionally high radial uniformity was obtained by rotating and oscillating the submerged heater. Although heavily doped, the crystals were free of unintentionally produced striae, even when grown under the oscillating submerged heater.

Single crystals were obtained in virtually all experiments. Intentionally produced striae indicate that the shape of the growth interface was planar or slightly convex in all experiments.
A flow visualization apparatus was set up to study the flow in the melt produced by rotation of the submerged heater. The visualization was conducted using the same crucible and submerged heater as used in the crystal growth experiments. Water was used as a model fluid. Fine aluminum powder mixed in water was used as a tracer substance to study the flow direction and to measure the maximum velocities in horizontal (angular) and vertical (axial) direction. The light from the light source was bundled in a planar beam in order to trace the light reflection from the metal particles that passed or moved within the illuminated plane.

Numerical analysis of the growth was performed using the code NEKTON and FIDAP. Simulations of the initial transient were carried on, while the shape of the solid-liquid interface was recalculated in each time step. Realistic boundary conditions are imposed on the melt.

**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Journals**


**Proceedings**

**Presentations**


Investigation of “Contactless” Crystal Growth by Physical Vapor Transport

Principal Investigator: Dr. Witold Palosz

Universities Space Research Association

Co-Investigators:

Sha, Y.-G.
Gillies, D.
Su, C.-H.
Lowry, S. (Associate)

Universities Space Research Association (USRA)

NASA Marshall Space Flight Center (MSFC)

CFD Research, Inc.

Task Objective:
The primary objective of this study is to evaluate the conditions required for growth of crystals without contact with the side walls by physical vapor transport (PVT) in closed ampoules. The potentials and limitations of this technique ("contactless" PVT - cPVT) with respect to crystal size, growth rate, crystal quality, and the potential benefits of the microgravity environment for this technique will be assessed. The conditions for growth of Pb(Se, Te) by PVT will be determined. The evaluation of capabilities and limitations of the existing facilities and recommendations for space crystal growth facilities with respect to this PVT technique will be made.

Task Description:
2. Development of thermochemical model of transport of Pb(Se, Te) by PVT.
3. Fabrication and/or modification of existing crystal growth furnaces.
4. Experimental investigation of transport of Pb(Se, Te) by PVT.
5. Performing test crystal growth experiments.

Task Significance:
The benefits of the microgravity environment for crystal quality may be offset by strains and related crystal defects caused by interaction of the crystal with the walls of the growth container. The cPVT technique offers improved growth conditions which may be beneficial for crystal growth both under ground and microgravity conditions. The study will provide a comprehensive assessment of the technique as opposed to only limited scope of the literature reports on the subject.

Progress During FY 1995:
Budgeting of project started in May 1995.
Theoretical thermochemical model of mass transport in Pb(Se, Te) PVT system has been developed. Fluid dynamic modeling of "contactless" crystal growth has been initiated. Construction of new crystal growth furnaces has started.

STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 2/95  EXPIRATION: 2/97
PROJECT IDENTIFICATION: 962-21-08-20
NASA CONTRACT NO.:  NAG8-1139
RESPONSIBLE CENTER: MSFC

Printed: 28 FEB 1996  II-693
Containerless Processing for Controlled Solidification Microstructures

Principal Investigator: Prof. John H. Perepezko
University of Wisconsin, Madison

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
The main research objective is the evaluation of the undercooling and resultant solidification microstructures in containerless processing including drop tube processing and levitation melting process of selected alloys as an experience base for microgravity experiments.

Task Description:
The degree of liquid undercooling attainable in a laboratory scale (3m) drop tube and levitation melting system can be altered through the variation of processing parameters such as melt superheat, sample size and gas environment. In a given sample, the competitive nucleation and growth kinetics between equilibrium and metastable phases controls microstructural development. The solidification behavior is evaluated through metallography, thermal analysis and x-ray diffraction examination in conjunction with calorimetric measurements of falling droplets and a heat flow model of the processing conditions to judge the sample thermal history.

Task Significance:
In the current program studies, solidification microstructures are being examined in selected Ni and Mn based systems. The specific alloy selection is based on a metastable phase diagram analysis that allows for the identification of unique microstructures and microstructural transitions that may be produced by microgravity containerless processing.

Progress During FY 1995:
In the current program studies, solidification microstructures are being examined in selected Ni, Co and Mn based systems. The specific alloy selection is based on a metastable phase diagram analysis that allows for the identification of unique microstructures and microstructural transitions that may be produced by microgravity containerless processing. A duplex partitionless solidification reaction involving fcc and bcc crystalline phases has been identified over a range of compositions near that of the eutectic in the Ni-V system. The reaction can be thought of as the limiting case of a eutectic transformation the solid phases each have the same composition as the liquid phase. Drop tube experiments are being conducted to characterize the competitive formation kinetics of the fcc and bcc phases. Near-equiaxative Ni-V alloys were solidified via containerless processing methods and studied with electron microscopy techniques. TEM has shown the presence of a duplex structure of fcc and bcc in large (c.100 mg) droplet samples. TEM analysis has suggested that the duplex structure is not the result of a solid state transformation and that the fcc and bcc phases have apparently nucleated independently from the liquid phase. A thermodynamic model has been applied to the Ni-V system to calculate T0 temperatures for the relevant phases to map compositions and temperatures for which the duplex partitionless reaction is possible. A kinetic model has also been forwarded which takes into account both the nucleation and growth rates of the competing fcc and bcc phases. The nucleation kinetics analysis is in reasonable agreement with experimental results. Further refinement of the model is underway to consider the effects of transient nucleation and heat flow effects. The study of the duplex partitionless reaction has been extended to Co-A1 alloys. Rapid solidification processing of alloys of near-eutectic composition has yielded a duplex partitionless structure of fcc and B2 (ordered bcc) phases. Drop tube processing experiments are in progress to assess the competitive phase selection in small droplet samples. The analysis of these alloys will build upon the foundation of experimental results and analysis developed for the Ni-V system.

Printed: 28 FEB 1996
Near equiatomic Mn-Al alloys represent an important class of permanent magnet materials. The key ferromagnetic phase is a metastable structure produced by solid state heat treatments. Recent drop tube and levitation melting studies have demonstrated for the first time that the metastable ferromagnetic $t$ phase can be produce from the liquid provided high undercooling is achieved. With specially prepared samples it has been possible to assess the thermodynamic driving forces involved in metastable $t$ phase solidification. It also has been determined $T_0$ temperature to help understand the metastable solidification reaction pathways which yield the metastable ferromagnetic $t$ phase during the containerless processing.

Building on the thermodynamic analysis a competitive nucleation model has been developed to account for the observed phase selection. As part of a test of the kinetics model a detailed statistical study of the metastable product yield as a function of sample size and processing gas such as He and Ar gases is underway. Furthermore, by optimizing the containerless processing conditions in levitation melting process it has been possible to obtain essentially single phase samples of the $t$ structure for scale-up sample size (mm size) to approach bulk levels. In the above studies a new calorimetric system is being used measure the temperature of falling drops during containerless processing to assess the complete thermal history.

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### Task Initiation: 12/91 Expiration: 11/95

**Project Identification:** 963-25-07-06

**Responsible Center:** JPL

### Bibliographic Citations for FY 1995:

#### Journals


#### Proceedings


#### Presentations


Containerless Processing of Composite Materials

Principal Investigator: Prof. John H. Perepezko
University of Wisconsin, Madison

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
The effects of containerless and low gravity processing are investigated in the synthesis of composite materials. Existing models predicting criteria for incorporation of particles dispersed in a melt are applied to metallic systems in a microgravity environment. The specific goals of the proposed research are to determine conditions necessary for an effective dispersion (i.e., uniform distribution of small particles), to characterize the bonding characteristics of particles with the matrix when processed under microgravity conditions; and to determine the implications of the results for effective solidification processing of composites. The ultimate goal of this work is to define conditions necessary for effective containerless or low-gravity processing as well as to determine characteristics which are unique to microgravity processing. Specifically, the results of this work will allow a critical test of existing theoretical models dealing with melt-particle interaction as well as processing conditions for particle incorporation. The outcome of these experiments will also further develop the basic understanding of relationships between microstructure and properties of metal matrix composites during microgravity processing.

Task Description:
The focus of this study is on a model system of pure nickel with a discontinuous dispersion of reinforcement particles. The effects of gravity and containment on the microstructural development in particle reinforced composites is studied using free fall to offset the linear acceleration of gravity. Given the extensive knowledge base of the solidification behavior of nickel, assessment of melt and particle interaction becomes possible through a variety of means. The evaluation of the microstructure obtained during containerless processing allows for understanding of the conditions necessary for a uniform distribution of particles without complications introduced by containing vessels such as chemical contamination or particle/mold interaction. When containerless processing is coupled with solidification during free fall, gravitational effects such as flotation, sedimentation, and density driven convection can be reduced. Variation of the magnetic field allows for vigorous mixing of the particles within the molten matrix, as well as solidification with or without external contact. The degree of undercooling may be controlled by monitoring the temperature with a two-color pyrometer and seeding crystallization with a needle when the desired temperature is reached. Through variation of the amount below the equilibrium freezing temperature a specimen is cooled, the driving force, and therefore the rate of solidification, can be directly influenced. The varied solidification front velocity allows comparison to both predictions made by theoretical models as well as experimental data obtained without containerless or microgravity processing.

Task Significance:
Processing of particle reinforced composites under microgravity conditions can result in more uniform distributions of particles. With the reduction of the effects of gravity, problems inherent to conventional fabrication of metal matrix composites such as flotation, sedimentation, and density-driven convection can be avoided. As has been demonstrated in directional solidification experiments, there is a critical rate at which solidification must occur for full incorporation (i.e. engulfment) of particles to occur. Particle engulfment into a forming solid in directional solidification experiments has been shown to depend on the temperature gradient, the solidification front velocity, and the size of the particles for a fixed chemical composition. The limitation in these experiments has been the container, which changes the shape of the front and which affects distribution of the particles through particle/surface interaction. Solidification under microgravity conditions permits a study of particle distribution without the effects of a container or gravity, which supplements information obtained from prior directional solidification experiments. As with all ground-based microgravity experiments, this work serves as a guideline of steps to be followed in subsequent work actually conducted in space.
Progress During FY 1995:

The primary experimental facility, a ground-based levitation system coupled with a laboratory scale drop tube, has been constructed and tested. Composite samples ranging from 1mm to 5mm diameter spheres are levitated, melted, and allowed to solidify during free fall. Alternately, the system also has the capability of melting and solidifying the specimen on a substrate, which allows the thermal history of the droplet to be recorded using a two-color pyrometer. Preliminary results indicate selection of nickel with aluminum oxide particles and nickel with titanium carbide particles are combinations at two extremes of surface energy balance. Aluminum oxide particles are typically excluded from the melt before solidification while the titanium carbide is almost exclusively incorporated. The cooling cycles which have been recorded are directly related to the resultant microstructures. Samples which cool below the equilibrium freezing temperature have a greater driving force for solidification and thus freeze at a higher rate than those which do not undercool. The changing rate of solidification is evident from the varied particle density across the composite sample upon metallographic examination of interior sections. The particle distribution as well as the cell morphology can indicate the comparative velocities during solidification of the droplet. These estimates of front velocity will be used to extend the experimental basis of support for existing models and possibly make additional constraints to their use.

An additional model system for microgravity composite study has been identified. The Cu-alumina system shows particular promise for undercooling studies through microgravity processing. The combination of high purity copper with low aspect ratio alumina particles is the only system found to date which produces undercooling of up to 100° K despite the presence of foreign particles in the undercooled liquid. Undercooling of this magnitude has not been seen in any previously studied composite system. A differential thermal analysis (DTA) system has been the primary means for establishing the relationship between sample microstructure and thermal history. Through DTA processing, a distribution of different values of undercooling is achieved ranging from 0° to 100° K below the equilibrium solidification temperature. The basis of behavior seen in these results is essential for estimation of thermal history in microstructures obtained under free fall conditions in which temperature measurement is not possible. Additional experimental support has been recently provided by the MSFC drop tube facility and staff. The longer duration of available free fall time with the 100 m tube provides additional cooling time necessary for larger samples, and accommodates the slower cooling rate of liquid copper which is not as fast as higher temperature liquids which cool predominantly through radiative heat loss. The complete set of results for Cu/Al2O3 composite processing is currently under analysis.

The current experience, in the second and last year of the program, has shown quite clearly that the proposed approach to testing the concept of a critical velocity for particle incorporation is valid. This experience is based on drop tube studies both in Madison and at MSFC (with the help of Tom Rathz). When the analysis of results is complete, it should be possible to demonstrate a new application for containerless processing in the study of basic solidification behavior.

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**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Proceedings**

**Presentations**
II. MSAD Program Tasks — Ground-based

**Comparison of the Structure and Segregation in Dendritic Alloys Solidified in Terrestrial and Low Gravity Environments**

**Principal Investigator:** Prof. David R. Poirier  
**University of Arizona**

**Co-Investigators:**
- Heinrich, J.C.  
- Tewari, S.N.  
- Hellawell, A.  
- Tandon, K.N.  
  
**University of Arizona**  
**Cleveland State University**  
**Michigan Technological Institute**  
**University of Manitoba**

**Task Objective:**
A primary goal of this research is to identify the growth conditions for microgravity experiments delineating the macrosegregation, microsegregation, and microstructure in comparison to the same features in directionally solidified alloys grown on earth with inevitable thermosolutal convection.

**Task Description:**
Growth conditions are being defined by combining mathematical modelling and simulation with terrestrial experiments on binary metallic and model alloys. The investigation is carried forward at three universities, the University of Arizona with D.R. Poirier and J.C. Heinrich, Cleveland State University with S.N. Tewari, and Michigan Technological University with A. Hellawell.

**Task Significance:**
This research relates directly to almost all casting of metals as almost all practical cast metals are alloys which solidify under conditions of directional heat transfer. Alloys develop microstructures of dendrite primary and secondary arms, with micro-and macrosegregation patterns influenced not only by the basic physical processes of solidification but also by convective currents. To separate out the convective effects, microgravity experiments are necessary. Results of this work will eventually be included in numerical models for casting of alloys and will thereby impact alloys and will thereby impact a major portion of the economy.

**Progress During FY 1995:**
Hypoeutectic Pb-Sn alloys, with compositions ranging from 10 to 58 wt. % Sn, have been directionally solidified at rates of 4 to 66 μm s⁻¹ in measured thermal gradients of 67 to 110 K cm⁻¹. As shown in Fig. 1, this results in macrosegregation along the length of the directionally solidified alloys. In the absence of thermosolutal convection, the tin content along the length would have been uniform, except for an initial length of the order of mushy zone length.

Using the solutal build up between the fraction solids corresponding to 0.2 and 0.6 as a measure of the macrosegregation, it has been observed that the extent of macrosegregation increases with increasing tin content, becomes maximum for 33.3 wt. % Sn, and decreases with further increase in the tin content.

The intensity of the segregation along the length of the samples (and hence the presumed thermosolutal convection) has been explained in terms of a parameter which includes the primary arm spacing, the volume fraction interdendritic eutectic, and the eutectic and tip compositions in the liquid. Alloy composition, thermal gradient and solidification rate control primary arm spacing, fraction eutectic, and liquid concentration at the dendrite tips.

To date numerical simulations have been done using the thermal conditions and composition of one of the experimental ingots exhibiting "freckles" (also called "channel segregates"). As early as 1000 s after the onset of
solidification, the numerical simulation shows upward flow next to the walls of the container, reminiscent of plumes that emanate upward from a remelted channel within a mushy zone. At 2000 s in the simulation, the channels along the walls are clearly evident and segregation is extensive, even though the leading part of the mushy zone has advanced only 1 cm. For example, the concentration of solute in the remelted channels is as much as 31.5% Sn and, of course, some of the Sn is transported from the mushy zone into the all-liquid zone by the upward flowing plumes. With no convection, the simulation shows essentially no macrosegregation except for an initial portion where solidification was effected in a temperature field with a variable gradient.

We now have many experimental DS castings that have been prepared in measured or known temperature fields. The macrosegregation in each has been measured. In addition, microstructures have been taken from quenched ingots, including regions near the leading part of the advancing mushy zone. With these microstructures, we have been able to discern whether the solid solidified with a dendritic or cellular morphology. This is important because the extent of macrosegregation increases with decreasing growth speed as the solid changes from a dendritic to cellular morphology.

An investigation into the influence of a transverse magnetic field (0.45) on the mush zone morphology and macrosegregation in directionally solidified hypoeutectic Pb-Sn alloys shows that the field has no influence on the morphology of dendritic arrays. The field does, however, cause severe distortion in the cellular array morphology. Cellular arrayed growth with the magnetic field results in an extensive channel formation in the mushy zone, as opposed to the well aligned and uniformly distributed cells formed in the absence of the field. The channels are produced due to the anisotropy in the thermosolutal convection caused by the magnetic field. Macrosegregation, however, along the length of the directionally solidified samples, is not influenced by this magnetic field for either the cellular or dendritic arrays. These experiments demonstrated that the low gravity environment is necessary to affect solidification with minimal convection because convection cannot be suppressed by the magnetic field.

STUDENTS FUNDED UNDER RESEARCH:

| BS Students: | 2 | BS Degrees: | 1 |
| MS Students: | 0 | MS Degrees: | 0 |
| PhD Students: | 2 | PhD Degrees: | 0 |

| TASK INITIATION: | 2/93 | EXPIRATION: | 1/96 |
| PROJECT IDENTIFICATION: | 963-25-05-09 |
| NASA CONTRACT NO.: | NAG3-1446 |
| RESPONSIBLE CENTER: | LeRC |

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals


Proceedings
II. MSAD Program Tasks — Ground-based

Kinetics of Phase Transformation in Glass Forming Systems

Principal Investigator: Dr. Chandra S. Ray
University of Missouri, Rolla

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
The objectives of this research are to: develop computer models for realistic simulations of nucleation and crystal growth in glasses, which would also have the flexibility to accommodate the different variables related to sample characteristics; and design and perform nucleation and crystallization experiments to verify these models. This research will lead not only to improved methods for the analysis of kinetic parameters for nucleation and growth determined from the peak profile studies of nonisothermal differential scanning calorimetry (DSC) or differential thermal analysis (DTA) measurements of crystallization, but also to determination of the relative merits and demerits of the theories presently used to study the phase transformations in glasses.

Task Description:
This research is to study and explain the critical issues for the nucleation and crystallization in glass-forming systems. The reported data for the kinetic parameters that determine the overall nucleation and crystallization mechanisms are often difficult to interpret on the basis of existing theory. The interpretation becomes more difficult when a variation in the characteristics of the glass, such as the thermal history, composition, particle size of the sample, and concentration of the nucleating agent, are taken into account. This is probably due to the fact that the theories that are presently used to analyze the isothermal and nonisothermal crystallization data for glass-forming systems are over simplified. Glasses are traditionally prepared by cooling a melt and are not in a state of stable equilibrium. Consequently, phenomena such as atomic mobility, cluster distribution, nucleation and crystal growth rate, and viscosity pertaining to the nonequilibrium state need to be accounted for to establish an accurate description of the phase transformations in glass forming systems.

Glasses (primarily silicate based) that devitrify polymorphically by homogeneous nucleation will be studied experimentally and by computer modeling. A lithium-disilicate (Li₂O.2SiO₂) glass will be used for most calculations and experimental measurements since the necessary thermodynamic and kinetic parameters are available for this system. Other glasses that will also be investigated to verify the general applicability of the model include soda-lime-silica (Na₂O.2CaO.3SiO₂) and barium-disilicate (BaO.2SiO₂). Crystallization experiments for the glasses will be conducted by DTA or DSC using the conditions used for computer modeling, such as the quench rate used to prepare the glass, sample weight, particle size of the sample, precrystallization heat treatment temperature and time, type and amount of nucleating agent, and DTA or DSC scanning rate. The experimental results will be compared with those predicted by the model. If flight opportunities become available, glasses of identical compositions will be prepared in space, and the same crystallization experiments as were conducted for the Earth-melted control samples will also be performed for the returned flight samples. The results from this research are anticipated to yield a realistic model for nucleation and crystal growth processes occurring in glass-forming melts which would provide not only an improved scientific understanding for these processes, but also allow a more accurate quantitative analysis of the thermal analysis data. This would help to explain several anomalous experimental results obtained for the kinetic parameters for crystallization and would lead to values that are more physically interpretable. The relative role of heterogeneous and homogeneous nucleation on glass formation can be determined, and the reported observation that a glass prepared in low gravity is more homogeneous and more resistant to crystallization than an identical glass prepared at 1-g can be explained.
Task Significance:
The results from this research are anticipated to yield a realistic model for nucleation and crystal growth processes occurring in glass-forming melts, which would provide not only an improved scientific understanding for these processes, but also allow a more accurate quantitative analysis of the thermal analysis data. This would help to explain several anomalous experimental results obtained for the kinetic parameters for crystallization and would lead to values that are more physically interpretable. The relative role of heterogeneous and homogeneous nucleation on glass formation can be determined, and the reported observation that a glass prepared in low gravity is more homogeneous and more resistant to crystallization than an identical glass prepared at 1-g can be explained.

Progress During FY 1995:
1. An experimental method that uses differential thermal analysis (DTA) was developed for determining the nucleation rates at different temperatures for a lithium disilicate (LS₂) glass. The DTA peak height for homogeneously nucleated LS₂ glasses was compared with that for glasses containing different amounts of platinum. The only assumption made in developing this method was that the crystal growth rate on a heterogeneous nucleus was essentially the same as that on a homogenous nucleus. Nucleation rates for the LS₂ glass determined by the present method are in excellent agreement with those reported in the literature. The validity of this method was tested using computer modeling developed by Dr. K. F. Kelton at the Washington University in St. Louis.

2. Heterogeneous nucleation and its effects on nucleation frequencies and induction times for a lithium-disilicate (LS₂) glass containing small amounts of Pt were investigated. A realistic computer model that describes phase transformation for heterogeneous nucleation was developed. An excellent agreement between the experimental results and computer modeling was observed.

3. The effect of particle size on DTA peak parameters (height, temperature and width) was experimentally established for the LS₂ glass. Analyzing the data as a function of particle size, it is possible to predict the dominant growth mechanism (bulk or surface) that occurs during crystallization of a glass. The DTA peak profiles for the LS₂ glass as a function of particle size indicated a considerable contribution of surface crystallization, which was also observed in the scanning electron microscopy (SEM) for the crystallized glass particles. The computer programs developed by Dr. K. F. Kelton were modified to take into account the effect of surface crystallization, and an excellent correlation between the experimental and theoretical results for the LS₂ glass were obtained.

4. An experimental method was developed to determine the nucleation rate-like curve for the LS₂ glass from dielectric constant measurements. The dielectric constant was measured at 1000 kHz for samples of about 2 mm thick and 8 mm in diameter after prenucleation and crystallization treatment at different temperatures for different times. The plot for the inverse of dielectric constant as a function of nucleation temperature yields a curve similar to that of the nucleation rate curve for the LS₂ glass.

**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Journals**

II. MSAD Program Tasks — Ground-based

**Discipline:** Materials Science


**Proceedings**


**Presentations**


II. MSAD Program Tasks — Ground-based

The Effects of Microgravity on Vapor Phase Sintering

Principal Investigator: Prof. Dennis W. Readey

Colorado School of Mines

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
The objective of this research is to develop an improved understanding of solid sintering by investigating the effects of reactive atmospheres and vapor transport on the sintering of ceramics. Ceramic systems that have been studied to date show a wide range of behavior that is thought to be due to the close proximity of particles in powder compacts appropriate for sintering and densification. The microgravity environment offers the unique opportunity to compare behavior of dispersed particles, or particles with minimal agglomeration, with those in a dense powder compact. These comparisons allow the separation of the relative contributions of particle interaction by gas phase diffusion and coalescence controlled by grain boundary mobility.

Task Description:
This research combines a study of the effects of vapor transport in powder compacts with those of dispersed particles. Ideal systems for space-based experiments will be determined from initial ground-based research. First, such an ideal system must exhibit significant microstructural effects of vapor transport. Second, the ideal system must show a difference in behavior between constrained particles in close proximity such as those in a typical ceramic powder compact compared to dispersed, unconstrained particles. Finally, an ideal system would be one that could be used at low temperatures, would not involve toxic materials or atmospheres, and would be amenable to short-time experiments. The latter would allow experiments to be performed in drop tower or aircraft parabolic maneuvers.

Task Significance:
The significance of this research is that it will lead to:

1. An improved understanding of solid state sintering of ceramics.

2. An improved understanding of the contributions that various transport processes make to microstructure development during the sintering of ceramics.

3. Will provide information for the design and production of porous ceramics through enhanced vapor phase sintering.

The latter is particularly important from a technical point of view since materials such as Al₂O₃ are being sintered to a controlled pore size and porosity for filter applications and for infiltration to form interpenetrating ceramic-metal composites.

Progress During FY 1995:
Prior work (see FY94 MSA Task Book) has shown that the main effect of enhanced vapor transport is significant particle coarsening with virtually no densification. This coarsening occurs over an extended period and is made difficult to model and understand by contacting particles, hence the need for extended microgravity experiments. Examination of traditional systems showed problems of high temperature, toxicity, and difficulties in measuring gas diffusion coefficients and solid surface energies.

Printed: 28 FEB 1996
II-703
INORGANIC SURROGATES

Various halides were examined in FY94. In this year attention was turned to sulfides. Zinc sulfide, ZnS, and cadmium sulfide, CdS, have high vapor pressures although at somewhat higher temperatures than desired. Never-the-less, these are harder materials and can be formed into powder compacts and behave, in general, similarly to other ceramic materials. In addition, different atmospheres can be used since hydrogen enhances the vapor pressure through the reaction:

$$\text{ZnS(s)} + \text{H}_2(\text{g}) = \text{Zn(g)} + \text{H}_2\text{S(g)}.$$  

Both of these materials were studied extensively and quantitative data on grain growth during sintering has been obtained. The data on these materials and the other inorganic surrogates investigated is has been incorporated into an M.S. thesis completed December 1995.

ORGANIC SURROGATES

An extensive survey of organic surrogates was conducted over the last two years. While these have low melting points and high vapor pressures, their particle size analysis is difficult. The research on organic surrogates has been incorporated into an M.S. thesis completed December, 1995.

VAPOR TRANSPORT IN DISPERSED SYSTEMS

In addition to the surrogate materials, another avenue that was to be investigated is the coarsening of more dispersed systems. This is being accomplished in two ways: 1) investigating coarsening in a multi-component system in which only one component coarsens; and 2) investigating coarsening in an extremely low density powder compact made by sol-gel techniques. Work has been initiated with Fe$_2$O$_3$ as one of the main constituents in HC1 since previous research has shown that this material is the most well behaved in terms of coarsening by vapor transport. Also, it coarsens in HC1 at temperatures between 900 and 1300°C so that the existing Crystal Growth Furnace could be used in shuttle experiments. Coarsening experiments have begun in the Fe$_2$O$_3$-Al$_2$O$_3$ system which show only limited solid solubility and only the Fe$_2$O$_3$ coarsens at low temperatures. Preliminary data show that the presence of the Al$_2$O$_3$ inhibits the coarsening of the Fe$_2$O$_3$. In contrast, preliminary experiments in the Fe$_2$O$_3$-NiO system in which both components coarsen, show that coarsening is not inhibited in this system. Finally, extremely low density (as low as 10 volume percent solids) power compacts of Fe$_2$O$_3$ are being prepared by sol-gel techniques to investigate coarsening in this lightly constrained system. This research is being performed by a Ph.D. candidate.

CONCLUSIONS

The necessity for long flights and non-toxic atmospheres has led to a search for suitable low temperature surrogates to study vapor phase sintering of ceramics. The most promising inorganic compound found to date is ZnS. Although ZnS requires temperatures in the neighborhood of 1000°C, it has a sufficiently high vapor pressure to produce the desired effects without the presence of a reactive, and possibly toxic, atmosphere. Cinnamic acid is a suitable low temperature (around 100°C) surrogate to investigate coarsening.

FUTURE WORK

The coarsening experiments in multi-component and low density systems containing Fe$_2$O$_3$ in HC1 will continue. Since Fe$_2$O$_3$ in HC1 is the one system studied that seems to fit the sintering and coarsening models most closely, transport processes and reactions in this system are being precisely characterized to answer questions about the effects of vapor transport during sintering of ceramics. Some of these questions include: 1.) "Are the forward and reverse surface reactions sufficiently fast to be ignored?" 2.) "How does the presence of a non-coarsening second phase (Al$_2$O$_3$) affect coarsening?" 3.) "What happens in a multi-component system in which all components are volatile (Fe$_2$O$_3$-NiO)?" and 4.) "How does the initial green density affect the shrinkage and coarsening in a powder
II. MSAD Program Tasks — Ground-based

Discipline: Materials Science

compact?" Finally, preliminary experiments on powder aerosol agglomeration both 1-g and in microgravity (DC-9) experiments will begin. The goal of these latter experiments will be to determine the degree of powder agglomeration and try to characterize the dimensionality (fractal geometry) of these agglomerates.

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PROJECT IDENTIFICATION: 962-26-05-08

NASA CONTRACT NO.: NCC3-289

RESPONSIBLE CENTER: LeRC

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Presentations
Modeling of Detached Solidification

Principal Investigator: Dr. Liya L. Regel
Clarkson University

Co-Investigators:
Wilcox, W.R.
Clarkson University

Task Objective:
The long term objective of this research is to develop techniques to achieve detached solidification reliably and reproducibly, in order to produce crystals with fewer defects. To achieve this objective, it is necessary to understand thoroughly the physics of detached solidification. It is the primary objective of the present project to achieve this understanding.

Task Description:
Ingots directionally solidified in space often have had diameters smaller than their containing ampoules, after correcting for the difference in thermal expansion coefficients. It is believed that such ingots have greater crystallographic perfection (i.e., fewer dislocations, twins, and grain boundaries). This is a better reason to grow crystals in space than the classical objective of achieving uniform doping.

Following is our mechanism for detached solidification in space. When solidification first begins, the solid is in intimate contact with the ampoule wall. As it cools from the freezing temperature, stress builds up between the ampoule and the solid because of the difference in their thermal expansion coefficients. Eventually this stress is sufficient to break the solid free from the ampoule. The gap between the solid and the ampoule is filled by evaporation of a volatile constituent from the melt. This constituent, probably residual gas that has dissolved in the melt, had accumulated near the freezing interface because of the usual impurity segregation. The gap width adjusts itself with subsequent solidification until transport of the volatile constituent to the meniscus is sufficient to provide the pressure difference across the meniscus required by surface tension.

The primary FY95 task was to derive and develop methods to solve the steady state equations relating the gap width, the meniscus curvature, the gap pressure, the velocity field, and the concentration field. We were also to have looked at the possibility of ground-based experiments on detached solidification.

Task Significance:
Solidification in space has often yielded crystals that were smaller in diameter than their containing ampoules. When this occurs, crystals of much higher perfection are produced. We now understand how this occurs and why crystal perfection is greater. This research project aims to put this understanding on a firm basis so that detached solidification can always be achieved. The research should also yield improvements in crystal growth on earth.

Progress During FY 1995:
1. Recruited Ph.D. student in Engineering Science in February 1995 (previous M.S. student failed his course work and had to leave).
2. Obtained an equation relating melt contact angle, growth angle, steady state gap width, and the pressure difference across the meniscus.
3. Derived the steady state differential equations and boundary conditions for convection and transport of a volatile species in the melt during detached directional solidification at zero gravity. To avoid a singularity where the meniscus contacts the ampoule wall, it was necessary to make the usual assumption of slip at the wall when the shear exceeds a critical value. The value chosen for this critical shear stress proved to have little effect on the flow field or concentration field.
II. MSAD Program Tasks — Ground-based

Discipline: Materials Science

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4. Developed finite difference numerical techniques to solve the above equations, with the equations of motion expressed in stream function and vorticity forms.

5. Began a parametric study of the influence of physical properties and operating parameters on the flow field, concentration field, and steady state gap width. Found, for example, that although Marangoni convection driven at the meniscus has a large influence on the nearby flow field, it does not have a large effect on the gap width.

6. Concluded that acceleration influences detached solidification primarily by causing buoyancy-driven convection, which perturbs the concentration field and inhibits the transport of volatile species into the gap. The pressure difference across the meniscus was found to be much larger than the hydrostatic pressure at Earth's gravity. Consequently, the experiments originally proposed were deemed to be irrelevant to our mechanism for detached solidification. We have developed a new concept for achieving detached solidification in earth.

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals


Presentations
Wilcox, W.R., and Regel, L.L. "Materials processing at low and high gravity (plenary lecture)." CAM '95, Canadian Association of Physicists Congress, Quebec, June 1995.
II. MSAD Program Tasks — Ground-based

Discipline: Materials Science

Drop Tube Operation

Principal Investigator: Dr. Michael B. Robinson  
NASA Marshall Space Flight Center (MSFC)

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
The objective is to maintain and upgrade the operation capability of and to conduct experiments in the MSFC 105-meter drop tube. The drop tube facility includes, in addition to the tube itself with the associated pumps and valves, such items as furnaces, levitators, other sample holding or handling devices, and data recording systems. This research includes the operation of a facility laboratory located at the facility. This laboratory includes furnace test facilities as well as pyrometer calibration platforms.

Task Description:
This research covers work in the area of defining, developing, and conducting experiments using the low-gravity capabilities of the drop tube. Such experiments may be in themselves complete investigations to develop new knowledge or to prove theories, or they may serve as precursors for more extensive experiments to be conducted in space. This research also includes studies and experiments to define the effects of various levels and durations of acceleration perturbations on microgravity experiments.

The research approach will be to:

• Study the limits of undercooling in a low-gravity containerless environment and ascertain if nucleation occurs homogeneously at the undercooling limits.

• Evaluate the effects of deep undercooling by containerless processing on resulting microstructure and define and understand the types of phases formed, their shapes and sizes, and their distribution, abundance, composition, homogeneity, and substructure.

• Study the formation of quasi-crystalline material through deep undercooling.

• Study the spreading of liquids as a function of undercooling in order to better understand the thermophysical properties of materials.

Task Significance:
This research activity is an essential part of a successful program of research in microgravity science and applications. Many experiments proposed for flight on the Space Transportation System (STS) can be developed and tested in preliminary form using drop facilities. This can result in significant savings through the proving of experiment concepts and equipment designs before proceeding to much more costly space flight hardware. It also provides additional data that can be compared with data obtained from the space flight experiments. And in some cases, the data obtained from the drop tube or aircraft experiments prove to be sufficient to satisfy the experimenter's requirements, thus obviating the need to proceed with an experiment on the STS. The result is an overall savings in the cost of conducting microgravity experiments while adding to the scope and quality of the results obtained.

Progress During FY 1995:
In FY95, the MSFC 105 Meter Drop Tube Facility supported six ground-based investigations and three flight programs. Recently a series of tests were conducted where the spreading of a liquid drop was recorded by high-speed photography with frames rates as high as 50,000 frames per second. This research is performed in conjunction with
II. MSAD Program Tasks — Ground-based

Dr. Julian Szekely of MIT. The purpose of this research is to determine the viscosity of undercooled metals by the rate and shape of spreading of the sample after impact with a heated copper plate.

Other drop tube research supported the study of nanocrystalline materials, the nucleation temperature distribution of undercooled high-purity metals, the undercooling of immiscible metal systems, and easy glass forming metallic systems. In addition, the drop tube supported the ground-based research supporting the recent flight of the TEMPUS program aboard the Second International Microgravity Laboratory (IML-2) space mission.

STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 1/78   EXPIRATION: 1/95
PROJECT IDENTIFICATION: 962-28-08-02
RESPONSIBLE CENTER: MSFC

Printed: 28 FEB 1996

II-709
Measurement of the Optical and Radiative Properties of High-Temperature Liquid Materials by FTIR Spectroscopy

Principal Investigator: Dr. Michael B. Robinson
NASA Marshall Space Flight Center (MSFC)

Co-Investigators:
Khrishan, S.
Rathz, T.
Workman, G.
CRI
University of Alabama, Huntsville (UAH)
University of Alabama, Huntsville (UAH)

Task Objective:
The objective is to fully develop the use of FTIR spectroscopy for the purpose of determining, at a high rate of speed, the normal and total hemispherical emissivities of deeply undercooled materials over the wavelength range of 2 to 20 microns. Due to the nature of the approach, the spectral emissivities can be determined quickly over a wide wavelength range and applied over a wide temperature range.

Task Description:
The task will involve development of the technique of measuring the radiative properties of high-temperature, highly reactive materials in the liquid and undercooled liquid state by use of FTIR spectroscopy. The sample will be positioned in a containerless environment by either electromagnetic or electrostatic positioners. The technique offers the advantage of fast measurement so that sample temperature stability will not present insurmountable problems.

Task Significance:
Optical property measurements are essential to the understanding of the behavior of liquids at high temperature. Accurate knowledge of these properties provide an ability to validate theories of nucleation, solidification, and undercooling, and provide the basis for accurate non-contact temperature measurement. They are particularly needed for accurate measurement of high temperatures so that existing and new thermophysical property measurements can be interpreted correctly.

Progress During FY 1995:
The FTIR system design has been completed and all necessary hardware purchased. Construction of the optical system and supports are underway. In addition, the uniquely designed electromagnetic levitation coils and system is nearly complete. The near-term goal is to characterize the optical properties, including emissivity, of a tungsten standard with a black-body source. Once the standard is completely characterized and the results published, the optical properties of unknown, undercooled alloys can be measured.

STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 9/94 EXPIRATION: 9/96
PROJECT IDENTIFICATION: 962-25-08-32
NASA CONTRACT NO.: NAG8-1069
RESPONSIBLE CENTER: MSFC

Printed: 28 FEB 1996

II-710
II. MSAD Program Tasks — Ground-based Discipline: Materials Science

Undercooling Behavior of Immiscible Metal Alloys in the Absence of Crucible Induced Nucleation

Principal Investigator: Dr. Michael B. Robinson NASA Marshall Space Flight Center (MSFC)

Co-Investigators:
- Frazier, D.
- Facemire, B.
- Rathz, T.
- Workman, G.

NASA Marshall Space Flight Center (MSFC)
University of Alabama, Huntsville (UAH)

Task Objective:
The objective of this effort is to determine if processing immiscible metals in a containerless environment would alter the critical point wetting mechanism and the extent to which this would lead to changes in the subsequent nucleation kinetics. If the nucleation kinetics are suppressed enough to allow bulk undercooling, the nucleation recalescence will be measured. In addition, the droplet surface conditions can be varied by processing in a vacuum versus a gas environment to alter the surface composition and thereby the wetting potential of the fluid phases.

Task Description:
Research in a containerless, low-gravity environment provides much information as to whether preferential wetting of the free surface occurs and more importantly whether it can be controlled. The MSFC 105 Meter Drop Tube Facility will provide the low-gravity, containerless environment necessary for this study.

Task Significance:
Cooling of monotectic alloys into the miscibility gap will lead to nucleation of droplets within the liquid matrix. Previous earth and space experiments have concentrated on the morphology of the bulk microstructure by attempting to control the convective-diffusive flow of these droplets with the proper selection of the crucible material and thermal fields. Immiscible metal systems have as yet not been studied in a containerless environment, eliminating crucible induced flows.

Progress During FY 1995:
Numerous samples have been processed in the MSFC 105 Meter Drop Tube Facility. Samples have been centered around the study of two immiscible systems, the Vanadium-Gallium system and the nickel-silver system. As yet complete metallurgical analysis has not been completed, with primary focus during FY95 being on the finding of a suitable system for study.

STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 9/94 EXPIRATION: 9/96
PROJECT IDENTIFICATION: 962-25-08-33
NASA CONTRACT NO.: H-15269D
RESPONSIBLE CENTER: MSFC

Printed: 28 FEB 1996 II-711
II. MSAD Program Tasks — Ground-based

Undercooling Limits in Molten Semiconductors and Metals: Structure and Superheating Dependencies

Principal Investigator: Dr. Frank G. Shi

Co-Investigators:

Rhim, W.K. Jet Propulsion Laboratory (JPL)
Rulison, A.J. Space Systems/Loral

Task Objective:
The objective of this work is to study the onset of nucleation of crystals and thus the undercooling limits for melts of semiconductors and metals in order to experimentally test and further develop a model for the onset of nucleation.

Task Description:
The undercooling experiments are performed using DSC-7 and the High Temperature Electrostatic Levitator (HTESL). The HTESL is fully operational and has proven to be effective in melting and undercooling metals (e.g., zirconium and nickel), semiconductors (e.g., germanium), and ceramics (e.g., silicon dioxide based glass-ceramics). The HTESL is fitted with a single color pyrometer which is used to measure the undercooling limits of all the samples. The DSC experiments which are not included in the original proposal are found to be necessary in elucidating the effects of container in determining the undercooling limits.

Task Significance:
The onset of nucleation in the undercooling liquids and thus the achievable undercooling level determines the selection of the final microstructures and formation of metastable amorphous phases. The ability to predict and control the onset of nucleation is therefore important in advanced materials processing.

Progress During FY 1995:

Experiments: Se, Li$_2$B$_4$O$_7$ and Sn$_{30}$Bi$_{50}$ have been selected to examine the dependence of the maximum undercooling on the temperature at which melts are annealed above their melting points. A series of experiments on Se have shown that, as our theory predicted, the maximum temperature of undercooling is indeed a function of the temperature at which the melt is annealed above the melting point. While continued DSC experiments have being done on Se, Li$_2$B$_4$O$_7$, and Sn$_{30}$Bi$_{50}$, parallel experiments have also begun to determine the undercooling limits using the HTESL.

Model: Our model for the onset of nucleation is being refined to predict the overheating dependence of undercooling limits. As an extension of the proposed research, we have developed a new approach for measuring the nucleation free energy barrier, which has been applied to various systems.

STUDENTS FUNDED UNDER RESEARCH:

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TASK INITIATION: 10/94 EXPIRATION: 9/96

PROJECT IDENTIFICATION: 962-21-08-22

NASA CONTRACT NO.: NAG8-1082

RESPONSIBLE CENTER: MSFC

Printed: 28 FEB 1996
BIBLIOGRAPHIC CITATIONS FOR FY 1995:

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Shi, F.G. Direct measurement of free energy barrier to nucleation of crystallites in amorphous silicon thin films. J. of Applied Physics, 76, 149-154 (1994).


Double Diffusive Convection during Growth of Lead Bromide Crystals

Principal Investigator: Dr. N. B. Singh
Westinghouse Electric Corporation

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
The main objective of this program is to evaluate, understand and eliminate thermosolutal convection during the crystal growth of PbBr$_2$-AgBr systems. The program will provide a quantitative understanding of convective effects and a correlation of experimental data with theories developed for thermosolutal convection will be carried out. For the PbBr$_2$-AgBr system less dense solute causes the convective (thermosolutal) instability in addition to morphological instability. Also, this system is optically transparent and we can monitor the interface shape to study the convective and morphological instabilities. The technical objectives of this program are to define the parameter at normal gravity to minimize the thermosolutal convection during growth of doped lead bromide crystals to achieve homogeneous distribution of dopant, significantly reduce the optical and acoustic scattering caused by convection during lead bromide crystal growth, and produce lead bromide crystals with unparalleled optical homogeneity for advanced device applications. This will be achieved by experimentally verified stability diagrams and direct observations of solid-liquid interface during crystal growth.

Task Description:
To achieve these objectives, crystal growth experiments will be conducted on earth and in space. Measurements involving Rayleigh number as a function of aspect ratio, and the radius of the growth tube to the length of the melt column, will be made. Experimental results will be compared with the stability diagram to test the validity of morphological and convective stability theories.

Task Significance:
The scientific objectives of this program are to understand the thermosolutal convection during the crystal growth of PbBr$_2$-AgBr system. This will be achieved by growing five crystals at five different concentrations, which will lead to different solutal convective levels. The experimental values of the concentration distribution will be compared with the theories based on pure diffusional growth to evaluate the effect of convection. Also, numerical studies will be carried out to study the convective and morphological instabilities, and to determine the critical concentration of dopant for a particular growth velocity and gravity level. Theoretical instability diagrams will be compared with the experimental studies. Relevant analytical characterization techniques are to be used to evaluate the effect of convection on crystal quality. These studies will provide basic data on convective behavior in doped lead bromide crystals grown by the commercially important Bridgman process.

Progress During FY 1995:
Direct observations were taken during crystal growth of lead bromide samples doped with 500 and 5000 ppm silver bromide in 1-g conditions. Stationary solid-liquid interface was flat. When we started growth by moving the ampoule at a velocity in the neighborhood of the critical velocity for interface breakdown, the interface got depressed and the shape of depressed pit varied with the velocity. It became sharply pointed and the instability then slowly formed by pulling down the central part of the interface. When the translation rate was increased, the interface broke down. The preliminary data showed that theoretically predicted stability curve agrees well with experimentally observed values. The flow patterns can be described as toroidal rolls. After many hours of sustaining the toroidal flow with the tube moving, we observed pinching of the interface at the node where radial inward flow converges and a line defect was formed. The instability could be explained as follows: When growth of doped crystal started, the radial gradients increased due to the effect of drawing the hotter material downward through the viewing block.
and due to latent heat emitted from the solid-liquid interface. The convective flow pattern was correspondingly altered.

Theoretical calculation on convective and morphological stability curve required diffusion coefficient and thermal conductivities for the solid and liquid. For this reason we measured diffusion coefficient of molten lead bromide-silver bromide system in a two chambered diaphragm cell separated by porous membrane of a sintered glass disk. The interdiffusion coefficient was determined to be $1.71 \times 10^{-3}$ cm$^2$/s above the melting temperature (400C) of lead bromide.

The ratio of the thermal conductivities of liquid and solid lead bromide was determined by measuring the temperature in the solid and liquid lead bromide and by using the heat flow equation. The temperature measurements provided the ratio of gradients in the liquid and solid lead bromide and we used $10.5 \times 10^4$ cal/K.cm.s as the value of thermal conductivity of liquid lead bromide from the literature. The ratio of thermal conductivities was determined to be 1.65 and thermal conductivity of solid lead bromide, $6.36 \times 10^4$ cal/K.cm.s, was calculated from the thermal gradient measurements.

The effect of thermal convection on crystal quality was studied by growing four crystals in different thermal convective conditions. The convective conditions were achieved by varying the thermal Rayleigh number by several orders of magnitude. The crystal grown at the lowest thermal Rayleigh number had full width at half maximum (FWHM) of 0.18°, the contour scan showed a variation of $2 \theta/\omega$ ranging from -0.7 to +0.7, and etch pit density of $1.9 \times 10^2$ cm$^2$, while the crystal with the largest thermal Rayleigh number had full width at half maximum (FWHM) of 0.52°, contour scan showed a variation of $2 \theta/\omega$ ranging from -1.5 to +1.5, and etch pit density of $3.3 \times 10^3$ cm$^2$. These data on x-ray rocking curves, x-ray contour scans and etchpit studies showed that crystals grown at lower Rayleigh numbers had the best quality. The effect of solutal convection on crystal quality was studied by growing crystals at different solutal Rayleigh numbers. For crystals grown at solutal Rayleigh numbers $5.83 \times 10^3$ and $2.7 \times 10^4$ FWHM were $0.2^\circ$ and $0.52^\circ$, $2 \theta/\omega$ ranged from -1.0 to +1.0, and -1.5 to +1.5, and chemical etchpits were $5.83 \times 10^3$ and $2.7 \times 10^4$ per cm$^2$ indicating that homogeneity was better for crystals grown at lower solutal Rayleigh number.

**STUDENTS FUNDED UNDER RESEARCH:**

**TASK INITIATION:** 10/93  **EXPIRATION:** 9/94

**PROJECT IDENTIFICATION:** 962-24-05-01

**NASA CONTRACT NO.:** NAS3-25811

**RESPONSIBLE CENTER:** LeRC

**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Journals**

**Presentations**


Crystal Nucleation, Hydrostatic Tension, & Diffusion in Metal and Semiconductor Melts

Principal Investigator: Prof. Frans A. Spaepen
Harvard University

Co-Investigators:
Aziz, M.J. Harvard University
Turnbull, D. Harvard University

Task Objective:
The objective is to develop basic understanding of the phenomena and processes that are central to the microgravity program: crystal nucleating, glass formation, and diffusion in the liquid state.

Task Description:
Crystal nucleation is studied in elemental metal, semiconductor, or quasi-crystal-forming droplets coated with different fluxes, droplets with clean surfaces in vacuum, and droplets solidified in a drop tube. The effect of hydrostatic stress on the nucleation kinetics is studied by dilatometry. The crystal-melt interfacial tension is studied experimentally and theoretically. The diffusivity in the liquid state is measured from the broadening of impurity profiles after pulsed laser melting.

Task Significance:
Studies of the undercooling of liquids and the kinetics of crystal nucleation are an important category of experiments that exploit the containerless environment provided by microgravity. Our work, ground-based, is aimed at advancing the understanding of the fundamentals of undercooling, nucleation, and glass formation; at exploring the potential and limitations of ground based alternatives such as fluxing and drop-tube processing; and at exploring the potential of ground-based containerless processing facilities provided by the microgravity program.

Progress During FY 1995:
The work on the undercooling of molten Si has continued. The SiO$_2$.BaO.CaO flux, developed to have the necessary fluidity and chemical compatibility to be used with molten Si, allowed us to achieve undercoolings up to 350K, even larger than those reported last year. This undercooling is 75K greater than that achieved in earlier experiments on bulk uncoated Si. The result implies that homogeneous crystal nucleation did not occur in the earlier experiments; most likely, it did not occur in our present experiments either. The crystal-melt interfacial tension of 0.38J/m$^2$ derived from the application of the classical theory for homogeneous nucleation is therefore a lower limit. A comparison with the value derived at even larger undercoolings from experiments on laser-melted thin films by Stifler et al. (0.34 J/m$^2$) indicates that the interfacial tension has a positive temperature coefficient. The temperature dependence can be accounted for by reasonable values of the interfacial entropy and enthalpy; the entropy drop in the liquid near the interface is similar to that in other metallic melts, such as mercury (F. Spaepen, Solid State Physics, vol 47 (1994) p.1).

To exploit the new flux further and to understand the relation between the undercoolings achieved in molten Si and Ge, we have started a series of experiments on the undercooling of Si-Ge alloys. So far, morphological observations of the solidification product have been made. These will be correlated with the undercooling measurements.

The volumetric behavior of Ni as a function of temperature indicates that it may be possible to undercool pure bulk liquid Ni to the glassy state. To explore this possibility, we have started a number of undercooling and glass-forming experiments, using a B$_2$O$_3$ flux, on Pd-Ni-P alloys. Starting from the composition Pd$_{40}$ Ni$_{40}$P$_{20}$, which is a well known easy glass former, progressively more Ni-rich alloys are being explored.
II. MSAD Program Tasks — Ground-based

All the work on bulk undercooling, both for the semiconductors and metals, is being carried out by a graduate student, Yan Shao.

In order to interpret quantitatively our drop tube experiments on the solidification of Ga-Mg-Zn alloys, in which the Frank-Kasper phase MgZn₂ appears to nucleate abundantly at low undercooling, we have started a detailed analysis of fluid flow and thermal transport in a drop falling in a gas atmosphere, in which we concentrate on the regime at low undercooling. So far we have obtained results for the fully liquid drop. At this time we are extending them to include the growth of a moving nucleus. This work has been the carried out by a postdoctoral fellow, Jan Fransaer.

**Students Funded Under Research:**

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**Bibliographic Citations for FY 1995:**

**Journals**


**Presentations**


Spaepen, F. "Interface between a crystal and its melt: Structure, tension and implications for nucleation." Department of Physics, Washington University, St. Louis, MO, 22 February 1995.


**Printed:** 28 FEB 1996
II. MSAD Program Tasks — Ground-based

Discipline: Materials Science

**Micro- and Macro-Segregation in Alloys Solidifying with Equiaxed Morphology**

**Principal Investigator:** Dr. Doru M. Stefanescu
University of Alabama, Tuscaloosa

**Co-Investigators:**
- Nastac, L.
- Curreri, P.
University of Alabama, Tuscaloosa
- NASA Marshall Space Flight Center (MSFC)

**Task Objective:**
The objective of this research is to extend the recently developed model for microsegregation from a closed system to an open system. This means that the new model should include the influence of (i) buoyancy-driven flow (thermosolutal convection), (ii) of convection caused by the relative motion of the liquid/solid interface, and (iii) of convection caused by density variation with change of phase, on both micro- and macro-segregation. The analysis will be conducted only for equiaxed dendritic and eutectic alloys.

Microgravity experimentation will be used to assess the relative value of the three factors affecting solute redistribution. The experimental work will be performed on the KC-135 aircraft for multi-directional solidification.

**Task Description:**
The theoretical work will consist of the following tasks:

1. Develop a formulation to describe the rheology of the particular two-phase equiaxed systems of interest (dendritic and eutectic);

2. Develop an analytical solution for microsegregation for the case of an open system;

3. Develop a formulation for thermosolutal and shrinkage flow that includes description of both macro- and micro-segregation;

4. Couple the macro-transport to micro-transport through description of solidification kinetics;

5. Develop a numerical code to solve the above algorithm;

6. Evaluate the validity of the closed system microsegregation model for the case of low-g environment.

The open system model will be used in validation in conjunction with both high-g and low-g experiments. In addition, by turning off the shrinkage flow and/or the solid-liquid relative motion flow, the relative effects of these flows can be evaluated. The closed system model should come reasonably close to the low-g experiments. Validation of the models will be done through experimental measurement of solute concentration at the macro- and micro-scale level, and of the temperature distribution.

The equipment that will be used for the experiments is NASA's Isothermal Casting Furnace that has a temperature range of 100 to 1350°C, and quenching rate capabilities of 1 to 50°C/s. This furnace will be flown on the KC-135 aircraft. The samples will be melted before the low-g period of the flight, and solidified during the 25 s of low-g. Parallel experiments will be conducted on ground.

**Task Significance:**
At the micro scale level, the assumption that mass transport is purely diffusive is quite reasonable, and thus, a closed system may be a good approximation for certain alloy systems solidifying under terrestrial gravity. This assumption is even more valid when considering low-gravity (low-g) solidification. However, for most alloy...
systems the influence of fluid flow caused by thermal and solutal convection cannot be ignored. During solidification of a multicomponent alloy, buoyancy-driven fluid flow occurs due to the temperature and concentration gradients. Even in upward directional solidification it is difficult to avoid horizontal temperature gradients since the container walls are not perfectly adiabatic. Accordingly, there is a need to further develop the model to address the open system case.

Progress During FY 1995:
During FY95 two compositions of Al-Cu alloys have been investigated: Al-2%Cu and Al-5%Cu. A total of 35 samples were cast and amongst them eight samples were chosen for ground experiments and KC-135 flights. Two samples each of Al-2%Cu and Al-5%Cu were solidified under the ground conditions and in the KC-135 parabolic flights under the low-g maneuvers.

Vertical sections of the cast ingots were polished and etched to examine the micro- and macrostructures of the samples. Preliminary results show some significant variation in the ingot macrostructures in the low-g and the ground solidified samples. The dendritic size and the different zones of the cast ingots are quite different in the two cases. This clearly shows the effect of gravity on the solidification behavior of the alloys. The effect is manifested both in the change of size of the dendrites and the area of different zones (chilled, columnar, and the central equiaxed).

Further studies are ongoing to investigate the change in the composition of the ingot in the different microstructural zones. This would give an idea of the effect of gravity on the microsegregation. These studies are being done on the EPMA by running composition line profile scans.

STUDENTS FUNDED UNDER RESEARCH:

BIBLIOGRAPHIC CITATIONS FOR FY 1995:
Presentations
The Impaction, Spreading, and Solidification of a Partially Solidified Undercooled Drop

Principal Investigator: Dr. Julian Szekely
Massachusetts Institute of Technology (MIT)

Co-Investigators:
Trapaga, G.
Massachusetts Institute of Technology (MIT)

Task Objective:
The purpose of this project is to examine the spreading of molten metal droplets and their solidification upon impacting onto a solid cooled surface.

Task Description:
This project is aimed at studying the splatting and solidification behavior of undercooled and partially solidified metallic specimens. The project consists of both an experimental component and a computational and analytical component.

In the experimental arrangement a levitated and inductively melted metallic droplet will be allowed to fall in the Marshall drop tube, cool during flight, and then impinge on a chilled solid substrate. The flattened droplets will be collected and analyzed. The thermal management of the system will be so arranged that upon approaching the chilled substrate, the droplets will be undercooled in the range of 10° - 800° C, although, for some control experiments we will impinge superheated droplets or droplets close to their melting temperature. Furthermore, a number of experiments will be carried out, such that undercooled droplets will be made to impinge on the flat surface of the substrate.

However, perhaps the most novel feature of the investigation is that for the experiments, immediately prior to impingement of the droplet on the target, nucleation of the solid phase will be triggered by contact with one or more of many spikes raised above the substrate. The height of the spikes will be such that the specimen will be only partially solidified before impact.

Experiments to be run include impacts on a flat target by non-undercooled drops for comparison to previous work and undercooled drops to study the effect of the degree of undercooling on the stability (splashing tendency) and final microstructure of the drop. The study will also include the effect of controlled nucleation on the liquid-solid phase transformation by contact with a spike raised above the chill block. The occurrence of nucleation at a controlled time before impact will add a new dimension to the study of the competition between solidification and spreading of the droplet.

The theoretical work will include the development of a new set of equations to describe the behavior of the partially solidified droplet upon impact and the calculation of the rate of cooling of the specimen as it falls and the velocity field in the specimen as it falls. In addition, we will address issues of impingement and splatting.

Task Significance:
The problem of the spreading and solidification of molten metal droplets is of both fundamental and practical interest. The fundamental interest is provided by the fact that the simultaneous spreading and solidification of metal droplets is a generic problem in materials processing. The practical interest is associated with the immediate relevance of these phenomena to spray forming and the formation of coatings.

Progress During FY 1995:
During the first year, two sets of experimental campaigns were performed using NASA's unique tower facility at the Marshall Space Flight Center. The experiments were performed in collaboration with NASA personnel and...
researchers from Caltech. The experimental component of the project was combined with a theoretical part in which computer simulations were employed to better understand and establish the conditions required to design such experiments. The MIT team taking part in this project included a master's student and a research associate who were involved with the principal investigator. The main accomplishments obtained in this period involve, the demonstration of the experimental approach to produce splats of different materials and a large number of recordings of splattings were obtained using state of the art high speed photography. Data analysis and comparison between experimental data and predictions is currently in progress.

Results to be published pending further data collection and analysis.

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<td>NASA CONTRACT NO.: NAG8-1069</td>
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Printed: 28 FEB 1996
II. MSAD Program Tasks — Ground-based

Discipline: Materials Science

Microporous Membrane and Foam Production by Solution Phase Separation: Effects of Microgravity and Normal Gravity Environments on Evolution of Phase Separated Structures

Principal Investigator: Dr. John M. Torkelson

Northwestern University

Co-Investigators:

No Co-I's Assigned to this Task

Task Objective:

The objective of this research is to develop a quantitative understanding of the microstructure formation in glassy, polymeric microporous membranes and foams produced via thermally induced phase separation. In particular, this study will delineate the role of gravity (resulting in buoyancy-driven flows resulting in macroscopic layering of phases) or the absence thereof in both the early-stage phase separation mechanisms, spinodal decomposition versus nucleation and growth, and the later effects of coarsening, by Ostwald ripening and/or hydrodynamic flow.

Coarsening effects will be studied quantitatively by experimental determination of the growth in pore size over time as a function of quench depth, polymer molecular weight and concentration, surface tension, etc., in near-critical and off-critical point phase separation processes. Comparisons to expectations based on Ostwald ripening and hydrodynamic flow mechanisms of coarsening will be made. By developing a quantitative understanding of the thermodynamics and kinetics of polymer solution phase separation processes, both in terms of gravitational effects and the more ordinary effects of polymer/molecular weight/solvent/concentration/quench temperature/quench time conditions, it may be possible to optimize microporous membrane and foam structure and performance.

Comparisons between coarsening effects in 3-dimensional and 2-dimensional environments, the latter expected to behave as if they are in a microgravity environment even if the polymer and solvent have differing densities and the experiments are done on earth, will also be undertaken.

Task Description:

A microgravity environment will be simulated by use of isopycnic (iso-density) solutions of polystyrene in diethyl malonate, which upon thermally induced phase separation do not exhibit any macroscopic layering effects on the time frame of a week. Comparisons will be made to conventional, nonisopycnic polystyrene solutions.

Early-stage spinodal decomposition effects will also be investigated by comparing the experimental temperature dependence of pore size to predictions from the linearized Cahn-Hilliard theory for spinodal decomposition. Measurements of the average pore size and pore-size distribution in membranes and foams produced by thermally induced phase separation will be characterized by scanning electron microscopy and mercury intrusion porosimetry. Comparisons of the growth rate in pore size observed from the three-dimensional membranes will be made to in situ measurements of phase separation and coarsening in polymer solutions by optical microscopy; the thin nature (10 microns between the base slide and cover plate) of the solutions yields two-dimensional coarsening effects when the phase separated droplets are equivalent to or larger than the film thickness.

Task Significance:

The effects of gravity in the production of microporous membranes and foams from phase separated polymer solutions will be studied quantitatively for the first time. By accessing microgravity conditions, it will be possible to study critically how the phase domain growth rate during coarsening compares to theories, including those related to the Ostwald ripening mechanism and the hydrodynamic flow mechanism of coarsening. From a scientific standpoint, by simulating microgravity conditions it will be possible to test for the first time the evolution of coarsened microstructure in polymer solutions over five to six decades in phase separation time and to determine how gravity-driven flows affect this evolution. From a technological standpoint, the microgravity environment using the isopycnic solution will allow determination of how space processing of microporous polymer membranes and foams may result in materials unobtainable or obtainable only for limited polymer species (where isopycnic solutions can be found) in a conventional terrestrial environment. These studies will also allow for a determination of how novel materials may be developed in 2-dimensional phase separation. As buoyancy driven flows should
occur only in 3-dimensional phase separation, 2-dimensional phase separation may be expected to simulate microgravity environments even on earth regardless of the solvent and polymer densities as buoyancy driven flows should occur only in 3-dimensional phase separation.

Progress During FY 1995:

Polymer solutions of various compositions have been phase separated in two dimensions in order to monitor the growth rate of the domain size, \( d \), associated with coarsening in restricted dimensions. These two-dimensional coarsening studies, which are not subject to gravity-driven macroscopic phase separation, have also been compared to three-dimensional coarsening of phase-separating polymer solutions, where gravity-driven buoyancy effects can become important on short time scales if the difference in polymer and solvent densities are significant enough. We completed the first experimental two-dimensional coarsening study of any type to demonstrate that the domain-size growth rates are dependent on overall composition (specifically, distance from the "critical" point) and that a crossover in growth rates from \( d \) scaling as the 1/3 power in time to \( d \) scaling as the 2/3 power in time for originally near-critical solutions. While such growth rates and crossover had been predicted by theory and numerical simulation, this was the first study to show such effects experimentally in two-dimensions. These growth-rate effects compare favorably to the results obtained by us in three-dimensions using similar polymer-solvent systems, where \( d \) scales as the 1/3 power in time with a crossover at longer times to a linear (1st) power in time. In both two-dimensional and three-dimensional cases, the short time effects (1/3 power) are consistent with coarsening by Ostwald ripening and/or coalescence while the long time effects (2/3 power in two-dimensional and 1st power in three-dimensional) are consistent with the hydrodynamic flow mechanism of coarsening. More detailed comparisons are underway with the effects of interfacial tension, quench depth, polymer molecular weight, and solvent viscosity receiving special attention.

**Students Funded Under Research:**

BS Students: 0  
MS Students: 0  
PhD Students: 1

**Task Initiation:** 6/94 **Expiration:** 6/96

**Project Identification:** 962-21-08-23  
**NASA Contract No.:** NAG8-1061  
**Responsible Center:** MSFC

**Bibliographic Citations for FY 1995:**

**Journals**


**Presentations**


II. MSAD Program Tasks — Ground-based

**Fundamentals of Mold-Free Casting Experimental and Computational Studies**

**Principal Investigator:** Prof. Grétar Tryggvason  
University of Michigan

**Co-Investigators:**
Ceccio, S.L.  
Jacqmin, Dr. D.  
University of Michigan  
NASA Lewis Research Center (LeRC)

**Task Objective:**
1. Provide the scientific knowledge necessary to operate a net-shape drop and spray casting facility by a computer designed and optimized process. In addition to its importance for earth based manufacturing, such knowledge will allow the operation of a space based facilities by computer programs developed on Earth.
2. Develop a detailed understanding of the fluid mechanics, heat transfer, and solidification of drops splatting on a solid surface or a layer of other drops, that can be used to predict the microstructure of solids formed by such depositions.

**Task Description:**
The project has both an experimental and a computational part. The experiment will consist of a facility that will allow drops of controlled size and velocity to be deposited on a flat substrata where they solidify. The numerical part will build on techniques developed under previous NASA support for accurate simulations of drop motion. By a careful comparison of the numerical and the experimental results, we expect to be able to identify the key physical aspects of the process that must be included in the numerical model so that it yields accurate results, yet is sufficiently fast to be of practical use. To scale-up the drops to allow more detailed observations of the process, we expect to eventually conduct the experiments in a low gravity environment where the drops can be made larger and the velocities smaller, while keeping the essential balance of physical effects the same as for the small drops that will be used in the actual production of artifacts.

**Task Significance:**
Moldfree casting by precision controlled deposition of drops of molten metal is an emerging manufacturing process with considerable promise for rapid prototyping and the production of high quality, custom artifacts. A detailed understanding of the process, and the ability to predict the effect of the controlling parameters, is essential if the technique is to reach its full potential.

**Progress During FY 1995:**
The project has both an experimental and computational part. The experiment will consist of a facility that will allow drops of controlled size and velocity to be deposited on a flat substrata where they solidify. The design of the facility is complete, and construction of the facility will be finished at the end of FY 1995. Already completed are the drop generator, the substrate holder and traverse, and the control electronics. A vacuum chamber is currently being built. A computer program has been developed that models solidification together with two phase (liquid-vapor) surface tension induced flow. Preliminary results have been obtained, but more testing is needed, particularly for the heat transfer and solidification part.

**STUDENTS FUNDED UNDER RESEARCH:**

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**TASK INITIATION:** 7/94  
**EXPIRATION:** 7/96  
**PROJECT IDENTIFICATION:** 962-25-05-28  
**NASA CONTRACT NO.:** NCC3-355  
**RESPONSIBLE CENTER:** LeRC
Electromagnetic Field Effects in Semiconductor Crystal Growth

Principal Investigator: Dr. Martin P. Volz  
NASA Marshall Space Flight Center (MSFC)

Co-Investigators:
Mazuruk, K.  
Universities Space Research Associates (USRA)
Watring, D.A.  
NASA Marshall Space Flight Center (MSFC)
Dulikravich, G.S.  
Pennsylvania State University

Task Objective:
The objectives of this investigation are the following:

1. To investigate the effects that combined electric and magnetic fields have on gravitationally-driven fluid flow processes during the bulk growth of selected semiconductor alloys.

2. To examine the criteria for the onset of thermal instability as a function of electric, magnetic, and gravitational field strength in electrically conducting liquids via theoretical analysis, computer calculations, and laboratory experiments.

3. To assess the possibility of using externally applied magnetic and electric fields to influence fluid motion in the melt, the rate of solid phase accrual, and the shape of the solid/liquid interface during solidification experiments both on earth and in a low gravity environment.

Task Description:
A series of specific tasks are planned to achieve the proposed objectives. A model growth cell will be developed which will allow for the passage of electric current through the melt simultaneously with the application of a magnetic field. In situ temperature measurements will be made using gallium as a model material. Both zone-melting and vertical Bridgman thermal profiles will be applied and the onset of thermal fluctuations will be measured as a function of electric and magnetic field strength. Crystal growth experiments will then be made on GaInSb and CdTe under various electromagnetohydrodynamic conditions. Computer modeling of the above systems will be accomplished by using a three-dimensional mathematical model which takes into account both magnetohydrodynamic and electrohydrodynamic phenomena. The model predictions will be compared with the experimental results from the model growth cell.

Task Significance:
Electromagnetic fields can interact with electrically conducting melts and substantially affect their fluid flow processes. Indeed, the intriguing possibility exists of using combined electric and magnetic fields to control fluid flow in the melt during the directional solidification of semiconductor alloys. The significance of this study is to gain a basic understanding of the possible interactions that combined electric and magnetic fields can have on fluid flow processes during semiconductor crystal growth.

Progress During FY 1995:
A cylindrical test cell has been constructed and placed in the center of a rotating magnetic field. The cell contains liquid gallium and thermistors inserted in the cell are used to measure temperature fluctuations. Critical Rayleigh numbers have been measured as a function of rotating magnetic field strength and several different stability regimes have been identified. These regimes are determined by the values of the Rayleigh and Hartmann numbers. For weak rotating magnetic fields and small Hartmann numbers, the experimental observations can be explained by the existence of a single non-axisymmetric roll rotating around the cylinder, driven by the azimuthal component of the magnetic field. The measured dependence of rotational velocity on magnetic field strength is consistent with the
existence of laminar flow in this regime. In conclusion, the model fluid cell experiments indicate that a flow regime exists in which the benefits of a rotating magnetic field on the crystallization process are not compromised by time-dependent flow.

A new theoretical formulation of combined electromagnetohydrodynamics has been developed and shows the inconsistencies and shortcomings of the existing separate electrohydrodynamic and magnetohydrodynamic theories. The new model is for three-dimensional, unsteady, viscous fluid flows involving electrically charged particles and electric polarization and magnetization effects. All interaction between externally applied and internally induced electric and magnetic fields are incorporated in the model. An eigenvalue analysis of the governing system of nine nonlinear coupled differential equations has been performed. For certain combinations of material properties, it is possible to obtain complex conjugate eigenvalues indicating that the hyperbolic-elliptic system could become inherently oscillatory. Derivation of characteristic and non-reflecting open boundary conditions were also performed, thus completing the analytical part of the research effort. Numerical algorithm development and computer coding will be performed during the next fiscal year.

**STUDENTS FUNDED UNDER RESEARCH:**

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**TASK INITIATION:** 9/94  **EXPIRATION:** 9/96

**PROJECT IDENTIFICATION:** 962-21-08-24

**NASA CONTRACT NO.:** NCC8-56

**RESPONSIBLE CENTER:** MSFC

### BIBLIOGRAPHIC CITATIONS FOR FY 1995:

**Proceedings**


**Presentations**


Containerless Liquid Phase Processing of Ceramic Materials

Principal Investigator: Dr. Richard Weber

Co-Investigators: Nordine, P.C.

Task Objective:

This research uses the control of chemistry and nucleation achieved by containerless liquid-phase processing to study non-equilibrium phase formation and crystal growth. The work is intended to advance the basic understanding of the high temperature chemistry of hard, refractory oxide and boride ceramics. Borides are of fundamental interest; they are a unique class of compounds which form highly covalent, complex crystalline structures.

Ground-based containerless experiments will enable non-equilibrium phase formation phenomena to be identified. This will allow candidate materials for more detailed investigation to be selected. Subsequent low gravity experiments will provide the high degree of control over molten specimens required for detailed studies and analyses of the liquids as well as crystal growth kinetics and solid-liquid phase relationships.

Task Description:

High temperature liquid-phase processing is achieved by aero-acoustic and aerodynamic levitation in combination with continuous wave CO$_2$ laser beam heating. Levitated materials are viewed by optical pyrometers and video cameras. Materials are being examined by optical and scanning electron microscopy, X-ray diffraction, nuclear magnetic resonance, Raman spectroscopy, and laser fluorescence measurements.

The effects of melt chemistry, temperature and process variables, including gravity-driven convection on solidification kinetics, metastable phase formation, and epitaxial growth onto isostructural seed crystals from undercooled melts is being investigated.

Task Significance:

The work provides insights into subtle chemical and transport effects on the solidification of complex oxide and boride melts. The investigation explores the limits of ground-based methods and identifies systems which require the additional control of transport which may be possible in low gravity.

Progress During FY 1995:

The first year of this project is an investigation of liquid-phase processing and properties of non-metallic materials. The principal experimental technique is containerless melting using the aero-acoustic and aerodynamic levitation methods in combination with cw CO$_2$ laser beam heating and non-contact diagnostics. Materials selected for the investigation were oxides based on the aluminum oxide-silicon dioxide system. Research results have been/will be published in refereed journals and relevant citations are presented in this report. In addition, results and progress have been reported to NASA and presented at international conferences and workshops on materials and microgravity materials science. Principal achievements of the research were:

1. Determined the limits for liquid-liquid phase separation in binary alumino-silicate melts. Undercooled melts from ca. 30-60 % SiO$_2$ formed two immiscible liquids which separated due to their density differences.

2. Investigated solidification of melts $f(pO_2)$, solidification and phase separation were influenced by ambient oxygen pressure in equilibrium with the melts.

4. Conducted collaborative investigations.

5. Developed new synthesis technique for high purity oxides.

6. Performed preliminary low-gravity melting experiments in KC-135 in collaboration with Canadian research team.

Future directions of the research include (i) continued investigation of liquid-liquid phase separation effects, and (ii) measurements of liquid surface energies and viscosity as a function of composition and process variables including melt temperature, thermal history, ambient oxygen fugacity, and bulk liquid composition.

Papers in preparation will present detailed interpretation of the results.

STUDENTS FUNDED UNDER RESEARCH: TASK INITIATION: 5/92 EXPIRATION: 5/95
PROJECT IDENTIFICATION: 963-26-07-03
RESPONSIBLE CENTER: JPL

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals

Proceedings

Presentations
II. MSAD Program Tasks — Ground-based

BSO/BTO Identification of Gravity Related Effects on Crystal Growth, Segregation, and Defect Formation

Principal Investigator: Prof. August F. Witt
Massachusetts Institute of Technology (MIT)

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
Knowledge gained from space experiments and the related ground-based program is expected to advance the science base for crystal growth of oxides and thus to narrow the still existing gap between theory and experiment. More specifically, the proposed program provides an approach to the deconvolution of the effects of largely uncontrolled, complex processing variables on crystal growth and segregation, leading to the identification of growth conditions by which device specific property requirements in oxides can be approached. Such growth conditions are expected to be realizable in a modified Bridgman growth geometry, the subject of development in the ground-based support program.

Task Description:
The on-going research program on growth and characterization of BSO places focus on a class of materials (optical and opto-electronic) with theoretical properties that are outstanding, but which have so far failed to reach their potential primarily because of our inability to adequately control during growth their stoichiometry, incorporation of functional minority constituents, crystal defect formation and confinement related contamination. Theoretical considerations indicate that the majority of existing growth deficiencies, which are responsible for our inability to produce viable device structures, are directly or indirectly related to gravitational effects. Thus it appears that an assessment of the true potential of selenites, which exhibit outstanding piezoelectric properties and exceptionally light optical rotative activity in device application, can at this time best be made on material obtained from controlled growth experiments in a reduced gravity environment. Under such conditions convective interference, otherwise unavoidable, is projected to be substantially suppressed and defect structures resulting during growth are expected to approach equilibrium values. Magnetic melt stabilization, found effective in growth of semiconductors, is ineffective in oxide systems.

Task Significance:
The research to date has focused on (a) the development of BSO single crystal growth capability by the Czochralski technique, (b) the characterization of growth and defect formation in BSO, and (c) the development of a Bridgman-type growth configuration for BSO that permits enhanced heat transfer control under quantifiable thermal boundary conditions and which is functionally compatible with NASA-generated existing hardware. The establishment of quantifiable growth conditions and the in situ growth characterization capability by means of current induced interface demarcation are considered essential in efforts directed at assessing the potential of reduced gravity environment for crystal growth research and development since the customary empirical approach is considered prohibitive in space experimentation. The approach taken is in addition also considered a key element in efforts to establish advanced crystal growth capabilities on earth, a prerequisite for the meaningful assessment of the potential of selenites in device applications.

Progress During FY 1995:
Crystal Growth of BSO
The heat pipe-based Czochralski system which can provide for controlled ambient atmosphere and current induced growth interface demarcation has been used extensively to grow single crystals of BSO for the development of characterization techniques, analysis of growth and segregation behavior in conventional operation and to provide for seeds and bulk material for the development of an advanced Bridgman growth facility.
Characterization
Existing facilities for optical transmission microscopy and for related computational image processing and analysis were upgraded.

To determine the basic growth characteristics of BSO, Ga-doped as well as undoped crystals, grown in this laboratory as well as commercial crystals were analyzed in detail. It was found that (1) rotational striations are absent in commercial material as well as in crystals grown with the heat pipe-based Cz system; (2) non-rotational striations, highly periodic in nature and exhibiting two distinctly different frequencies, are present in both core and off-core regions; the intensity of striations is greatest in the crystal periphery and most pronounced in facet growth regions; (3) the frequency of striations in the crystals is a weak function of the aspect ratio of the charge, independent of the rate of seed rotation and not noticeably dependent on the rate of crystal pulling; (4) the primary macroscopic crystal deficiencies are gaseous inclusions -- their appearance is a function of both the rate of crystal rotation and the rate of crystal pulling; (5) generation of dislocations is found to be predominantly related to inclusions, to gas bubbles and to precipitates; (6) dislocations have been made "visible" through decoration by annealing in a reducing atmosphere; and, (7) current induced interface demarcation has been successful - its formation is more consistent with the action of Joule heating than with that of a Peltier effect, generally assumed.

Design of an advanced Bridgman growth facility
A heat pipe-based Bridgman facility has been designed and constructed. The system is equipped for growth interface demarcation and can provide growth rate as well as thermal gradient stabilization. It is currently being subjected to extensive testing in efforts to optimize its performance characteristics and the predictability of growth conditions based on limited data input, a prerequisite for the effective application of computational growth modeling.

SUMMARY
The ground-based research phase on growth and segregation of BSO, considered essential for the conduct of related space experiments, is considered as completed. The basis for informative growth experiments in a reduced gravity environment has been established. In the course of this work, fundamental deficiencies in commercial BSO have been identified and approaches for their elimination and/or control have been developed. It is anticipated that space experiments to be conducted in the follow-up phase will provide experimental data which will establish the potential of materials with induced refractivity for device applications.

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals

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Task Initiation: 1/93 Expiration: 1/96

Project Identification: 962-21-08-19

NASA Contract No.: NAG8-949

Responsible Center: MSFC

Printed: 28 FEB 1996
II. MSAD Program Tasks — Advanced

Free Float Trajectory Management ATD

Principal Investigator: Mr. A. P. Allan
University of Delaware

Co-Investigators:
O'Donoghue, D.
Nyma, Inc.

Task Objective:
Many researchers perform experiments that are sensitive to the effects of gravity aboard aircraft flying low gravity trajectories. The aircraft is flown in a ballistic trajectory during which a period of free-fall is attained, providing the desired low gravity environment. These trajectories are characterized by an initial trajectory entry phase which can be very dynamic, followed by the stabilized low-g period and finally, an exit phase. Ideally, the entry phase should be minimized to allow the longest possible low-g time.

The low-g environment experienced by an experiment attached to the aircraft is degraded by vibrations from the aircraft as well as directly imposed disturbances from acoustics, air flow or that are self-induced. However, an experiment package allowed to free-float during the stabilized low-g phase will only be affected by the direct disturbances. The drawback of free-floating an experiment is that the package typically contacts the walls of the aircraft after only a few seconds due to the initial velocity of the package at release and the rotation of the aircraft during the parabolic trajectory. Longer free-float times are achievable, but are not predictably reproducible.

The objective of this work is to develop the technology for an extended, consistently reproducible acceleration environment during the stabilized low-g phase of the trajectory, specifically for free-float packages. The goal is to extend the free-float time to 10 seconds or longer and obtain stable accelerations of 0.001 g or lower in a consistent, reproducible manner.

Task Description:
Improving the low-g environment for free-float packages requires the optimal control of the aircraft trajectory and the release of the package. The control of the trajectory is dependent on the limitations of the specific aircraft used and the feedback the pilots use to maneuver through the trajectory. The definition of the trajectory in terms of air speed and pitch angle when entering the trajectory, the acceleration level during pull-up, and the air speed and pitch angle when exiting the trajectory is the first step in maximizing the overall trajectory time and, in particular, the stable low-g time.

To control the aircraft maneuver during the low-g phase, specific commands for pitch, roll and throttle must be provided to the pilots. This requires parameter identification of the aircraft and the development of a control law to develop the appropriate commands to maintain the stable low-g environment for the longest time possible. The control law will be based on the states of the aircraft and the relative position, velocity and acceleration of the experiment package. For a free-float package, the motion detection will be accomplished non-intrusively.

The controlled release of the experiment package once the stable low-g environment has been established is the next step in achieving the maximum duration free-float time. A technique or mechanism will be developed to optimally release the package during the low-g phase.

The test bed for the developed technology will be the NASA LeRC DC-9 aircraft.

Task Significance:
Numerous researchers utilize low-g aircraft trajectories to perform scientific investigations in the fields of combustion, fluid physics and materials processing. For some, an extended, high-quality low-g free-float environment, as is the goal on this ATD, would be sufficient for most or all of their testing needs without the need for more expensive, time-consuming suborbital or orbital carriers.
Although the developed system will only be applicable to a specific aircraft, the NASA LeRC DC-9 in this case, the technology to develop the system will be applicable to other aircraft.

Progress During FY 1995:

This ATD project was initiated in fiscal year 1995 and is planned to continue through fiscal year 1997. To date, the NASA LeRC DC-9 trajectory profile was defined through flight testing. Development of two data acquisition systems, a rack-mounted system and a free-float system, has been completed. The rack-mounted system will record the accelerations of the aircraft and the free-float system will record the accelerations of the package during free-float. These systems will be used to measure the performance of the aircraft and free-float environment for the duration of the program.

Initial flight testing will be performed to obtain baseline data on the performance of the aircraft and quality of the trajectories. Aircraft parameter identification studies will begin during the last quarter of 1995. Development of the pilot command guidance system will start in 1996, followed by flight testing on the DC-9. Concept development for a free-float package release system will begin in 1996.
II. MSAD Program Tasks — Advanced

Stereo Imaging Velocimetry

Principal Investigator: Dr. Mark Bethea

NASA Lewis Research Center (LeRC)

Co-Investigators:

No Co-I's Assigned to this Task

Task Objective:

The objectives of this project are to develop a stereo imaging velocimeter that will:

1. Measure quantitatively and qualitatively velocities up to 10.0 cm/sec with an accuracy of 2.0% of full-field for 150-micron seed particles in a 2.0-inch field of view.

2. Have streamlined data processing which processes 100 time steps of consecutive stereo images to obtain 3D velocity fields in less than ten minutes.

3. Be able to track at least 100 particle pairs per frame.

4. Require minimal apriori assumptions about the flow.

5. Initiate tracking and matching out automatically.

Task Description:

The approach to successful implementation is to develop robust and efficient 3D camera calibration, edge finding/centroid determination, overlap decomposition, particle tracking, and stereo matching algorithms that will be used in the Stereo Imaging Velocimetry system. These five tasks are the basis for the velocimeter and will determine its final processing speed and accuracy. We will then test the prototype for accuracy on particles with known trajectories. A user interface for both the front-end and post-processing will be created. The velocimeter will be tested on real fluids experiments. This testbed experiment will be done with a water tunnel experiment that is currently under development. Using the water tunnel experiment, we can set the flow rate and compare the results with the SIV system results.

Task Significance:

Stereo Imaging Velocimetry will permit the collection of quantitative and qualitative, three-dimensional flow data from any optically transparent fluid which can be seeded with tracer particles. This includes such diverse experiments as the study of multiphase flows, bubble nucleation and migration, pool combustion, non-contact measurements, and crystal growth -- all of which are part of NASA's Microgravity science program.

Progress During FY 1995:

A neural network approach to particle tracking has been invented, coded, and tested. The results show a substantial improvement over our existing particle tracking scheme. This work is significant in that we use a globally optimum neural network instead of a locally optimum one. This increases our particle tracking yield by as much as 10%.

A 3D camera calibration technique has been developed and tested for the SIV system. We are now able to take two 2D calibration (orthogonal views) and extract 3D information to produce a robust 3D camera calibration routine capable of calibrating our fluid volume to within 0.17% of full-field.

The Neural Network implementation of the overlapping particles reported in FY 93 has been combined with our edge finding-centroid processing algorithm to form one robust algorithm.

Printed: 28 FEB 1996
II. MSAD Program Tasks — Advanced

The 3D camera calibration technique, overlap decomposition with edge-centroid determination, along with particle tracking and stereo matching are currently being combined in one SIV algorithm. This will produce our SIV software system package and accuracy and speed will be analyzed.

All 1995 activities have been concentrated on identifying potential users of SIV technology. We have space act agreements with two local companies and two possible flight projects.

We are also concentrating on a user-friendly interface for users of the SIV system.

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PROJECT IDENTIFICATION: 963-70-OC-00
RESPONSIBLE CENTER: LeRC

Printed: 28 FEB 1996
II. MSAD Program Tasks — Advanced

Real-Time X-ray Microscopy for Solidification Processing

Principal Investigator: Dr. Peter A. Curreri NASA Marshall Space Flight Center (MSFC)

Co-Investigators:
Kaukler, W. University of Alabama, Huntsville (UAH)

Task Objective:
The objective of this ATD consists in the development of an X-ray Transmission Microscope (XTM) for the in situ and real-time observation of interfacial processes in metallic systems during freezing or solid-solid transformation. The XTM should have the following capabilities:

1. Provide a resolution for specimen features of 10-100 μm;
2. At solidification rates of 0.1 to 20 μm/sec;
3. Temperatures up to 1100°C with temperature gradients up to 50 C/cm;
4. With contrast sensitivities sufficient to detect 2-5% difference in absorptance;
5. Offer 1, 2, and 4 in exposure times of a few seconds; and,
6. Permit recording of stereo pairs for depth information.

Task Description:
The purpose of this ATD is the development of a high resolution x-ray either during freezing or solid-solid transformations. We will use the X-ray Transmission Microscope (XTM) to view, in situ and in real-time, interfacial processes in metallic systems. XTM will operate in the hard x-ray range (10 to 100 KeV) and achieve magnification through projection.

Task Significance:
Physical processes which occur at, or near, interphase boundaries during solidification, or other phase transformations, play a major role in the determination of many of the technologically important properties of solids. To date, interfacial morphologies and particle-interface interactions in the respective metallic, optically opaque systems have been deduced from post-process metallographic analyses of specimens. Thus, little information is obtained about the detailed dynamics of the processes. These investigations have been considerably augmented by real-time observations of transparent materials; yet, since some of the interfacial and transport properties of these materials differ greatly from those of metals and semiconductors the results are not necessarily representative of these opaque systems.

Progress During FY 1995:
The main efforts for FY95 were the construction of the x-ray furnace, evaluation and selection of the technology for the advanced x-ray camera, the establishment of criteria for and selection of peripheral equipment, solidification experiments with Al alloys and Al-Zirconia composites in the prototype furnace, evaluation of the specimens for the "Particle Pushing at Interfaces" flight definition experiment (Dr. Stefanescu, UA, P.I.), measurements of emitted spectra from x-ray source, and testing of higher resolution x-ray targets.

Many of the goals of the Project objectives have already been met. Cellular structures have been observed during solidification of Al-Ag alloys with dimensions of 50 micrometers width or less. Imaging of solidifying interface with growth rates of 0.2 μm/sec to 34 μm/sec has been demonstrated. A 3.5% contrast sensitivity was achieved using the high definition x-ray source and detection system with large features.

Four technologies, all based on CCD's, were evaluated for the advanced x-ray camera/converter. A cooled CCD with a fiber optic faceplate and a CsI phosphor material 1242-by-1152 pixels and 16-bit gray scale resolution was found to provide the optimum performance.

Printed: 28 FEB 1996
An advanced x-ray transparent furnace has been designed and is being constructed. The prototype furnace enables a maximum real-time magnification of a solidifying sample of about 50 X. The advanced furnace is designed to allow a magnification of 200 X, which could allow real-time imaging of growth of fine microstructural features (such as fibers in eutectic and monotectics) in these opaque metallic alloys for the first time.

**STUDENTS FUNDED UNDER RESEARCH:**
**TASK INITIATION: 10/94**
**EXPIRATION: 10/97**
**PROJECT IDENTIFICATION: 963-70-04**
**RESPONSIBLE CENTER: MSFC**

**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

**Proceedings**

**Presentations**
II. MSAD Program Tasks — Advanced

Advanced Heat Pipe Technology for Furnace Element Design

Principal Investigator: Dr. Donald C. Gillies
NASA Marshall Space Flight Center (MSFC)

Co-Investigators:
Palosz, W.
USRA/NASA Marshall Space Flight Center (MSFC)

Task Objective:
The principal objectives for this study are to improve the capabilities of heat pipe technology for use as isothermal liners, by (1) fabricating liners to operate at up to 1500°C, (2) to determine the feasibility and establish the protocol for the incorporation of liquid metal heat pipes as furnace liners in a man tended environment in space, and (3) to develop a furnace with no moving parts which can solidify or cool materials with a high degree of control. The performance of this device, the Moving Gradient Heat Pipe Furnace (MGHPF), will be extensively characterized.

The rest of the first part should be transferred to the Task Description - next section. I suggest that the sentence starting “In conjunction with . . .” should end with the phrase “. . . processed in the MGHPF, . . .” The material not used in FY95 Task Objective would then go in, and the phrase about the “possibility of fabricating a high temperature version of the MGHPF will be explored” could go in at the end.

Task Description:
A major consideration of the high-temperature heat pipe is the selection of materials for its successful fabrication. The first year's effort will focus on reviewing possible materials and examining their known history in conventional heat pipes. The isothermal furnace liner has a more complex geometry than the heat pipe traditionally used for heat transfer in that there is a central bore in which the load is processed. This puts considerably more strain on the welding of the end caps to the main body. With the anticipated high temperature differential between the inner and outer surfaces of the pipe, the potential for the welds to crack is much higher. Suitable test articles will be fabricated and extensive testing will be initiated. The aim is to produce heat pipes capable of operating within 0.75-BOC of the required temperature over long periods of time (a matter of days) and withstanding many cycles between room and the operating temperatures. These tests will culminate in the growing of crystals in a new facility. In conjunction with the high-temperature heat pipe effort, materials will be processed in the MGHPF, and the possibility of fabricating a high-temperature version of the MGHPF will be explored.

Task Significance:
Each of the instruments presented here will impact materials science. The MGHPF, primarily aimed at flight experiments, simplifies a furnace facility by dispensing with the need for a translation device for moving the sample during processing. The equipment also operate with a much simpler heating and control system by being able to operate with only one heating element and controller system. Other benefits will include saving space, positioning the sample accurately in the furnace, and maintaining the position of the sample during processing.

While specifically aimed at manned flight applications, ground-based benefits are equally important. Since a high temperature heat pipe of the specifications described does not exist for crystal growth, it will immediately produce dividends for individuals growing crystals and conducting other materials science work in the temperature range 1100-BOC - 1500-BOC. In flight experiments, where the investment per mission is so high, it is essential that thermal conditions for experiments be optimized to maximize science returns. The heat pipe is a major step toward this goal.

Progress During FY 1995:
This project started in FY 1995 and was planned as a four-year effort, but a later decision trimmed the effort back to only two years. During the first six months progress was made in identifying the most cost-effective materials and
II. MSAD Program Tasks — Advanced

design for fabricating the high-temperature heat pipe. The first three heat pipes were designed and fabrication initiated in FY 1995. Installation and testing is planned for FY1996. Meanwhile, dialog to establish the protocol for flying liquid metal heat pipes was also begun.

The Moving Gradient Heat Pipe Furnace (MGHPF) was installed at the Marshall Space Flight Center (MSFC) and initial thermal testing begun. It was found that interface translation rates of 1 through 50 mm/hr could readily be achieved and controlled. Additionally, during the first year a crystal of indium antimony (InSb) was grown. Other materials, including seeded germanium, were grown to demonstrate the capability of the equipment. Additional instrumentation, such as a current pulsing system, will be added during FY 1996.

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**BIBLIOGRAPHIC CITATIONS FOR FY 1995:**

Presentations
II. MSAD Program Tasks — Advanced Technology Development

Microgravity Combustion Diagnostics

Principal Investigator: Dr. Paul S. Greenberg  
NASA Lewis Research Center (LeRC)

Co-Investigators:
Griffin, Dr. D.W.  
Vander Wal, Dr. R.L.  
Weiland, Dr. K.J.  
Piltch, Dr. N.D.  

NASA Lewis Research Center (LeRC)  
NYMA, Inc.  
NASA Lewis Research Center (LeRC)  
NASA Lewis Research Center (LeRC)

Task Objective:
Currently available diagnostic instrumentation for achieving these objectives has been extremely limited, consisting primarily of conventional film-based imaging systems and intrusive temperature and velocity probes, such as thermocouples and hot wire anemometers. This situation has arisen primarily because of the unique and severe operational constraints which are inherent in the conduct of reduced-gravity experimentation. It is the recognition of this pressing need to provide diagnostic systems of greater sophistication that has motivated the existence of this particular development program.

Task Description:
For a variety of reasons, predominant emphasis has been placed on the development of optical diagnostic techniques. Principal among these is the relative fragility of the physics and chemistry of reduced-gravity systems relative to their 1-g counterparts. The action of buoyancy-induced convection is vigorous when compared with the dominant mechanisms associated with reduced-gravity phenomena, such as surface tension and thermal and concentration driven diffusion processes. The essentially nonperturbative nature of optical measurement techniques is therefore extremely appropriate in this context.

Optical measurement techniques are, in general, well-suited to the acquisition of multidimensional data fields (e.g., two- and three-dimensional imaging). This is an important consideration in the present state of understanding of microgravity science, since a clearer understanding of basic phenomenology, including the verification of fundamental length and time scales and dominating physical mechanisms in still being developed.

Task Significance:
The success in achieving a significant scientific return from existing and proposed microgravity fluid physics and combustion science experiments depends substantially on the availability of diagnostic systems for the collection of the required scientific data.

Progress During FY 1995:
In the area of full-field infrared emission spectroscopy, calibration of the IR sensitive staring array camera has been conducted. This was accomplished through the use of a blackbody radiation source and several infrared bandpass filters. An end-to-end calibration is required in this case because of the varying response of the detector array and the transmission characteristics of the lens and bandpass filters. All of these elements exhibit behavior that is wavelength dependent; the detector response may exhibit nonlinearities with respect to absolute intensity as well. The response of the detector array has been observed to be nearly proportional to blackbody intensity when narrow band filters are employed. As a first step in exploiting this calibration, images of radiating thin filament fibers suspended in jet flames have been obtained. The resulting data will be compared to thermocouple measurements of the hot gases above the flame that were obtained simultaneously.

During this year, a blackbody source needed for camera calibration was recalibrated. This is necessary to characterize the camera response for quantitative measurements. Also, an infrared monochromator with lead salt detector was used...
configured to acquire wavelength resolved emission scans. Future plans include the acquisition of bandpass filtered images, and multi-point temperature measurement using thin-filament pyrometry.

A preliminary literature search was made investigating the inverse problem wherein infrared emission measurements are inverted to provide temperatures and concentrations of selected species. The present search focused on references comparing modelling efforts and experimental data for an experimentally tractable (soot-free, relatively simple chemistry, straightforward geometry) combustion system. Published infrared spectra of some laboratory flames, such as a Bunsen-type natural gas flame, have also been found.

In addition, a new Phase I SBIR work entitled "Analysis Tools for Spectrally-Resolved and Broad-band Infrared Imaging Data" was successfully proposed by Aerodyne, Inc., with much of the proposal being based on discussions with ATD personnel regarding the data analysis requirements for both bandpass filtered and spectrally resolved infrared imaging data obtained by several microgravity combustion experiments. The proposed work involves a survey of available optimizer routines, an evaluation of their adaptability to the Aerodyne Radiation Code, and the fitting of synthetic data with and without synthetic noise added in preparation for the fitting of real data. It is anticipated that the contractor will propose for a Phase II SBIR contract. The results of the competition will be announced during the upcoming year.

Independent of but complementary to this ATD, the Phase II SBIR contract with SSG, Inc. for the development and delivery of an infrared imaging spectrometer capable of supplying spatially and spectrally resolved images made much progress and is nearing its end. All requirements for the instrument have been finalized and the hardware components are complete. The design of the system is such that the system can be accommodated in an aircraft rack for reduced-gravity experimentation and the components have been ruggedized as much as possible. An aircraft rack is under construction. Integration of the hardware and software is being done now, after replacement of the flawed Pentium processor in the computer. The PI was invited to present a talk on the contract results at the Third International Microgravity Combustion Workshop. All hardware and software systems have been delivered to LeRC and are presently undergoing acceptance testing in the laboratory.

Efforts supporting two-dimensional species and temperature measurements were initiated with the completion of the procurement process for the titanium:sapphire laser. The laser, built by Continuum, Inc., was installed into a new laboratory room in the Space Experiments Laboratory. Through frequency doubling, tripling, and mixing of the titanium:sapphire fundamental output, light in the blue and ultraviolet has been successfully generated. These lines cover the wavelength regions of 431 nanometers and 308 nanometers, providing the ability to perform laser-induced fluorescence measurements of the CH and OH radicals, respectively. This laser system is currently being characterized as to its operational capabilities. Optics and other support equipment (burners, chamber, etc.) to perform Rayleigh scattering, laser-induced fluorescence, and other optical diagnostics have been obtained and are presently being assembled.

Characterization of this new device began by looking at water, which is optically active throughout the fundamental wavelengths of the laser and allowed familiarization of the laser system without the worry of frequency conversion. A photoacoustic cell and associated electronics were constructed. Excitation of various absorption bands of water at room temperature can be detected, thus serving as a frequency marker to reference the laser tuning process. Synchronization of the laser pulses, data collection systems, and wavelength tuning has also been implemented.

Next, the frequency doubling capabilities of the laser were characterized. Several millijoules of light could be produced at 390 nm, which is in the range of one of the CH radical electronic transition. The laser was set up to automatically scan the frequency conversion crystal angle as the wavelength is scanned. The scan rate for auto-tracking was determined to be at least 0.1 nm/sec. The tuning range of the laser when optimized at a particular wavelength is greater than 60 nm, although the auto-tracking alignment must be slightly realigned for good tracking at roughly 20 nm intervals.

A Bunsen-type methane/air flame was set up and a point, laser-induced fluorescence signal of the CH radical present in the inner cone of the flame was obtained with the use of a monochromator and photomultiplier tube detection.
During the past year, the solid-state laser system was relocated to a new laboratory to minimize conflicts with the extensive soot production from the laser-induced incandescence (LII) ATD which lead to the damage of several optical components. The laser system was characterized at 308 nm, which allows the detection of the OH radical, one of the most important flame front markers. The pulse energy as a function of wavelength or the tuning curve, and the shot-to-shot stability were measured in this wavelength range. The pulse energy and stability are both within specifications and sufficiently good for OH detection experiments. Writing of a computer program for the control of wavelength and multi-channel data acquisition has begun.

In preparation for imaging laser-induced fluorescence experiments, a demonstration of the synchronization of the pulsed laser, gated image intensifier camera, and digital frame-store capabilities was conducted. The first demonstration was for the fluorescence imaging of an acetone-seed, non-reacting jet. The image was obtained at an excitation wavelength of 266 nm using the new laser for the LII/ATD. The synchronization set-up is identical for a combusting system, but is easier to accomplish for a system with essentially zero background, as there is no flame. The jet had a Reynolds number of roughly 2000 based on the nitrogen flow.

Also in support of the laser-induced fluorescence experiments, several OH and CH chemiluminescence images were obtained for burning methanol and decane fiber-supported droplets. These experiments were performed at low pressure in an attempt to simulate low-gravity conditions and obtain an approximate signal level of the OH and CH chemiluminescence intensity to guide potential reduced-gravity measurements of the same. The methanol images reveal significant emission from OH A-state and CH A-state electronically excited radicals. The CH emission may be used as an alternative to the OH emission in determining the position of the flame front. Since emission is seen from CH A-state radicals, this hints that other reactions may produced CH in the C-state, whose emission overlaps that from the OH radical. Detailed high resolution spectroscopic measurements are needed to discern the relative contribution of the CH emission to the light collected near 308 nm. Previous modelling by Prof. Dryer does not predict the formation of CH in the combustion of methanol droplets. These observations provide an alternative method of spectroscopically determining the flame front position during the burning droplet lifetime, provide input for the theoretical modelling of the combustion chemical mechanism for methanol droplets, and provide a caution against the assumption that light emission centered at 308 nm is due solely to OH radical emission. The decane droplet chemiluminescence images provide a point of comparison for the methanol droplet.

Future work in this area will be to conduct OH point laser-induced fluorescence experiments. The successful demonstration of the synchronization of the laser, camera, and computer digitizer permits the extension of the laser-induced fluorescence measurements to line and imaging configurations. Rayleigh scattering experiment will also be conducted.

During this period of performance, a related effort was undertaken to determine oxygen spatial extent and concentrations profiles in flames, using non-intrusive optical techniques. Absorption in the ultraviolet Schumann-Runge bands was selected based on sensitivity, compactness of the apparatus, and ability to measure hot oxygen without interference from ambient oxygen. In addition, the same technique would similarly be able to measure ambient oxygen by suitable choice of wavelength.

The absorption spectra for these bands were modeled with a simulation program obtained from Princeton University, over a temperature range from ambient to 2000 K. Several groups of transitions were identified that promised high sensitivity and minimum interference by ambient oxygen. Since several excited vibrational levels are populated and can be probed in this region, temperature can be determined from these measurements. The simulated spectra at 300 K and 1500 K show spectral regions, such as that near 195 nm, where hot oxygen absorbs but room temperature oxygen does not.

Also considered are the Franck-Condon factors as a function of wavelength and vibrational quantum number. The Franck-Condon factor is the vibrational contribution to total transition probability. This figure shows that there is a four order of magnitude increase from \( v' = 0 \) to \( v'' = 4 \). The product of Franck-Condon factor and population of that
state is nearly constant within a factor of ten for states from \( v'' = 0 \) to \( v'' = 4 \) at flame temperatures. Therefore vibrationally excited states can be detected at nearly the same sensitivity as \( v'' = 0 \), while also allowing temperature determination from the spectra. The vibrationally excited states have the additional advantage of being shifted toward longer wavelengths where transmission through optical elements will be improved.

Measurements are planned in line-of-sight absorption, point fluorescence, and planar imaging. A lightweight, compact, low power xenon arc lamp has been purchased with a repetition rate of 300 Hz and sufficient intensity in the sub-200 nm region for these measurements. UV optics (lenses, filters, and a monochromator grating) have also been obtained. Initial absorption measurements are in progress.

Exciplex thermometry experiments are being performed utilizing a small pulsed nitrogen laser operating at 337 nanometers. The objective is to utilize this method to measure the temperature of a vaporizing droplet, and extend the technique to a burning droplet. Exciplex thermometry utilizes the ratio of fluorescent intensities of an excited state monomer and exciplex compound (formed through the interaction of an excited state monomer and ground state partner). By referencing this ratio of fluorescence intensities to calibration measurements in which the intensity ratio is measured at known temperatures, the temperature can thus be determined. This technique has been applied to measure the volume average temperature of a fiber suspended droplet in an inert atmosphere in response to heating by a hot wire coil surrounding the droplet. The system in this case is hexadecane doped with PYPYP. Application of exciplex thermometry to burning droplets remains a challenge as the fluorescence from the monomer and exciplex are quenched at different rates in the presence of oxygen. Current efforts are aimed at addressing this issue.

Several hardware modifications were accomplished to enable these investigations. These include: a) a refurbishment of the existing droplet combustion chamber to serve as a glove-box for performing experiments in an oxygen-free environment, b) the design and fabrication of a glass vacuum system for degassing solutions of dissolved oxygen, (the capabilities of this system have since been adopted for use by the DCE program) c) the design and fabrication of a new calibration cell, d) dual fluorescence detectors for measuring the fluorescence of the monomer and exciplex compounds simultaneously, independent of the experimental geometry, e) an improved hot-wire ignition mechanism, and f) the identification and procurement of a new potential potential exciplex compounds based on a survey of the open literature.

Several new exciplex compounds and fuel combinations were tested for their temperature dependence and fluorescence signal levels. The two most promising systems were identified as PYDMAP/Decane and PYPYP/Decane. The PYDMAP was previously unavailable and neither exciplex compound had been tested in combination with the fuel decane.

Most significantly, these compounds are intramolecular compounds which form an exciplex compound through an intramolecular rearrangement. Since the two 'parts' of the molecule are in relatively close proximity to each other, far lower concentrations can be used. It has been routinely observed that intermolecular compounds require significantly higher concentrations for an exciplex compound to form. The resulting drawback is that with such high concentrations, not only is the burning rate constant of the droplet altered (a significant perturbation), but the droplet is frequently difficult, if not impossible to ignite. Furthermore, the differences in vapor pressure between the fuel and dopant dictate that the relative concentration of the dopant varies throughout the burning process. Establishing experimental procedures to produce repeatable and robust calibrations is an extremely daunting task. The overall lower concentrations required by the use of intramolecular compounds tends to reduce this undesirable effect.

The fluorescence of an exciplex compound and monomer is also sensitive to the presence of oxygen. While adequate signal levels are attainable in the presence of oxygen, its quenching effect on the fluorescence differs for both the monomer and exciplex compound. Calibration experiments are performed steady-state, where in order to bring the solution temperature to a predetermined level, a period of several minutes is required. In contrast, the ignition of a fuel droplet occurs within a few seconds. As the temperature of the droplet varies both temporally and spatially (which is what is desirable to measure experimentally) during the ignition and burning stages, the oxygen...
concentration in the solution will vary as well, owing to the temperature dependence of the dissolved oxygen concentration. Since the fluorescence intensity of exciplex to monomer is a strong function of both temperature and oxygen concentration, the inferred temperatures based on fluorescence intensity ratios will not necessarily accurately reflect the true temperature of the droplet.

It does not appear feasible to begin with an oxygen free fuel droplet (for a single fiber-supported droplet) and accomplish ignition in air before significant changes in the fluorescence intensities occur due to oxygen dissolving in the solution. Given the rate of diffusion of oxygen to the droplet, it is observed that the dissolving of oxygen into the solution occurs almost instantaneously.

The temporal and spatial variation in the oxygen concentration essentially renders a suitable calibration procedure exceedingly difficult, if not impossible. Thus our approach involves a comparison of droplet temperatures prior to ignition for an oxygen-free fiber-supported decane droplet (doped with either of the two indicated exciplex forming compounds) residing in an oxygen free environment, and an identical aerated decane droplet residing in air. The calibration for each experiment will be performed in the appropriate oxygen-free or air environments. Comparison of the temperatures of the decane droplet during the ignition process for both sets of experimental conditions (with calculated temperatures being based on the appropriate calibration experiment) will be made for both experiments to empirically determine how much error may be incurred in an actual experiment performed in air referenced to a calibration procedure performed under similar conditions. If similar temperatures are indicated, then the technique should also be applicable to burning droplets. At a minimum, the technique is clearly applicable to studying droplet temperature prior to ignition, a situation of expressed concern to several MSAD funded investigators.

During the course of these experiments the nitrogen laser failed, preventing a direct comparison of the two sets of experimental conditions. It was possible, however, to perform the calibration experiments in air. Using the resulting functional fit describing the calibration curve, the experimentally determined fluorescence intensity ratio of a fiber-supported oxygen-free decane droplet in an oxygen-free environment was used as an input. The ignition point is based on the observed time to ignition for an identical droplet in an air environment. Rough measurements have indicated that the very low concentrations of these intramolecular exciplex compounds do not change the burning characteristics of the host fuel. Additionally, the ratio of thermal conductivity of air to pure nitrogen is nearly unity, thus validating the determination of the ignition point in the oxygen-free environment based on measurements performed in air. Most importantly, the inferred radially averaged droplet temperature is significantly lower (~90 Celsius) at the point of ignition than the boiling point of decane (~175 Celsius). Preliminary results from the PYDMAP exciplex compound in decane yield similar results. Thus our initial conclusion indicates that hot-wire ignition does not bring the bulk droplet temperature near the fuel boiling point. The accuracy of these results however, will require a comparison to the oxygen-free experiments as previously discussed.

Point-wise and imaging velocity measurements are also in progress. Addressing the former, compact, solid-state laser doppler velocimeter modules have been obtained on loan to supplement the module being completed under contract. This module is implemented for coaxial backscatter measurements, utilizing common optics for simultaneous transmission and detection. This simplifies the experimental geometry, and eliminates the requirement for precise alignment of separate transmission and collection sample volumes. Acceptance tests of the optics module were performed during September of 1993 at EG&G's facility in Montreal. The results of these tests indicate that the unit performed as per specification. A comparative basis for the resulting performance was afforded by the availability of a pair of similar units presently in use in our laboratory. These units were loaned from the David Taylor Institute of the Naval Research Laboratory. Both the EG&G and NRL units employ diode laser sources operating at 780 nanometers, delivering approximately 15 milliwatts at the sample volume. Both units also employ DC coupled, avalanche photodiodes operating in the sub-geiger module (i.e. analog detection as opposed to photon counting). For comparable optical power present at the sample volume, the EG&G module affords an improved detection sensitivity by roughly a factor of four. A two-fold improvement is evident directly from the indicated data, whereas the additional factor of two results from F# considerations; both modules employ a collection aperture of 50 millimeters, but the EG&G module has a working distance which is 1.4 times as long. Also visible in the data is the considerable improvement in the spatial definition afforded by the EG&G module. The radial profile of the sample volume is very close to the predicted value of 100 microns, and much more closely.
II. MSAD Program Tasks — Advanced Technology Development

resembles the $1/e^2$ profile associated with the source. The NRL unit, in contrast, has inferior beam shaping optics, resulting in a series of unwanted "wings" which contribute to the overall scattered signal.

Upon receipt of the EG&G optics module at LeRC these tests were repeated. Unfortunately, two anomalies in the performance of the system were observed which were not seen previously. The first corresponds to a misalignment between the transmitted and detected sample volumes. Data was collected to indicate the output of the avalanche photodiode detector as a partially reflective target was translated along the optical axis. It is seen from the resulting plot that the detection sensitivity peaks approximately 20 millimeters behind the sample volume as defined by the transmitter. Because the detection optics operate at a conjugate ratio on the order of 10:1, this is indicative of a misalignment of the detector by roughly two millimeters. This distance was later revealed to correspond to a shift in the distance between the entrance window and sensor surface that had been altered in the final version of the detector. This change had evidently not been indicated in the final configuration drawings, and hence had become inadvertently misaligned in the final cleaning, purging, and sealing of the module which occurred prior to shipment. The second anomaly relates to 50 KHz bleed-through from the detector high voltage power supply, and was traced to a leaky by-pass capacitor. The module has subsequently been returned to EG&G, where these problems were addressed.

When the module was received from EG&G, these same tests were revisited. The results were favorable, indicating that the identified problems had been adequately rectified. A number of additional tests were then conducted, serving to both validate the module's performance, and as a basis for further comparison with the existing NRL units. The actively cooled APD utilized by EG&G affords a net increase in detection sensitivity of eight fold. A similar increase in signal to noise ratio (SNR) is not achieved, however, due to the larger noise floor attributable to the initial design goal of incorporating the high voltage generation circuitry internal to the module. Factoring in this consideration provides an improvement in SNR of roughly 3:1. This improvement affords the use of smaller seed particles, and offers the possibility of confocal masking to improve the axial spatial resolution. Also evident is an improvement in sample volume geometry. The anamorphic prisms effectively circularize the output, providing a $1/e^2$ diameter that corresponds well with the design specification of 150 microns. This is not to say, however, that true diffraction limited performance has been achieved. A circular stop preceding the beam splitter does result in some observable diffraction at the sample volume; this is manifest by the occurrence of modulation fringes external to the nominal $1/e^2$ envelope. Scattered signals resulting from particles traversing these regions are normally rejected by frequency domain filtering. The modulation in regions external to the sample volume resulting from residual diffraction effects reduces the effectiveness of this filtering process. The increase in detection sensitivity, however, affords the possibility of enhanced axial rejection through confocal masking. Because confocal masking is achieved through the use of a central obscuration, a loss in effective collection efficiency results.

The difference in detection sensitivity also influences the minimum resolvable scattering cross section (i.e. particle diameter for a specified seed material) that is required. Velocity field data from a droplet seeded jet was obtained from both the TSI and EG&G optics modules. The droplet seeder used for these measurements produces a broad size distribution, particularly in the range of 0.5 micron and below, and thus provides a useful demonstration of this specific point. In the more laminar core flow, the impact of the droplet size distribution is diminished regarding the ability to accurately follow the flow. At larger radii, the ability to track increasing levels of turbulence intensity becomes biased towards smaller droplet diameters. The relative decrease in detection sensitivity afforded by the non-cooled APD begins to underpredict the level of turbulence intensity, and biases the estimate of the mean towards lower velocities. This is also reflected in the corresponding data rates, wherein the more sensitive APD is seen to more adequately resolve a larger percentage of the net droplet flux. The more sensitive detector provides velocity estimates over a broader range of SNR, produces higher overall data rates, and a broader velocity probability density function.

Also acquired during this period of performance is a novel LDV signal processor. This processor belongs to a relatively new generation referred to as burst-resolved, and implements the calculation of complete frequency spectra at a maximum rate of ten thousand spectra per second. This particular device possess several advantageous features useful for the conduct of reduced gravity experiments, most notably its configuration as a pair of stand-alone circuit cards which plug into the bus of a conventional PC. Also of significance is the ability to operate as a digital
II. MSAD Program Tasks — Advanced Technology Development

The pulsed Nd:YAG laser and video was successfully demonstrated, resulting in the capability for flow analysis. During this last reporting period, the synchronization of the video system and laser must be properly configured so as to be compatible with operation in either the NASA Learjet or KC-135 aircraft.

In the area of Particle Image Velocimetry, a specially configured, pulsed Nd:YAG laser was received. This laser was designed to emit a 4 pulse burst every second; shots in the burst are repeated at a rate of 1KHz. It also can produce single pulses with a 30Hz repetition rate, although the energy produced is much less. The output is frequency doubled to 532 nanometers via an internal doubling crystal. This laser uses a recently developed folded-resonator configuration, resulting in an extremely compact optics package. Although some "tweaking" of this system was required by the manufacturer, subsequent acceptance testing of this unit has validated that the specified performance has indeed been achieved. In addition to the rugged, compact packaging of the optical head, this laser system is configured so as to be compatible with operation in either the NASA Learjet or KC-135 aircraft.

Following receipt of the laser, a cyclonic insertion fluidized bed seed chamber was designed and fabricated. This seeder is intended for use with gas jets which are identical to those in use by investigators currently funded under the MSAD program. Clear acrylic was used as the material so that the flow structure in the interior could be viewed. Testing revealed that the injection velocities were too low to entrain sufficient numbers of particles due to the diameter of the inlet port and the relatively small mass flow rates employed in these experiments. Additionally, the flow tended to "tunnel" through the particle bed which further precluded entrainment. Based on these results, a new design was developed, with the goal of developing fully-turbulent flow inside the seeder plenum. The diameter of the burner, which forms the outlet of the chamber is 1.65 mm. With an inlet diameter of approximately 0.1 mm, a Reynolds number of roughly 2000 inside the seeder was achieved. This design was tested using a new type of seed particle known as "Microspherical Feathers," manufactured by Osaka Gas and discovered by Paul Greenberg while working on the LDV portion of this project previously described. These particles are essentially hollow spheres of silicon dioxide, and are extremely monodisperse. They possess extremely large scattering cross sections and small effective hydrodynamic diameters, both properties being advantageous for this application. Initial testing showed that the particle entrainment was excellent and velocity vectors were determined using images captured both live and from a pre-recorded sequence recorded in Sony BetaCam format.

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In order to measure velocity maps, the video system and laser must be properly synchronized to insure that an image is recorded on all frames. Additionally, particle images must appear as points and not streaks so that the software can accurately locate the centroid of each particle image. During this last reporting period, the synchronization of the pulsed Nd:YAG laser and video was successfully demonstrated, resulting in the capability for flow analysis in a range between 5 and 150 cm/sec. Given that the sub-buoyant velocity range is 5-40 cm/sec, the current setup can easily accommodate most phenomena of interest. Velocity maps were recorded with the limitation on measurement accuracy being determined by the resolution of the array currently being used.
II. MSAD Program Tasks — Advanced Technology Development

Previous analysis of particle images was performed using software that was written using support from the previous ATD award. However, that software required 5 images to accurately determine velocity vectors. As a result, flows with curvilinear characteristics were difficult to accurately quantify. More recent versions of the software have incorporated fuzzy logic algorithms, which reduce the image requirement to only two. This software has been used with data generated from the Droplet Combustion Experiment (DCE) in the 5.2 Second Tower to compute velocity vectors for the large soot aggregates generated during the burn. Due to the illumination system used, these numbers were two-dimensional projections of a three-dimensional velocity. Based on the results to date, MCD personnel are assessing designs for an improved illumination system to better record the flow.

As PIV technology is transferred to investigators external to the MCD group, investigators frequently want to know the appropriate design parameters required to properly engineer a PIV system. To develop an intelligent design, the intensity of the scattered light and sensitivity of the array must be known. While the latter is provided by the manufacturer, the former must be computed from light scattering theory. To that end, software was acquired to calculate intensity factors for Mie scattering. These results are used directly to determine intensity distributions at the receiving optics, or integrated using MCD-developed software to determine the scattering cross section. Use of these two codes allows direct engineering of PIV systems in terms of required source strengths, scattering cross sections, integration times, and fields of view. The capability has since been made available to the general research community. Also derived and disseminated is a program that describes the propagation of Gaussian beams and allows the design of optical systems to generate points, lines and sheets of arbitrary dimensions using a variety of laser sources.

While the laboratory development is still in progress, the transition to reduced gravity platforms has begun. The initial effort involves a collaboration with Fletcher Miller, technical analyst/monitor for NRA awardee, Fokion Egolfopolous of the University of Southern California. MCD personnel have designed and tested a particle seeder that will be used in the 2.2 Second Drop Tower tests. This seeder is a scaled-up version of the seeder that MCD personnel have been using in the laboratory over the past year.

In addition to assisting Professor Egolfopolous, MCD personnel have taken steps to provide PIV capability in the small drop tower and on the DC-9 aircraft. In the former facility, PIV and LIF would be facilitated by using a large diameter optical fiber to transmit 10-20 nanosecond laser pulses with energies on the order of tens of millijoules. Typical fibers suffer laser damage at those levels, but initial testing with a new fiber type revealed a transmission capability of 15 millijoules at 532 nm. Exhaustive testing could not be completed in time for this report due to a failure of the flash lamp and associated electronics in the compact Nd:YAG laser. Upon completion of repairs, testing will resume.

Finally, fabrication was begun on a frame to accommodate the use of the compact Nd:YAG laser on reduced gravity aircraft. This frame will be used as a platform for the laser and will be used in conjunction with a rig from the 2.2 Second Drop Tower to demonstrate gas phase PIV in reduced gravity. We anticipate completing these experiments at the end of the present performance period.

STUDENTS FUNDED UNDER RESEARCH:      TASK INITIATION: 10/92      EXPIRATION: 9/96
PROJECT IDENTIFICATION: 93-2
RESPONSIBLE CENTER: LeRC

BIBLIOGRAPHIC CITATIONS FOR FY 1995:

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II. MSAD Program Tasks — Advanced Technology Development

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Weiland, K.J. Imaging invisible flames without additives. NASA Tech Brief, (Accepted, 1995).

Proceedings


II. MSAD Program Tasks — Advanced

Small, Stable, Rugged Microgravity Accelerometer

Principal Investigator: Dr. Frank T. Hartley
Jet Propulsion Laboratory (JPL)

Co-Investigators:
Zavracky, P.  
Dolgin, D.  
Northeastern University  
Jet Propulsion Laboratory (JPL)

Task Objective:
The objective of this ATD task is to build a novel micromachined accelerometer that is capable of measuring accelerations from $10^{-2} \text{g}$ to $10^{-8} \text{g}$ with a better than $10^{-6} \text{g}$ accuracy ($<10^{-6} \text{g/Hz}$) for the frequencies from $10^{-4}$ to 20 Hz. The device should have low temperature sensitivity and have a built-in calibration. The accelerometer must withstand the launch environment. The task objectives include: 1) development of micromachined flexures; 2) development of an electronic parking mechanism; 3) development of active controls for the accelerometer, and 4) development of electronics for data acquisition and control.

Task Description:
The most important parameters are independent measurements of all three spatial components of acceleration, high accuracy, and low-frequency measurements. The sensitivity, accuracy and frequency domain are determined by the different acceleration sources that are found aboard the Space Shuttle and other spacecraft. The lowest frequency is determined by the low Earth orbit period. The "as designed" device should be able to measure accelerations at frequencies lower than $10^{-4} \text{Hz} - 1/180$ minutes, but the accuracy may decrease. Inertial navigation requires precise positioning in space as well as the orientation tracking (sets of six or more accelerometers working as a three-dimensional gradiometer). Seismology applications require high sensitivity in the presence of a constant acceleration (g). All of these applications are possible with a flexure suspension and electrostatic actuation design of the accelerometer. The flexure permits precise measurements of very small accelerations in the presence of a large cross-axial acceleration. In other words, the accelerometer can measure the components of the acceleration independently. The electrostatic actuation reduces temperature dependence and permits in situ calibration.

Task Significance:
The accelerometer will find applications in microgravity research, inertial navigation, seismology, geophysics, planetary physics, and DoD programs. The device under development is optimized for a microgravity application, and additional applications should require only minor modifications.

Progress During FY 1995:
Custom multi silicon wafer micro machined accelerometers have been built and tested. Each of the three operational modes of the accelerometer sensor have undergone testing. The re-deployable electrostatic 'caging' mode was used for shipping accelerometers and evaluated in a load jig. The transitional mode, between caging and tunneling tip control, was implemented with both low and high frequency controllers. Finally closed loop tunneling tip control was implemented in a one-g field on a small displacement test stand and found to exhibit comparable sensitivity to a collocated QA3000. Low 'g' (horizontal) acceleration measurement trials, which will be more sensitive, are awaiting further funding. A Space Acceleration Measurement System (SAM) interface was designed, built and flight tested. Precision voltage reference, filters and accurate analog to digital converter systems were developed that provided ppm accuracy. The accelerometer controller, interface and metrology circuit modules were designed and are ready for flight electronics fabrication.
## II. MSAD Program Tasks — Advanced

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| PROJECT IDENTIFICATION:        | 93-1                     |
| RESPONSIBLE CENTER:            | JPL                      |

Printed: 28 FEB 1996
II. MSAD Program Tasks — Advanced

High Resolution Pressure Transducer and Controller

Principal Investigator: Dr. Ulf E. Israelsson
Jet Propulsion Laboratory (JPL)

Co-Investigators:
Michelson, Prof. P.
Lipa, Prof. J.
Barmatz, Dr. M.
Stanford University
Stanford University
Jet Propulsion Laboratory (JPL)

Task Objective:
The objective of this project is to develop high resolution pressure transducers and controllers with many orders of magnitude better performance than presently commercially available. The main use of these devices will be for fundamental science investigations in the area of low temperature physics at helium temperatures.

Task Description:
Two types of pressure transducers will be developed for use in the 1 to 10 bar pressure range. The first will utilize a capacitive readout technique and will be capable of operating at any temperature from 300K to 2K. The second will utilize inductive readout by means of a SQUID and will operate at helium temperatures only (2K to 7K). The expected resolution of these transducers are about one part in $10^{10}$, and they will dissipate negligible amounts of heat when operated. The pressure controller will be based on similar techniques as the transducers and will be capable of controlling pressures in the 1 to 10 bars range over a limited pressure swing to near one part in $10^{10}$.

Task Significance:
The current capability of the MSAD program in fundamental low temperature physics is limited to controlling and reading out one experimental variable, temperature, to sub-nano kelvin resolution at helium temperatures. By providing a similar capability for a second variable, pressure, many new important flight investigations can be performed. An example would be to test the validity of the Universality hypothesis in the theory of second order phase transitions by performing high resolution measurements of some experimental quantity at several fixed pressures.

Progress During FY 1995:
A prototype capacitive pressure transducer operating in the 2.5 PSI range was constructed using silicon micromachining techniques. It employs a flexible silicon membrane which is sealed to a helium sample chamber with an indium seal. Attached to the back side of the membrane is a thin film capacitor plate. A second thin film capacitor plate is located a short distance away on a fixed block of silicon. Pressure variations in the sample cell will flex the membrane thereby adjusting the relative capacitance of the circuit. The transducer was tested on an existing high resolution thermal platform at JPL. The resolution was determined by making use of the known temperature dependence of the saturated vapor pressure of helium and by applying known temperature changes measured with a high resolution thermometer. With a room temperature reference capacitor, a resolution of better than one part in $10^7$ was obtained, thereby demonstrating the viability of the concept. The pressure stability of the transducer was found to be less than desirable. It is believed that this is due to relaxation problems associated with the indium seal. A new transducer for operation at the desired higher pressures of 1 to 10 bars has been fabricated. Because of it's higher operating pressure, the new device will be less sensitive to relaxation problems associated with the indium seal. If need be, harder metal seals can also be used in the current design to reduce the drift to acceptable levels. In order to improve the resolution of the device further, a cold reference capacitor has been fitted to the apparatus.

A prototype inductive pressure transducer has also been constructed. It employs a niobium diaphragm and a superconducting coil in close proximity. The diaphragm serves as a ground plane for the coil. A pressure change
II. MSAD Program Tasks — Advanced

will bend the diaphragm thereby changing the inductance of the coil. The inductance change is detected with a DC SQUID sensor at low temperature. The initial test results are very encouraging. At low pressures, a SQUID-limited resolution of about 0.1 billionth of an atmosphere has been demonstrated. The actual resolution is about 1.5 times worse, probably limited by vibration noise. If the measured performance holds up at high pressures, it is likely that the goal performance on resolution of about 0.01 parts per billion at 10 bars can be achieved.

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BIBLIOGRAPHIC CITATIONS FOR FY 1995:

Journals

NASA Tech Briefs

Presentations
**II. MSAD Program Tasks — Advanced**

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**Single Electron Transistor (SET)**

**Principal Investigator:** Dr. Ulf E. Israelsson  
Jet Propulsion Laboratory (JPL)

**Co-Investigators:**  
LeDuc, Dr. H.G.  
Jet Propulsion Laboratory (JPL)  
Maker, Dr. P.D.  
Jet Propulsion Laboratory (JPL)  
Bozler, Prof. H.  
University of Southern California

**Task Objective:**

The objective of the proposed effort is to develop an electrometer (Single Electron Transistor) capable of a resolution of $10^6$ e/Hz$^{1/2}$, where e is the electron charge, operating at 4.2K using Nb/Al$_2$O$_3$/Nb technology. The objective of FY95 task period is to develop the Single Electron Transistor with relaxed size constraints.

**Task Description:**

A mesoscopic single electron transistor (SET) is formed from two tunnel junctions whose capacitances are small, of the order of $10^{-13}$ Farad or smaller. The common electrode is capacitively coupled to the source of the charges to be measured. Changes in the capacitively coupled charge by fractions of a single electron charge will modulate the current passing through the junctions. The main technological problem is fabrication of the junctions. The junctions will be formed using e-beam lithography. The technological basis is developed in JPL's Microdevices Laboratory.

This task will result in a design for the charge amplifier that will take into account the feasibility of the processing steps involved in it, such as the limits of electron-beam lithography and the verticality of etch during the reactive ion etching steps, for example. The size constraints at the first period of the task, to be concluded in FY95, will be relaxed to facilitate fabrication and thus decrease the turn-around time for each design version. The devices fabricated will be tested at 4.2 K and at sub-Kelvin temperatures. The success criteria of the task will be obtaining devices with satisfactory current-voltage characteristics.

**Task Significance:**

Development of technologies required for production of a SET will allow making working SET in the next task’s period. This in turn will open new possibilities for controlling and reading out different experimental variables. SET is suitable for measuring many parameters of the materials under investigation. Examples include measurements of dielectric constant, pressure, density, temperature, charge, voltage, etc. The parameters whose measurement can be reduced to capacitance measurements are especially suitable for measurements with SET. Unlimited dynamic range of SET allows precise measurements even in cases when a capacitive method with present techniques does not lead to sufficient sensitivity.

For example, it is feasible that SET-based thermometers will have resolution comparable to or exceeding that of High-Resolution Thermometers, presently used in several flight experiments, designed or supported by JPL. In addition, SET is compact in size. This small size will allow miniaturization of SET-based devices and creating arrays of them, accelerating information collection and in many cases decreasing flight time required to conclude an experiment. Miniaturization of the experiments is also very much in line with JPL’s goal of creating miniature spacecraft.

**Progress During FY 1995:**

Two different approaches to the design were taken. The first approach is based directly on miniaturization of the technology already existent at the Microdevices Laboratory for fabrication of small tunnel junctions. The second approach is a departure from the "traditional method" and involves a different technology of making junctions called...
"edge junctions." Each method has its advantages and disadvantages. In each approach roughly three designs were tried, each solving problems encountered in the previous version.

Tests of the current-voltage (I-V) characteristics of the devices fabricated were performed at JPL. Single junctions fabricated using the two approaches showed reasonably good I-V characteristics. Full devices (that is, two tunnel junctions connected in series capacitively coupled to the external world via a gate) showed good I-V characteristics for the second method ("edge" junctions) so far. The I-V characteristic displayed small subgap leakage current and gap voltage corresponding to that of the bulk niobium, indicating good quality of both the electrodes and of the oxide barrier forming the junctions.

The devices fabricated using the "edge" method that showed good I-V characteristics at 4.2K were tested at dilution refrigeration temperatures. No charge amplification behavior was observed in any of them so far. This is probably due to a parallel path for conductance that is inherent to the "edge" junctions method and is being circumvented in another redesign. In the redesigned "edge" junction SETs the effect of this parallel path is minimized. Tests at 4.2K revealed good I-V characteristics.

As part of this task, a new measurement setup with significantly lower noise was designed and built at USC. This setup also has the capability of voltage biasing the devices very precisely using a feedback scheme. Improved low-temperature microwave filters were installed at USC as well to ensure that measurements are not affected by ambient electromagnetic noise.
II. MSAD Program Tasks — Advanced

Surface Light Scattering Instrument

Principal Investigator: Dr. William V. Meyer
Ohio Aerospace Institute

Co-Investigators:
Mann, Prof. J.A.  Case Western Reserve University
Tin, Dr. P.  Case Western Reserve University
Rogers, R.B.  NASA Lewis Research Center (LeRC)

Task Objective:
We are developing a miniature, non-contact instrument which examines laser light scattered from fluid/fluid interfaces to measure surface tension, viscosity, true surface temperature, and their gradients. The fluid/fluid interface can be either liquid/liquid or liquid/vapor, and it may include a monolayer. Our work springboards from traditional designs which are bulky and difficult to use because of their extreme sensitivity to vibration. For these reasons, surface light scattering instruments have never been commercially available. The new generation of hardware developed under this program is significantly improved and has widespread applications, so that commercialization is the next logical step.

Task Description:
While individual surface light scattering (SLS) instruments have been built and used successfully by experienced researchers in isolated labs around the world, the method has been unavailable to non-specialists wishing to use it in their applications. To bring surface light scattering instrumentation out of the lab and into the worlds of space research, the natural environment and the commercial sector, we had to address the problems of vibration sensitivity, size, and optical alignment. We have designed, built, and successfully tested a new anti-slosh cat’s eye optical train which makes the instrument immune to vibration. Previously, the instrument could only be used if extreme measures were taken to isolate the experiment from ambient vibrations. We have used the new instrument to accurately measure the surface properties of water, ethanol, acetone, silicone oils, and liquid gallium the presence of ambient laboratory vibration. We have greatly reduced the size and simplified the optical alignment by developing a fiber optics version of the instrument. With these improvements, surface light scattering instruments are ready to graduate from the research lab and enter the world of applications. Brookhaven Instruments Corporation (among others) has already expressed an interest in making this happen, and has designed and provided us with a custom version of their next-generation correlator with this in mind.

Task Significance:
Surface tension is an elusive phenomena which controls many everyday processes. It is the two-dimensional analog of pressure, and it attempts to maintain the smallest possible surface area. It affects cooking, cosmetics (improved formulation), tertiary oil recovery (20-30% more oil can be pumped from the ground), detergents (better wetting of fibers), controlled release and targeted drug delivery, and materials processing such as steel making. Accurate surface tension data is vital in the development of lead-free solders. The multi-billion dollar solder industry is actively searching for lead-free solders because lead leaches from discarded soldered pieces in landfills and contaminates water supplies.

In addition to surface tension data, surface light scattering instruments provide viscosity and, for pure substances, temperature information. Viscosity is the internal friction of fluid (how sticky it is). This affects liquid crystal displays such as the flat screens used in computers and other applications. Surface temperature is useful because it indicates heat transfer rates and the presence of buoyancy driven flow.

The study of surface tension driven phenomena is often masked by gravitational forces which are not present in the reduced gravity environment of a space station or space shuttle. Because of this, a number of space experiments...
have been proposed and flown which examine the effects of surface tension in near-weightless conditions. Despite their stated interest in surface tension, these experiments have lacked a means to measure it. The surface light scattering instrument described here is a unique and valuable addition to space science in that it permits accurate, non-invasive, in-situ measurements of surface tension and viscosity. From these, one can extrapolate the local surface temperature of a known, clean sample. It has also been developed to be rugged, power efficient, miniature, and flexible in set-up. The knowledge gained with its application will have diverse impacts in many fields of endeavor.

Progress During FY 1995:
The anti-slosh cat's eye optical train has been refined and successfully tested in both the transmission and reflection modes as well as on the front end of the high-amplitude laser vibrometer we have developed for JPL. The SLS instrument has also been tested with good results on a Langmuir trough, which permits tests of controlled thin films (monolayers, etc.). The fiber optics SLS instrument has been built and tested on a variety of dielectric materials. For all versions of the instrument, the overall errors have been within 0.1% of established surface tension values for dielectrics and room temperature liquid gallium (with an oxide layer). We are testing the SLS instrument on electromagnetically levitated molten aluminum (m.p. 660 °C) and copper (m.p. 1083 °C). The high amplitude laser vibrometer development has been completed, and the instrument was delivered to JPL for their use in studies of high amplitude turbulence and chaos. Our work on this surface light scattering instrument has prompted many new ideas which we will be exploring in another Advanced Technology Development project.

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Project Identification: 92-4
NASA Contract No.: NCC3-419
Responsible Center: LeRC
The Laser Feedback Interferometer: A New, Robust, and versatile Tool for Measurements of Fluid Physics Phenomena

Principal Investigator: Dr. Ben Ovryn

Co-Investigators:

Task Objective:

The objective of this Advanced Technology Development (ATD) project is the evaluation, adaptation, and delivery of a novel form of interferometry, based upon laser-feedback techniques, which will provide a robust and versatile, state-of-the-art diagnostic instrument applicable to the ground measurement of a wide variety of microgravity and ground-based fluid physics and transport phenomena.

Task Description:

Laser feedback interferometry (LFI) differs from conventional interferometry by using the laser as both a light source and as a phase detector. Either a cavity or a semiconductor (diode) laser can be used. LFI can be used either in direct reflection of the interrogating beam can pass through the sample and then be reflected into the laser.

The instrument developed under this ATD will be a robust, phase-measuring device (with simultaneous imaging of the sample) which can determine both slowly time varying and dynamic phenomena over a microscopic and macroscopic field-of-view. Since the interferometer can be used to count fringes, there is no upper limit to the size of the measured displacement. Additionally, the direction of the displacement can also be obtained. The apparatus will accommodate both small and large working distances (many cms). For a microscopic field-of-view, the laser feedback interferometer will be incorporated with an optical microscope.

Because LFI utilizes a common-path geometry (the incident and reflected light traverse the same path), and utilizes only a few optical components, requirements on stability, optical alignment, and sensitivity to external disturbances are much lower than other forms of interferometry.

This ATD consists of a multi-year effort to accomplish three specific goals: (1) build an interferometer based upon a continuous wave HeNe laser with stationary imaging optics and a translated sample, quantify the random and systematic measurement errors (including sensitivity to external perturbations) and calibrate the technique; (2) modify the two-dimensional scanning technique so that the sample remains stationary and the optics translate; and (3) investigate the LFI response with semiconductor diode lasers and incorporate a diode laser into the previously designed instrument.

Task Significance:

Often the primary science requirements of experiments of relevance to the microgravity Science and Applications Division (MSAD), both ground-based and those requiring reduced gravity, involve the measurement of phenomena which can be evaluated by measuring the spatial variations of the path length of an interrogating light ray. These variations can arise either from changes in the index of refraction (a function of temperature, pressure, mass density, and concentration) along the path of the ray or from changes in the distance a ray travels.

Some examples of science requirements from experiments of relevance to the Fluid Physics and transport Dynamics Discipline which can be determined from accurate measurements of the change in the optical path length include the determination of: (1) the location and orientation of a contact line and interface shape between two fluids; (2) the evolution of the thickness of a thin film; (3) deformation of a free surface due to evaporation or vibration; (4) resonant mode shapes in bubbles; (5) fundamental fluid parameters such as surface tension and viscosity; (6) variations in temperature and density in a fluid; (7) diffusion coefficients; and (8) fluid velocity.
The developed technology is likely to be attractive to many users outside the aerospace community. In fact, LFI will augment the ubiquitous uses of interferometry which have demonstrated applicability to nearly every scientific discipline including: physics, materials science, mechanical engineering, chemistry, and biomedical sciences.

Progress During FY 1995:
The first goal of the project has been achieved; a laser feedback interferometer has been constructed and calibrated by measuring the cantilever bending of a piezoelectric bimorph; static bending of less than a nanometer has been observed. This instrument has also been used in fringe counting mode to measure several microns of displacement. The systematic and random errors have been quantified.

Presently, the interferometer is being incorporated into an optical microscope. The instrument will be calibrated by measuring the three-dimensional surface profiles of static liquid drops. This instrument will incorporate piezoelectric stages to scan the sample underneath fixed optics. Once calibration tests are completed, the sample will remain stationary and scanning beams optics will be incorporated.

A demonstration of the technique was presented at Technology 2005 in Chicago, IL (October, 1995). the instrument and software performed flawlessly in a noisy environment and demonstrated the feasibility of using this instrument to measure static and oscillatory optical path length changes with high accuracy.

One spin-off application of this project, currently being pursued at NASA Lewis, is the application of the technology to emission sensing in jet engines.

Students Funded Under Research:

Task Initiation: 10/94
Expiration: 9/98
Project Identification: 
Responsible Center: LeRC

Printed: 28 FEB 1996
II. MSAD Program Tasks — Advanced

Crystal Growth Instrumentation Development: A Protein Crystal Growth Studies Cell

Principal Investigator: Dr. Marc L. Pusey  NASA Marshall Space Flight Center (MSFC)

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
The goal is to design and construct prototype cells which will permit study of the growth process of protein crystals by a variety of techniques. Two cell designs are proposed. The first and most important will be for studies of the solution concentration gradients surrounding a growing crystal. The second design will be to study interfacial features of protein crystals under growth and etching conditions. A second goal of the proposed work will be to develop practical methods for protein storage prior to use in crystal growth (and other) experiments. The characteristics desired for a Protein Crystal Growth Analysis Chamber system for μg growth studies are:

1. Several types of growth cells to accommodate specific study goals
2. Temperature control of the growth cell from 0 - 40° C (±0.05° C)
3. Control of nucleation and growth of crystals at a defined location
4. A fluidic system to accurately prepare crystal growth solutions from stock solutions of protein, precipitant, buffer, etc. and deliver them to the growth cell
5. The cells should be accessible for additions to and/or modification of the solution
6. The ability to do follow-up experiments based upon preceding growth runs
7. Some cell adjustment to bring selected crystal faces in line (perpendicular or parallel) with the optical axes
8. Easy accessibility to other solution measurement systems (pH, conductivity, etc.) for maximum data return
9. Remotely operable from the ground to the maximum extent possible
10. Ability to maintain proteins in a viable state prior to use

Task Description:
Current microgravity (μg) protein crystal growth (PCG) hardware systems are hybrid, attempting to both acquire data about the processes of crystallization while growing crystals suitable for x-ray diffraction studies. This leads to compromises in the design, with the result that neither approach is successfully accomplished. The proposed hardware will be designed solely for studying the growth of protein crystals, which requires fewer experimental growth cells, but each having the maximum amount of data return and control of solution physico-chemical parameters.

For the upcoming year’s effort the primary emphasis will be shifted from growth cell design to methods suitable for protein storage during long term (three months) μg missions. This will employ 8-12 different proteins, each stored in two forms (in solution or as a freeze-dried powder) at three different temperatures (20° C, 4° C, and -20° C). At periodic intervals the stored material will be sampled and tested for changes in crystallizability, turbidity, and structural and functional integrity. These measurements will determine if the net charge, molecular size, or shape, of the protein have altered during storage. Concurrently with the above we will begin designing and testing potential storage containers suitable for g use.
II. MSAD Program Tasks — Advanced

Task Significance:
In usage on board an orbiting platform ground-based researchers will be able to monitor and change selected solution parameters within the growth cells, and conduct follow-on experiments based upon recently acquired data. This will enable the realistic studying of how μg affects the protein crystal growth process, and how it can be improved both in space and on Earth. Long-term protein solution storage and stability will become a dominating concern as the time scale of g experiments increases. Developing methods for long-term protein storage will improve both this and future g-based instrumentation.

Progress During FY 1995:
Due to advances in interferometric methods, we now believe that a single cell design can be used for all the experimental goals of this project. The current cell design is a 25 mm x 10 mm x 0.5 mm chamber enclosed by a circulating water jacket, with all surfaces made from high optical quality glass. Thermal control will be provided by a small circulating water bath now under development.

The fluids preparation system has been delivered by NRL, and found to work according to specifications. However, the solutions in this system are each delivered by a separate line, necessitating additional syringes and hardware to functionally integrate it with the cell. Accordingly, we decided to build a second fluids preparation system based upon the NRL system. At this point, working with only a single syringe and valve system, we are able to accurately take up, mix, and dispense a 50:50 mixture of a 20% PEG (chosen for its high viscosity) and dH2O. Mixing is accomplished by passing the solutions back and forth to an inert (non-motorized) "take up" syringe. Current work, while awaiting the delivery of additional motorized valves, is centered on passing the solution back and forth to a second motorized syringe. In the final configuration we anticipate that this will be a smaller volume syringe for accurate measurement of small amounts of protein and other solutions.

In connection with Dr. Alex McPherson and William Withrow we responded to the FY95 Glovebox solicitation and were selected for the initial trial period. The tasks assigned to this laboratory are the delivery of a working growth cell, a thermal control system, and a manual solutions delivery system. The cell design and temperature control system are identical to those being developed above. The solutions delivery system, under development, are a simple hand-held syringe array to deliver protein crystal nucleation and growth solutions at predetermined concentrations.

Stability testing of protein solutions was initiated this year, starting with lysozyme and ovostatin at 20°, 4° and -20° C, in the lyophilized and solution states at each temperature. Stability was measured by sitting drop crystallization trials carried out at two week intervals, with the data collected being the numbers and sizes of crystals in the sitting drops. Most surprising was the result that the least stable method of storing lysozyme was as a frozen solution. Ovostatin rapidly degraded in the 20° C conditions, but was viable at 4° and -20° C.

Students Funded Under Research: Task Initiation: 10/94 Expiration: 10/96
Project Identification: 963-70-02
Responsible Center: MSFC
II. MSAD Program Tasks — Advanced

High Resolution Thermometry and Improved SQUID Readout

Principal Investigator: Dr. Peter Shirron
Goddard Space Flight Center (GSFC)

Co-Investigators:
DiPirro, Dr. M.J.
Tuttle, Dr. J.G.
Goddard Space Flight Center (GSFC)
Hughes/STX

Task Objective:
The objective of this work is to develop two technologies which are becoming increasingly central to low temperature microgravity research: high resolution thermometers and Superconducting Quantum Interference Devices (SQUIDs). A penetration depth thermometer (PDT) which uses thin film superconductors is being developed which can meet the performance of existing paramagnetic salt thermometers, while offering greater flexibility and immunity to particle radiation. Advanced SQUID sensors have been manufactured with far better intrinsic energy resolution than commercially available sensors. These sensors use a second stage array of 100-200 SQUIDs to amplify the signal from a single input SQUID. SQUID arrays and control electronics will be developed and evaluated for potential use in spaceflight applications.

Task Description:
The two efforts mentioned above have until now proceeded along independent paths. The emphasis during FY95 for the PDT development was to fabricate superconducting films with a transition temperature (T_c) in the range of 2.3-2.4 Kelvin. This would result in a thermometer with peak sensitivity at 2.17 Kelvin and make it suitable for use in both microgravity and ground-based studies of the lambda transition in liquid helium. Different geometries and film deposition techniques have been explored to determine which factors most significantly impact the sensitivity and noise of a thermometer. Measurements of temperature sensitivity and intrinsic resolution have been made using commercial SQUID system. The emphasis for the SQUID development has been the fabrication of flight quality control electronics and the continued evaluation of SQUID array chips which have been supplied by HYPRES, Inc. System level measurements of noise, power dissipation, bandwidth, and sensitivity to operating temperature and external magnetic fields are underway. Following this evaluation, the SQUID array system will replace the commercial system for reading out the PDT sensors.

Task Significance:
When studying critical phenomena (for example, the lambda point of liquid helium), the focus is usually on how certain parameters such as density or heat capacity vary with temperature close to the critical point. It is crucial for such experiments to determine or control the thermodynamic state to very high precision. Particularly in the microgravity environment, the precision to which temperature and/or pressure can be measured sets the limit for science return. Advances in current state-of-the-art for thermometry and SQUID sensors therefore translate into improvements in the quality of microgravity experiments. The SQUID technology will be particularly beneficial since SQUIDs have a wide range of uses, both in spaceflight applications and in ground-based laboratories. The PDT concept also can be adapted to allow non-contact temperature measurement. By not physically contacting the sample, parasitic heat inputs are minimized (important for heat capacity measurements) and mechanical dissipation is eliminated.

Progress During FY 1995:
• A PDT sensor fabricated with a 70-μm thick aluminum film having a T_c of 1.72 K was assembled and tested. The peak sensitivity was observed at 1.65 K, and corresponded to an intrinsic resolution of 1.0 nanoKelvin/√Hz. Preliminary tests showed the device, operated in a vacuum, had an equivalent thermal noise background of approximately 15 nanoKelvin/√Hz. While this is better than any previous thermometers which use superconducting films, it is still shy of the intrinsic resolution. A stable thermal platform for further testing is being fabricated.
II. MSAD Program Tasks — Advanced

which will allow the PDT to be immersed in liquid helium, to minimize statistical thermal fluctuations.

- Concentrated efforts at increasing the \( T_c \) of aluminum films by progressively reducing the film thickness were not successful. Using a different technique, in which oxygen is admitted into the chamber during the aluminum deposition to produce small aluminum grains in the film, a transition temperature of 2.23 K was achieved. This film could provide useful thermometry from 0 to 2.14 K. Additional effort is still needed to tune the \( T_c \) into the 2.3-2.4 K range. An unexpected benefit of this approach is an increase in the effective penetration depth in the films, and possibly greater temperature sensitivity.

- Improvements in the design of the SQUID array chips have reduced noise levels markedly. At signal frequencies above 300 Hz, the energy resolution (the figure of merit for SQUIDs) is less than 500 h (h is Planck's constant), which is a factor of two better than for commercial systems. However, below 25 Hz the noise rises as the inverse of frequency. This \( 1/f \) noise is typical in electronic systems, but has an onset at a higher than desirable frequency. Recent modifications to the chip design were made to address this. Testing is in progress.

- Control electronics for the SQUID arrays with a "flux-locked loop" feedback circuit have been manufactured and delivered. Preliminary tests indicate overall system noise levels are 50% higher than the open-loop measurements mentioned above, however it appears the noise is due to the chip under test and not the electronics. A more careful study of the open loop characteristics will confirm this. Nevertheless, the system noise levels are well below the design criteria.

- Measurements of the sensitivity of the SQUID output signal to changes in electronics box and SQUID sensor temperature are in progress, and will be followed by EMI sensitivity tests.

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II. MSAD Program Tasks — Advanced

_Determination of Soot Volume Fraction Using Laser-Induced Incandescence_

Principal Investigator: Dr. Randall L. Vander Wal  
NASA Lewis Research Center (LeRC)

Co-Investigators:

No Co-I's Assigned to this Task

Task Objective:
This project will develop a laser-based optical diagnostic technique, laser-induced incandescence (LII), for the measurement of soot volume fraction. The major objectives are to obtain the hardware and choose a combustion system of interest that yields relatively high soot concentrations, verify and develop the technique in the laboratory, including establishing LII on a quantitative basis, demonstrate LII in a laboratory flame under a variety of conditions and geometries, conduct reduced-gravity tests with LII and other complementary optical measurements, and assess the usefulness of LII as a measure of soot volume fraction.

Task Description:
We intend to develop laser-induced incandescence (LII) as a two-dimensional imaging diagnostic for the measurement of soot volume fraction for microgravity combustion research. LII, in conjunction with other optical imaging techniques, would provide unparalleled temporal and spatial resolution, and therefore, yield insight and sensitivity into soot formation and oxidation processes. Present methods of measuring soot volume fraction are limited to line-of-sight methods. These methods offer poor temporal and spatial resolution and require assumptions about the path length and soot physical properties. LII utilizes the spatial and temporal properties of pulsed laser excitation to heat soot to far greater temperatures than ordinary flame temperatures and exploits the resultant blue-shifted incandescent emission from the laser-heated soot. The incandescence is predicted theoretically to be a measure of soot volume fraction, which is the feature of soot that governs many physical processes such as radiative heat transfer from flames. This technique will be extended to two-dimensional imaging applications and will be calibrated against gravimetric sampling.

Task Significance:
Successful development of this diagnostic technique will provide detailed data on soot formation, growth, and oxidation processes for a variety of combustion systems, which may lead to a better understanding and control of sooting processes in flames.

Progress During FY 1995:
1. Laboratory Hardware Setup
Five manufacturers of gated intensified array cameras were contacted along with several researchers using similar devices, regarding manufacture of a cooled intensified array camera. One responded positively and an order has been placed with the manufacturer. The thermophoretic sampling device was completed, tested and utilized in a variety of investigations described below. A gravimetric sampling device was also designed, fabricated and used both in measuring the dependence of the LII signal upon soot volume fraction and also in quantification of the LII signal. A premixed flat flame produced on a McKenna burner stabilized by means of a chimney above the burner proved suitable for investigating through thermophoretic sampling, the effects of the pulsed laser light upon the soot. Stabilization of the flame proved challenging, however. A burner to facilitate the investigation of soot aggregate size upon the LII signal has been fabricated and will be tested this summer. Design of a variable pressure chamber is near completion.

2. Technique Verification and Development
Successful development of any laser-based combustion diagnostic requires characterization of the physical phenomenon being used to probe the flame. For laser-induced incandescence, the dependence of the signal upon a

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Excitation and detection wavelength, b) excitation intensity, c) soot aggregate size effects and d) soot volume fraction must be studied. Our primary goal was to determine how to minimize laser-induced and natural interferences, maximize the laser-induced incandescence signal, and ensure a known dependence of the signal upon soot volume fraction. Investigation of the physical basis for the observed LII signal dependence upon laser intensity and the dependence of the LII signal upon soot volume fraction was the central focus of this year's studies and resulted in two archival journal submissions.

**Excitation laser intensity dependence**

As illustrated in last years annual report, the dependence of the LII signal upon laser intensity has been investigated at 1064 and 532 nm. In both cases, the curves exhibited a steeply rising portion, next a 'plateau' region where the signal is relatively independent upon the laser intensity followed by a falling portion where the LII signal decreases with laser intensity. The 'plateau' region is still considered the optimum region for performing LII measurements slight differences in the laser intensity across the spatial profile of the beam and small attenuations of the laser light by the soot will not affect the LII signal in either 'point' or planar measurements. The utility of such curves is that for a specific beam area and temporal duration, the curve is readily interpreted as laser energy. Hence laser energies necessary for different experimental configurations in which the laser beam may be formed into a sheet may be estimated.

Our efforts this year have focused on understanding the physical basis for the observed excitation laser intensity dependence. Depending upon the nature of the laser interaction with the soot during the heating process, laser intensities other than those in the 'plateau' region may be preferred. The issue of the laser effect upon the soot is vital to discern the appropriate laser excitation intensity and signal collection duration in LII measurements. To examine then issue of the appropriate excitation laser intensity and the effect(s) of the laser upon the soot, we began a series of investigation in collaboration with Prof. Mun Y. Choi of UIC last summer further experiments to be conducted this summer.

In our experiments, soot was collected on 200-mesh copper grids for observing the laser-heated soot particles using transmission electron microscopy (TEM). Unlike non-laser-heated soot, unusual shell structures were observed. These changes in the soot structures are similar to the morphological changes observed during high-temperature oxidation of carbon black. In some particles, small substructures or rosettes within the shell are observed. In others, the structures appear to have a void or a porous material (less dense glassy or amorphous carbon) in the inner core. Energy dispersive x-ray analysis confirms that the chemical composition of the soot exhibiting shell structure is carbon with negligible amounts of other elements and thus not an artifact of some possible contamination. Variations in the temperature-time history of the soot particles relative to irradiation by the laser and thermophoretic collection and flow streamlines within the flame may account for the variable degree to which shell-like structures are found in the experiments. On the basis of tests described in the references, these alterations appear to arise through a combination of thermal annealing and interior pyrolysis of the soot particles. While different researchers have found encouraging agreement between the laser-induced incandescence signal and soot volume fraction these results caution use of laser-induced incandescence without careful consideration of excitation laser intensity and possible variation in soot composition at different measurement locations.

**Excitation wavelength dependence**

Preliminary investigation of the usefulness of different laser excitation wavelength such as 532 nm light has found significant photochemical interferences to be produced at laser intensities suitable for LII. Similar interferences are expected for other available excitation wavelengths such as 355 and 266 nm. Visible or ultraviolet wavelengths offer far greater ease of alignment and allow lower intensities to be used due to the higher absorption coefficient of soot at the shorter wavelength. Testing of the visible wavelengths will aid in determining the systems in which simultaneous LII and scattering measurements will be made. In contrast, using 1064 nm light for laser heating of the soot generally does not produce photochemical interferences. Furthermore, commonly used detectors of the LII signal such as photomultiplier tubes and gated intensified array cameras are incapable of detecting this wavelength, thus scattered laser light by the soot is not a problem. Even though higher excitation intensities are necessary at this wavelength along with alignment difficulty of the invisible laser light for the purpose of performing only LII measurements, 1064 nm is the optimum excitation wavelength.
Optimal spectral imaging wavelength region

The optimal spectral imaging detection region will be determined by avoidance of spectral interferences, availability of commercial bandpass interference filters and sensitivity of the PMT or gated intensified array camera. In general, the optimal spectral range for collecting the LII signal appears to be application specific and hence needs to be investigated for each new combustion process. We will continue to document results for each combustion system we study.

Quantification of the LII signal via light extinction

Absolute soot volume fraction measurements may be obtained by calibration of the LII signal against a known value. The LII signal in two different flame systems has been quantified by comparison to light extinction and by referencing the LII signal to that observed in a gravimetric chimney. The calibration via the technique of gravimetric sampling conveys several advantages to the accuracy of svf determination via LII. Both calibration methods and results are briefly described below and in full detail in the cited references.

For calibration of the soot volume fraction in fiber-supported burning droplets, a small coflow burner with a soot volume fraction previously measured via full-field laser extinction replaced the droplet. The laser sheet geometry, detector gating, and optical collection system are identical for obtaining the LII signal for both the droplet and this 'reference' flame. Comparison of LII intensites arising from the coflow burner and the droplet flame allowed assignment of absolute soot volume fractions to the LII images of the burning fuel droplets. Our peak value of 6.5 ppm produced by a burning decane droplet is consistent with the lower sooting tendencies of alkanes compared to aromatic compounds as has been found in gaseous diffusion flames.

Quantification of the LII signal via gravimetric sampling

As an alternative method of calibrating the LII signal, a second technique involves comparison of the LII signal with soot volume fraction determined through the technique of gravimetric sampling. Gravimetric sampling involves measuring the mass of soot within a measured volume of flame gas. Calibration of the LII signal via this method avoids any reference to the optical properties of soot. We observed excellent linearity between the LII signal and the soot volume fraction determined via gravimetric sampling under a variety of conditions.

Imaging Characterization of the gravimetric chimney and application to a turbulent gas-jet flame

To serve as a calibration device for imaging applications, both temporal and spatial uniformities of soot volume fraction over the entire imaged region are critical. Thus before calibration was performed, the spatial and temporal fluctuations in the soot volume fraction within the chimney were characterized by LII. Although single laser-shot LII images illustrate a nonuniform and chaotic spatial and temporal variation in soot volume fraction. A temporal average of individual LII images, however, rapidly converges towards a final uniform pattern in a time short compared with the GS sampling time (~30 individual LII images obtained at 10Hz compared to minutes of GS time) thus validating the assignment of soot volume fraction to the average pixel intensity obtained by spatially and temporally averaging several LII images. Averaging greater than 30 images in this manner assured invariance of the calculated average pixel intensity upon additional averaging. This calibration procedure was then performed for a variety of flame conditions. Each measurement yielded an identical calibration factor within 10%. The average of these three measurements was then used to assign soot ppm levels to pixel intensity values. Independent measurements demonstrated the linearity of the camera detection system with soot volume fraction. With this calibration, a series of single laser-shot LII images of a turbulent ethylene gas-jet flame (Re = 5000), at three different heights above the nozzle were calibrated for absolute soot volume fraction. Further details are provided in the cited references.
II. MSAD Program Tasks — Advanced

3. Laboratory demonstration of LII in laminar diffusion flames:

*Geometric versatility*

Although the geometric versatility of LII has been demonstrated last year, better quality data have been obtained in the interim. Single laser-shot LII images were obtained of the soot distribution within an ethylene/air diffusion flame surrounded by an air coflow. LII images revealing the soot distribution and concentration were obtained with the laser sheet oriented both vertically and horizontally. The vertically oriented laser sheet reveals the convergence of the soot towards the jet centerline with increasing height above the burner. The LII images obtained with the horizontally oriented laser sheet illustrate 'rings' clearly locating the soot containing regions of the diffusion flame as being a thin annular region centered on the gas-jet centerline. Such images reveal the capability of the technique for determining svf in diffusion flames.

**STUDENTS FUNDED UNDER RESEARCH:**

**TASK INITIATION:** 10/93  **EXPIRATION:** 9/97

**PROJECT IDENTIFICATION:** 94-2

**NASA CONTRACT NO.:** NAS3-27186

**RESPONSIBLE CENTER:** LeRC

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**Presentations**


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II. MSAD Program Tasks — Advanced


Multi-Color Holography

Principal Investigator: Mr. William K. Witherow
NASA Marshall Space Flight Center (MSFC)

Co-Investigators:
Trolinger, Dr. J. 
Vikram, Dr. C. 
Metrolaser 
University of Alabama, Huntsville

Task Objective:
A noncontact method of simultaneously determining concentration and temperature variations in fluid systems is underway. An additional benefit will be the additional simultaneous data acquisition capability and thus a possible reduction in the number of experiment runs required per mission.

Task Description:
In this system, two fluid parameters will be varied simultaneously, and this technique will measure the variations by using two different frequency lasers.

Task Significance:
More complete multivariable research on fluid science experiments will be enabled by this new capability.

Progress During FY 1995:
The KC-135 flight unit has been delivered to MSFC/NASA. Prospective PI's and experiments will be sought that might make use of the technology that was developed for this project. The final report has been written and is in the process of being printed for delivery to NASA Headquarters. The final report will also be published as a NASA TM for wider distribution.

STUDENTS FUNDED UNDER RESEARCH:

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II. MSAD Program Tasks — Advanced

Ceramic Cartridges via Sintering and Vacuum Plasma Spray

Principal Investigator: Dr. Frank R. Zimmerman
NASA Marshall Space Flight Center (MSFC)

Co-Investigators:
No Co-I's Assigned to this Task

Task Objective:
The objective of this development work is to provide a manufacturing process for containment cartridges used in high temperature (1200° to 2000° C) crystal growth furnaces. A thermal spray process will be used to build up refractory metals and ceramics into a containment cartridge for high temperature, single crystal semi-conductor growth experiments. These plasma spray-formed materials will be evaluated for mechanical properties, density, microstructure, and resistance to liquid metal attack. Forming techniques and the resultant mechanical and metallurgical properties will be identified.

Task Description:
A thermal spray process is being used to build up refractory metals and ceramics into a containment cartridge for high temperature, single crystal semi-conductor growth experiments. This process uses high energy plasma inside a low pressure (100-200 torr) inert environment to apply layers of material onto a removable mandrel. A variety of materials are being characterized and evaluated against a demanding set of requirements, including high service temperature (1700° C), oxidation resistance, and resistance to liquid metal attack. Techniques to spray form refractory metals (tungsten, molybdenum, niobium, tantalum) and ceramics (alumina, boron nitride) are being developed in the Plasma Spray Cell at Marshall Space Flight Center.

Task Significance:
NASA's Crystal Growth Furnace (CGF) has flown on the United States Microgravity Laboratory missions (USML-1) and -2) to conduct single crystal growth work on a variety of semiconductor materials. These semiconductor crystals (GaAs, CdZnTe, HgZnTe, HgCdTe) are grown in quartz ampoules surrounded by insulation, thermocouples, and other instrumentation. The entire experiment is contained within a Sample Ampoule Cartridge (SAC) which facilitates handling and installation into the furnace. The SAC also functions as a containment system, which is required to protect the flight crew from the highly toxic materials used in these crystal growth experiments. If the ampoule containing the crystal growth material should rupture, these cartridges provide a necessary level of containment. The containment requirement is complicated by the fact that most of these crystal growth compounds are known to aggressively attack most common structural metals at furnace operating temperatures. Currently, these cartridges are made of materials which are attacked at a rate that will not fully penetrate the cartridge within the duration of the experiment. For experiments of longer duration, a more chemically resistant material is required to assure no breach in the containment will occur. Regardless of the material used, the current manufacturing sequence is labor intensive, dependent upon product availability, and contains several fabrication steps and joining processes.

Progress During FY 1995:
Pre-machined adjustment blocks were successfully brazed to tungsten and molybdenum/rhenium cartridges. Units passed helium leak and proof pressure testing.

Addition of 1-3% nickel to tungsten was found to improve VPS product ductility, and verified to resist attack by gallium. Nickel was shown to diffuse into tungsten matrix, eliminating concerns about local chemical attack.

Introduction of non-contact failure detection system into the VPS formed cartridge. Objective is to embed sensor in the cartridge that will reliably detect ampoule failure without interfering with the ongoing science.
II. MSAD Program Tasks — Advanced

STUDENTS FUNDED UNDER RESEARCH:  

TASK INITIATION: 10/93  EXPIRATION: 10/95
PROJECT IDENTIFICATION: 963-70-05
RESPONSIBLE CENTER: MSFC

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Proceedings
Advanced High Brilliance X-Ray Source

Principal Investigator: Dr. Daniel C. Carter

Co-Investigators:
Gibson, W.
Kumakhov, M.
Ho, J. (X. He)

NASA Marshall Space Flight Center (MSFC)
State University of New York, Albany
I.V. Kurchatov Institute of Atomic Energy
NASA Marshall Space Flight Center (MSFC)

Task Objective:
The primary objective of this research is to produce the first x-ray generator and Kumakhov lens system optimized in design for 8.0 KeV x-rays.

Task Description:
The x-ray unit will be integrated with existing x-ray diffraction equipment at Marshall Space Flight Center to produce a diffraction facility with the most advanced laboratory x-ray source for application in crystallography in the world. The approach to complete the task will be to produce intense small cross section parallel x-ray beams for structural analysis using third-generation Kumakhov optics.

Task Significance:
Protein crystallography is currently the most powerful method for the determination of the three-dimensional structure of proteins and other macromolecules. This method usually requires crystals which are relatively large in size and which possess a reasonably high degree of internal order. This research is concerned with the development of an extremely bright x-ray source for application in the evaluation and determination of the atomic structure of crystalline matter from both ground-based and current flight experiment activities.

Progress During FY 1995:
1. Advanced x-ray multicapillary optics have been successfully designed, constructed and evaluated by a team of scientists at MSFC, SUNY, and X-ray Optical Systems, Inc. to produce high intensity x-ray sources.

2. Initial tests of Stage II lenses surpass the original performance estimates and tests conducted in the MSFC Laboratory for Structural Biology indicate that the brightness of lens/x-ray source combinations should exceed by a factor of 160 those currently obtainable by a Rigaku RU200 rotating anode.

3. Low power systems operating at 37 watts have been demonstrated to exceed brightness of the current rotating anode generator by a factor of 2. The low power requirements and high brilliance of this prototype system have important implications for the development of a future orbiting diffraction facility on Space Station (i.e., feasibility has now been demonstrated), and in addition, also has applications in numerous other areas including medical imaging.

STUDENTS FUNDED UNDER RESEARCH:

TASK INITIATION: 4/93   EXPIRATION: 3/96
PROJECT IDENTIFICATION: 963-40-20
RESPONSIBLE CENTER: MSFC
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III. MSAD Program Tasks — FY1995

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Bibliographic Citations


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Bibliographic Citations


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Bibliographic Citations


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Bibliographic Citations


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Lochhead, R.Y., and Rulison, C.J. "An investigation of the ability of poly(acrylic acid) microgels to stabilize oil-in-water emulsion systems under shear flow conditions."


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Bibliographic Citations


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ATD

III. MSAD Program Tasks — FY1995


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Weiland, K.J. Imaging invisible flames without additives. NASA Tech Brief, (Accepted, 1995).
Appendix

A  Table of Acronyms ......................................................... A-1
B  Guest Investigator Index .............................................. B-1
C  Principal Investigator Index ......................................... C-1
The following list of acronyms, though by no means complete, includes those used in this document as well as some that are often found in text associated with Microgravity Science and Applications research and which may be encountered when reviewing references cited in the bibliography herein.

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<tr>
<th>Acronym</th>
<th>Description</th>
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<tbody>
<tr>
<td>AADSF</td>
<td>Advanced Automated Directional Solidification Furnace</td>
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<tr>
<td>AAL</td>
<td>Aero-acoustic Levitation</td>
</tr>
<tr>
<td>ACRT</td>
<td>Accelerated Crucible Rotation Technique</td>
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<tr>
<td>AO</td>
<td>Announcement of Opportunity</td>
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<tr>
<td>AOMS</td>
<td>Advanced Optical Monitoring Systems</td>
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<tr>
<td>APCF</td>
<td>Advanced Protein Crystallization Facility</td>
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<td>APCG</td>
<td>Advanced Protein Crystal Growth</td>
</tr>
<tr>
<td>ATD</td>
<td>Advanced Technology Development</td>
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<tr>
<td>BDPU</td>
<td>Bubble, Drop and Particle Unit</td>
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<tr>
<td>BTF</td>
<td>Biotechnology Facility</td>
</tr>
<tr>
<td>CADAP</td>
<td>Computer-Aided Dendrite Analysis Program</td>
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<tr>
<td>CAST</td>
<td>Casting and Solidification Technology</td>
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<td>CBE</td>
<td>Chemical Beam Epitaxy</td>
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<td>CCD</td>
<td>Charge-coupled Device</td>
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<tr>
<td>CFLSE</td>
<td>Critical Fluid Light Scattering Experiment</td>
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<tr>
<td>CFTE</td>
<td>Critical Fluid Thermal Equilibration Experiment</td>
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<tr>
<td>CFVME</td>
<td>Critical Fluid Viscosity Measurement Experiment</td>
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<td>CGF</td>
<td>Crystal Growth Furnace</td>
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<tr>
<td>CM-1</td>
<td>Combustion Module</td>
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<tr>
<td>CNES</td>
<td>Centre Nationale d’Études Spatiales [The French National Center for Space Studies]</td>
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<tr>
<td>CoDR</td>
<td>Conceptual Design Review</td>
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<tr>
<td>CPF</td>
<td>Critical Point Facility</td>
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<td>CSA</td>
<td>Canadian Space Agency</td>
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<td>CVD</td>
<td>Chemical Vapor Deposition</td>
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<td>CVTE</td>
<td>Chemical Vapor Transport Experiment</td>
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<tr>
<td>CW</td>
<td>Continuous Wave</td>
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<tr>
<td>DARA</td>
<td>Deutsche Agentur für Raumfahrtangelegenheiten [German Space Agency]</td>
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<tr>
<td>DARFire</td>
<td>Diffusive and Radiative Transport in Fires</td>
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<tr>
<td>DLR</td>
<td>The German Aerospace Research Establishment</td>
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<tr>
<td>DPM</td>
<td>Drop Physics Module</td>
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<tr>
<td>DSC</td>
<td>Differential Scanning Calorimetry</td>
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<td>DSF</td>
<td>Directional Solidification Furnace</td>
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<tr>
<td>DTA</td>
<td>Differential Thermal Analysis</td>
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<tr>
<td>EDM</td>
<td>Engineering Development Model</td>
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Appendix A: MSAD Program—FY1995

Table of Acronyms

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<tr>
<th>Acronym</th>
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<tbody>
<tr>
<td>EDS</td>
<td>Energy Dispersive Spectroscopy</td>
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<td>EHD</td>
<td>Electrohydrodynamic</td>
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<tr>
<td>ESA</td>
<td>European Space Agency</td>
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<td>FDSMM</td>
<td>Fluid Dynamics and Solidification of Metallic Melts</td>
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<tr>
<td>FES</td>
<td>Fluids Experiments System</td>
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<tr>
<td>FES/VCGS</td>
<td>Fluid Experiment/Vapor Crystal Growth System</td>
</tr>
<tr>
<td>FFEU</td>
<td>Free-Flow Electrophoresis Unit</td>
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<tr>
<td>FFDF</td>
<td>Fluid Physics and Dynamics Facility</td>
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<tr>
<td>GaAs</td>
<td>Gallium Arsenide</td>
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<tr>
<td>GAS Can</td>
<td>Get-away Special Canister</td>
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<tr>
<td>GBX</td>
<td>Glovebox</td>
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<tr>
<td>GFFC</td>
<td>Geophysical Fluid Flow Cell</td>
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<tr>
<td>HRT</td>
<td>High-Resolution Thermometer</td>
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<tr>
<td>ICE</td>
<td>Interface Configuration Experiment</td>
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<tr>
<td>IDGE</td>
<td>Isothermal Dendritic Growth Experiment</td>
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<tr>
<td>IML</td>
<td>International Microgravity Laboratory</td>
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<td>IWG</td>
<td>Investigator Working Group</td>
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<td>JPL</td>
<td>Jet Propulsion Laboratory</td>
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<tr>
<td>JSC</td>
<td>Johnson Space Center</td>
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<td>KSC</td>
<td>Kennedy Space Center</td>
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<td>LaRC</td>
<td>Langley Research Center</td>
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<td>LDV</td>
<td>Laser Doppler Velocimetry</td>
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<tr>
<td>LeRC</td>
<td>Lewis Research Center</td>
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<tr>
<td>LIF</td>
<td>Large Isothermal Furnace</td>
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<td>LPE</td>
<td>Lambda Point Experiment</td>
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<tr>
<td>MCF</td>
<td>Modular Combustion Facility</td>
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<tr>
<td>MEPHISTO</td>
<td>Matériel pour l'Étude des Phénomènes Intéressants de la Solidification sur Terre et en Orbite [Interesting Phenomena of Solidification on Earth and in Orbit]</td>
</tr>
<tr>
<td>MGM</td>
<td>Mechanics of Granular Materials</td>
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<tr>
<td>MMSL</td>
<td>Microgravity Materials Science Laboratory</td>
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<tr>
<td>MP</td>
<td>Microgravity Pressure</td>
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<tr>
<td>MPA</td>
<td>Microgravity Pressure, Ambient</td>
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<tr>
<td>MSA</td>
<td>Microgravity Science and Applications (Program)</td>
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<td>MSAD</td>
<td>Microgravity Science and Applications Division</td>
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<td>MSFC</td>
<td>Marshall Space Flight Center</td>
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<td>MSL</td>
<td>Microgravity Science Laboratory</td>
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<td>NASA</td>
<td>National Aeronautics and Space Administration</td>
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<td>NASDA</td>
<td>National Space Development Agency of Japan</td>
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### Table of Acronyms

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<th>Acronym</th>
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<td>NIST</td>
<td>National Institute for Standards and Technology</td>
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<td>NLO</td>
<td>Nonlinear Optical</td>
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<td>NRA</td>
<td>NASA Research Announcement</td>
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<td>NRC</td>
<td>National Research Council</td>
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<td>OARE</td>
<td>Orbital Acceleration Research Experiment</td>
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<tr>
<td>OLMSA</td>
<td>Office of Life Sciences and Microgravity Applications</td>
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<tr>
<td>OMCVD</td>
<td>Organometallic Chemical Vapor Epitaxy</td>
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<td>OPCGA</td>
<td>Observable Protein Crystal Growth Apparatus</td>
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<td>PBE</td>
<td>Pool Boiling Experiment</td>
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<td>PCC</td>
<td>Protein Crystal Growth</td>
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<td>PDA</td>
<td>Phase Doppler Pneumometer</td>
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<td>PIV</td>
<td>Particle Image Velocimetry</td>
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<tr>
<td>PMZF</td>
<td>Programmable Multizone Furnace</td>
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<tr>
<td>POCC</td>
<td>Payload Operations Control Center (at MSFC)</td>
</tr>
<tr>
<td>PVT</td>
<td>Physical Vapor Transport</td>
</tr>
<tr>
<td>RAPIV</td>
<td>Radiographic Particle Image Velocimetry</td>
</tr>
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<td>RDR</td>
<td>Requirements Definition Review</td>
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<td>RPCVD</td>
<td>Remote Plasma-enhanced Chemical Vapor Epitaxy</td>
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<tr>
<td>RSD</td>
<td>Rainbow Schlieren Deflectometry</td>
</tr>
<tr>
<td>RTE</td>
<td>Radiative Transfer Equation</td>
</tr>
<tr>
<td>QSAM</td>
<td>Quasi-steady Acceleration Measurement System</td>
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<tr>
<td>SAA</td>
<td>South Atlantic Anomaly</td>
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<tr>
<td>SAMS</td>
<td>Space Acceleration Measurement Systems</td>
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<td>SCF</td>
<td>Supercritical Fluid</td>
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<td>SEM</td>
<td>Scanning Electron Microscopy</td>
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<tr>
<td>SL</td>
<td>ESA Spacelab (in Space Shuttle cargo bay; e.g., IML and USML)</td>
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<td>SQUID</td>
<td>Superconducting Quantum Interference Detector</td>
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<td>SSCE</td>
<td>Solid Surface Combustion Experiment</td>
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<td>SSFF</td>
<td>Space Station Furnace Facility</td>
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<tr>
<td>STDCE</td>
<td>Surface Tension Driven Convection Experiment</td>
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<td>STEP</td>
<td>Satellite Test of the Equivalence Principle</td>
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<tr>
<td>STS</td>
<td>Space Transportation System</td>
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<tr>
<td>SVP</td>
<td>Space Vehicle Pressure</td>
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<tr>
<td>TEM</td>
<td>Transmission Electron Microscopy</td>
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<tr>
<td>TEMPUS</td>
<td>Electromagnetic Containerless Processing Facility</td>
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<tr>
<td>TGDF</td>
<td>Turbulent Gasjet Diffusion Flames</td>
</tr>
<tr>
<td>TME</td>
<td>Test and Measurement Equipment</td>
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<tr>
<td>TMS</td>
<td>Thermal Maneuvering System</td>
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<td>USML</td>
<td>United States Microgravity Laboratory</td>
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<tr>
<td>Acronym</td>
<td>Description</td>
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<td>---------</td>
<td>--------------------------------------------------</td>
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<tr>
<td>USMP</td>
<td>United States Microgravity Payload</td>
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<td>VCGS</td>
<td>Vapor Crystal Growth System</td>
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<td>VDA</td>
<td>Vapor Diffusion Apparatus</td>
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<td>VIBES</td>
<td>Vibration Isolation in a Box Experiment System</td>
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<tr>
<td>XRD</td>
<td>X-ray Diffraction</td>
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</table>
University of Alabama Guest Institutions (c.f. p. II-11)  
(Note: L. Delucas is PI)

**Academic, Domestic:**

Medical College of Buffalo  
Rutgers University  
University of Alabama, Birmingham  
University of Houston  
University of Minnesota  
University of Texas Medical Branch at Galveston

**Academic, Foreign:**

Kyoto University (Japan)  
McMaster University (Canada)  
University of Hamburg (Germany)  
University of Saskatchewan (Canada)  
University of Toronto (Canada)

**Government, Domestic:**

NASA Marshall Space Flight Center

**Government, Foreign:**

EMBL (Germany)

**Industry:**

Eli Lilly & Company  
SmithKline Beecham Pharmaceuticals  
Bristol-Myers Squibb  
Schering Plough Research Institute  
Vertex Pharmaceuticals
University of Alabama Guest Institutions (c.f. p. II-11)

(Note: L. Delucas is PI)
## Principal Investigator Index

<table>
<thead>
<tr>
<th>Name</th>
<th>Institution</th>
<th>Office Ph</th>
<th>Fax</th>
<th>Page Number</th>
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<tbody>
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<td>904 392-6359</td>
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<td>S. Levy Incorporated 3425 South Bascom Avenue Campbell, CA 95008-4870</td>
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<td>408 371-6804</td>
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<tr>
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<td>405 744-5796</td>
<td>405 744-6811</td>
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<tr>
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<td>405 325-1088</td>
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<td>805 893-4170</td>
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<tr>
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<td>205 895-6050</td>
<td>205 895-6791</td>
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<tr>
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<td>Department of Chemistry University of Tennessee 575 Buehler Hall Knoxville, TN 37996-1600</td>
<td>423-974-3399</td>
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<td>Mr. A. P. Allan</td>
<td>University of Delaware 1200 Sycamore Street Wilmington, DE 19805</td>
<td>302 757-1991</td>
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<tr>
<td>Prof. Robert A. Altenkirch</td>
<td>Dean, College of Engineering and Architecture Washington State University 146 Dana Hall Pullman, WA 99164-2714</td>
<td>509 335-5593</td>
<td>509 335-9608</td>
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<tr>
<td>Prof. C. D. Andereck</td>
<td>Department of Physics</td>
<td>Ohio State University</td>
<td>614 292-2360</td>
<td>614 292-7557</td>
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<tr>
<td>Prof. John L. Anderson</td>
<td>Department of Chemical Engineering</td>
<td>Carnegie Mellon University</td>
<td>412-268-6986</td>
<td>412-268-7139</td>
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<td>Dr. Timothy J. Anderson</td>
<td>Department of Chemical Engineering</td>
<td>University of Florida</td>
<td>904-392-0882</td>
<td>904-392-9513</td>
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<tr>
<td>Dr. J. B. Andrews</td>
<td>Department of Materials Science &amp; Engineering</td>
<td>BEC 360, University of Alabama, Birmingham</td>
<td>205-934-8452</td>
<td>205-934-8485</td>
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<tr>
<td>Prof. Robert E. Apfel</td>
<td>Robert Higgin Professor of Mechanical Engineering</td>
<td>Yale University</td>
<td>203-432-4346</td>
<td>203-432-7654</td>
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<tr>
<td>Prof. Stephen Arnold</td>
<td>Department of Physics</td>
<td>Polytechnic University</td>
<td>718-260-3085</td>
<td>718-260-3139</td>
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<tr>
<td>Prof. Arvind Atreya</td>
<td>Department of Mechanical Engineering and Applied Mechanics</td>
<td>University of Michigan</td>
<td>313 747-4790</td>
<td>313 747-3170</td>
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<tr>
<td>Prof. C. T. Avedisian</td>
<td>Sibley School of Mechanical and Aerospace Engineering</td>
<td>Cornell University</td>
<td>607 255-5105</td>
<td>607 255-1222</td>
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<tr>
<td>Prof. Portonovo S. Ayyaswamy</td>
<td>Department of Mechanical Engineering &amp; Applied Mechanics</td>
<td>University of Pennsylvania</td>
<td>215 898-8362</td>
<td>215 573-2065</td>
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Appendix C: MSAD Program — FY1995

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