Microgravity Science and Applications

Program Tasks and Bibliography for FY 1993

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OBJECTIVES AND FOCUS FOR FY1993

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 keystone 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 Sciences and Microgravity 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.

Fiscal year 1993 was an important year for microgravity science in general and for MSAD in particular. Not only was the ground research program enhanced, several new investigators and research areas were added to the program through maturation of experiments in a flight development phase. The on-orbit research carried out in FY 1992 with four microgravity Space Shuttle missions is currently coming to fruition as the results from these missions are gathered and evaluated. Scientific data from the first International Microgravity Laboratory (IML-1), January 1992; the first United States Microgravity Laboratory (USML-1), June 1992; the Japanese Spacelab (SL-J) mission, September 1992; and the first United States Microgravity Payload (USMP-1), October 1992, provided valuable insight into each of the five discipline fields in FY 1993. The processing and evaluation of these results have provided a solid basis for the planning of future microgravity missions, such as USMP-2 scheduled in March 1994 and IML-2 in July 1994, thus beginning the research cycle anew.

This document, NASA Technical Memorandum 4569 [1994], The Microgravity Science and Applications Program Tasks and Bibliography for Fiscal Year 1993 (October 1992 – September 1993), includes research projects funded by the Office of Life Sciences and Microgravity 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 The Bionetics Corporation, and in particular recognizing Dr. Stephen Davison (MSAD task review process administrator), Ms. Celia Griffin (process manager), and Mr. Duke Reiber (data system development and publishing manager) for their lead efforts in the development, compilation and publishing of this report, and for data processing assistance provided by Mr. Bill Wilcox and Ms. Sandra Freeburn. Gratitude is also expressed for significant data processing support at the responsible Centers for MSAD task management, recognizing: Angela Belcastro and Karla Miller, JPL; Leanne Ward, JSC; Pam Williams, LaRC; Janet Jedlicka and Debbie Sedlak, LeRC; and Jessie Emerson and Mary Wood, MSFC.
## FY1993 PROGRAM RESEARCH TASK SUMMARY: Overview Information and Statistics

**Total Number of Principal Investigators:** ................................................................. 196

**Total Number of Co-Investigators:** ................................................................. 268

**Total Number of Bibliographic Listings:** ................................................................. 767
  - Proceedings Papers: ........................................................................................... 111
  - Journal Articles: ............................................................................................... 449
  - NASA Tech Brief Articles: .................................................................................. 6
  - Science/Technical Presentations: ....................................................................... 201

**Total Number of Patents Applied for or Awarded:** ....................................................... 7

**Number of Graduate Students Funded:** ................................................................. 329

**Number of Graduate Degrees Granted Based on MSAD-funded Research:** ............... 61

**Number of States with Funded Research (including District of Columbia):** ................. 32

**FY1993 Microgravity Science & Applications Budget:** ............................................. $170.3 Million

### Microgravity Science & Applications Research Tasks and Types

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<th>Centers, Types of Research</th>
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<th>Flight</th>
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II. Microgravity Science & Applications Program Tasks for FY 1993

- **Flight Research Tasks**
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- **Ground Research Tasks**
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  - Biotechnology ........................................................................ II-168
  - Combustion Science ............................................................... II-216
  - Fluid Physics ........................................................................... II-251
  - Materials Science ................................................................... II-364

- **ATD**
  - Advanced Technology Development ..................................... II-474
Critical Dynamics in Microgravity

**Principal Investigator:** Dr. Robert V. Duncan  
**Co-Investigators:** Talso C.P. Chui, U.E. Israelsson

**Sandia National Laboratory**  
**Jet Propulsion Laboratory (JPL)**

**Task Objective, Description & Significance:**

**Objective:**
The objective 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 DRGT;
   - Explore boundary suppression of fluctuations near criticality;
   - $0.3 \text{nW/cm}^2 \leq Q \leq 10 \mu\text{W/cm}^2$ with no convection in a 2 cm cell.

2. Measure the temperature profile very near, and through, the HeI-HeII interface:
   - Measure $\Delta T_s(Q)$ and explore "supercooled" HeI 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 DS: $w = w_j/\sqrt{Q}$;
   - Measure the superfluid $\Delta T_s$, hence HeII vorticity.

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

**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.

**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 1993:**
DYNAMX was funded for flight definition by the Microgravity Science and Applications Department of NASA, HQ in February 1993. A strong effort is underway to design a thermal conductivity cell capable of making very demanding measurements near the superfluid transition in $^4$He. This new all-aluminum cell is designed to be as
immune as possible to cosmic radiation in low Earth orbit. Two such cells have now been constructed, and extensive thermal modeling has been done to verify that this cell design will not substantially perturb the measurements. Extensive electron beam welding technique has been developed in order to precisely weld aluminum alloys to 99.999% pure aluminum, and to make all niobium welds on small parts of the pressure servo system described below.

In support of this cell development effort, we have been developing a ground-based cryostat, and a four meter tall shielded room, at the University of New Mexico, Department of Physics and Astronomy. This cryostat will use four high-resolution thermometers (HRT) currently under development at JPL. These thermometers will be integrated into the cryostat during the first two weeks of January 1994. Following successful testing of these thermometers the cryostat shall be used to measure (under gravity) the thermal transport properties of 4He near its superfluid transition, using procedures under development for the future microgravity studies. This will provide a direct test of the adequacy of the new cell design to specially resolve very small temperature changes near the superfluid transition. Although a full study of these predicted nonlinear thermal transport effects very close to the superfluid transition shall require microgravity conditions. These ground-based measurements shall be used to both verify the cell, and to demonstrate that Earth's gravity creates the only remaining experimental limitation. Machining of the DYNAMX cryostat is nearly complete and its assembly and wiring has started. The large, home-brew shielded room at UNM is complete, and the Dewar and mu-metal shield have been installed in it. The next major step shall be the HRT integration in January. All room temperature electronics, including thermometry bridges, cabling, and controllers have been assembled and tested. The main data acquisition computer is on order. It will be identical to the computer used by JPL during the HRT development.

An ultra-precise pressure servo system is under development in collaboration with Dr. Martin Barmatz at JPL. This pressure servo consists of a 70 μm thick Nb superconducting membrane with the helium pressure on one side, and with magnetic (Meissner Effect) pressure on the other. The helium pressure may be controlled by varying the magnetic pressure, and the helium pressure is detected by the variation in the membrane's displacement from equilibrium, which is read out with a SQUID. The machining of this pressure servo system is nearly complete. It shall be installed within Dr. Barmatz's cryostat at JPL, permitting the pressure servo testing to be conducted in parallel with the flight definition studies in the DYNAMX cryostat.

In addition to the technical activities described above, the principal investigator has been involved with staff at JPL funded through DYNAMX on activities related to the preparation of the Pre-SCR and the SCR. One unscheduled review, namely the Pre-SCR, has been requested by NASA HQ. There has been a minimal amount of work spent on project management details and initial flight engineering concerns which must be considered early in the flight definition process. Clearly the DYNAMX project is science driven; however, basic engineering constraints must be clearly understood early in the design process.

**Students Funded Under Research:**

PhD Students: 1

**Task Initiation:** 1/93  **Expiration:** 12/95

**Project Identification:** 694-24-04-04

**Responsible Center:** JPL

**Bibliographic Citations for FY 1993:**

**Journals:**

II. MSAD Program Tasks — Flight Research

Discipline: Benchmark Science

Presentations:
Critical Dynamics of Fluids

Principal Investigator: Prof. Richard A. Ferrell

University of Maryland

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

Task Objective, Description & Significance:

Objective:
The objective is to advance the understanding of the approach to equilibrium in near critical fluids.

Description:
The work involves three critical fluid investigations: 1) ground-based definition of an acoustic attenuation in a pure liquid-vapor system space experiment, 2) principal investigator activities for a fast adiabatic thermal/density relaxation space experiment to fly on IML-2 in 1994, and 3) theoretical work on second sound attenuation near the lambda transition of $^4$He.

Items 1 and 2 are experimental with lab work occurring at NASA Lewis Research Center. Item 3 is covered entirely by the principal investigator with the aide of a post-doc, a graduate student, and a visiting faculty fellow.

Progress During FY 1993:

1. Reviewed CFTF IML-1 results in the context of the Onuki-Ferrell adiabatic fast equilibration description. It is concluded that more data is needed to establish the basis for theoretical investigation.

2. Submitted to Physics A a paper extending the adiabatic fast equilibration theory to systems experiencing temperature and pressure pulses along with the consequences for sound attenuation and dispersion in very compressible critical fluids.

3. Supported cell design for IML-2 AFEQ experiment.

Students Funded Under Research:

PhD Students: 1

Task Initiation: 12/92
Expiration: 12/95
Project Identification: 694-24-05-15
Responsible Center: LeRC
Critical Fluid Light Scattering Experiment (CFLSE)

PRINCIPAL INVESTIGATOR: Prof. Robert W. Gammon
University of Maryland

CO-INVESTIGATORS:
No Co-I's Assigned to this Task

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

OBJECTIVE:
The goal of the Critical Fluid Light Scattering Experiment (ZENO) is to measure, in a low-gravity environment, the decay rate and correlation length of density fluctuations in xenon very near (1 K to 100 μK above) the liquid-vapor critical point.

RESEARCH APPROACH:
The measurement of decay rates and correlation lengths will be achieved by using laser light scattering (time correlation spectroscopy) and turbidity measurements. Light is scattered from a near-critical fluid due to density fluctuations, which increase in scale as the critical temperature is approached, and the fluid exhibits a characteristic phenomenon known as critical opalescence. The low-gravity environment of USMP-2 and the precision of both optical alignment and temperature control will allow measurements to be made much closer to the critical temperature than is obtainable on Earth.

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.

PROGRESS DURING FY 1993:
The engineering development model (EM) was completed by Ball Aerospace and delivered to the University of Maryland in January 1992. The procurement and fabrication of flight hardware were completed in FY93. All elements had been integrated except the thermostat and sample cell from the University of Maryland. Functional checkout of the system electronics showed noise levels are below requirements.

The thermostat design was modified and verified to allow for STS launch survivability. The design of optical mounts was modified and verified to resolve alignment stability problems. Groundbreaking measurements of optical heating effects were made by University of Maryland. A concept to allow for dual laser intensities at the sample cell was developed and is being implemented. A flight-worthy sample cell was constructed. The hardware development schedule is consistent with flight on USMP-2 on February 8, 1994.

STUDENTS FUNDED UNDER RESEARCH:
Total Students: 5  Total Degrees: 1

TASK INITIATION: 12/88  EXPIRATION: 2/95
PROJECT IDENTIFICATION: 694-24-05-02
RESPONSIBLE CENTER: LeRC
BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:


Heat Capacity Measurements Near the Lambda Point of Helium

Principal Investigator: Prof. John A. Lipa
Stanford University

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

Task Objective, Description & Significance:

Objective:
Central to condensed-matter physics is the phenomenon of second-order phase transitions. These come about when a wide class of interactive terms is added to the simple ideal gas picture of matter. To understand condensed matter in general, it is necessary to address the phase transition issue, since phase transitions are involved in nearly all interesting effects observed. Our goal is to perform the most stringent test currently feasible of the present theory of second-order phase transitions in the asymptotic limit as the transition is approached. To do this we will measure the heat capacity of helium very close to its lambda transition at 2.177 K.

Description:
To perform the heat-capacity measurements, two main requirements must be met: first, we must have sufficient temperature resolution to establish the temperature scale, and second, we need to control the energy input to the sample to determine its heat capacity.

To these ends we have been developing a new high-resolution thermometer and an advanced, multilayer thermal control system. The thermometer makes use of superconducting technology to achieve a resolution of about $3 \times 10^{-10}$ K in a 1-Hz bandwidth, and the thermal control system can achieve a power resolution approaching $10^{-12}$ W.

Significance:
These two systems give us the capability to make measurements to the limits imposed by the Space Shuttle environment. A third requirement is to achieve an operating temperature near the lambda point. To do this we make use of the superfluid helium research facility previously flown on Spacelab-2 by JPL.

Progress During FY 1993:
Launch occurred on October 22, 1993. The experiment operated flawlessly throughout the mission. All systems, including the High Resolution Thermometers (HRTs) were found to operate perfectly after launch. Close to 100 high resolution heat capacity sweeps across the lambda point were performed which promise to yield outstanding science results. The influence of cosmic ray events was found to be slightly worse than expected, resulting in a larger than expected thermometer noise, while the impact of passing through the South Atlantic Anomaly (SAA) and the influence of acceleration noise was less than predicted. Heat capacity data was collected from 20 mK below the transition to 1 microkelvin above. The data above the transition constitutes a bonus which was not guaranteed prior to launch. It will yield important new information about the thermal conductivity in the normal phase in a region totally inaccessible on the ground.

The operation of the experiment was initially in the automatic mode with little commanding from the ground. After the transition region was reached, and the lambda temperature was well characterized, the science team began operating the instrument almost entirely in the interactive mode, with commands being sent up from the ground to
optimize data collection and initiate heat capacity data sequences during periods of minimum disturbance. More than 5,000 commands were successfully uplinked to the experiment.

The experiment also quantified the heating effects of passing through the SAA at two different orbiter altitudes. The detailed mapping of the radiation environment of the shuttle orbit by the LPE charged particle monitors will serve as an important guide for operations planning of future planned low temperature experiments and other investigations.

Experiment data analysis was nearing completion at the end of FY '93. After developing models accounting for calorimeter and thermometer effects, the lambda-point location was consistently located to within 0.3 nano-kelvin. The data is now being prepared for averaging in order to extract the heat capacity exponent. Thermal conductivity data collected above the lambda-point was consistently analysed to within 10 nano-kelvin of the lambda-point. These thermal conductivity results represent a significant enhancement in the measurement of dynamic properties near a critical point.

<table>
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<th>STUDENTS FUNDED UNDER RESEARCH:</th>
<th>TASK INITIATION: 10/86</th>
<th>EXPIRATION: 12/93</th>
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| RESPONSIBLE CENTER: JPL |

**BIBLIOGRAPHIC CITATIONS FOR FY 1993:**

**Journals:**


Confined Helium Experiment (CHeX)

Principal Investigator: Prof. John A. Lipa

Stanford University

Co-Investigators:
T.C.P. Chui
U.E. Israelsson
F.M. Gasparini

Jet Propulsion Laboratory (JPL)

Jet Propulsion Laboratory (JPL)

State University of New York, Buffalo

Task Objective, Description & Significance:

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, it is not possible to vary the film thickness in most cases without totally changing the sample, making it difficult to keep track of intrinsic changes in the parameters. Near the lambda transition of helium the correlation length diverges, magnifying the effects of the confinement while simultaneously decreasing the importance of substrates. 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°C below the transition the correlation length is about 0.1 mm. If we can access the temperature 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.

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.

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 1993:
In the past year, most of the effort has been centered on rebuilding the Lambda Point Experiment (LPE) flight instrument and computer system to meet the needs of the new experiment. All the calorimeter parts have been made and are awaiting the completion of plate stack that is used to generate the confinement effect. This stack consists of about 250 silicon wafers, each of which is 100 microns thick and 4 cm in diameter, separated by 100 micron spacers. Difficulties were encountered getting the required separation uniformity, but these have been overcome. The engineering model of the flight computer is now under test and software development is proceeding. The lifetime of the liquid helium cryostat has been extended to meet the new launch hold criterion. A new cryopump capable of substantially improving the vacuum in the instrument has been built and is under test.
II. MSAD Program Tasks — Flight Research

STUDENTS FUNDED UNDER RESEARCH:

PhD Students: 1

TASK INITIATION: 12/92   EXPIRATION: 12/97

PROJECT IDENTIFICATION: 694-24-04-02

RESPONSIBLE CENTER: JPL
Studies in Electrohydrodynamics

**Principal Investigator:** Dr. Dudley A. Saville

**Co-Investigators:**

No Co-I's Assigned to this Task

**Task Objective, Description & Significance:**

**Objective:**
The purpose of this work is to test and extend models of electrohydrodynamic processes involving fluid interfaces. Particular attention is given to the behavior of fluid globules and cylinders in systems with poorly ionized solutes at high (applied) field strengths.

The long-term objective of the research is the development of new ways to manipulate fluids, in particular, fluid interfaces. Externally applied electric fields offer a particularly attractive means of doing this because the electric stress is applied directly to the interfacial region.

**Description:**
The research has involved construction of mathematical models and experiments involving steady or oscillating fields with fluid globules and cylinders. The modeling work is intended to take specific account of charge transport processes. Experimental work involves use of digital imaging and analysis of steady and transient shapes of fluid bodies stressed by steady and transient electric fields.

**Significance:**
A microgravity environment may be necessary to test the theoretical models because experiments on Earth must be carried out with isopycnic systems to avoid sedimentation. The need to use isopycnic systems severely limits the range of fluid properties and phenomena that can be studied to test and extend theory.

**Progress During FY 1993:**
The sample cell and high voltage electronics were used to study the behavior of a fluid cylinder stressed by an axial field. We had found that an axial field can either stabilize or destabilize the cylinder, depending on the properties of the two fluids. For example, using an electric field we were able to stabilize a cylinder whose length is more than 7 times as large as its radius; recall that the plateau limit is 3.14. An extensive study was carried out with both steady and oscillatory fields and a paper describing our work was accepted for publication in the *Physics of Fluids A*. We are currently analyzing our results in the context of G. I. Taylor's leaky dielectric model.

Our proposal to continue the work and prepare a science requirements document for a flight experiment was approved for funding.

**Students Funded Under Research:**

Total Students: 1

**Task Initiation:** 3/92  
**Expiration:** 4/95

**Project Identification:** 674-24-05-25  
**Responsible Center:** LeRC
II. MSAD Program Tasks — Flight Research

**Critical Fluid Thermal Equilibrium Dynamics**

**Principal Investigator:** Dr. R. Allen Wilkinson  
**Co-Investigators:**  
Dr. R. Berg, National Institute of Standards and Technology (NIST)  
Dr. M. Moldover, National Institute of Standards and Technology (NIST)  
Prof. R. Gammon, University of Maryland  
Prof. J. Straub, Technical University of Munich, DE

**Task Objective, Description & Significance:**

**Objective:**
The objective of this study is to examine the thermal relaxation and density profile versus time, after a temperature perturbation, of SF$_6$ near its liquid-vapor critical point, in a low-gravity environment.

**Description:**
The SF$_6$ sample is to be observed using interferometry, visualization, and transmission. The scientific objectives are, first, to observe or determine the following: large-phase domain homogenization with and without stirring of the fluid; time evolution of heat and mass transfer after a temperature step is applied; phase evolution and configuration after transition from one-phase equilibrium to a two-phase state; and the effects of stirring on a two-phase low-gravity configuration; and secondly, to quantify the mass and thermal time constant of a one-phase system under logarithmic temperature steps.

**Significance:**
Critical-point experiments generally depend on achieving thermal equilibrium to within a specified tolerance and on knowing how phases develop or disappear. Data from this experiment will be used to determine the practical time scale needed to execute meaningful critical fluid space experiments and to characterize the location and dynamics of density or phase domains within the sample.

**Progress During FY 1993:**
The fiscal year began with a flurry of mission timelines and mission training. The CITF experiment successfully flew on the European Space Agency's Critical Point Facility aboard IML-1 in January 1992. The experiment logged 60 hours of digital and videotaped interferometric fringes images, which provide a changing density map following a heat pulse.

Following the mission, activity was directed at an analysis of the information contained in these fringe patterns. A commercial software package was adapted to automate the location of fringe centers and to characterize the fringe patterns. Analytical fitting functions mapped the phase of the fringe patterns. The amplitude of the phase was tracked in time and was found to undergo exponential decay. The time constant for this decay provides a simple parameter to describe the slowing down of heat diffusion as the critical point is approached to within a few milliKelvin. Preliminary results agree with theoretical predictions and the earlier 1-g measurements. The software techniques used were proven to be viable for this type of analysis.
II. MSAD Program Tasks — Flight Research

Task Initiation: 1/89  Expiration: 9/92
Project Identification: 694-24-05-06
Responsible Center: LeRC

Bibliographic Citations for FY 1993:

Presentations:
II. MSAD Program Tasks — Flight Research

Protein Crystal Growth in a Microgravity Environment

PRINCIPAL INVESTIGATOR: Dr. Charles E. Bugg

CO-INVESTIGATORS:
L.J., DeLucas
L.C. Cook

University of Alabama, Birmingham

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

OBJECTIVE:
The objectives of this research are: to produce larger, high quality protein crystals in microgravity; and to understand the dynamics and processes of protein crystal growth. The research will utilize the protein crystal growth (PCG) vapor diffusion apparatus (VDA) flight hardware that will be capable of determining protein molecular structures for applications in medicine, drug design, agriculture, and the biological sciences.

DESCRIPTION:
The flight investigations utilized proteins from a number of co-investigators at universities and pharmaceutical companies. These proteins selections were based on a number of criteria, including: availability of extensive ground-based, 1-g visual and x-ray data; crystal growth time; crystal stability; and inherent problems with crystal quality that might benefit from exposure to a microgravity environment (e.g., crystal size, internal crystal quality, crystal sedimentation, etc.).

Proteins were selected by a committee composed of co-investigators from academia, industry, and NASA's Marshall Space Flight Center (MSFC). Several months of ground-based experiments were performed in flight-like hardware by each investigator whose protein had been selected for upcoming space shuttle flights. Crystallization conditions were optimized in this hardware in an effort to maximize the success rate during the flight experiment.

SIGNIFICANCE:
During the 1992 time frame, protein crystal growth experiments were flown on IML-1, SL-J, and USML-1. For the first United States Microgravity Laboratory mission, new hardware was developed that utilized the Glovebox flown in the Spacelab module. The Glovebox hardware allowed, for the first time, optimization of protein crystal growth experiments and provided better mixing procedures for viscous protein/precipitant solutions.

In addition, a micromanipulator system was developed to allow seeding experiments and crystal mounting procedures to be undertaken. A special adapter for the Glovebox video system, as well as a binocular microscope, allow crystals observed by the crew to be downlinked for analysis by co-investigators in the POCC.

PROGRESS DURING FY 1993:
Through the series of missions flown in 1992, and in conjunction with the co-investigator group, it has been demonstrated that the microgravity environment provided by the space shuttle can enhance the size and/or quality of protein crystals in a significant fashion. Resolution enhancements as great as 0.6 Å have been observed, and, in several instances, crystal volume increased twofold. In addition, it was clearly demonstrated on USML-1 that crystal optimization can increase the success of these experiments and that crystal mounting via a micromanipulator is straightforward, thereby providing a method to collect data on crystals once an x-ray facility is available on Space Station Freedom.
Ground-based work carried out at UAB, MSFC, and MSU has continued to provide a better understanding of the fundamental mechanisms involved during crystal nucleation and subsequent growth phases. Using both a hanging drop (vapor diffusion) configuration or batch/temperature-induced crystallization system, it has been demonstrated that static light scattering can be an effective tool to detect the onset of crystal nucleation so that the growth phase can be dynamically controlled.

**BIBLIOGRAPHIC CITATIONS FOR FY 1993:**

**Journals:**


II. MSAD Program Tasks — Flight Research


Proceedings:


PATENTS:
Patent Status: Pending U.S. Patent #: Undetermined
DeLucas, L.J., and Volanakis, J. "Crystals of Factor D."

Patent Status: Pending U.S. Patent #: Undetermined

Patent Status: Pending U.S. Patent #: Undetermined

Patent Status: Pending U.S. Patent #: Undetermined
II. MSAD Program Tasks — Flight Research

Protein Crystal Growth Vapor-Diffusion Flight Hardware and Facility

PRINCIPAL INVESTIGATOR: Dr. Daniel C. Carter  
NASA Marshall Space Flight Center (MSFC)

CO-INVESTIGATORS:
X. He  
Universities Space Research Association (USRA)
T. Miller  
NASA Marshall Space Flight Center (MSFC)

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

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.

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 (RIM) and thermal environment system (TES) temperature control hardware 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.

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 1993:
Preliminary studies were conducted with plastic vapor diffusion plates available from CrysChem Corporation. Several experiments have been conducted which allow the individual vapor-diffusion experiments to approach nucleation before flash-freezing to -150 °C. Vibration tests were conducted at the Marshall Space Flight Center test lab facility on a tray fully loaded with protein samples under conditions equivalent to those currently experienced by the VDA and RIM in the middeck lockers.

The crystallization conditions for human serum albumin have been thoroughly investigated over the past six years. A complete atomic model is currently in the refinement stage. Optimized experimental conditions for the VDA and VDT have been determined in the past, and crystals have been successfully obtained on all occasions.
II. MSAD Program Tasks — Flight Research

Discipline: Biotechnology

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TASK INITIATION: 05/93  EXPIRATION: 11/97
PROJECT IDENTIFICATION: 694-23-08-08
NASA CONTRACT NO.: In-house
RESPONSIBLE CENTER: MSFC

BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:


Books:

PATENTS:
Patent Status: Pending U.S. Patent #: Undetermined
II. MSAD Program Tasks — Flight Research

Discipline: Biotechnology

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, DESCRIPTION & SIGNIFICANCE:

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.

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.

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 1993:
During the past year, this laboratory has made significant progress in developing a system that utilizes laser light scattering to detect the onset of nucleation in both the hanging drop and a batch crystallization configuration. In addition, we have developed software to control the vapor equilibration process using dry nitrogen, a single humidity sensor that is multiplexed to ten crystallization chambers, an individual TEU temperature control system, and a software-driven system that will dynamically control the rate and time for temperature changes based on information gathered via the light scattering system. The next step will be to utilize these ground-based systems to grow crystals of a variety of different commercially available proteins so that ground-based data can be obtained to verify the beneficial effects, if any, of these dynamically-controlled systems.

STUDENTS FUNDED UNDER RESEARCH:
MS Students: 1
PhD Students: 3

MS Degrees: 1

PHD Students: 3

TASK INITIATION: 04/93  EXPIRATION: 03/98
PROJECT IDENTIFICATION: 694-23-08-06
NASA CONTRACT NO.: NAS8-397
RESPONSIBLE CENTER: MSFC

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II. MSAD Program Tasks — Flight Research

Discipline: Biotechnology

BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:


Proceedings:


PATENTS:

Patent Status: Pending U.S. Patent #: Undetermined
DeLucas, L.J., and Volanakis, J. "Crystals of Factor D."

Patent Status: Pending U.S. Patent #: Undetermined

Patent Status: Pending U.S. Patent #: Undetermined
II. MSAD Program Tasks — Flight Research

Discipline: Biotechnology

Crystal Observation System (COS) and Advanced Crystal Observation System (A/COS)

Principal Investigator: Dr. Lawrence J. DeLucas
University of Alabama, Birmingham

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

Task Objective, Description & Significance:

Objective:
The Crystal Observation System (COS) provides a means for undisturbed visual observation of protein crystals that are growing in a smaller version of the Vapor Diffusion Apparatus (VDA), which is currently being flown on space shuttle missions. Visual observation of the growing crystals will provide valuable information on the physical aspects and the kinetics of protein crystal growth in a microgravity environment. Video will also enable scientists to observe the mixing of solutions in microgravity and design new or improved mixing geometries to provide improved mixing of solutions.

In the Advanced Crystal Observation System (A/COS), a "traveling microscope" arrangement is planned which will allow a large number of crystal growth of cells to be observed without disturbing the growth environment of the crystals. This system will utilize technologies proven in the COS, allowing visual observation of numerous experimental chambers.

Description:
The COS is comprised of the following components: a six-chamber VDA tray, a microscope objective lens and miniature charge-coupled device (CCD) camera for each camera, a primary and back-up camera control unit, camera head switching unit, two voltage regulation circuits, dual input fiber optic light strip, two miniature halogen incandescent lamps, focusing mechanism, a battery pack containing 15 D-cell batteries, and a front panel containing appropriate switches and connectors. The COS will be installed inside a Thermal Enclosure System (TES), which is an upgraded design of the Refrigeration/Incubation Module (RIM). The TES will provide more finite temperature control and significantly-increased experiment volume. In addition, the TES will be hermetically sealed to provide a level of containment which will generally simplify the safety/verification requirements placed on the experiment hardware contained within it.

In the A/COS, one "traveling microscope" will be "sandwiched" between two VDA-2 or L-LDA trays and will be capable of imaging experiments in both trays. It is comprised of the following components: reflective prism, microscope objective lens, and miniature color CCD camera and controller.

Significance:
This system will overcome a limitation of the present VDA hardware and is a necessary step in the development of a fully-automated protein crystal growth system.

The A/COS offers the advantage of observing many more experiments (up to 80 total) compared with the six available in the COS.
II. MSAD Program Tasks — Flight Research

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Progress During FY 1993:
The successfully functioning COS system has been developed as part of an on-going PCG project.

Task Initiation: 05/93  Expiration: 03/96
Project Identification: 694-23-08-06
NASA Contract No.: NAS8-397
Responsible Center: MSFC
Electrophoretic Separation of Cells and Particles from Rat Pituitary and Rat Spleen

**Principal Investigator:** Dr. Wesley C. Hymer  
**Co-Investigators:**  
A. Mastro  
R. Grindeland  
R. Snyder

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

**Task Objective, Description & Significance:**

**Objective:**
The objectives are to separate pituitary cells as well as pituitary, hormone-containing granules by free-flow electrophoresis, using the Japanese Free-flow Electrophoresis Unit (FFEU).

**Description:**
To accomplish these aims it is necessary to: optimize conditions for growing pituitary cells in the Japanese cell-cultural kits; remove cells from the growth chamber and fractionate them by electrophoresis; and lyse the cells grown in the chamber and fractionate the lysate to obtain the hormone-containing fractions. These procedures must be done in such a way as to be executable in flight. It is also necessary to develop the technologies to assay each sample and fraction for growth hormone, prolactin, and heat shock protein (HSP). The hormones will be assayed by immune and bioassays. A screen to detect the HSP immunocytochemically will be developed. Because of limited sample size and because of the buffers required for electrophoresis, procedures used routinely for ground-based research must be modified.

**Significance:**
This experiment is designed to probe the mechanism by which the biological activity of pituitary hormones (growth hormones and prolactin) may be modified by exposure to microgravity.

**Progress During FY 1993:**
In order to be able to separate pituitary cells and their hormone-containing granules by electrophoresis in microgravity, it is necessary to use a specially-developed cell chamber and free-flow electrophoresis unit developed by the Japanese Space Agency for this experiment. In 1992 we established that several of the cell procedures could be used. We were also able to do a limited number of runs with the FFEU unit, which is in Japan.

We established that the cells can be lysed in buffer compatible with electrophoresis and that lysate can be concentrated and separated on the FFEU. Growth hormone and prolactin were each assayable by at least one assay. We also established that pituitary cells could be maintained and recovered from a chamber similar to that to be used with the FFEU. We also found that the culture medium will have to be modified to be compatible for bioassays and electrophoresis as well as for growth.

**Task Initiation:** 12/89  
**Expiration:** 6/95  
**Project Identification:** 694-23-01-01  
**NASA Contract No.:** GN8-953  
**Responsible Center:** MSFC
An Observable Protein Crystal Growth Flight Apparatus

PRINCIPAL INVESTIGATOR: Dr. Alexander McPherson, Jr. University of California, Riverside

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

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

OBJECTIVE:
The task objective was 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).

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.

SIGNIFICANCE:
The efforts described have required the solution of a vast number of technical problems. It is anticipated that the "spin-off" value of many of the novel approaches that have, and are being developed, will alone be of substantial value to the crystallographic community.

PROGRESS DURING FY 1993:

In the past year, more than sixty macromolecules have been screened as potential flight samples and conditions identified for the reproducible crystallization of approximately fifty-five of these. These include three transfer RNA molecules, six different viruses, three different peptides, and forty-three proteins. From this set, plus perhaps as many as a dozen additional candidates, a final ensemble for flight experiment purposes will be defined and their crystallization conditions refined in the next year.

Time lapse video microscopy has been transferred from a tape-based video recorder of low resolution to a computer (PC) controlled system that is capable of recording single frames at a relatively rapid pace with maintenance of quality and resolution, and transferring these directly to an SGI computer at high transfer speed. We are now able to display successive frames in time lapse video mode on an SGI graphics system and accumulate the images in mass storage. We are currently in the process of developing methods and procedures for the editing and improved display of the image stream.

A vibration-isolated optics table has been installed in a specially-prepared laboratory at UCR and the optical components assembled for Mach-Zender interferometry of concentration fields in aqueous fluids. A subsystem for Michelson interferometry of crystal surfaces, to be used for the characterization of crystals and their kinetics of
growth, is currently being constructed. The construction of this optics system, with the guidance and assistance of Teledyne-Brown Engineering (TBE), has only recently been completed, and the first measurements are only now being made at UCR.

An identical optics system to that at UCR was constructed at the beginning of last year in the laboratories of TBE in Huntsville, Alabama. This system will be utilized in parallel with the identical system at UCR and experiments duplicated at the two facilities. Because of the differences in expertise of the UCR and TBE scientists, both will generate ideas, procedures, and results, and each will be cross-checked by the other. The focus in both laboratories will be the design and, ultimately, the fabrication of an observable protein crystal growth apparatus (OPCGA).

Useful measurements providing evidence that the fundamental concepts driving development of OPCGA are technically sound have already been obtained at TBE. Interferometry has been used to measure accurately protein concentration gradients and refractive index changes in solution, and this data is of a quality that presentation in the scientific literature is presently in preparation. The early indications are quite positive in all regards, and we feel confident that in the next year we will indeed be able to demonstrate the technical feasibility of OPCGA.

In addition to the interferometric measurements, a number of inelastic light scattering studies have been carried out at UCR relevant to the kinetics and thermodynamics of macromolecular crystal growth and to the identification of stable nuclear size and intermediate aggregate character. Several papers have now been published detailing these studies, and several more are in press. In particular, we have focused on the larger crystallizable particles, primarily T=1 icosahedral viruses (170nm diameter), and are currently extending the studies to T=3 icosahedral viruses of significantly greater diameter (280nm diameter). Coupled with our studies of proteins of smaller size, we hope to complete an investigation of the entire size range of macromolecular species from 3nm to 280nm.

BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:


II. MSAD Program Tasks — Flight Research

Discipline: Biotechnology


Proceedings:

Enhanced Hybridoma Production Using Electrofusion

PRINCIPAL INVESTIGATOR: Dr. David W. Sammons
University of Arizona

CO-INVESTIGATORS:
No Co-I's Assigned to this Task

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

OBJECTIVE:
The objectives of the hybridoma flight are to determine the extent to which sustained microgravity environment increases electrofusion frequencies and hybridoma yields compared to ground-based controls, and secondly to enhance working knowledge of lymphocyte activation and cell culture.

DESCRIPTION:
The hardware is being developed by Zimmerman et al., in Germany and furnished to Drs. Sammons and Neil under an international agreement. The NASA investigators will furnish B cells for activation and subsequent data analysis. Nonactivated, preselected B cells will be activated in flight and through a high-efficiency process fused to an SP2 cell line for the production of specific hybridomas.

SIGNIFICANCE:
Typically, electrofusion protocols allow the generation of antigen-specific hybridomas with an efficiency of 1 in 10,000, using 106 SP2 cells in the profusion chamber. The object of this experiment is to identify the effect of microgravity on fusion frequency and specificity.

PROGRESS DURING FY 1993:
Investigators completed ground-based investigations and critical design review. The experiment is manifested for STS-55, which will fly in February 1993.
II. MSAD Program Tasks — Flight Research

Discipline: Biotechnology

Electrophoresis Technology

PRINCIPAL INVESTIGATOR: Dr. Robert S. Snyder

NASA Marshall Space Flight Center (MSFC)

CO-INVESTIGATORS:

P. Rhodes
T. Miller
G. Roberts

NASA Marshall Space Flight Center (MSFC)

Roberts Associates, Inc.

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

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.

DESCRIPTION:
The electrophoresis separation process can be considered to be simple in concept, but flows local to the sample filament produced by the applied electric field have not been considered. These electrohydrodynamical flows, formulated by G. 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, while 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 plane 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 will 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 will permit part of the dielectric dependency to be determined. Two different frequencies will be used to vary the dielectric constant of the samples. Samples will not be collected, but the cross-section illuminator will be used to show the sample filament cross section, and the observed shapes will be recorded photographically.

The higher frequency 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.

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
II. MSAD Program Tasks — Flight Research

Discipline: Biotechnology

Scientific phenomena are a critical part of electrophoresis in space. The severity of sample distortion due to dielectric constant variations is not known in the laboratory to date because of the concurrent sample concentration effect.

Progress During FY 1993:

Most activities during FY92 were directed toward adapting our requirements to the RAMSES design. The borate buffer preferred by the European principal investigators (PI's) is not optimum for our polystyrene latex samples. The cross-section illuminator will be a laser rather than single-filament lamp, and this also introduces problems of resolution. We are building a prototype chamber for conducting critical tests. Laboratory experiments have recently showed the dielectric effect on electrohydrodynamics. The separate roles of electrical conductivity and dielectric constant require more precision of measurement than previously suspected, and the failure of earlier experiments to observe the separate contribution of dielectric constant has now been identified.

Task Initiation: 9/89  Expiration: 9/95

Project Identification: 694-23-08-04

NASA Contract No.: In-house

Responsible Center: MSFC

Bibliographic Citations for FY 1993:

Journals:


Presentations:
Scientific Support for an Orbiter Middeck Experiment on Solid Surface Combustion

PRINCIPAL INVESTIGATOR: Prof. Robert A. Altenkirch Mississippi State University

CO-INVESTIGATORS:
No Co-I's Assigned to this Task

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

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 identifies 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.

DESCRIPTION:
The Solid Surface Combustion Experiment (SSCE) is to be built and flown to perform a minimum of eight 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.

SIGNIFICANCE:
The spreading of flames over solid fuels is a fundamental combustion problem that has practical interest in the prevention and control of fires. Flame spreading in normal gravity is usually dominated by buoyant airflow which introduces a significant complexity into fundamental model 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 1993:
The modeling effort in the program has been extensive and productive. A steady state model of flame spreading in opposed flow has been developed that includes a complex calculation of gas-phase radiative interactions between the flame, fuel surface and the surroundings. The model has been modified creating a separate version that is capable of calculating transient behavior, including the effects of a thick fuel. The models provide details of predicted flame spread phenomenon including complete two-dimensional temperature fields, heat transfer estimates and flame spread rates and structure.

The Solid Surface Combustion Experiments has flown aboard the Space Shuttle (aboard each of the four orbiters) a total of six times. The flight missions included four flights in the Shuttle Middeck (STS-41, STS-43, STS-47 and STS-54) and two flights in the Shuttle/Spacelab module (STS-40, and STS-50). Test results for all of the thin-fuel samples and the single thick-fuel sample have been successfully obtained and reduced, providing detailed images of
the spreading flames, time and spatially resolved profiles of flame and fuel temperature, and estimates from the temperature data of the heat transfer between the flame, the fuel and the surrounding environment. These data have been compared to the numerical calculations with substantial agreement. However, some differences have been identified that have motivated further theoretical work.

Modifications in the numerical model have been made to account for the differences between the computational and flight test results including extending the computational domain to include the region beyond the tail of the flame. This modification was necessary in order to calculate additional mass diffusion in that region that finally accounted for the observed flame shapes. The model has confirmed the hypothesis of the importance of radiative heat transfer effects to accurate prediction of experimental results. Differences between fuel temperatures calculated for the thick fuel and the flight test results have been attributed to uncertainty in either the knowledge of the fuel pyrolysis kinetics or the correct interpretation of the flight data.

| STUDENTS FUNDED UNDER RESEARCH: |
| PhD Students: 2 | MS Degrees: 2 | TASK INITIATION: 12/84 | EXPIRATION: 12/94 |
| PhD Degrees: 2 | PROJECT IDENTIFICATION: 694-22-05-01 | RESPONSIBLE CENTER: LeRC |

BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:


Presentations:


II. MSAD Program Tasks — Flight Research

Discipline: Combustion Science

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

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

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

Task Objective, Description & Significance:

Objective:
The objective of the work is to uncover the underlying physics of the mechanisms that cause flames to propagate over solid fuels against a low velocity of oxidizer flow in a low-gravity environment, where flame and fuel surface radiative effects and details of the diffusional processes are important.

Specifics objectives are:

1. To analyze experimentally observed steady-flame shapes, measured gas-phase field variables, spread rates, radiative characteristics, and solid-phase regression rates for comparison with theoretical prediction capabilities; and

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

Description:
To meet the objectives of this research program, a sounding rocket experiment package (DARTFire- Diffusive and Radiative Transport in Fires) is under development 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, the external radiant flux directed to the fuel surface, and the oxygen concentration of the environment. A minimum matrix of tests is defined to obtain the information needed to meet the scientific objectives. Experimental results will be compared with unsteady, computational modeling results that are able to track the flame from ignition onward. Through such comparisons, the physics important to the flame spread process may be identified.

Significance:
Although the work is fundamental in nature, it has clear applications to fire safety in space and on Earth.

Progress During FY 1993:

During the past year, the a Delta-CoDR was successfully completed for the sounding rocket concept for DARTFire. The SRD was updated for this review, to address some of the requirements specific to sounding rockets. Additionally, a PIC was held with Wallops Flight Facility. Preparations are underway for an RDR.

At Mississippi State University, the unsteady flame spread model has been used to predict results for each DARTFire matrix point. Flame spread rate, gas phase temperatures, and vaporization temperatures all increase with increasing opposed flow, increasing oxidizer, or increasing radiant heat flux. The computational results have been animated for better comparison with experiment.
At San Diego State University, a code has been developed which will take the species and temperature fields predicted from the steady flame computations (including fuel vapor) and predict emitted infrared intensity fields for direct comparison with the infrared images obtained from the sounding rocket flights. Additionally, the steady code has undergone rigorous parametric studies of the 16 non-dimensional parameters. Lewis number is found to have a dramatic effect on spread, while radiation parameters were found to have a marginal effect on the baseline case of 10 cm/s flow at 50% oxygen and 1.5 atm pressure. Surface radiant loss is found to be important at all values of flow for thick fuels, whereas it was only important at flow velocity for thin fuels. Other parameters (mostly kinetic) were found to have expected effects. SDSU also examine the transition from a thermally-thin fuel to a thermally-thick fuel, and found that as flow decreases, the depth of penetration of the thermal wave increases due to the decreased burning rate, but spread rate is established by gas-phase preheating, not solid-phase forward conduction.

At Lewis, four Learjet test series were conducted this past year to evaluate hardware configurations and diagnostics for the sounding rocket experiment: the xybion intensified array camera, the infrared camera, fuel and radiant heater radiometers, sample width and insulation effects, and radiant heater performance. These were all conducted to demonstrate feasibility for the RDR coming up in December 1993. A journal article on the results from the Learjet testing has been drafted, and the numerical prediction comparison section is currently being added.

"Color" images were generated by superimposing Red, Green and Blue filtered images of the learjet flames agree well with color film images. OH and CH free radical chemiluminescent images were found to agree qualitatively with the visible flame, with OH being present more uniformly over a broader region than CH. Additionally, infrared images of CO, CO₂, H₂O, MMA, and soot were obtained that are consistent with each other and with the color images of the flames. The extent of the product fields was larger than expected. The images were processed to obtain intensity contours for comparison with the model predictions.

In order to better select filters for the infrared camera, a spectrum analysis was performed at LeRC on the burning pmma sample. Once a visibly blue flame at low pressure was established only the typical combustion product emission bands were recorded (CO, CO₂, H₂O); there were no additional bands or background emissions found, which would have corresponded to soot and/or partially-burned hydrocarbons.

Surface temperatures of pmma were successfully measured using a bead-embedding technique. Heat flux calculations with learjet pmma thermocouple data reveal that approximately 1.3 W/cm² is imposed by the leading edge of the flame (@50% O₂), and most of that is Initially directed into solid phase conduction. This agrees well with the range of the radiant heater (0-2 W/cm²). Radiative heat loss increases until the in-depth solid conduction and radiative heat loss terms are of comparable size. The energy required for fuel vaporization is only a fraction of either of these other terms. Thus heat losses dominate the surface energy balance for these conditions.

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**BIBLIOGRAPHIC CITATIONS FOR FY 1993:**

**Journals:**

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

Presentations:


West, J., Bhattacharjee, S., and Altenkirch, R.A. Surface radiative effects on flame spread over thermally thick fuels in an opposing flow, Presented at the ASME Summer 1992 Meeting, also accepted for publication in Journal of Heat Transfer.
Gravitational Effects On Laminar, Transitional, and Turbulent Gas-Jet Diffusion Flames

II. MSAD Program Tasks — Flight Research

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

PRINCIPAL INVESTIGATOR: Dr. M. Yousef Bahadori

Science Applications International Corporation (SAIC)

CO-INVESTIGATORS:

Dr. U.G. Hegde

Sverdrup Technology, Inc.

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

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, and (iii) revealing phenomena which may be masked by buoyant convection.

DESCRIPTION:

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

(a) Investigate 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 will be conducted in normal-gravity and in the ground-based facilities, including the Drop Tower, Zero-Gravity Facility, and/or KC-135 aircraft. 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.

SIGNIFICANCE:
The two parallel studies of parts (a) and (b) will provide a detailed database and associated predictive capabilities to not only understand the structure and characteristics of microgravity laminar/transitional/turbulent gas jet diffusion flames, but also the fundamental processes that govern the observed classical behavior of their normal-gravity counterparts.

PROGRESS DURING FY 1993:

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 efforts: 1. M.Y. Bahadori, Principal Investigator (SAIC); 2. U.G. Hegde, Co-Investigator (Sverdrup Technology); 3. L. Zhou, Postdoctoral Fellow (UC Berkeley); 4. D.P. Stocker, Project Scientist (NASA Lewis).
II. MSAD Program Tasks — Flight Research

During the past year, accomplishments included:
(a) Following the acceptance of the Glovebox-2 proposal on pulsed laminar flames, it was concluded that the experiment could be more appropriately be conducted in a GAS can. Due to this change the investigators were requested to prepare for a formal science review.

(b) This program was selected by NASA as a fast-track space flight project subject to a successful science review, a Requirements Design Review (RDR) scheduled for October 1993.

(c) The Science Requirements Document for the RDR was submitted to NASA Lewis in September 1993.

(d) Tests on high-momentum laminar, transitional, and turbulent gas jet diffusion flames were conducted in the 2.2-Second Drop Tower by L. Zhou. These tests were conducted for a variety of fuels, nozzle sizes, and a wide range of Reynolds numbers extending well into the turbulent regime. The tests provided significant new findings on the effects of buoyancy on laminar-to-turbulent transition of diffusion flames, as well as their turbulent behavior. These included: (i) the dominant effect of large-scale structures on flame development; (ii) significant reduction in the values of effective transport properties in microgravity compared to normal gravity; (iii) extended Reynolds-number range for transitional flames in microgravity; (iv) substantially larger flame lengths (up to twice) in microgravity versus normal gravity for the turbulent regime, even for Froude numbers in excess of 400; (v) smaller stand-off distances in microgravity transitional/turbulent flames (approximately half that in normal-gravity); (vi) significantly higher blow-off Reynolds number in microgravity (approximately 20% higher than that for normal gravity).

(e) The results were presented at the following meetings and generated a lot of interest in the peer community: (i) three papers at the Western States Section of The Combustion Institute, Berkeley, California, October 1992; (ii) one paper at the AIAA 31st Aerospace Sciences Meeting, Reno, Nevada, January 1993; and (iii) two papers at the Joint Central and Eastern States Meeting of The Combustion Institute, New Orleans, Louisiana, March 1993.

(f) In addition to the presentation papers, several articles were submitted to peer-reviewed journals (5), books (1), and proceedings (1). At this time, papers have been accepted by the AIAA Journal (1), Pergamon Press (1), and the Lunar and Planetary Institute (1).

(g) The final report of the previous contract (NAS3-22822) was submitted to NASA Lewis, and appeared as NASA CR-191109 in April 1993.

(h) Work continued on the analytical and numerical modeling of laminar, turbulent, and pulsed flames. An improved radiation model was incorporated in the laminar-flame framework; the results are currently being compared with the measurements of thermal field and radiation data from normal-gravity and microgravity tests, and the preliminary comparisons show satisfactory agreement. Extensive new post-processing capabilities for the computed data have been developed including contour mapping and 3-D projection plots. An analytical model was developed for the pulsed-flames; predictions of flame response and structure characteristics show good qualitative agreement with the transitional flame observations. The numerical model of laminar flames is currently being extended to include the time-dependent capability needed for the pulsed-flame studies. In addition, a k-e-g model is being developed for the study of turbulent diffusion flames under arbitrary gravitational levels.

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II-42
BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:


Proceedings:


Presentations:


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-I's Assigned to this Task

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

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.

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.

SIGNIFICANCE:
The experimental results, combined with theoretical modeling, 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. This will have a significant impact on understanding of the spread of unwanted fires, and on the design of jet engines and large-scale boilers.

PROGRESS DURING FY 1993:
Considerable progress has been made in study of soot processes in weakly buoyant flames. The flames that have been studied are in air at reduced pressure and include acetylene, propylene, propane, butadiene at 1/8, 3/16 and 1/4 atm. Measurements include: thermophoretic sampling, gas sampling, and laser extinction measurement, thermocouple measurements, a traversable detector for 4 color soot pyrometry, imaging 4 color soot pyrometry, traversable and imaging soot volume fraction measurement.

The results show competition between soot nucleation and growth processes in these flames. Initially, growth is as rapid as nucleation so primary particles sizes are quite large but numbers are small. Further up in the flame, nucleation dominates so particle number increases rapidly and particle size decreases slowly. Following this, in the soot oxidation region, primary particle sizes decrease rapidly. At 0.25 atm for acetylene for regions near the flame center, they see slow growth of soot primary sizes from 13 to 15 nm whereas the number of particles per aggregate increases from 120 to 260. The data to date indicate that the effect of pressure on state relationships is small for the pressure range of 0.125-1.000 atm. Evaluation of the soot nucleation and growth mechanisms indicates that they are roughly first and second order, respectively, in acetylene.

Comparison to model predictions have been successful, with laser velocimetry measurements agreeing with model predictions within 10%.
II. MSAD Program Tasks — Flight Research

To verify the imaging diagnostic systems for flight the P.I. conducted a considerable body of tests to compare the imaging soot volume fraction system to a standard, single-point traversable system and to compare imaging multicolor soot pyrometry to thermocouple measurements and to a standard, single-point traversable system.

Use of a flying thermocouple system proved unsatisfactory for regions with large soot concentrations, consequently a traversable detector was used for 4 line soot pyrometry in these regions. The results to date are very encouraging and it appears that the system is now adequately demonstrated for RDR. Using the video camera and 4 different line filters at 632.8, 700, 800 and 900 nm they have deconvoluted the results and calculated the temperature for three wavelength pairs. They have found good agreement between all of the measurements. These measurements also extrapolate nicely with flying thermocouple measurements in soot free portions of the flame.

Testing of the imaging soot volume fraction system was very successful. The imaging system provides superior results to the traversing system that they had been used previously in their laboratory.

Preparations for RDR (in October) are complete with a final version of the SRD distributed to the review panels.

A series of tests on the KC-135 have been conducted to support the flight experiment and to evaluate gas jet smoke height behavior in low-gravity. The results have been very interesting. Contrary to predictions by other workers, smoke heights have been shown to exist for non-buoyant flames and to be quite different from those for normal gravity flames. The tests will continue into the next fiscal year.

STUDENTS FUNDED UNDER RESEARCH:
PhD Students: 3

TASK INITIATION: 2/92  EXPIRATION: 2/98
PROJECT IDENTIFICATION: 694-22-05-04
RESPONSIBLE CENTER: LeRC

BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:

Presentations:


II. MSAD Program Tasks — Flight Research

Fundamental Study of Smoldering Combustion in Microgravity

PRINCIPAL INVESTIGATOR: Prof. A. Carlos Fernandez-Pello University of California, Berkeley

CO-INVESTIGATORS: No Co-I's Assigned to this Task

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

OBJECTIVE:
This project is an experimental and theoretical investigation of the mechanisms of smoldering combustion in porous media, specifically in polyurethane foam. The final objective is a set of flight experiments in a GAS Can facility in the orbiter cargo bay.

DESCRIPTION:
Several types of experiments have been conducted. The majority have been in normal gravity, studying the influence of gravity by changing the direction of smolder propagation with respect to gravity. In addition, experiments have been conducted on NASA's low-gravity aircraft, (the KC-135 at JSC and the Lear Jet at LeRC). To date, the majority of the work has concerned one-dimensional configurations with the smolder front propagation tracked using thermocouples. Study of transition to flaming in three-dimensional configurations has recently begun.

SIGNIFICANCE:
These experiments, combined with ground-based low and normal gravity experiments, will increase the current fundamental understanding of smoldering combustion, and this will have a significant impact on understanding of the initiation of unwanted fires both on the ground and in space.

PROGRESS DURING FY 1993:
Considerable progress has been made in both modeling and experiments. The one-dimensional work was largely completed in 1992. Papers discussing the influence of gravity in these configurations will be submitted in FY 1993. The program successfully passed CoDR in December 1991 and was approved to continue to RDR with an initial test matrix of four tests. These tests will consist of two quiescent tests at different oxygen concentrations and two flow tests at different flow rates.

Owing to the lack of outstanding science or design issues at the CoDR for the Microgravity Smoldering Combustion (MSC) Experiment, a programmatic review was held at Lewis in lieu of RDR. Subsequently, the program successfully passed through PDR.

The final SRD for the MSC experiment has been submitted to and signed by headquarters and the preliminary issue of the Experimental Data Management plan was submitted to headquarters for review.

A graduate student (J. Torero) has completed his Ph.D. dissertation titled "Buoyancy Effects on Smoldering of Polyurethane Foam."

Continued work has been conducted to determine the minimum conditions for smolder ignition and propagation. Among the parameters considered are ignitor temperature and power level, sample diameter, gas flow rate and direction, and sample insulation.
II. MSAD Program Tasks — Flight Research

The conditions for transition to flaming have also been established and work is underway to evaluate the mechanisms that control the process.

The theoretical model has been expanded to consider natural and forced convection smolder and allows correlation of data at different flow rates, including buoyant flows. The resulting smolder velocity is proportional to the buoyant or forced flow velocity for both downward and upward smolder.

In support of the Smoldering Combustion in Microgravity (SCM) glovebox investigation, J. Torero conducted normal-gravity tests in the flight hardware at NASA LeRC in January 1993. The post-combustion gases from the flight and normal-gravity tests were analyzed via GC and GC/MS at NASA JSC. They revealed a dramatically larger production of combustion products, especially carbon monoxide, in all microgravity tests compared to the normal-gravity tests. The polyurethane fuel samples have been cut open and showed significant smolder propagation; this was unexpected based on the low temperature measurements in the tests. The fuel samples and available temperature data gave similar results in both flight and normal-gravity tests, which is somewhat surprising considering the dramatic differences in the gas analyses. The results indicate that the smolder process was primarily controlled by heat losses from the reaction to the surrounding environment.

STUDENTS FUNDED UNDER RESEARCH:

Total Students: 4  Total Degrees: 1

TASK INITIATION: 2/92  EXPIRATION: 2/93

PROJECT IDENTIFICATION: 694-22-05-05

RESPONSIBLE CENTER: LeRC

BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Presentations:

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, Description & Significance:

Objective:
The objective of this work is to study the effects of gravity-induced buoyancy on the combustion limits of premixed gas flames. Four subtasks have been pursued:

1. Radiation effects on premixed gas flames;
2. Flame structure and stability at low Lewis number;
3. Flame propagation and extinction in cylindrical tubes; and
4. Experimental simulation of combustion processes using autocatalytic chemical reactions.

Description:

For task 1, we are studying the effects of the addition of inert, radiant particles to gas mixtures to increase the absorption coefficient of the gas. This enables us to study both the optically thin and optically thick radiation regimes in a single experiment. Drop towers and the KC-135 aircraft are used to obtain microgravity (μg) conditions.

For task 2, we are studying flames in a variety of gas mixtures having low Lewis number (Le) in KC-135 aircraft tests. The goal of these studies is to determine under what conditions, if any, the instabilities which occur in low-Le mixtures may lead to the development of stable, stationary spherical flames called "flame balls."

For task 3, we have conducted experiments at Earth gravity on flame propagation in vertical tubes of varying diameter, at varying pressures and with mixtures having varying fuels, inert gases, and Le, and have measured the flame propagation rates just inside the extinction limit (S_b,lim). Since this quantity is predicted by most relevant theoretical models, the relative importance of buoyancy, flame stretch, heat loss to the tube wall, radiation loss, etc., may be assessed.

For task 4, we have introduced the use of aqueous autocatalytic propagating chemical fronts (in particular, the arsensous acid-iodate system) for the experimental simulation of premixed combustion in nonuniform and unsteady flows. These fronts more nearly match the assumptions made by most relevant theoretical models that do gaseous flames; for example, constant density, constant thermodynamic and transport processes, and no heat losses. We have studied propagation in a Taylor-Couette (TC) flow, that is, in the annulus between two rotating concentric cylinders, and in capillary-wave (CW) flow in a thin layer of fluid in a vibrating dish.

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 mechanisms of combustion limits in Earth-based combustion 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 1993:

For task 1, experiments have shown that at small particle loadings, burning rates are reduced, peak pressures in constant-volume combustion are lower, and thermal decay rates in the burned gases are increased. These results indicate that the significance of radiative loss is enhanced by the addition of particles to the gas. With sufficient seeding, the burning rates are practically the same as those found in particle-free mixtures, the peak pressures are comparable, and the thermal decay rate is smaller than in particle-free mixtures. All of these observations are consistent with the hypothesis that at sufficiently high particle loadings, radiation is reabsorbed within the combustible medium, and thus may not constitute a fundamental limit in very large systems. Tests on the KC-135 did not produce as dramatic an effect, possibly due to the lower quality of reduced gravity on the aircraft.

A model of the effects of reabsorption of emitted radiation on propagating flames and flame balls (described below) was developed. It demonstrates that there are no flammability limits because there is no loss mechanism in the problem.

For task 2, we have observed flame balls in all mixtures with sufficiently low Le and near flammability limits, independent of the chemical mechanism, which are stable for at least the 20 seconds of low-g available. The lean $\mu$ flammability limits are leaner than any which can be observed at Earth gravity. By comparison with analytical models, it has been concluded that radiation from the combustion products, along with diffusive-thermal effects in low-Le mixtures, is probably the stabilization mechanism which allows flame balls to exist at $\mu$. The drift velocity which results from the finite g-levels in the KC-135 aircraft leads to the formation of two types of quasi-cylindrical flame structures. Another type of flame string has been observed in high pressure flames having sulfur hexafluoride as the diluent gas. These strings are uncorrelated with buoyancy effects and seem to form much faster than any conventional diffusional or hydrodynamic timescales would allow. Their formation is conjectured to be a result of reabsorption of emitted radiation, which is most likely to occur in these mixtures because of their short Planck mean absorption length.

A flight experiment is under development to determine if the apparent stability of flame balls can be confirmed in experiments where both the duration of $\mu$ is long (unlike drop-tower experiments) and the quality is significantly better than that in aircraft tests.

For task 3, we have found that the characteristics of the limits can be described in terms of the effect of the Grashof number $Gr \sim g d^3/\alpha$ on the limit Peclet number $Pe = S_{\alpha,lim} (d/a)$, where $g$ is gravity, $d$ the tube diameter and $a$ the thermal diffusivity at room temperature. At low-Gr the results are consistent with theoretical predictions for flame extinguishment by conduction loss to the tube walls. At higher Gr, results are consistent with buoyancy-induced extinction mechanisms, in the upward propagating case $Pe \sim Gr^{1/2}$ and in the downward case $Pe \sim Gr^{1/3}$.

Because of the difference in extinction mechanisms, we have found that the flammability limit can actually be wider for downward propagation than for upward propagation. In the upward case, at $Gr > 2.0 \times 10^8$ (corresponding to a pipe-flow Reynolds number of 2000) turbulent flow is exhibited; the flame behavior can be either flamelet-like or distributed-like depending on Gr and Le.

For task 4, we have found that in TC flows under conditions where the flow is essentially a linear array of steady vortices, the experimental values of the propagation rate agree well with a model we developed to describe the interaction of a propagating front with a one-scale flow field. In TC and CW flow characterized by a broad range of scales, at low turbulence intensities ($u'$) our results agree with a model developed by Yakhot in 1988. At higher $u$, where the thin front is disrupted, a different type of model we developed, based on turbulent transport models in these flows, describes our results well. However, our results at small $U = u'/S_L$ are compromised by buoyancy effects, since even the very small fractional density change across the aqueous front leads to significant buoyancy influences because of the very low $S_L$. The fractal dimensions of the aqueous fronts were measured at values of $u'/S_L$ at least an order of magnitude higher than any result obtainable in gaseous flames. The linear fractal plots gave a fractal dimension close to 7/3, which agrees with a number of theories and experimental studies in gaseous flames.
in 3-d turbulence. Numerical studies of propagating fronts at very high $U/S_L$ have begun and are in good agreement with the experiments and previous theoretical predictions.

**STUDENTS FUNDED UNDER RESEARCH:**

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**BIBLIOGRAPHIC CITATIONS FOR FY 1993:**

**Journals:**


Ronney, P.D. Laser versus conventional ignition of flames. *Optical Eng.*, (accepted).


**Presentations:**


Ronney, P.D. Studies of premixed laminar and turbulent flames at microgravity. Final Report for Grant, NAG3-1252, August 31, 1993.


**Presentations:**


II. MSAD Program Tasks — Flight Research

Discipline: Combustion Science

Ignition and Flame Spread of Liquid Fuel Pools

PRINCIPAL INVESTIGATOR: Dr. Howard Ross

NASA Lewis Research Center (LeRC)

CO-INVESTIGATORS:

W.A. Sirignano
University of California, Irvine

F.J. Miller
Case Western Reserve University

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

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.

Flame spread over liquid fuel pools is most commonly characterized by the relationship of the initial pool temperature to the fuel’s idealized flash point temperature, with four or five separate characteristic regimes having been identified. In the uniform spread regime, control has been attributed to (a) gas-phase conduction and radiation, (b) gas-phase conduction only, (c) gas-phase convection and liquid conduction, and most recently (d) liquid convection ahead of the flame. Suggestions were made that the liquid convection was owed to both buoyancy and thermocapillarity. In the pulsating regime, complicated flow structures have been observed in both the gas and liquid phases, with circulation around several centers; these flows were attributed to combined thermocapillary and buoyant effects.

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 level.

SIGNIFICANCE:
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. One possible mechanism for pulsating spread in microgravity is if the “premixed gas diffusive burning” pulsations are due to periodicity between gas-phase conductive and liquid-phase convective control. A second possibility, which will be determined by these investigations, is whether pulsations may be induced in low gravity by the presence of slow, forced, gas-phase flow.

PROGRESS DURING FY 1993:
In support of the sounding rocket experiment, several development tests were conducted by the investigator team: (a) Fuel tray filling tests conducted aboard the KC-135 in December 1992 determined that the tray inlet port should be a 2 mm wide slot running the full length of one tray end as the filling port. (b) Several sizes of diamond particles were tested as fluid seeding candidates in propanol for the Particle Image Velocimetry system. Particles above 16 μm settled too quickly for use in normal gravity, but smaller ones were still bright enough to be useful for
F. Miller recorded more liquid-phase flow patterns for 1-propanol at two temperatures in the uniform regime using the particle image velocimetry (PIV) system. Flow ahead of the flame was found in most cases, but the length and time scales were such that video framing rates (30 f/sec) are not adequate to definitively capture the motion. Nevertheless, the data that was obtained agreed well with recent modeling efforts by Dr. David Schiller at UC Irvine which predict flow only about 2 mm ahead of the flame. Initial tests with a borrowed Kodak EktaPro EM high-speed video system were conducted to determine if it was possible to use it in the PIV system. Good images were obtained at 125 f/sec, but problems with data transfer prevented the images from being saved on the computer. In general, the high-speed video shows promise for being able to capture the small, rapid liquid movements ahead of the uniformly spreading flame and will be pursued further.

D. Schiller and W. Sirignano of the University of California at Irvine and H. Ross of NASA Lewis reviewed the latest computer simulation results of flame spread across liquid pools. These showed excellent quantitative agreement with the shallow pool 1g and 0g experiments at Lewis, and the literature's value for the transition temperature between pulsating and uniform spread. The simulations show conclusively that liquid-phase buoyancy plays no role in pulsating or uniform flame spread over propanol; this is opposite the conclusions of a recent article in the literature, but consistent with our previously held beliefs and publications. This was accomplished by comparing predicted spread and mass burning rates and characteristics in tests in 1g to tests in 0g in the liquid phase only, the gas phase only, and in both phases. In addition the simulations show how critically important gas-phase buoyancy and hot-gas expansion are in the spread process. Finally, they ran a series of tests to assure themselves that the convergence criterion used in their code was adequate. Disagreements remain between the UCI model and (a) the Lewis deep pool experimental results as well as (b) the predictions of Di Blasi et al on the importance of liquid-phase convective flow in the uniform flame spread regime. Upon completion of this set of runs, a journal article was prepared.

A successful Requirements Definition Review (RDR) was held for the SAL project and we gave a presentation about our current research on flame spread over liquids and answered panel members' questions about the experiment as they arose. The project was well received both on its scientific merits as well as its cost, schedule, and engineering attributes.

We completed a series of flame spread experiments over a pool of 1-propanol in a He/O₂ environment in normal gravity. All experiments were carried out with 21% O₂ (thus the He simply replaced the N₂ in normal air), and the initial fuel temperature ranged from 10° C to 30° C. In this range the flame spread in air moves from the pulsating to the uniform to the superflash regimes, yet with a helium diluent the flame spread is always in the pulsating regime. The pulsation frequency is much higher than for this fuel in air and is nominally constant at 15 hz, while the spread rate itself only increases from 5 cm/s to 11 cm/s. Preliminary rainbow schlieren data revealed no appreciable liquid-phase temperature gradients under the flame and no vortical motion as with air. The apparent lack of liquid-phase participation, the much higher pulsation frequency, and the fact the flame actually retreats in the case of helium leads us to believe that the pulsation mechanisms is different in helium. One scenario is that the flame flashes through a premixed region and then extinguishes due to heat-loss or lack of fuel vapor then has to pull back. That would imply that the flash and fire points are different in helium ( they are the same in air for 1-propanol). With a new spark ignitor we continued to try measure the flash point, and did get one flash at 48° C (compare to 25° C in air) at a stand-off distance of 4 mm (compare to 0.3mm in air). The ensuing flame immediately extinguished, and other runs resulted in no flash at all.
The computer code from UCI was then installed on the Goddard Cray supercomputer and run from NASA Lewis. It was used to predict the flame spread over 1-propanol in air and He/O₂ (21%) environments. The code was modified to allow for an atmosphere containing two diluents and an interpolation program was written to display full field properties such as temperature and velocity. In the air runs, the temperature distribution indicated a hot cloud of gas sitting ahead of and above the flame, which is as yet unexplained. From the results of the helium run the temperature map seemed to indicate that the program considered the initial fuel/gas mixture above the pool to be flammable, which accounts for the high spread rate. This is not observed in the experiments perhaps because we do not start with a uniform fuel/gas mixture. The flash point (either closed or open) of propanol in this atmosphere is currently unknown, and initial attempts to measure it were unsuccessful.

**BIBLIOGRAPHIC CITATIONS FOR FY 1993:**

**Journals:**

**Proceedings:**

**Presentations:**

II. MSAD Program Tasks — Flight Research

Droplet Combustion Experiment

PRINCIPAL INVESTIGATOR: Prof. Forman A. Williams
University of California, San Diego

CO-INVESTIGATORS:
Prof. F.L. Dryer
Princeton University

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

OBJECTIVE:
The objective of this research effort is to provide scientific support, in collaboration with Professor F. Dryer of Princeton University, for a droplet burning experiment to be performed on board a space platform. This support will include performance of theoretical analyses relevant to the experiments, execution of drop-tower experiments to acquire additional data, analysis of experimental data obtained in ground-based experiments, and identification of test conditions for experiments in space.

DESCRIPTION:
The objectives stated above will be pursued through the use of the 2.2-second drop tower with measurements made on heptane and methanol burning in atmospheres of normal air and in diluted atmospheres and through the analysis of data from this tower, and also from the 5-second drop tower with these fuels as well as decane. The data will be analyzed for droplet diameter and extinction diameter by use of high-precision analysis systems. Flame diameters also will be obtained by suitable digital image analysis procedures.

The theoretical approach will employ asymptotic methods to relate observed extinction conditions to elementary rate parameters. Treatment of the data by use of the theory will help to identify experiments that need to be done in space. Additional experiments will also be performed to address the effects of relative droplet-gas convection on burning rates. In the theoretical part of the project, a spherosymmetric, time dependent, finite element-based numerical model with detailed gas phase kinetics, and variable property effects will be extended to study the effects of soot formation in droplet burning. Extension of the existing one-dimensional code to two-dimensional axisymmetric geometry will be evaluated.

SIGNIFICANCE:
The overall purpose of the research is to achieve fundamental advances in the science of droplets.

PROGRESS DURING FY 1993:
Significant progress has been made concerning the extinction of heptane diffusion flames. Extinction diameters of heptane droplets were predicted by an asymptotic analysis that systematically reduced the chemistry to two-step, three-step, and four-step mechanisms. This reduction is not empirical or semi-empirical modeling, but instead is a logical reduction from underlying elementary rates of the multitude of steps involved. This is the first time that such a reduction has been accomplished for a fuel as complicated as heptane. The predicted extinction conditions are quite reasonable, but strongly dependent on fuel chemistry, and the magnitudes of uncertainties involved were estimated and shown to exceed a factor of ten for the extinction diameter. Necessary steps in improving the estimates were identified for future research. There is a possibility of a significant advance in understanding through this work.

A significant portion of the progress involves improvements to the numerical code to include soot shell formation due to the influence of thermophoretic effect and Stefan flow in droplet combustion and to include 92-step Warnatz
II. MSAD Program Tasks — Flight Research

Discipline: Combustion Science

kinetic mechanism for heptane. Some preliminary calculations were also carried out for methanol using a skeletal mechanism at different ambient pressures.

During this reporting period, a successful CoDR for the Droplet Combustion Experiment (DCE) was also completed in collaboration with NASA personnel.

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Scientific Support for a Space Shuttle Droplet Burning Experiment

PRINCIPAL INVESTIGATOR: Prof. Forman A. Williams
University of California, San Diego

CO-INVESTIGATORS:
Prof. F.L. Dryer
Princeton University

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

OBJECTIVE:
The objective of this research effort is to provide scientific support (in collaboration with Professor F. Dryer of Princeton University) for a droplet burning experiment to be performed on board a space platform. This support will include performance of theoretical analyses relevant to the experiments, execution of drop-tower experiments to acquire additional data, analysis of experimental data obtained in ground-based experiments, and identification of test conditions for experiments in space.

DESCRIPTION:
The objectives stated above will be pursued through the use of the 2.2-second drop tower with measurements made on heptane and methanol burning in atmospheres of normal air and in diluted atmospheres and through the analysis of data from this tower, and also from the 5-second drop tower with these fuels as well as decane. The data will be analyzed for droplet diameter and extinction diameter by use of high-precision analysis systems. Flame diameters also will be obtained by suitable digital image analysis procedures.

The theoretical approach will employ asymptotic methods to relate observed extinction conditions to elementary rate parameters. Treatment of the data by use of the theory will help to identify experiments that need to be done in space. Additional experiments will also be performed to address the effects of relative droplet-gas convection on burning rates. In the theoretical part of the project, a spherically symmetric, time dependent, finite element-based numerical model with detailed gas phase kinetics, and variable property effects will be extended to study the effects of soot formation in droplet burning. Extension of the existing one-dimensional code to two-dimensional axisymmetric geometry will be evaluated.

SIGNIFICANCE:
The overall purpose of the research is to achieve fundamental advances in the science of droplets.

PROGRESS DURING FY 1993:
This is a computational complement to the task presented on page II-54 and 55. It is not a separate flight program.

Numerical and asymptotic analyses of droplet combustion continued during this reporting period. The focus of these studies rests primarily on time-dependent droplet-burning characteristics and on extinction phenomenon. Extinction diameters were calculated for methanol in nitrogen-oxygen mixtures and helium-oxygen mixtures using a two-step reduced chemical kinetic mechanism. In the near future these calculations will be extended to a three step mechanism where the current restriction of H-atom steady state will be removed. Also, effort is underway to include water dissolution effects in the rate ratio asymptotic model.

Computational studies were performed to model fully transient vaporization process of liquid methanol fuel droplet in dry and humid microgravity environments at ambient temperature. These studies were performed to determine the instantaneous gas phase composition surrounding the liquid droplet after deployment but prior to ignition so that
optimal igniter location could be determined. Results for methanol show that the local fuel to oxidizer ratio of the gas phase is within flammability limits at distances less than 1 radius away from the droplet surface for a short period of time interval which increases with increasing initial droplet diameter. Following this short period, local cooling of the droplet surface due to the high latent heat of vaporization of methanol results in an entire gas phase which is outside the lean flammability limit.

The CRFM1D model developed earlier at Princeton for droplet combustion with detailed chemistry was used to calculate extinction diameters for methanol in O₂/N₂ and O₂/He environments. These results along with the asymptotic results are used to develop the test matrix for Droplet Combustion Experiment.

A successful Delta Conceptual Design Review for the Droplet Combustion Experiment was held during this reporting period with participation from UCSD, Princeton, and Lewis Research Center.

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**BIBLIOGRAPHIC CITATIONS FOR FY 1993:**

**Journals/Publications:**


**Proceedings:**


II. MSAD Program Tasks — Flight Research

Surface Controlled Phenomena

PRINCIPAL INVESTIGATOR: Prof. Robert E. Apfel

Yale University

CO-INVESTIGATORS:

R.G. Holt

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

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.

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.

Four sample materials will be investigated: triply distilled water, octyl beta-D-glucopyranoside (a nonionic surfactant), Triton X-100 (a nonionic, fast-sorption surfactant), and bovine serum albumin (a nonionic, sorption-inhibited surfactant). Five concentrations of each surfactant will be used. A range of drop sizes from 2 cc up to 8 cc will be investigated for each concentration.

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 1993:

Progress to Date:
Oscillating drops bearing surfactants were investigated in DPM on USML-1. By measuring the diameter of a drop as a function of time, the frequency and damping constant of the oscillations can be inferred by Fast-Fourier-Transform (FFT) and nonlinear-fitting techniques. The frequencies of oscillation for various concentrations of surfactants were obtained. Determining the damping constants has been difficult due to an unexpected rotation present during all sequences. The shift in the natural-oscillation frequency due to the distortion of the equilibrium shape was measured.
II. MSAD Program Tasks — Flight Research

STUDENTS FUNDED UNDER RESEARCH:

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BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:

Presentations:


II. MSAD Program Tasks — Flight Research

**Discipline:** Fluid Physics

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

**Principal Investigator:** Prof. Paul M. Chaikin

Princeton University

**Co-Investigators:**

Prof. W.B. Russell

Princeton University

---

**Task Objective, Description & Significance:**

**Objective:**
The objective is to understand the fundamental nature of the liquid-solid transitions.

**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.

**Significance:**
The ideal system for such study is a set of hard spheres that theoretically should order as their density is increased past ~50%. The problem is that sedimentation prevents setting the density or attaining equilibrium. The answer is microgravity, but the preflight questions are concerned with how the measurements can be done and determining the equilibration times.

**Progress During FY 1993:**

We have made great progress in studying a variety of different colloids suspended against gravity by the counterflow of the solvent - i.e., with a fluidized bed. We have found the variation in sedimentation velocity resulting from hydrodynamic interactions and its effect on particle dispersion. We have observed crystallization of fluidized particles due to their Coulomb repulsion.

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**Task Initiation:** 4/90  **Expiration:** 7/95

**Project Identification:** 694-24-05-10

**Responsible Center:** LeRC
Study of Two Phase Flow Dynamics and Heat Transfer at Reduced Gravity

**Principal Investigator:** Prof. A. E. Dukler

**University of Houston**

**Co-Investigators:**

J.B. McQuillen

NASA Lewis Research Center (LeRC)

**Task Objective, Description & Significance:**

**Objective:**

Theoretical models will be developed and experimentally verified to predict two-phase flow characteristics, such as flow regime, pressure drop, void fraction, and liquid film thickness. In addition, models for the prediction of heat transfer in two-phase flows will also be developed and verified.

**Description:**

Reduced-gravity experiments will be conducted in NASA aircraft to measure the previously listed 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.

For larger diameter tubes and for heat-transfer experiments, the transient times become longer than the microgravity time available in aircraft trajectories. Thus the long-term microgravity duration of a spaceflight is necessary to obtain important two-phase flow data. The spaceflight experiment design will be an extension of the aircraft experiments: water mixtures and air will flow together in a tube, with sensible heat transfer to the subcooled liquid effected by heating a section of the tube.

**Significance:**

The purpose of this study is to achieve a better understanding—better predictability—of two-phase (gas-liquid) flow in pipes.

**Progress During FY 1993:**

Learjet tests that were conducted during May, June and August 1993 used water as the test liquid to further characterize annular flow in a 1.27 cm diameter tube. A new data acquisition and control system was installed on the Learjet rig, and software was written and tested for these flights. New probes were installed to measure wall shear stress in annular flow. Difficulties with an interaction between two valves masked out most of the differential pressure drop data. Valid liquid film thickness data was obtained around the circumference of the test section during these tests, however. These flights will be repeated.

A KC-135 rig will be used for test section diameters ranging from 2.54 to 5.1 cm is being built and is nearing completion. Checkout of the various subsystems has been initiated, but testing of the entire rig is dependent on completion of the wiring and delivery of a calibrated test section.

Work is proceeding on the Science Requirements Document although there are several aspects of the report that cannot be written until some preliminary low-gravity tests have been completed with the KC-135 rig.
II. MSAD Program Tasks — Flight Research

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BIBLIOGRAPHIC CITATIONS FOR FY 1993:

**Journals:**

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

Discipline: Fluid Physics

Kinetics of Diffusional Droplet Growth

**Principal Investigator:** Dr. Donald O. Frazier

**Co-Investigators:**
- Dr. M. Glicksman
- Dr. J. Rogers
- Dr. J. Downey

**Task Objective, Description & Significance:**

**Objective:**
The flight objective is to determine droplet growth rate constants and changes in size distributions using optical techniques for recording droplet data with time.

**Description:**
A transparent model binary liquid/liquid miscibility-gap type solution is the system of choice for the flight. The experimental approach is to deploy a fixed array of droplets of one liquid phase into a cell filled with the conjugate phase. This approach will greatly reduce any residual gravity effects (from "g-jitter" or maneuvers) and eliminate the possibility of coalescence. Ground-based studies determine the experimental parameters required to maximize the amount of data on "pure" Ostwald ripening obtainable during the flight experiment. Such parameters as volume fraction of droplet phase, initial droplet number, droplet spacing and sizes, the time required for the experiment, and ideal concentrations for supersaturation and growth must be determined.

**Significance:**
Ostwald ripening is the process by which larger droplets grow at the expense of smaller ones by diffusion of mass away from droplets below a critical radius toward ones above this critical size. The phenomenon is important in many disciplines.

**Progress During FY1993:**

Observation of Ostwald ripening in an isopycnic mixture of succinonitrile and water over a period of about four months, using holography, revealed a decreasing droplet number and average radius increase in excellent agreement with theory. The predicted scaling behavior for droplet number decay involving 2-D diffusion fields of 3-D spherical caps is $t^{-0.75}$. This accurately models the experiment, droplets of succinonitrile-rich phase stuck to the wall of the experiment cell, with a droplet number decay of $t^{-0.73}$. Average droplet radius scales as $<t>^{0.25}$, also consistent with the model. A manuscript reporting these results in the peer-reviewed literature is in preparation. A proposal to extend the ground-based aspect of this work to include eventually long-term quench experiments on carriers orbiting for extended periods of time, e.g., retrievable rockets or space station, has been submitted to NRA-93-OSSA-12. This approach will differ from the "tethered droplet" approach to allow for a large population of droplets with time to clarify for holographic applications.

The tethered droplet approach continues to be under development for further ground-based work and potential "short-term" shuttle flights if opportunities become available. Dr. Rogers is preparing for KC-135 flights, commencing in January, 1994, to test syringe tips for microgravity operations.

We have completed materials property measurements which include interfacial tension, density, and viscosity measurements as functions of temperature and composition. A separate publication will present some of this data.
II. MSAD Program Tasks — Flight Research

Discipline: Fluid Physics

Task Initiation: 12/89   Expiration: 12/93
Project Identification: 694-24-08-01
NASA Contract No.: In-house
Responsible Center: MSFC

BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:


Presentations:


Microscale Hydrodynamics Near Moving Contact lines

PRINCIPAL INVESTIGATOR: Prof. Stephen Garoff Carnegie Mellon University

CO-INVESTIGATORS:
M. Weislogel NASA Lewis Research Center (LeRC)

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

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.

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.

SIGNIFICANCE:
The essential points of the investigation are:

1) Are the viscous stresses near a moving contact line appropriately described by present phenomenological models?
2) Can predictive models for spreading be developed for complex systems (rough surfaces, non-Newtonian fluids, etc.) if the flow fields and interface shapes near a contact line are known?
3) What is the nature of the unique fluid dynamical processes near a moving contact line? How do these processes depend on the properties of the fluids and the solid surface involved? How do these processes determine the true dynamic contact angle?

PROGRESS DURING FY 1993:

Static interface calculations have been used to simulate outer scale interface shapes which will be seen during the space experiment and to design fluid fill and velocity step cycles for the experiment. In addition, the static interface calculations have been used to investigate potential interface instabilities during experiment and to determine the optimal radii of the test cell.

Lighting improvements on image analysis technique have been made which minimize the effects of pixel noise on information gained about interface shape. In order to take advantage of this improvement a new dynamic contact angle measurement system is being installed.

Interface images have been taken and analyzed and the preliminary indication is that the present analytical expansions work to a dimensionless capillary length of 0.5. The first images of the interface in capillary depression have been taken and preliminary indications are that the analysis matches the experimental data to Ca=0.1. As a side benefit, microscopic bubbles near the contact line provided velocity field information about the moving contact line for an interface in depression.
II. MSAD Program Tasks — Flight Research

Discipline: Fluid Physics

Materials studies of polydimethylsiloxanes have continued and have shown the sensitivity of inner scale physics to polymer end group chemistry.

Studies of the effect of vibrations on the interface shape and stability have been performed in order to determine the transverse size of the test cell.

Preliminary evidence has been obtained that UV cleaning may produce surfaces which remain clean for weeks under inert gas storage. Design of the next phase of testing has begun.

A postdoctoral fellow has been recruited and will start November 15, 1993.

BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:

Proceedings:

Garoff, S. Dynamic contact angles of non-newtonian fluids, American Chemical Society meeting, Toronto, June 1993.
II. MSAD Program Tasks — Flight Research

Plasma Dust Crystallization

Principal Investigator: Dr. John A. Goree
University of Iowa

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

Task Objective, Description & Significance:

Objective:
The objective of this research is to demonstrate that a crystal can be grown in a charge neutral plasma environment.

Description:
The investigator will assemble and test apparatus for radio-frequency (RF) plasma generation/particulate confinement. This will include working with German scientists in designing microgravity experiments.

Significance:
This investigation will begin a new genre of microgravity research that will provide a much needed test of the dusty plasma theories used in space physics and astronomy. Regardless of the results, this investigation will open the doors to new areas of research with strongly-coupled plasmas and non-quantum lattices.

Progress During FY 1993:
The scientists developed a theory of fluctuations of the charge of a particulate in a plasma and found that these fluctuations could inhibit the formation of a crystal if the particulates are too small or the neutral gas pressure in the plasma is too low. These results will help define the parameter space needed for experiments.

Collaboration with German scientists has yielded the most convincing laboratory evidence to date of the feasibility of forming a crystalline lattice of charged grains suspended in a plasma. The lattice structure is ~100 layers horizontal and ~5 to 10 layers vertical. Earth's gravity is thought to account for the limited vertical extent of this lattice. An image of the lattice is formed by illuminating the particulate cloud with a horizontal sheet of laser light, photographing the cloud, and digitizing the image.

A laboratory platform designed especially for conducting further laboratory definition experiments is now under construction at the University of Iowa.

Students Funded Under Research:

MS Students: 2

Task Initiation: 04/93  Expiration: 04/96
Project Identification: 694-24-08-02
NASA Contract No.: GN8-292
Responsible Center: MSFC

Bibliographic Citations for FY 1993:

Journals:


**Presentations:**


II. MSAD Program Tasks — Flight Research

Evaporation from a Meniscus within a Capillary Tube in Microgravity

PRINCIPAL INVESTIGATOR: Prof. Kevin P. Hallinan
University of Dayton

CO-INVESTIGATORS:
Dr. J. Ervin
University of Dayton

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

OBJECTIVE:
The primary objective of this study is to improve our understanding of evaporation from a porous or grooved media, such as that found in phase change heat transfer devices such as heat pipes and thermosyphons.

DESCRIPTION:
In order to effectively study the thin film region of a capillary interface, experiments will be conducted in a large evaporation/condensation loop in a low-gravity environment. The experiments will include simultaneous particle image velocimetry (PIV) and interferometry in order to measure the fluid velocities and interface shape in the thin film region. Non-intrusive temperature measurements of the surface will be correlated to PIV data for determination of thermocapillary flows.

SIGNIFICANCE:
The essential points of the investigation are:

1. Determine the effect of surface curvature on the local evaporation rate;
2. Determine the effect of solid-liquid intermolecular forces on the evaporation rate and the fluid flow in the thin film region;
3. Quantitatively examine the interface instability mechanisms of an evaporating capillary interface; specifically, thermocapillarity and vapor recoil.

From this study we should gain an improved understanding of the fundamentals of evaporation from a capillary interface. This improved understanding will not only be used to refine failure criteria for phase change heat transfer devices, but may be applied to other phase change processes such as nucleate boiling and spreading of thin films. In addition, a PIV technique for thin film fluid flows will be developed which may have use in future research.

PROGRESS DURING FY 1993:
The efforts this year have focused upon the following specific tasks:

• Application of a 3-D scattering particle image velocimetry technique to thin film velocity field measurement.

A great deal of effort has been directed toward the application of the 3-D scattering particle image velocimetry technique, being developed by B. Overyn at LeRC, toward the measurement of the velocity field within the evaporating thin film of liquid adjoining the bulk meniscus. Efforts have focused on i) optimization of the light source, ii) determination of the sensitivity of the technique to particle size, shape, and velocities, and iii) selection of suitable particles. A calibration rig is under development for the PIV technique.

• Modeling the thermo-fluid behavior of the evaporating meniscus.
II. MSAD Program Tasks — Flight Research

A model of the thermo-fluid behavior of the evaporating thin film adjoining the bulk capillary meniscus has developed for large tube diameters in 0-g. This model has been developed to obtain an estimate of the heating conditions which can be tolerated in 0-g before the onset of thermocapillary flows on the liquid-vapor interface. Also, a model has been developed to gauge the required accuracies for measurement of the thin film velocity field, film thickness, power input, and temperature measurements. The model development is now complete and it has been solved for the idealized case of a uniform wall temperature.

- Designing the evaporator/condenser configuration.

This task is primarily aimed at providing passive control of the evaporating meniscus position in the test cell during low-gravity testing. Several test cell configurations have been constructed and are being evaluated.

- Low-gravity experimentation to study evaporation from a capillary interface.

Low-gravity testing of an evaporating capillary interface was conducted at the 2.2 Second Drop Tower at LeRC in order to examine the relative effects of buoyancy and thermocapillary driven convection on evaporation. Unfortunately, before acceptable data could be gathered the 2.2 Second Drop Tower facility was shut down for upgrading. The facility should be on-line this spring and testing will resume.

- Investigation of the effect of vibrations on the stability of the meniscus.

The final task has aimed at quantifying the effect of vibrations on the meniscus stability for both evaporating and non-evaporating conditions within both single tubes and the evaporator/condenser configuration. After completion of the 1-g tests, similar experiments are planned for low-g.

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BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:

Proceedings:
II. MSAD Program Tasks — Flight Research  

Geophysical Fluid Flow Cell

PRINCIPAL INVESTIGATOR: Dr. John E. Hart  

University of Colorado

CO-INVESTIGATORS:
- F. Leslie  
- T. Miller  
- J. Toomro  
- D. Ohlsen

NASA Marshall Space Flight Center (MSFC)  
University of Colorado, Boulder

Task Objective, Description & Significance:

Objective:
The Geophysical Fluid Flow Cell Experiment (GFFCE) 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 geophysical fluid flow cell uses electrostatic fields 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 rotation, where the resulting buoyancy forces are radially directed, as in planetary atmospheres and stars.

Description:
The research has two components: hardware development, and basic theoretical and numerical study of spherical convection in preparation for USML-2. 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. This permits interactive experiments that can identify and focus on important flow regimes.

Theoretical and computational simulations of the expected flows are carried out in advance of the flight in order to suggest particular experiments that can critically advance our basic understanding. Such models usually involve various approximations or assumptions that must be checked experimentally.

Significance:
This fundamental configuration is significant because large-scale motions of the atmospheres of planets and many stars are strongly constrained by rotation, under the action of Coriolis forces, and by gravity, which is manifest in the buoyancy forces that drive thermal circulations. The GFFC experiments will provide fundamental laboratory data that can be applied to problems of jet streams on the giant planets, differential rotation on the sun, and motions in the Earth's core and mantle.

Progress During FY 1993:
The most exciting results are from a fully nonlinear computational model of thermal convection in the presence of strong basic rotation which varies with latitude. The resulting flows are composed of banana cells, as observed on the Spacelab-III flight of the GFFC, but the computation is simplified by using an approximation to the full sphere, the "equatorial annulus" geometry. The turbulence at high heating rates is found to be associated with extremely strong, even dominating, mean zonal jets.

A scaling of the model results with the observed heat flux of Jupiter; for example, loads to zonal jets with velocities of order 100 meters per second. This number is similar to Voyager observations. The turbulence can have isolated
spots embedded in the self-induced zonal jets. When the basic rotation is sufficiently large the whole system
pulsates. The resulting differential rotation and the turbulent convection pulses in almost periodic bursts. If an
effective eddy viscosity appropriate to solar convection is used, the pulsation period is about 13 years—nearly the
observed period of the solar cycle. These model results suggest a new and potentially very important regime of
convection that will be investigated by the GFFC on USML-2.

BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:
II. MSAD Program Tasks — Flight Research

II-7.3

Discipline: Fluid Physics

Interfacial Phenomena in Multilayered Fluid Systems

Principal Investigator: Prof. Jean N. Koster
University of Colorado

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

Task Objective, Description & Significance:

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 is induced by 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 will also be studied.

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 1-g data will be used to design and predict the results of the flight experiment on the IML-2 mission.

Significance:
The results are expected to significantly advance our knowledge in the area of surface-tension-driven and in multilayered fluid systems and are, in addition, relevant to applications related to encapsulated float zone processing.

Progress During FY 1993:

An analytical model has been developed to study multiple immiscible liquid layers. The thermocapillary flow of the triple layer configuration of FC40 2cst. silicone Oil FC75, selected for the space flight experiment, has been predicted for zero-gravity environment using asymptotic theories. Interfacial tension gradients of the chosen fluorinerts and silicone oil have been measured and could be included in the models for prediction.

Numerical simulations of the single and double layers of fluorinerts and silicone oils have been done and compared to experiments. Numerical efforts are also planned to estimate the effects of g-jitter on the interfacial layers.

In ground-based experiments, the combination of fluorinerts and silicone oils have been proven to be immiscible fluids that exhibit mechanical coupling and generate interfacial roll cells. Both FC75 and FC40 have been tested with a variety of silicone oils. The flight triple layer configuration, however, could not be tested on the ground due to the relatively large density of the FC fluids (with a specific gravity of about 2). A replacement for the upper fluor inert, with a lower density than the middle layer, although not yet defined, remains a goal for the one-g experiments.

There were also some activities relative to the flight experiment preparation. There were numerous discussions conducted and various documents prepared for ESA regarding the flight design of the cell. Issues discussed include: materials relative to the cell design (knife edge and seal); concerns regarding the cleaning procedures for the cell; and concerns regarding the design and placement of the thermocouples in the cell. Discussions were also initiated on the
II. MSAD Program Tasks — Flight Research  

Discipline: Fluid Physics

subjects of application software and the man-machine interface. The Functional Objectives (FO's) documents were revised several times during this period.

A videotape was prepared describing the flight experiments. This was done for the benefit of the crew and copies can be made available to those interested. In addition, the Principal Investigator and Project Scientist personally briefed the crew regarding the conduct of the experiments and crew involvement. This was done in various meetings in Houston, Torino (Italy), and Boulder.

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BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:


II. MSAD Program Tasks — Flight Research

Discipline: Fluid Physics

Proceedings:


II. MSAD Program Tasks — Flight Research

Discipline: Fluid Physics

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, Description & Significance:

Objective:
The objective of this work is to determine the extensional viscosity of fluid systems such as dilute polymer solutions, emulsions, and suspensions, in steady uniaxial elongation.

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 an 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.

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.

Progress During FY 1993:
The first draft of the Science Requirements Document has been submitted. The ground-based experimental apparatus is now being constructed; interlocking aluminum tubes for use in it are being fabricated at NASA Lewis Research Center and will be completed soon. The particle image velocimetry apparatus has been built and is being tested.
II. MSAD Program Tasks — Flight Research

Future plans include further development and construction of the experimental apparatus and submission of the second draft of the Science Requirements Document.

STUDENTS FUNDED UNDER RESEARCH:                TASK INITIATION: 12/92  EXPIRATION: 12/95
PhD Students: 1                                       PROJECT IDENTIFICATION: 694-24-05-12

RESPONSIBLE CENTER: LeRC
II. MSAD Program Tasks — Flight Research  

Discipline: Fluid Physics

Pool Boiling Experiment

PRINCIPAL INVESTIGATOR: Prof. Herman Merte, Jr.  
University of Michigan

CO-INVESTIGATORS:
No Co-I's Assigned to this Task

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

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.

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 at 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. For the initial flight, a total of nine tests are proposed, with three levels of heat flux and three levels of subcooling.

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;
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Discipline: Fluid Physics

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.

PROGRESS DURING FY 1993:
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/cm$^2$), 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 also flown on STS-57 mission in June 1993. Eight of nine test points were successful.

The Pool Boiling Experiment will be flown again on the STS-60 mission, scheduled for February 1994.

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BIBLIOGRAPHIC CITATIONS FOR FY 1993:

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

PRINCIPAL INVESTIGATOR: Prof. Simon Ostrach Case Western Reserve University

CO-INVESTIGATORS:
Prof. Y. Kamotani Case Western Reserve University

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

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.

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 39 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
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Discipline: Fluid Physics

bulk temperature distributions, and the free surface deformations will be correlated to support the proposed physical mechanism for the oscillatory phenomenon.

Progress During FY 1993:

STDCE-1: The significant milestone was achieved with the successful completion of the STDCE-1 during the USML-1 mission in July 1992. Thirty-eight tests of STDCE-1, operated for the first time in-orbit in Spacelab, were conducted in 12 1/2 hours to obtain thermocapillary flow data. The flight hardware provided better video flow data than expected, as well as infrared images, some of which were downlinked for observation and analysis by the PIs. Preliminary evaluation corroborates the Principal Investigator’s theory that oscillatory flows require lower viscosity liquids and smaller test chambers. Operations were controlled by a team of NASA Lewis Research Center engineers located in the Payload Operations Control Center at NASA’s George C. Marshall Space Flight Center.

The STDCE-1 data analysis is in progress. Because the volume of data is large the plan for analysis and presentation of the results calls for reducing, analyzing and publishing specific areas of the data separately. After these parts are complete more global conclusions will be drawn in additional publications.

The temperature fields in the one-hour CF and CT tests have been analyzed and compared with the results of the numerical computations. Good agreement was shown. The work will be presented at the ASME Winter Annual Meeting in New Orleans in December 1993 and has been submitted to the Journal of Heat Transfer.

The flow fields from the one-hour CF and CT tests are being analyzed. Preliminary comparison with numerical predictions also show good agreement. This data was presented at the 1993 Gordon Conference on Gravitation Effects on Physio-Chemical Systems.

The downlinked video and the SAMS data have been analyzed to study the effects of g-jitter on the STDCE. The effect on the fluid motion was very small but g-jitter continuously caused small free surface disturbances which could affect the free surface measurement in the STDCE-2. The result of the analysis was presented at the First International Workshop on G-jitter at Clarkson University, Potsdam, New York in June 1993.

STDCE-2: The STDCE-2 is scheduled to fly on USML-2 in September 1995. The MHRR was held in December 1992; Sub-system design reviews were held between April and June 1993 and a Critical Design Review was held in September 1993.

Ground-based Work: The oscillatory thermocapillary flow experiment with CO₂ laser heating, used to determine the critical heat fluxes at the transition from steady to oscillatory flow, has been completed. These data were used to confirm the STDCE-2 design parameters and will compared with the space data. This work was reported as a Ph.D. thesis. Some results from this work were presented at the International Symposium on Microgravity Science and Application which was held in Beijing, China in May 1993. An M.S. thesis reported the results of CT tests using 1.2, 2 and 3 cm diameter cylindrical containers. The main objective was to study the effect of buoyancy on oscillatory thermocapillary flow. The experiment to analyze the free surface behavior has been completed. The observed free surface behavior supported earlier conjecture regarding the free surface motion during oscillations. And, a surface deformation parameter appropriate for the CF configuration has been derived and has been shown to correlate well the above experimental data.

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PROJECT IDENTIFICATION: 694-24-05-04
RESPONSIBLE CENTER: LeRC
BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:


Proceedings:


Presentations:
Kamotani, Y. Effects of g-jitter on the surface tension driven convection experiment aboard USML-1, International Workshop on G-jitter, Clarkson University, Potsdam, NY. June, 1993.


II. MSAD Program Tasks — Flight Research

Discipline: Fluid Physics

Modeling and New Experiment Definition for the VIBES

PRINCIPAL INVESTIGATOR: Prof. Robert L. Sani

University of Colorado

CO-INVESTIGATORS:

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

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

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

OBJECTIVE:
The Vibration Isolation Box Experiment System (VIBES) is an IML-2 flight experiment being 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 will contain 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.

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.

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.

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PROGRESS DURING FY1993:

During this reporting period, the following have been accomplished:

1. A semi-consistent mass finite element projection algorithm for 2D and 3D Boussinesq flows has been implemented on Sun, HP and Cray platforms. The algorithm has better phase speed accuracy than similar finite difference or lumped mass finite element algorithms, an attribute which is essential for addressing realistic g-jitter effects as well as convectively-dominated transient systems.

2. The projection algorithm has been benchmarked against solutions generated via the commercial code FIDAP. The algorithm appears to be accurate as well as computationally efficient.

3. Optimization and potential parallelization studies are underway. PI's implementation to date has focused on execution of the base algorithm with a concern for vectorization.

4. The initial time-varying gravity Boussinesq flow simulation is being set-up. The mesh is being designed and the input file is being generated. Some preliminary small mesh cases will be attempted on the PI's HP9000/735 while the request from the PI to MSAD for supercomputing resources is being addressed.

5. The Japanese research team for VIBES was visited by the PI. The current set-up and status of the physical experiment obtained and an ongoing E-mail communication link was established.

TASK INITIATION: 1/93 EXPIRATION: 1/96
PROJECT IDENTIFICATION: 694-24-05-14
RESPONSIBLE CENTER: LeRC
**Studies in Electrohydrodynamics**

**Principal Investigator:** Dr. Dudley A. Saville  
Princeton University

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

**Task Objective, Description & Significance:**

**Objective:**  
Experiments are proposed to strengthen the experimental foundation of electrohydrodynamics theory through extension studies of the deformation of drops and the stability of fluid cylinders in strong electric fields.

**Description:**  
The proposed study consists of experimental and (a limited amount of) theoretical work necessary to prepare a science requirements document (SRD) for microgravity experiments.

**Significance:**  
Strong static or quasi-static electric fields can be used to manipulate fluids and fluid interfaces in unique ways since they provide a means to apply stress at places where there are gradations in electrical properties. Electrohydrodynamics is the body of knowledge dealing with the behavior of fluids in electric fields. The leaky dielectric model, used currently to explain electrohydrodynamic phenomena, is largely untested. This work provides a stringent test of the leaky dielectric theory.

**Progress During FY 1993:**  
At present we are studying the behavior of almost neutrally bouyant fluid bridges to aid the design of a "breadboard" experiment for a KC-135 platform. The stability of castor-oil (doped with eugenol) in silicon oil or vice versa has been examined in axial fields of different strengths. We found that bridges with aspect ratios (length/circumference) larger than \( \pi \) (the plateau limit for neutrally bouyant bridges in the absence of a field) can be stabilized with a suitably large field. However, the size of the steady field is smaller than that required when the field is oscillatory. We interpret this as evidence that the fluids behave as leaky dielectrics and that conduction processes are important in establishing the dynamics of the column. Moreover, we found that the critical field is extremely sensitive to the mismatch in the density, illustrating the desirability of a microgravity environment. Present efforts are focused on finding ionic compounds that can be used to control the conductivity.

**Students Funded Under Research:**  
PhD Students: 1

**Bibliographic Citations for FY 1993:**

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

Discipline: Fluid Physics

Presentations:
II. MSAD Program Tasks — Flight Research

Mechanics of Granular Materials

PRINCIPAL INVESTIGATOR: Dr. Stein Sture
University of Colorado

CO-INVESTIGATORS:
N. Costes
NASA Marshall Space Flight Center (MSFC)

Task Objective, Description & Significance:

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

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.

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.
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PROGRESS DURING FY 1993:

The Science Requirements Document (completed in 1991), experiment design, management issues, and schedules were discussed in several meetings held during the year between HQ, Sandia National Laboratories (which is fabricating the MGM apparatus), and University of Colorado personnel. The specimen and test configurations have been changed to accommodate physical requirements, and additional sets of experiments have been conducted to verify feasibility and to verify that the science is not compromised. Ground-based experiments are about to begin on the latest specimen configuration. Techniques for specimens preparation, handling, and dissection have been refined. Analysis and modeling procedures, especially in the area of evaluating material instability, have also been refined. Techniques for optically measuring displacements on the specimen’s surface are also undergoing improvement.

BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:


Proceedings:

II. MSAD Program Tasks — Flight Research

Thermocapillary Migration and Interactions of Bubbles and Drops

**PRINCIPAL INVESTIGATOR:** Prof. R. Shankar Subramanian
Clarkson University

**CO-INVESTIGATORS:**
Dr. R. Balasubramanian
NASA Lewis Research Center (LeRC)

**TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:**

**OBJECTIVE:**
The objectives of the research are to experimentally measure 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.

**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 shapes. The theories and numerical models developed and verified with the 1-g data will be used to design and predict the results of the flight experiment.

**SIGNIFICANCE:**
These results 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.

**PROGRESS DURING FY 1993:**
The FY93 activities concentrated on flight experiment preparation. The Principal Investigator and the Project Scientist conducted numerous discussions and prepared various documents for ESA regarding the flight design of the cell. Issues discussed include: materials for the cells; the actual design of the cell and supporting equipment; concerns regarding the filling and cleaning of the reservoirs, lines, and cells; injection and extraction of bubbles and drops; low-gravity testing of the performance of the injectors and plans for ground-based tests using the engineering model. Also discussed in substantial depth was the application software for performing the flight experiments and the man-machine interface. The Functional Objectives (FO's) documents were revised several times, and the question of the impact of disturbances from vernier thruster firings on the experiments was addressed briefly.

A videotape was prepared describing the flight experiments. This was done for the benefit of the crew and copies can be made available to those interested. In addition, the Principal Investigator and Project Scientist personally briefed the crew regarding the conduct of the experiments and crew involvement. This was done in various meetings in Houston, Torino (Italy), Cleveland, and Friedrichshafen (Germany).

Calculations were made regarding the effects of background convection in the cell during the flight experiment. For this purpose, idealized two-dimensional models are being used. These calculations will continue in FY94.

Ground-based research is continuing. Work was performed on identifying a suitable liquid-liquid pair for flight experiments and on the interaction of pairs of bubbles in a temperature gradient.
II. MSAD Program Tasks — Flight Research

**STUDENTS FUNDED UNDER RESEARCH:**

Total Students: 4

**TASK INITIATION:** 1/90  **EXPIRATION:** 12/93

**PROJECT IDENTIFICATION:** 694-21-08-02  **RESPONSIBLE CENTER:** LeRC

**BIBLIOGRAPHIC CITATIONS FOR FY 1993:**

**Journals:**


**Presentations:**


II. MSAD Program Tasks — Flight Research

Discipline: Fluid Physics

Drop Dynamics Investigation

**Principal Investigator:** Dr. Taylor G. Wang

**Vanderbilt University**

**Co-Investigators:**

C.P. Lee
A.V. Anilkumar
E.H. Trinh

**Vanderbilt University**

**Jet Propulsion Laboratory (JPL)**

**Task Objective, Description & Significance:**

**Objective:**
The objective of this program is to understand the behavior of free 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 behavior and dynamics of simple and compound drops as well as of liquid shells. Equilibrium shapes and the stability of rotating and nonrotating drops, their associated internal flow patterns, and the centering force associated with shape oscillatory dynamics of rotating compound drops—will be the principal scientific areas of interest.

**Description:**
A variety of experiments will be performed in space. The most important of these is the study of oscillating drops—to verify the established models for small-amplitude behavior and to provide observations at large amplitudes to motivate improvements in the theory. Experiments to verify the location of the bifurcation point between the axisymmetric and two-lobed shape families for rotating drops will be performed and the dynamics of fission explored. The oscillation-mode spectrum of rotating drops will be measured for a variety of drop parameters. Compound drops and liquid shells will be formed to study their oscillation modes and their effectiveness between the acoustic field and the fluid will be studied: the drops' static shape, the stability of distorted shapes, and the generation of any flows in the liquid.

On USML-2, there are three objectives:

1. **Internal flow study**
   
   Existing calculations have assumed the internal flow in a drop undergoing large-amplitude oscillations or deformation is basically laminar or at best weakly nonlinear. However, our preliminary data suggests otherwise. Therefore, on this flight, the internal flow of an oscillating drop will be systematically measured with emphasis on its effect on resonant frequency shift and mode coupling.

2. **Dynamics of fission**
   
   USML-1 data has shown that the fission process is very sensitive to changes in acoustic pressure and drop viscosity. On this flight, the dynamics of the elongated drop, while still an equilibrium shape, will be studied as a function of surface tension, centrifugal force, acoustic deformation, and viscosity. A special interest is the internal flow in the fissioning drop. Assuming that the internal pressure of the liquid depends on position along the axis, a numerical model will be developed to compliment the experiment.
3. Compound drop
As a compound drop undergoes capillary oscillations, the theory predicts core-centering will take place. In the USML-1 data, we have observed centering in most of cases but off-centering in some. A detailed study of the centering mechanism will be performed as a function of the core-shell volume ratio, viscosity, oscillation amplitude and interfacial tension.

**PROGRESS DURING FY 1993:**

**Progress to Date:**
In preparation for the flight of USML-1, ground-based studies of drop stability when the fluid is distorted by intense sound pressure were performed; a complimentary theoretical study on bubble shapes was undertaken. Observations on the effects of the relative size of two coalescing drops on their subsequent mixing were made and reported.

The flight of USML-1 provided several interesting results. Several high-quality runs to study the shape of drops in solid-body rotation were performed. The data for drops with minimum acoustic distortion agree with theory; drops flattened by the acoustic field deviate in the same way as the Spacelab-3 data. The outstanding discrepancy between theory and experiment appears to have been resolved; these results are being submitted for publication. An unexpected torque caused the drops to rotate throughout the mission; this complicates the analysis of the oscillating-drop experiments. However, by extrapolating data obtained for small rotation rates back to zero rotation, the forced response of the drops to oscillating acoustic pressure can be compared to existing theory. The ability of forced oscillations to center the inner fluid in a compound drop was demonstrated with both shells and two-liquid compound drops.

The anomalous torque is being investigated on the ground; it highlights the complexity of the acoustic-drop interactions inside the DPM.

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**Colloid Physics in Microgravity**

**PRINCIPAL INVESTIGATOR:** Dr. David A. Weitz  
**EXXON Research and Engineering Co.**

**CO-INVESTIGATORS:**  
Prof. P.N. Pusey  
The University of Edinburgh

**TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:**

**OBJECTIVE:**
The 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.

**DESCRIPTION:**
The work within this effort will be 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 will be the study of colloidal superlattices formed from mixtures of different-sized colloidal particles, the in-depth study of the formation of fractal colloidal aggregates, and the study of granular particles fluidized in a gas. 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.

**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 1993:**
The necessary contractual arrangements are still being processed to fund this proposed research which was accepted under the 1991 NRA on Microgravity Fluids.
II. MSAD Program Tasks — Flight Research

Task Initiation: 3/94  Expiration: 3/97
Project Identification: 694-24-05-13
Responsible Center: LeRC
In Situ Monitoring of Crystal Growth Using MEPHISTO

**Principal Investigator:** Prof. G.J. (Reza) Abbaschian

**University of Florida**

**Co-Investigators:**
- A.B. Gokhale
  **University of Florida**
- S.R. Coriell
  **National Institute of Standards and Technology (NIST)**
- J.J. Favier
  **CENG (France)**

**Task Objective, Description & Significance:**

**Objective:**
The objective is to determine the morphology stability of S/L interface 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.

**Description:**
Investigate the solidification behavior and stability of solid/liquid interface 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 are defined such as to subsequently 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.

**Significance:**
The first flight studied morphological stability in Sn alloyed with Bi (non-facet forming material), conducted by French scientists as part of this collaborative study. The results of the second flight experiment will therefore complement the results and findings of the first flight.

**Progress During FY 1993:**
Test and improve many current solidification theories, particular interplay between prophotogical stability and kinetics. Develop an analytical model of prophotogical stability and interface kinetics.

**Task Initiation:** 1/90  **Expiration:** 9/94

**Project Identification:** 694-25-05-04

**Responsible Center:** LeRC
II. MSAD Program Tasks — Flight Research

Coupled Growth in Hypermonotectics

PRINCIPAL INVESTIGATOR: Dr. J. Barry Andrews
University of Alabama, Birmingham

CO-INVESTIGATORS:
A. Sandlin
University of Alabama, Birmingham
S. Coriell
National Institute of Standards and Technology (NIST)

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

OBJECTIVE:
It is the objective of this investigation to quantify the applicability of the Jackson and Hunt model for eutectic and off-eutectic solidification to coupled growth in monotectic and off-monotectic composition alloys. The study will entail minor modifications to Jackson and Hunt's analysis to make it more suitable for monotectic systems, and then verification of the model through experimentation. In order to make possible the coupled growth process in the hypermonotectic composition range, experimentation must be carried out under low-g conditions.

DESCRIPTION:
The proposed project includes the major research tasks of theoretical modeling, selection of sample materials, selection of ampoule materials, and development of ampoules for processing, experimentation, and data analysis. In addition, this project will cover the development of a final draft of a Science Requirements Document, evaluation and recommendation of flight hardware, preparation of a project plan, and preparation and presentation of a MSAD Requirements Definition Review (RDR). As an option, design and fabrication of a flight type sample ampoule assembly for preliminary ground tests in the Ground Control Experiment Laboratory (GCEL) hardware is included. The proposed study will involve the development of a more detailed model and the verification of the Jackson and Hunt model. Verification will require directional solidification of immiscible alloys (aluminum-indium alloys in the current investigation) under low-g conditions in order to avoid convective instability and promote steady-state coupled growth.

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.

The premier work on modeling of eutectic solidification was carried out by Jackson and Hunt in 1966. Jackson and Hunt's analysis addressed diffusion-controlled, coupled growth of the two solid phases, α and β, from the liquid in both eutectic and off-eutectic composition alloys. At least one attempt has been made to modify Jackson and Hunt's analysis in order to produce a model for directional solidification in immiscible systems.

PROGRESS DURING FY 1993:
The major task in this project involves experimentation which will investigate coupled growth in hypermonotectics. One major area associated with this task is the final selection of an ampoule material for the flight experiment. There are several candidate materials being considered as well. These materials include graphite and pyrolytic boron nitride. It is necessary to investigate both the wetting characteristics of the immiscible alloy with these ampoule materials and the chemical compatibility. Wetting characteristics are being determined by melting the immiscible alloy to be utilized in this study (aluminum-indium) in small crucibles made of the
candidate materials. Processing is carried out so that phase separation between L₁ and L₂ occurs. The contact angle between the L₁-L₂ interface and the crucible wall is then measured to determine the wetting tendencies. The initial tests in this area are almost completed. Promising materials will be further investigated using long-term compatibility testing.

Efforts are also currently underway to model the redistribution of constituents at the solidification front during processing. This work is being carried out by Dr. Sam Coriell at the National Institute of Standards and Technology.

**STUDENTS FUNDED UNDER RESEARCH:**

PhD Students: 2

**TASK INITIATION:** 01/93  **EXPIRATION:** 01/96

**PROJECT IDENTIFICATION:** 694-25-08-09

**NASA CONTRACT NO.:** NAG8-397

**RESPONSIBLE CENTER:** MSFC

**BIBLIOGRAPHIC CITATIONS FOR FY 1993:**

**Journals:**


**Proceedings:**


**NASA Technical Memorandums:**


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:

W. Hofmeister

Vanderbilt University

M. Robinson

NASA Marshall Space Flight Center (MSFC)

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

OBJECTIVE:
The primary scientific objective is to further the understanding of nucleation of solids from their melts. Within the envelope is a concern for the role of melt agitation on nucleation. The practical objective is to determine, in qualitative terms, the necessity for containerless processing in low Earth orbit, with regard to nucleation studies.

DESCRIPTION:
Comparisons of the data from the ground-based techniques give evidence 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. Since nucleation is a statistical process, approximately one hundred undercooling measurements are recorded for each type of sample. This number of measurements allows for statistical assumptions to be made, thereby easing the interpretation of results. In addition, considerable effort on contactless temperature measurement techniques is being made. Increasing the precision of temperature measurement is an important part of the experiments because of its dramatic effect on the results.

SIGNIFICANCE:
All possible ground-based methods are being investigated for applicability to containerless processing of bulk samples of pure metals.

PROGRESS DURING FY 1993:

Nucleation frequency experiments on zirconium were conducted using the Marshall Space Flight Center Drop Tube Facility and the electromagnetic levitation apparatus at Vanderbilt University. Temperature measurement at both locations was done by optical pyrometry. Experiments were done on 99.5% pure stock and 99.95% pure stock.

Over the entire sample set, maximum undercoolings ranged from 13% to 15% of the equilibrium freezing temperature, but higher-purity samples tended to produce higher undercoolings. Also, a fundamental difference in the distribution of undercoolings was seen in the different environments. A low undercooling tail was present in the levitator undercooling distribution, whereas a similar tail was not observed in the drop tube. In both cases, calculations based on the Skripov approach to the analysis of nucleation experiments yielded values in the heterogeneous regime for the exponential and pre-exponential factors in the nucleation rate equation.

STUDENTS FUNDED UNDER RESEARCH:

PhD Students: 1  PhD Degrees: 1

TASK INITIATION: 04/90  EXPIRATION: 06/95

PROJECT IDENTIFICATION: 694-25-08-06

NASA CONTRACT NO.: GN8-978

RESPONSIBLE CENTER: MSFC
II. MSAD Program Tasks — Flight Research

Discipline: Materials Science

BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:


Presentations:


II. MSAD Program Tasks — Flight Research

Alloy Undercooling Experiments in Microgravity Environment

PRINCIPAL INVESTIGATOR: Prof. Merton C. Flemings
Massachusetts Institute of Technology (MIT)

CO-INVESTIGATORS:
Y. Wu
Massachusetts Institute of Technology (MIT)

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

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.

DESCRIPTION:
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, collect thermal history, nucleation and growth history, and resulting solidification microstructures.

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 1993:
Specimens were prepared for and delivered to DLR and Intersonic for calibration and property measurements. Specimens were prepared and delivered for flight experiment and backups. Some of the thermophysical properties of alloys were determined using differential thermal analysis, including onset temperatures and heats of phase transformations during heating from room temperature to liquid temperatures. The high-vacuum facility was installed for further ground-based work on Ni-base alloys to follow (work is in progress). Progress has been made in ternary Fe-Ni-Cr alloys to test the feasibility and effectiveness of additional alloys for inclusion in follow-on microgravity experiments.

STUDENTS FUNDED UNDER RESEARCH:

PhD Students: 2

TASK INITIATION: 04/90 EXPiration: 06/95
PROJECT IDENTIFICATION: 694-25-05-03
NASA CONTRACT NO.: GN8-971
RESPONSIBLE CENTER: MSFC
BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Proceedings:
Compound Semiconductor Growth in Low-g Environment

**PRINCIPAL INVESTIGATOR:** Dr. Archibald L. Fripp

**NASA Langley Research Center (LaRC)**

**CO-INVESTIGATORS:**
- R.K. Crouch
  - NASA Headquarters, Code UG (MSAD)
- W.J. Debnam
  - NASA Langley Research Center (LaRC)
- I.O. Clark
  - NASA Langley Research Center (LaRC)

**TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:**

**OBJECTIVE:**
The objective of the Langley flight program is to determine the effects of gravity-driven convection on the growth conditions and crystal properties of the compound semiconductor alloy, lead tin telluride which is a substitutional alloy that is miscible over the entire compositional range.

**DESCRIPTION:**
The electronic properties of this material are dependent on the ratio of the two 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. The 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 similar phase diagram to other compound semiconductors of interest such as mercury cadmium telluride and mercury zinc telluride.

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.

**SIGNIFICANCE:**
Lead tin telluride is 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 that, in turn, produce driving forces for convection when coupled with gravity.

Earth-based 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 a 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.

**Progress During FY 1993:**
This year's effort concentrated on finalizing the AADSF furnace configuration and calibration. Lead tin telluride has been grown in the flight configuration. The newly designed calibration cartridge has been used for extensive...
characterization of the AADSF furnace. Doped germanium samples, with interface demarcation, have been grown in
the prototype AADSF for comparison to the calibration data. Numerical modeling of the furnace and samples is
presently underway for subsequent extrapolation to modeling of lead tin telluride in the furnace.

A cartridge with a miniature internal pressure gauge was designed and tested in furnaces both at Langley and
Marshall. These tests not only measured the pressure as a function of set point temperatures (yes, the gas law still
applies) over the different thermal gradients but also measured the integrity of the cartridge seals and the rigidity of
the cartridge as a function of temperature and differential pressure.

We have also continued with compatibility tests between the lead tin telluride and potential cartridge materials. The
Inconel cartridge does not contain molten lead tin telluride but tests with WC-103 indicate that this material is
impervious to the molten charge for at least the length of time of a typical growth run.

<table>
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Journals:
Rosch, W., Debnam, W., Fripp, A., Jesser, W.A., Pendergrass, T.K., and Woodell, G.A. A technique for measuring the

Rosch, W., Debnam, W., Fripp, A., Simchick, R., and Sorokach, S. Damage of fine diameter platinum sheathed type R
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Sears, B., Anderson, T.J., Fripp, A.L., and Narayanan, R. The detection of solutal convection during electrochemical


Simchick R.T., Barber, P.G., Berry, R.F., Debnam, W.J., Fripp, A.L., and Sorokach, S. Gamma ray and X-ray imaging
studies of the location and shape of the melt-solid interface during Bridgman growth of germanium and lead tin telluride.

Presentations:
Hubert, J.A., Fripp, A.L., and Welch, C.S. Comparison of melt-solid interface position by radiography and temperature
AIAA 93-0475.

Knuteson, D.J., Debnam, W.J., Fripp, A.L., and Woodell, G.A. Time dependent convection in a Bridgman system, ACCG,
Baltimore, MD, August 1-3, 1993.
Melt Stabilization of PbSnTe in a Magnetic Field

**Principal Investigator:** Dr. Archibald L. Fripp  
**NASA Langley Research Center (LaRC)**

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

**Task Objective, Description & Significance:**

**Objective:**
The objective of this research is to further elucidate the gravity-driven physical phenomena on the growth of the alloy compound semiconductor, PbSnTe.

**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 1994 AADSF 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.

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.

**Significance:**
This work, coupled with the past microgravity experiment with the MEA and the existing flight program to grow PbSnTe in the AADSF, will form the most comprehensive set of space processing experiments performed to date. This 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 FY 1993:**
The progress within this first year of the research on the magnetic stabilization of PbSnTe consists primarily of preparations for future quantifiable, modelable experiments.

The ampoule configuration has been design for ground based tests in the five Tesla magnetically stabilized furnace at the Marshall Space Flight Center. Ampoules have been fabricated and the starting crystal boules have been cast, loaded and one sample directionally solidified in the Marshall furnace. Analysis of this crystal is presently underway.
One of the key thermophysical parameters that determine the potential for magnetic stabilization of any material is the electrical conductivity of the melt. This value is known for the end materials, PbTe and SnTe, but only estimates exist for the ternary compound. Agreements for a joint effort with the Thermophysical Division of the National Institute of Standards and Technology (NIST) have been made to perform these measurements. Measurement samples have been cast and delivered to NIST for these measurements.

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.

TASK INITIATION: 10/93  EXPIRATION: 9/95
PROJECT IDENTIFICATION: 694-80-07-05
RESPONSIBLE CENTER: LaRC
II. MSAD Program Tasks — Flight Research

Gravitational Role in Liquid-Phase Sintering

PRINCIPAL INVESTIGATOR: Prof. Randall M. German
Pennsylvania State University

CO-INVESTIGATORS:
No Co-l's Assigned to this Task

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

OBJECTIVE:
The purpose of this research is to establish how gravity influences the macro and microstructural development of tungsten heavy alloys during liquid phase sintering (LPS).

DESCRIPTION:
The investigation is in the definition phase where science requirements will be established. The investigation may then be confirmed for the execution phase if it is approved for flight. If flown, the experiment will be conducted in the Japanese-developed large Isothermal Furnace (LIF) on the IML-2 mission.

SIGNIFICANCE:
Experiments in space will determine grain growth kinetics without settling and gravity-induced liquid convection.

PROGRESS DURING FY 1993:
The P.I. Prof. German, completed a successful Flight Science Readiness Review (FSRR) in June 1992. All of the samples sintered in the Large Isothermal Furnace (LIF) engineering model facility at the IHI Mizuho plant in Tokyo, Japan, have now been returned to Penn. State. During this reporting interval the analyses of the "1-minute" and the "120-minute" samples were finished.

TASK INITIATION: 1/90 EXPIRATION: 9/94
PROJECT IDENTIFICATION: 694-25-05-05
RESPONSIBLE CENTER: LeRC
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, Description & Significance:

Objective:
The objective of the Isothermal Dendritic Growth Experiment (IDGE), developed for two of the United States Microgravity Payload (USMP) flights starting with USMP-2, is designed to provide microgravity data on dendritic growth for a critical test of theory.

Description:
The first IDGE flights, scheduled for the USMP-2 mission in February 1994, will measure dendritic growth events in succinonitrile (SCN; CN-(CH2)2-NC), a transparent, body-centered-cubic plastic crystal that freezes with characteristics similar to cubic metals, and therefore acts as an analog for metal solidification. Furthermore, SCN's thermophysical properties are well characterized; it is transparent to visible light, has a convenient (for experimentation purposes) melting temperature, and therefore serves as an excellent test substance for this investigation.

Ground-based measurements have clearly delineated the influences of gravity on the dendritic growth process. Specifically, gravity-driven convection (natural or buoyancy-driven convection) starts to affect the growth rate and crystal morphology of SCN dendrites when the supercooling is less than about 1.2 K. At large supercoolings (> 1 K) gravity plays a minor role, because convection provides weak transport relative to thermal diffusion. Buoyancy-driven convection arises spontaneously from the interaction of the gravitational body force with the microscopic density gradients produced in the fluid phase by the thermal transport field responsible for the flow of latent heat from the solid to the supercooled melt. Then buoyancy-driven convection becomes an increasing and then an overwhelming kinetic factor as the supercooling decreases. Unfortunately, it is only at small supercoolings—where gravity effects dominate—that the kinetic conditions are suitable for checking dendritic growth theories.

Thus, the first IDGE flight will perform photographic observations at a variety of supercoolings in the 0.1 K - 1.0 K range. A novel growth chamber, millikelvin thermostat, and optical system have been developed to accomplish this task, for operation in orbital microgravity, under semi-autonomous conditions. In addition, IDGE science and engineering teams have developed a photographic analysis system and automated techniques to extract data from the IDGE photographs.

The IDGE growth chamber permits a single sample of SCN (greater than 5-9's purity) to be repeatedly melted, supercooled, nucleated, and photographed on orbit. Dendritic growth velocity and tip radii measurements can be derived from optical measurements for each of the selected supercoolings. IDGE provides two independent optical axes for stereographic correction. Up to 250 35 mm photographs can be taken, which allows up to 37 independent dendritic growth sequences at the preselected supercoolings, where 10 photographs (one for each 1.5 mm of growth) record each supercooling and each optical axis.

On-board accelerometry (derived from the SAMS) temperature measurement and control, and data for other relevant experimental conditions, including slow-scan video down-link of the dendritic growth events, are all available during
flight in near real time to the payload operations control center from the IDGE. Up-linking of a restricted set of commands for the adjustment of the experimental protocols, including experiment interruption, repeated runs, shift of supercooling, etc., are also communication features of the IDGE.

Future integrated ground-based test using the flight hardware will provide both pre- and post-flight ground-based data sets. Any difference between the microgravity and terrestrial data sets, using the same growth chamber, material and ancillary apparatus, will demonstrate conclusively the effects of microgravity on dendritic growth and provide a critical test of theory.

SIGNIFICANCE:
Dendrite crystallization is common in most industrial casting and welding processes, and it remains a subject of interest to both scientists and engineers attempting to understand the factors that control dendritic patterns and microstructures and nonlinear dynamic patter formation in general. Current theories of dendritic pattern formation are based primarily on diffusive transport, with convection either ignored altogether or added as a separate phenomenon.

In actuality, the simultaneous presence of convection and diffusion during dendritic growth in the presence of Earth’s acceleration field results in coupled transport that in turn, alters the dendritic “operating state,” which is often expressed as the speed and size scale of the crystals for a given supercooling or thermal driving force. The ability to perform a critical test of dendritic growth theory depends on the availability of a suitable experimental system that provides precise, quantitative, thermodynamic driving forces acting on a well-characterized material. IDGE is designed to meet these specifications.

PROGRESS DURING FY 1993:
In FY 1992 the project has achieved considerable success. The Principal Investigator, his science and engineering team, and the NASA LeRC IDGE engineering team have spent considerable efforts on the continued integrated tests of the IDGE prototype engineering hardware and the subsequent analysis and data reduction. This work was incorporated into the final design and manufacture of the flight hardware and analysis tools.

The results of this work, both at Rensselaer and LeRC, have contributed to the latest Science Verification Review (SVR) results. In these tests, the dendritic velocity data have little scatter, and are consistent with the historical (conventional laboratory) data set.

The radii data exhibit more scatter than do the velocity data. The differences between the IDGE and historical data are due, primarily, to the differing chamber sizes supporting different states of natural convection. At present, for supercoolings equal to or greater than 0.77 K, we are unable to analyze shadowgraphic photographs for dendritic radii and maintain an uncertainty of less than 5%, which is our goal. However, the lack of determinacy in the radii data at the larger supercoolings is not an insurmountable problem, because we can use the velocity data to correlate the historical, the IDGE ground-based, and the IDGE space-flight data sets. At the lower supercoolings, both the radii and velocity data are easily measured, and the differences between space-flight and ground-based data should be clearly distinguishable.

These improved SVR results were achieved in many ways. Extensive numerical modeling showed that the differences between the Science Verification Review (SVR) data and the historical data could not be explained by gaussian scatter in the photographic edge function. Nor could the gaussian scatter explain the radii in the SVR data sets. These tests also indicated how the gaussian scatter in the edge function could contribute to the radii scatter in the data, and how to minimize this by analytical methods. Curve-fitting tests yield information on how to compare differing data sets.
Detailed examination of the photographic negative subsequently revealed three sources of scatter: (a) some dendrites grow so close to neighboring dendrites that they are no longer isothermal; (b) some dendrites grow so that a crystallographic [110] profile is presented to the optic system, rather than a [100] profile, and whereas the [110] profile has a parabolic shape for a much smaller distance from the dendrite tip, the tips can appear to be too small; (c) some dendrites have not achieved steady-state growth by the time they leave the field of view. The problems associated with nonisothermal and non-steady-state dendrite tips have hardware fixes that will be described later. For the situation of the [110] profiles, they will simply have to be recognized and separated from the rest of the data set. We also discovered that the background illumination, attributed to the flash unit in the engineering unit, provided a nonuniform light intensity that was detrimental to the photographic analysis. This too has been fixed through the modification of the optic system on the flight unit.

During FY 1992 we have taken the results for the SVR tests on the engineering hardware, incorporated improvements to the flight hardware design, constructed MOD III or flight quality growth chambers, filled one flight quality chamber with 5-9's pure SCN, completely tested and retested that chamber, and delivered it to LeRC. We also filled and partially tested a second flight quality chamber, and are waiting for the delivery of an improved and sturdier thermoelectric cooler unit to use in completing the assembly of this second chamber.

The most critical and obvious hardware design change has been to the stinger. It has been moved higher into the chamber so that a dendrite emerging from the tip has the entire field of view of the photographic frame in which to grow. In addition the stinger has been notched, much like the tip of a fountain pen, so as to isolate the dendrite tips better and increase the probability of isothermal growth conditions. Tests at Rensselaer show that this latter change was successful.

Many less obvious changes have also been made to the chambers and in the way we handle the manufacturing process. The windows have been polished; the thermistors have been aged to reduce their drift. Our laboratories at Rensselaer have been changed so as to separate and isolate the laboratory-based growth-chamber work from the data analysis operations, thereby reducing the possibility of contaminating the growth chambers. The piece parts to the chambers have been, at several intermediate stages, cleaned and bagged in a microelectronic clean room facility at the Center for Industrial Innovation (CII) here at Rensselaer. Finally, all parts of the cleaning, filling, and testing procedures were fully documented and followed to insure rigorous reliability and quality assurance.

At the end of FY 1992 we had fully implemented a computer aided dendrite analysis program (CADAP), version 1.2, running on a 486-based computer. This program evolved from the work at both Rensselaer and LeRC over the last several years. CADAP, however, is not stand-alone, but is used best in conjunction with the precision optical state (ROI) that we have used the last few years. The ROI now has a black-and-white camera with variable gamma function and on/off automatic gain control. Furthermore, CADAP is only used for measuring radii. Velocities, magnification of the flight optic system, dendritic growth angle for the stereographic correction, transmission data for the image processing, etc. are measured on the ROI or other instruments, and the results are input to CADAP if required. We intend to test and complete the development of CADAP and ancillary apparatus and methods early in FY 1993, with the arrival of the first few integrated mission simulations using the flight chamber with the flight hardware.

Finally, we have continued to make progress for the second and third IDGE flights. We have filled and begun testing a pivalic acid (PVA) engineering growth chamber, and are continuing to seek improvements in the day-to-day operation of our laboratories. Our new vacuum distillation system has produced enough SCN for the current and future flights, and we are beginning a production run to produce high-purity PVA.
II. MSAD Program Tasks — Flight Research

Discipline: Materials Science

STUDENTS FUNDED UNDER RESEARCH:
Total Students: 5 Total Degrees: 1

TASK INITIATION: 12/88 EXPIRATION: 12/98
PROJECT IDENTIFICATION: 694-25-05-01
RESPONSIBLE CENTER: LeRC

BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:


Proceedings:


Presentations:
Glicksman, M.E. Evolution of dendritic crystal structures, ICI Explosives Group Technical Centre, McMasterville, Quebec, Canada, May 7, 1993.


Glicksman, M.E., and Koss, M.B. How to measure the tip size of a dendritic crystal, Ninth American Conference on Crystal Growth, Baltimore, MD, August 2, 1993.


II-110
II. MSAD Program Tasks — Flight Research

**Discipline: Materials Science**

**Evaluation of Microstructural Development in Undercooled Alloys**

**Principal Investigator:** Dr. Richard N. Grugel
Vanderbilt University

**Co-Investigators:**

W.F. Flanagan
Vanderbilt University

**Task Objective, Description & Significance:**

**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 ($\Delta T$), composition ($C_o$), and volume ($V_o$).

**Description:**
Nucleation of the undercooled alloy samples will be induced, and the extent of dendritic growth will be evaluated as a function of undercooling ($C_o$ and $V_o$ constant), composition ($\Delta T$ and $V_o$ constant) and volume ($T$ and $C_o$ constant). Varying the composition will in effect vary the volume fraction of the primary dendrites and, consequently, the initial amount of heat released due to recalescence. Varying the sample size will affect the rate of cooling after recalescence. Transitions in the microstructure (for example, a change from columnar dendritic to equiaxed grains) are also an important consideration and will be noted as a function of the above parameters. Critical evaluation of solute distribution and primary dendrite trunk diameters will provide information with regard to the internal dendrite growth velocity and will allow for comparison with those measured on the sample surface. The internal solidification history will be further understood through evaluation of the evolution of the structure of interdendritic eutectic, and not merely the change in spacing.

**Significance:**
Following microstructural evolution from the site of nucleation, this will provide valuable information with regard to maintaining the integrity of desired microstructure and the nature of abrupt morphological transitions. Irrefutable trends in microstructural development will be evaluated in terms of present theory, which will be amended as necessary. Finally, evaluation of the ground-based experiments will ascertain the feasibility of processing undercooled bulk melts in microgravity.

**Progress During FY 1993:**

Nucleation was induced in lead-61.9 wt pct Sn (eutectic) alloys which were undercooled from $\Delta T=5$ to 25K below the equilibrium freezing temperature. Microstructural development was then characterized from the point of nucleation with significant variations found. In view of these experimental results and predictions from a model, the implications for processing such alloys, particularly in a microgravity environment, are considered.

A solidification model for undercooled melts which is Newtonian in nature and follows that of Levi and Mehrabian has been developed. Assuming the interface to grow radially from the point of nucleation the extra degree of freedom introduced by the non-isothermal condition can be accounted for and the equation is solved by iteration using the Newton-Raphson technique at each time step, starting with the nucleation temperature and with appropriate parameters and material properties from the literature.
Results of the model show the solid fraction to increase rapidly during the first 1-2 seconds after which it increases at a slower rate. It is also seen that the temperature reaches to just under that of the eutectic (183°C) before the first tenth of the solid forms. Subsequently, the calculated velocity of the solidification interface as a function of the fraction solid drops off very quickly.

Experimentally for ΔT's of 5, 10, 15, and 20K a columnar structure is seen to emanate from the point of nucleation in a fan-like manner with the extent of columnar eutectic growth increasing with increased undercooling. Growth of the columnar eutectic is interrupted by competing equiaxed grains. In contrast, at ΔT=25K eutectic growth is not apparent as is evidenced by a dendritic structure about the point of nucleation.

In view of the model and experimental results, it is not obvious that processing of undercooled "bulk" alloys in a microgravity environment will be advantageous.

STUDENTS FUNDED UNDER RESEARCH:
Total Students: 1

TASK INITIATION: 12/91  EXPIRATION: 11/94
PROJECT IDENTIFICATION: 694-25-07-03
RESPONSIBLE CENTER: JPL
Thermophysical Properties of Metallic Glasses and Undercooled Alloys

**Principal Investigator:** Dr. William L. Johnson  
California Institute of Technology (Caltech)

**Co-Investigators:**  
D. Lee  
California Institute of Technology (Caltech)

**Task Objective, Description & Significance:**

**Objective:**
The objectives are to study physical properties of undercooled metallic alloy melts that relate to glass formation and develop calorimetric methods to investigate the specific heat and thermal conductivity of alloy melts, both in the equilibrium and undercooled state.

**Description:**
In the experiment, ground-based measurements of the specific heat of glass-forming liquid alloys are made using a differential scanning calorimeter and a drop calorimeter. A noncontact AC calorimetry method for high melting-point transition metal alloys will be implemented using the TEMPUS facility on the IML-2 mission. Samples chosen for study with the TEMPUS hardware and software are Zr$_{70}$Ni$_{30}$ and Nb$_{80}$Ni$_{20}$. Dr. Shankar Krishnan at Intersonics Corporation in Chicago will examine these samples for spectral emissivity measurements at 633 nm, and the data will be used to calibrate temperature measurement on TEMPUS.

In addition, a ground-based blackbody bolometer will measure the total hemispherical emissivity of levitated liquid samples that will calibrate the heat flow in the flight experiments. A complete set of software input parameters for carrying out the AC, specific heat measurement using the TEMPUS facility will be developed. Liquid Zr-Ni and Nb-Ni alloys will undergo specific heat measurements both above and below the thermodynamic melting point of the alloys during the IML-2 flight of TEMPUS. This data will be used to calculate the entropy and free-energy functions for the equilibrium and undercooled liquids, and the Kauzmann isentropic temperature of the undercooled liquids will then be determined, comparing it to the observed glass transition temperature of the alloys.

**Significance:**
After data obtained during the flight experiments are analyzed, plans are to assess the results in terms of classical nucleation theory, and relate the results to the glass-forming ability of the alloys.

**Progress During FY 1993:**
We have completed the design and testing of a ground-based bolometer for measuring the total hemispherical emissivity of liquid metal samples. We have done extensive finite element analysis modeling of the design and of the heat flows in the levitation system. We are currently integrating this device, which was delivered to us in early November, 1993, onto an RF levitation system in the laboratory of Dr. Ken Ohsaka at JPL. Measurements of total hemispherical emissivity will begin by early 1994.

We have also carried out ground-based specific heat measurements on Ni-Zr and Ni-Nb alloys using several techniques: differential scanning calorimetry on rapidly-quenched samples, drop calorimetry on undercooled liquid samples, and cooling curve measurements on electrostatically-levitated samples. We are continuing to analyze these results in an effort to determine the optimum experimental parameter set for the TEMPUS software. We are working with the group of Professor Fecht in Germany on maturing the AC calorimetry technique and in Köln, Germany. The AC calorimetry method has been successfully demonstrated in the development module at DLR.
We have carried out AC calorimetry measurements on solid Nb, Zr, Ni-Zr, and Ni-Nb (liquids cannot be levitated in the lab model of TEMPUS). We are continuing these experiments and are also investigating the total hemispherical and spectral emissivities of our flight sample compositions both at DLR and at Intersonics Corporation, where Dr. Shankar Krishnan is measuring the spectral emissivity of our samples at 633nm. Finally, we have determined the evaporation rates for all the alloys in the TEMPUS IML-2 flight and have delivered flight samples to Dornier GmbH, which were then integrated into the flight model and delivered to NASA.

**STUDENTS FUNDED UNDER RESEARCH:**

<table>
<thead>
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<th></th>
<th>MS Students</th>
<th>PhD Degrees</th>
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**PhD Students:** 2

**TASK INITIATION:** 02/92  **EXPIRATION:** 06/95

**PROJECT IDENTIFICATION:** 694-25-08-07

**NASA CONTRACT NO.:** GN8-954

**RESPONSIBLE CENTER:** MSFC
Orbital Processing of High Quality Cadmium Telluride

PRINCIPAL INVESTIGATOR: Dr. David J. Larson

Co-Investigators:
A. Levy
DiMarzio
F. Carlson
J. Alexander
D. Gillies
J. Moosbrugger

Grumman Corporate Research Center
Grumman Corporate Research Center
Clarkson University
University of Alabama, Huntsville
NASA Marshall Space Flight Center (MSFC)
Clarkson University

Task Objective, Description & Significance:

Objective:
The objective of this research is to investigate the influence of gravitationally dependent phenomena on the growth and quality of cadmium zinc telluride (CdZnTe). The family of II-VI compound semiconductors, of which CdZnTe is a member, is used in the fabrication of nuclear detectors, gamma ray detectors, laser optics, electro-optic modulators, and infrared detectors.

Description:
The CdZnTe samples will be grown in microgravity using the seeded Bridgman-Stockbarger method of crystal growth. Bridgman-Stockbarger crystal growth is accomplished by establishing isothermal hot-zone and cold-zone temperatures with a uniform temperature gradient between. The thermal gradient spans the melting point of the material (1,095 °C). After sample insertion, the furnace's hot-and cold-zones will be ramped to temperature establishing a thermal gradient of 25 °C/cm and melting the bulk of the sample. The furnace will then move farther back on the sample, causing the bulk melt to come into contact with the seed crystal, thus "seeding" the melt. The seed crystal prescribes the growth orientation of the crystal grown. Havingseeded the melt, the furnace translation is reversed and the sample is directionally solidified at a uniform velocity by moving the furnace and the thermal gradient over the stationary sample.

Significance:
Orbital processing offers a unique opportunity to advance toward the goal of increased structural perfection within the bulk crystals and of more uniform specific lattice parameters within the substrate.

Progress During FY 1993:
During USML-1, the crew removed processed samples from the Crystal Growth Furnace (CGF) and inserted new samples, safely demonstrating sample exchange procedures required for handling potentially toxic materials in a closed life support system. This operation served as an effective demonstration for space station, and, as a result of the crew interaction, two GCRC samples were processed instead of the one planned, significantly increasing scientific yield. The samples were processed instead of the one planned, significantly increasing scientific yield. The Crystal Growth Furnace performed to expectations in every respect during its first mission.

Significant portions of the samples processed in microgravity grew without contact with the sample containment ampoule. In these "dewetted" regions, no twinning defects were recorded, suggesting that much twinning (a common crystalline defect in these materials) is surface nucleated, and that ampoule wall contact and stiction, which greatly increased local stress, are responsible. Later in the solidification process, portions of the sample experienced...
II. MSAD Program Tasks — Flight Research

Discipline: Materials Science

partial contact with the ampoule wall. In the regions of partial wall contact, twinning incidence was frequent, but localized, reinforcing the conclusion from the dewetted region. Internal twinning, not surface-generated, was invariably found to originate in regions of high mechanical deformation, usually in regions that evidenced cross-slip. This suggests the possibility that in regions of cross-slip the interacting partial dislocations form a stacking fault that serves to nucleate the twin. Though this is not yet confirmed, the concept offers the prospect of reducing twin density by reduction in the resolved shear stresses imposed during crystal growth. This concept might prove valuable as an approach to twin control (reduction) terrestrially. Twin control is highly significant to increasing the producibility of detector crystals.

Synchrotron white beam topography of the various flight sample surface regions clearly showed that slow growth, with low-imposed thermal gradients and without wall contact, produced the lowest strain and fewest defects. Quantitative comparative analyses of the bulk flight material with identically-processed ground truth samples revealed that the flight samples had significantly improved rocking curve widths in the best regions of the samples (10/20-arc-sec FWHM vs. 25/35 arc-sec FWHM), and significantly reduced dislocation defect densities ($\leq 10^3$ EPD vs. $5 \times 10^5/5 \times 10^3$ EPD) throughout. These results were quantitatively confirmed from transmission topographs imaged using monochromated synchrotron radiation.

Comparison of the flight results with the ground baseline and high fidelity process models developed for this program is underway. These comparisons were complicated by the thermal and gravitational asymmetries encountered during the process of the flight specimens. Ground-based results for vertical Bridgman-processed crystals have demonstrated for the first time, qualitative and quantitative correlation between the stress distributions predicted by the models and morphologies, subgrains and dislocations, imaged within the samples. This result suggests that substantive improvement of crystalline quality and yield is possible within this class of materials by applying these validated models.

<table>
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<th>STUDENTS FUNDED UNDER RESEARCH:</th>
<th>TASK INITIATION: 8/90 EXPIRATION: 12/93</th>
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<td>RESPONSIBLE CENTER: MSFC</td>
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BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:


II. MSAD Program Tasks — Flight Research

Discipline: Materials Science


Proceedings:


Presentations:


Larson, Jr., D.J. Bulk crystal growth and process modeling of VI semiconductor compounds, University of Alabama at Birmingham, February 2, 1993.

Larson, Jr., D.J. Reduced defect density in microgravity processed CdZnTe, International Symposium on Microgravity Science and Application, Beijing, China, May 10-13, 1993.

Larson, Jr., D.J. CdZnTe process model validation, ARPA/University of Virginia, Charlottesville, Virginia, June 24-25, 1993.


Larson, Jr., D.J. Microgravity crystal growth of II-Vi and III-V semiconductors, 9th American Conference on Crystal Growth, MD, August 2-6, 1993.


Larson, Jr., D.J. CdZnTe bulk crystal growth and process modeling, University of Alabama in Huntsville, September 24, 1993.


Crystal Growth of II-IV Semiconducting Alloys by Directional Solidification

**Principal Investigator:** Dr. Sandor L. Lehoczky  
**CO-INVESTIGATORS:**  
F. Szofran  
C. Su  
R. Andrews

**NASA Marshall Space Flight Center (MSFC)**  
**Universities Space Research Association (USRA)**  
**University of Alabama, Birmingham (UAB)**

**Task Objective, Description & Significance:**

**Objective:**  
The objective of this research is to investigate the effects of microgravity during the crystal growth of mercury zinc telluride (HgZnTe) on its compositional, metallurgical, electrical, and optical properties.

**Description:**  
The HgZnTe alloy for this experiment will be prepared by reacting 99.9999% pure, elemental constituents in evacuated, sealed, fused-silica ampoules. The HgZnTe will be agitated while molten to achieve complete reaction and homogenization, and then cast in the tapered end of an ampoule. The ampoule will then be installed in the hot zone of the growth furnace. The hot zone will be heated above the liquids temperature of the alloy, and the cold zone will be maintained at a temperature low enough to provide a gradient sufficient to prevent constitutional supercooling. The directional crystal growth will be accomplished by raising the temperature gradient through the ampoule. The crystal nucleates in the bottom tip of the ampoule and grows as the gradient is raised.

**Significance:**  
The anticipated results of this study are both scientifically and technologically significant. 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 liquids and solids 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 1993:**

One HgZnTe sample was planned to be processed for approximately 148 hours in the crystal growth furnace (CGF), which was flown in the pressurized Spacelab module in the habitable environment of the Space Shuttle Columbia during the first United States Microgravity Laboratory (USML-1) in June of 1992. Unfortunately, because of the loss of power to the CGF, the experiment terminated prematurely at about 39 hours into the experiment timeline. About a 5.5 mm sample was grown during this time period. The initial microstructural, compositional, and x-ray radiographic and diffraction results indicate that preprocessed alloy crystals can be successfully quenched, back-melted, and regrown maintaining nearly steady-state compositions. Further analysis of the sample is continuing.
II. MSAD Program Tasks — Flight Research

Discipline: Materials Science

Task Initiation: 12/92  Expiration: 09/96
Project Identification: 694-21-08-04
NASA Contract No.: In-house
Responsible Center: MSFC

BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:

Proceedings:


Presentations:


Volz, M.P., Sha, Y-G, and Lehoczky, S.L. Compositional distributions and electrical properties of Hg_{1-x}Cd_xTe grown by CVT, Ninth American Conference on Crystal Growth, Baltimore, MD, August 1-6, 1993.

GaAs Crystal Growth Experiment

PRINCIPAL INVESTIGATOR: Dr. David H. Matthiesen  
Case Western Reserve University

CO-INVESTIGATORS:
Dr. J.A. Kafalar  
GTE Laboratories, Inc.
Dr. B.M. Ditchek  
GTE Laboratories, Inc.

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

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

DESCRIPTION:
Selenium-doped (~10e-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.

PROGRESS DURING FY 1993:
The GaAs/GAS payload flew on STS-40 (June 1991) and on STS 45 (March 1992). Dr. Matthiesen presented results of characterization and analysis of national meetings at the AIAA, the American Soc. of Crystal Growth, and the Gordon Research Conference. Sample characterization continued subsequent to Dr. Matthiesen's move to Cleveland, his appointment to CWRU Materials Science and Department of Engineering and the construction of his new laboratories at the university.

Results to date emphasize the complexities of dynamic crystal growth. The shape of the interface has required modification of existing theory for analyses and the distribution of dopant near the interface cannot be explained by existing theory.

STUDENTS FUNDED UNDER RESEARCH:
BS Students: 1

BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Presentations:
**Diffusion Processes in Molten Semiconductors**

**Principal Investigator:** Dr. David H. Matthiesen  
Case Western Reserve University

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

**Task Objective, Description & Significance:**

**Objective:**  
One of the primary objectives of the Phase A Definition Plan is to perform experiments that identify any dependence of the measured diffusion coefficient on capillary diameter and, thus, the need to perform diffusion experiments in space. This influence is predicted to be due to convection with larger capillary diameters and due to wall effects when the capillary is small.

**Description:**  
The research in the Phase A definition consists of four areas of concentration: shear cell experiments, numerical modeling, physical modeling, and molecular modeling. The numerical modeling will be used to optimize the experiments, to understand the fluid dynamics of the shearing, and to access convective and wall effects. The physical modeling will be used to investigate phenomena that cannot be numerically modeled, such as the fluid-fluid shear that occurs during shearing. The molecular dynamics techniques will be used to calculate diffusion coefficients as functions of temperature and impurity concentrations.

**Progress During FY 1993:**

◊ **Experiments:**  
A fast and accurate technique for preparing shear cell charges has been developed. Many shear cell modifications have been done, including overfills. The overfills have been designed and tested. The shearing mechanism has been implemented in existing furnace hardware. We have demonstrated our capability to melt, shear and remove Ga-doped Ge samples from a graphite shear cell.

◊ **Numeric Modeling:**  
The numerical modeling has given us insight into the fluid dynamics of the shearing action. The minimum aspect ratio for shear cell segments has been determined. Convective effects have been quantified in regards to the measured diffusion coefficient. Wall effects were included in some of the simulations and the effect on the measured diffusion coefficient quantified. A first estimate of the error bar size on the measured diffusion coefficient has also been determined through numerical modeling.

◊ **Physical Modeling:**  
A physical model has been developed, fabricated, and tested. The physical model will be used during spring 1994.

◊ **Molecular Modeling:**  
A molecular dynamics program has been written and tested for calculating the diffusion coefficients. Potentials for liquid Ge and Si have been determined, and empirical Ga-Ga and Ga-Ge potentials have been obtained to treat Ga
impurities in liquid Ge. Using these potentials, self-diffusion coefficients have been calculated for liquid Ge and Si, and for Ga impurities in liquid Ge, both as functions of temperature.

**BIBLIOGRAPHIC CITATIONS FOR FY 1993:**

**Journals:**


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**TASK INITIATION:** 1/93  **EXPIRATION:** 1/94
**PROJECT IDENTIFICATION:** 694-21-05-04
**RESPONSIBLE CENTER:** LeRC
The Study of Dopant Segregation Behavior During the Growth of GaAs in Microgravity

**Principal Investigator:** Dr. David H. Matthiesen  
**Case Western Reserve University**

**Co-Investigators:**

J. Kafalas  
**Viable Systems, Inc.**

**Task Objective, Description & Significance:**

**Objective:**
The objectives of this program are to investigate gravitational and thermal techniques for obtaining complete axial and radial dopant uniformity of the selenium dopant during crystal growth of gallium arsenide (GaAs). These techniques include controlling the thermal conditions to obtain a flat interface shape and a steady-state growth rate and, most important, growth in the microgravity environment afforded by the Crystal Growth Furnace (CGF) in the first United States Microgravity Laboratory (USML-1).

**Description:**
Crystals of GaAs, 16.5 cm long by 1.5 cm in diameter, were supplied by GTE Laboratories to NASA for growth in CGF on USML-1. These crystals were doped with selenium to approximately $1 \times 10^{17}$ cm$^{-3}$. As supplied to NASA, these crystals were hermetically sealed in a specially designed fused quartz ampoule. This ampoule was sealed into an experiment cartridge, which on orbit was loaded unto the furnace system for growth. After flight, extensive characterization of the electrical and structural properties was done.

**Significance:**
The large hot zone length of the CGF (20 cm) should allow, for the first time in microgravity, the achievement of steady-state growth rates.

**Progress During FY 1993:**

Three ampoules containing GaAs have been tested at GTE Laboratories. In addition, two alumina thermal probes and three GaAs samples have been tested in the Ground Control Experiment Laboratory (GCEL). The GCEL contains a ground-based equivalent of the flight unit, which has been delivered to the Kennedy Space Center for integration into the United States Microgravity Laboratory.

The flight samples have been returned to CWRU and analysis is proceeding. Preliminary infrared measurements indicate that the flight sample initially began growth in a diffusion controlled regime and then subsequently was driven into a complete mixing regime. These measurements are currently being verified with Hall effect measurements. In addition, the acceleration data is being investigated, as it is made available, to determine the cause of this result.

**Task Initiation:** 5/92  
**Expiration:** 9/93  
**Project Identification:** 694-21-05-01  
**NASA Contract No.:** NAS8-397  
**Responsible Center:** MSFC
II. MSAD Program Tasks — Flight Research

Discipline: Materials Science

BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:


Presentations:
Matthiesen, D.H. The total pressure of arsenic over GaAs at 1260°, American Conference on Crystal Growth, Baltimore, August, 1993.

Matthiesen, D.H. and Majewski, J.A. The study of dopant segregation behavior during the growth of GaAs in microgravity, USML-1 L+1 meeting, Huntsville, September, 1993.

Matthiesen, D.H. Crystal growth results from the steady state growth of selenium doped GaAs in microgravity: The crystal growth furnace on USML-1, American Conference on Crystal Growth, Baltimore, August, 1993.
Temperature Dependence of Diffusivities in Liquid Metals

**PRINCIPAL INVESTIGATOR:** Prof. Franz Rosenberger  
**University of Alabama, Huntsville**

**CO-INVESTIGATORS:**  
R. M. Banish  
**University of Alabama, Huntsville**

**TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:**

**OBJECTIVE:**
The technological and scientific objectives of the proposed work are:

- Development of a technique for dynamic *in situ* measurements of diffusivities and their temperature dependence in melts with much higher efficiency than current approaches permit;

- Development of a flight-centered hardware package (GAS container) to perform such diffusivity measurements automatically, with relative high flight frequency;

- Establishment of a large definitive data base for the temperature dependence of self-diffusivities in liquid metals and alloys in order to further the development of the theory of diffusion in liquids and our understanding of numerous diffusion processes underlying materials processing; and

- Exploration of the possibility of approximating low-gravity diffusion conditions in conducting liquids on Earth through the application of magnetic fields.

**DESCRIPTION:**
All earlier liquid diffusion experiments conducted under low-gravity conditions were based on the establishment of step-function-like initial concentration distributions and required solidification of the samples for post-flight deduction of the diffusivities. Thus, only one data point for the diffusivity at one temperature can be obtained per sample. In order to simplify the requirements for the initial conditions, to obtain more insight into the dynamics of the diffusion process, and to increase the data yield per sample, we have developed a different approach. Originally, this approach was conceived for interferometric determinations of diffusivities in transparent solutions. But, utilizing a different physical mechanism for the concentration monitoring, the concept is applicable to liquid metals as well.

**SIGNIFICANCE:**
The diffusion of species in liquids governs many materials preparation processes of great technological importance. In particular, a detailed understanding of diffusion in liquid metals and alloys is pivotal for the interpretation of numerous phenomena encountered in metallurgical and semiconductor manufacturing processes. In gases and crystalline solids, studies of pure diffusion can readily be performed with great accuracy at normal gravity. In liquids, such measurements are hampered by difficult-to-control contributions from convection.

As a consequence of this experimental difficulty and the complex structures of liquids, our fundamental understanding of diffusion in melts is very limited. For instance, there are currently various theoretical models for the temperature dependence of diffusivities in liquids, ranging from a pure Arrhenius-type behavior (exponential increase with temperature) to combinations of Arrhenius laws with power laws and pure power laws. Due to the limited accuracy of available liquid diffusion data, the theoretical development is at an impasse. Accurate liquid diffusion data are urgently needed to stimulate further development of both fundamental diffusion theory and
transport models essential for materials processing. Our flight definition/development program will provide a broader database for liquid diffusivities and their temperature dependence.

Diffusivities will be determined in situ through multi-channel detection of radioactive tracer emission. The calculation of the diffusivities from the evolving concentration profiles will be facilitated by a novel algorithm, which is not limited to the simple initial conditions traditionally used, but enables data deduction from any consecutive smooth concentration distributions. Hence, diffusivity data can be gathered over a range of temperatures in a single experiment, which was not possible in earlier diffusivity measurement approaches.

**Progress During FY 1993:**

During the first six months of this study, the following tasks were accomplished. The steady-state sample temperature nonuniformity in a silica-enclosed sample inside a heated gold cell was estimated using an axisymmetric model with conductive heat transfer only. An estimation of the radioactive dose (weight of radioactive material deposited by electroplating) required was made based on geometrical, material (including heat transfer/insulation estimates), and detector considerations. We began to set up a model to carry out a systematic system performance analysis. Detectors, radioactive calibration standards, interface boards and software, and heater cartridge have been specified and ordered. Detectors and their electronics were checked for statistical noise fluctuations at different bias voltages, threshold discriminator levels, source energies, counting times, and temperatures. A heater cartridge which consists of a graphite heater enclosed in a pyrolytic boron nitride support was tested with a copper liner. Milestones obtained from GSFC have been worked into the schedule of activities.

**Students Funded Under Research:**

| MS Students | 1 |

**Bibliographic Citations for FY 1993:**

**Journals:**


II. MSAD Program Tasks — Flight Research

**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, Description & Significance:**

**Objective:**
Lead bromide is a highly attractive material for acousto-optic devices. Hardening of the crystal by dilute doping has been investigated. This is necessary in order to prevent damage caused by post-processing operations. However, bulk doping is difficult to achieve due to thermal-solutal convection. This work will test convection effects in normal and reduced gravity and relate the results to the growth of device-grade crystals.

**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.

**Progress During FY 1993:**
Minutes of the March 1992 Flight Science Readiness Review have been published. Dr. Singh is taking action based on the recommendations of the review.

**Task Initiation:** 10/93  **Expiration:** 9/94
**Project Identification:** 694-21-05-03
**Responsible Center:** LeRC
II. MSAD Program Tasks — Flight Research

Discipline: Materials Science

Particle Engulfment and Pushing by Solidifying Interfaces

PRINCIPAL INVESTIGATOR: Dr. Doru M. Stefanescu
University of Alabama

CO-INVESTIGATORS:
P. Carreri
NASA Marshall Space Flight Center (MSFC)

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

OBJECTIVE:
The scientific objective of the present proposal is twofold: to enhance the fundamental understanding of the physics associated with the solidification of liquid metals/ceramic particles mixtures; and to investigate aspects of melt processing of particulate metal matrix composites in the unique microgravity environment which will yield some vital information for terrestrial applications.

It is proposed to develop further the present model into a numerical model that takes into account convection effects. Anticipated results include development of an enhanced model for particle/interface interaction that will address convection in the liquid and interface morphology, as well as further contributions to the experimental data base for the understanding of solidification of metals/ceramic particles mixtures.

DESCRIPTION:
All these interactions are influenced by gravitational acceleration. The third one is the most complex one and has, therefore, been a subject of research over the past two decades. A uniform distribution of particles in the matrix cannot be achieved without the control of particle behavior at the liquid-solid interface during solidification. Understanding the particle/interface interaction is of great fundamental, as well as practical, importance.

To understand further the physics associated with the behavior of insoluble particles at the liquid-solid interface, directional solidification experiments will be conducted with metal matrix and transparent organic matrix doped with ceramic particles. Planar and cellular interfaces will be produced by appropriate selection of the solidification variables so that a clear pushing/engulfment of the particles could be achieved. Both ground and microgravity experiments will be conducted.

SIGNIFICANCE:
The project will enhance the fundamental understanding of the physics associated with the solidification of liquid metals/ceramic particles mixtures. One of the main issues in the fabrication of particulate metal matrix composites by melt processing techniques is that of uniform distribution of particles in the solid matrix. The distribution of reinforcing particles depends on the interaction between various phases in three different stages of processing: during transfer of particles from gas to liquid, during particle-particle interaction in the liquid, and during transfer of particles from liquid to solid.

PROGRESS DURING FY 1993:
Through combined thermal field and force field calculation, a model for prediction of particle behavior at the liquid-solid interface was developed. The model is based on the hypothesis that both thermal equilibrium and force equilibrium must be achieved at the interface during steady state pushing of the particle when the particle is pushed at a distance higher than the atomic distance. If thermal equilibrium cannot be achieved, the particle can still be pushed, but the separation distance between the particle and the interface is of the order of the atomic distance. Two
basic equations were derived. They were used for the calculation of the thermal and force equilibrium distance as a function of the solidification velocity. By coupling these equations, a unique value can be found for the critical velocity.

The thermal field equation derived predicts the formation of bumps and troughs on the melt interface behind the particles. However, it demonstrates that the condition $K_p/K_L < 1$ is not sufficient for bump formation. Limited effects of the thermal gradient on the critical velocity are also predicted. The conclusions of earlier researchers are thus confirmed. The occurrence of the bending of the interface was documented through experiments on transparent matrix materials (succinonitrile) containing SiC and on polystyrene particles.

Model validation through calculation with data from the present experiments as well as from experiments performed by other researchers on water—tungsten and aluminum—SiC systems showed reasonable agreement. The model allows an approximate evaluation of the exponent used in the definition of the repulsive force based on surface energy.

The present model is applicable to particulate metal matrix composites. It can be used to predict particle distribution in such composites solidifying with equiaxed structure and inclusion distribution in castings.

**STUDENTS FUNDED UNDER RESEARCH:**

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**TASK INITIATION:** 2/93  **EXPIRATION:** 2/96

**PROJECT IDENTIFICATION:** 694-25-08-11

**NASA CONTRACT NO.:** NAS8-387

**RESPONSIBLE CENTER:** MSFC

**BIBLIOGRAPHIC CITATIONS FOR FY 1993:**

**Journals:**


**Proceedings:**


**Presentations:**

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

PRINCIPAL INVESTIGATOR: Dr. Ching-Hua Su

Universities Space Research Association

CO-INVESTIGATORS:

R. Brebrick
M. Volz
Y. Sha
D. Noever
S. Sanghanitra
S. Johnson

Marquette University
NASA Marshall Space Flight Center (MSFC)
Universities Space Research Association (USRA)
NASA Marshall Space Flight Center (MSFC)
Santa Barbara Research Center
Santa Barbara Research Center

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

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.

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.

SIGNIFICANCE:
The materials to be investigated are ZnSe and related ternary semiconducting alloys, e.g., ZnS, Se, and ZnCdSe. 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 1993:

During the first six months of this study, the following tasks were accomplished. A zone refiner was set up and a zone refining process was performed in order to purify the starting Se materials. Twenty zone passes were made and then five slices of the ingot were chemically analyzed in order to study the effectiveness of the zone refining process. Heat treatment was performed on a ZnSe ampoule in which the zone-refined Se was utilized. All ZnSe starting material used in the first six-month period was heat treated by either baking out the material under dynamic vacuum.
condition or distilling it by subliming it from one end of the ampoule and depositing it on the other end under dynamic vacuum condition. A three-zone clam shell furnace was set up to conduct the transport rate measurements. Using an optical absorption technique for partial pressure measurements, a Se calibration run, a Zn calibration run, and a ZnSe calibration run were completed at Marquette University. Six heat-treated ZnSe ampoules were constructed and crystal growth by physical vapor transport was conducted on each of these ampoules. The characterization work on the grown crystals was initiated using x-ray (Laue) diffraction, atomic force microscopy (AFM), differential scanning calorimetry (DSC), and synchrotron radiation.

**STUDENTS FUNDED UNDER RESEARCH:**

PhD Students: 1

**BIBLIOGRAPHIC CITATIONS FOR FY 1993:**

**Journals:**


**Presentations:**


II. MSAD Program Tasks — Flight Research

Discipline: Materials Science


Measurement of Viscosity and Surface Tension of Undercooled Melts

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

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

**Task Objective, Description & Significance:**

**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 Arrhenius-type or a power-law relationship.

**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.

**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 1993:**
We have improved our mathematical model of the free surface shape of an electromagnetically-shaped molten metal droplet by including the effects of fluid flow; this had previously been neglected. We accomplished this using the electromagnetics code we developed with the FIDAP computational fluids package, which has the ability to include electromagnetic body forces and surface shapes and affords excellent graphics. Using our FIDAP-based model, we have also achieved preliminary success in what was thought to be one of the most difficult aspects of the modeling effort: simulation of the oscillations that the electromagnetically-positioned and perturbed samples undergo.

**Students Funded Under Research:**
PhD Students: 1

**Task Initiation:** 12/90  
**Expiration:** 06/95

**Project Identification:** 694-25-08-08

**NASA Contract No.:** GN8-970

**Responsible Center:** MSFC
BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Jourmals:


II. MSAD Program Tasks — Flight Research


Proceedings:


Presentations:


II. MSAD Program Tasks — Flight Research

Test of Magnetic Damping of Convective Flows in Microgravity

**PRINCIPAL INVESTIGATOR:** Dr. Frank R. Szofran

**NASA Marshall Space Flight Center (MSFC)**

**CO-INVESTIGATORS:**

S. D. Cobb  
M. B. Robinson  
M. P. Volz  
S. Motakef  

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

**TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:**

**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 transverse and axial magnetic fields 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 1g;
- To test the validity of magnetohydrodynamic modeling predictions in characterizing the effectiveness of reducing gravity for suppressing convective flows in a modest (about 0.1T) magnetic field; and
- To characterize as completely as possible any material that is grown in space to determine the effects of reduced gravity on the heat and mass transfer processes that occur during growth.

**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 four solid solutions—Ge-Si, InSb-GaSb, Cu-Ni, and Ag-Au—with liquid density ratios of 2.18, 1.07, 1.012, and 0.538, 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, Cu-Ni is even less stabilizing, and Ag-Au is unstable. The Ag-Au system will be solidified in both vertical orientations in the ground-based part of this study. All five systems will be processed by the Bridgman-Stockbarger 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.

**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
II. MSAD Program Tasks — Flight Research

Discipline: Materials Science

mixing is generally very intensive and interferes with segregation of melt constituents at the growth front. 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 1993:

Several Ga-doped Ge samples have been grown both in zero field and with a 5 T axial magnetic field. Preliminary Ga distribution analysis by electrical resistivity of two samples indicates complete mixing in both the sample grown with an applied field and in the zero-field sample. Nonetheless, the growth interfaces in both samples is virtually flat except near the ends or near bubbles. The informal part of the Science Concept Review was held on August 3, 1993.

TASK INITIATION: 10/92  EXPIRATION: 10/95
PROJECT IDENTIFICATION: 694-21-08-10
NASA CONTRACT NO.: in-house
RESPONSIBLE CENTER: MSFC
II. MSAD Program Tasks — Flight Research

Measurement of Liquid-Liquid Interfacial Tension

PRINCIPAL INVESTIGATOR: Dr. Michael C. Weinberg
University of Arizona

CO-INVESTIGATORS:
G.F. Nelson
E.H. Trinh
R.S. Subramanian

University of Arizona
Jet Propulsion Laboratory (JPL)
Clarkson University

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

OBJECTIVE:
The objective of this task is to perform measurements of small liquid-liquid interfacial tension in microgravity. This test will establish the feasibility of using the same technique at high temperatures to determine the interfacial tension between two phases of liquid glass and thereby assess the role of gravity in phase separation kinetics. Using two slightly miscible organic fluids, the Drop Physics Module creates a drop of one fluid inside a drop of the other. This compound drop is rotated and photographed from several angles. From the shapes of the two interfaces at a given rotation rate the interfacial tension will be determined.

DESCRIPTION:
To prepare for the flight, appropriate sample-fluid pairs were identified and their relevant physical properties measured; existing theory was used to select the optimal volumes and rotation rates to provide the most sensitive test matrix for determining the interfacial tension.

PROGRESS DURING FY 1993:

During the USML-l mission, the astronauts and DPM were unable to form the desired compound drops. Images of drops containing both fluids are being studied.

Discolored fluid returned from the mission is being analyzed. The tendency of the organic solutions which are saturated at one temperature to become separated as the temperature changes has been studied experimentally. The sensitivity of the fluids to absorbing small amounts of water was also examined. Analysis of the discolored droplets by IR spectroscopy has not provided conclusive answers.

STUDENTS FUNDED UNDER RESEARCH:

Total Degrees: 1

BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:
II. MSAD Program Tasks — Flight Research

Discipline: Materials Science

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, DESCRIPTION & SIGNIFICANCE:

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

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.

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.

PROGRESS DURING FY 1993:

Two HgCdTe samples were processed in the crystal growth furnace (CGF), which was flown in the pressurized Spacelab module in the habitable environment of the Space Shuttle Columbia during the first United States Microgravity Laboratory (USML-1) in June of 1992. After return to MSFC, the ampoule/cartridge assemblies were examined by x-ray radiation. In both cases, the thermocouples were in the proper locations along the ampoules. The mechanical integrity of the fused silica ampoules was confirmed after their removal from the cartridge. Based upon visual inspection of the flight ampoules, the surfaces of the source materials in both ampoules showed some recrystallization, which is typical for these procedures and conditions. The epitaxial layers grown on the substrates were nearly mirror smooth and showed a high degree of spectral reflection. This reveals the very high surface quality of the layer. Under comparable ground conditions, the surfaces of the epitaxial layers typically show a wavy morphology. Based on this qualitative comparison, the surface morphology of the epitaxial layers grown in a microgravity environment demonstrates a significant improvement relative to ground specimens. In addition, the epitaxial layers grown during the USML-1 mission have a very homogeneous appearance with respect to their overall surface morphology. Further characterization and further analysis of these flight experiments are proceeding.

STUDENTS FUNDED UNDER RESEARCH:

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BIBLIOGRAPHIC CITATIONS FOR FY 1993:
Journals:


Wiedemeier, H. and Ge, Y.R. Crystal defects and interdiffusion behavior of Hg$_{1-x}$Cd$_x$Te/(100)CdTe epitaxial layers grown by CVT. J. Electronic Materials, (in press) 1993.
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, Description & Significance:**

**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.

**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.

**Significance:**
We expect that our earth-bound measurements will yield information about possible advantages to be gained from micro-gravity 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 1993:**
The major issue to be addressed with support from this grant is whether a heat current renders the superfluid transition of $^4$He first-order. We built a special apparatus and search experimentally for hysteresis at the $\lambda$-transition in a heat current and in the gravitational field, and found none. With the temperature controlled by a thermometer with 3 nK (rms) resolution, the transition temperature was crossed from below and above at ramp rates from 1.14 to 10.42 nK/s in the presence of heat currents from 1.0 to 37.0 $\mu$W/cm$^2$. In all cases, the hysteresis was less than 0.1 $\mu$K. This is an order of magnitude less than the theoretical estimates of 1.6 or 3.1 $\mu$K at the larger currents of the experiment.

In these experiments we found that the temperature difference across the cell was considerably smaller than that calculated from the zero-current conductivity of the fluid. We are presently exploring whether this experimental observation is a manifestation of the heat current dependence of the conductivity predicted by Haussmann and Dohm. To this end, it is necessary to carry out a detailed numerical calculation of the two-dimensional temperature field in our cell. We have nearly completed the computer code for this project.

During the coming year, we expect to arrive at a definitive analysis of our data and a comparison with existing theoretical predictions and/or models for the heat current dependent conductivity. We also expect to obtain additional data for an experimental cell of a different thickness.
II. MSAD Program Tasks — Ground-based Research

Discipline: Benchmark Science

TASK INITIATION: 1/93  EXPIRATION: 12/95
PROJECT IDENTIFICATION: 674-24-07-17
RESPONSIBLE CENTER: JPL

BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:
Liu, F-C. Ahlers, G. The $\lambda$-transition of $^3$He in a heat current and under gravity. Physica B. (in press).
II. MSAD Program Tasks — Ground-based Research

Discipline: Benchmark Science

Microgravity Test of Universality and Scaling Predictions Near the $^3$He Critical Point

**Principal Investigator:** Dr. Martin B. Barmatz

**Jet Propulsion Laboratory (JPL)**

**Co-investigators:**

U.E. Israelsson

J. Rudnick

**Jet Propulsion Laboratory (JPL)**

**University of California, Los Angeles**

**Task Objective, Description & Significance:**

**Objectives:**
The objective of this task is to test the universality and scaling laws at the liquid-gas critical point of $^3$He in microgravity environment. The task objectives will include 1) precision measurements of the isothermal compressibility along the critical isochore to determine the critical exponent $\gamma$ and 2) precision measurements of the constant volume specific heat along the critical isochore to determine the critical exponent $\alpha$.

**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 ($10^{-3} g$) 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 ($\Delta p/p = 10^{-11}$), density ($\Delta p/p = 10^{-9}$), and temperature ($\Delta T/T = 10^{-9}$).

**Significance:**

The ability to perform these simultaneous measurements in microgravity should provide a very stringent test of the universality predictions.

**Progress During FY 1993:**

During this last year, we have fabricated a new cryostat based on the microgravity Lambda Point platform design capable of $10^3$ Kelvin temperature stability. This cryostat will be used to evaluate a new pressure transducer with a resolution of one part in $10^{11}$. A new low temperature pneumatic valve was also developed and tested for microgravity sample transfer.

**Task Initiation:** 12/92  **Expiration:** 11/95

**Project Identification:** 674-24-04-07

**Responsible Center:** JPL

II-144
II. MSAD Program Tasks — Ground-based Research

Measurement of the Heat Capacity of Superfluid Helium in a Persistent-Current State

Principal Investigator: Dr. Talso C. P. Chui
Jet Propulsion Laboratory (JPL)

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

Task Objective, Description & Significance:

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.

Description:
The flow will be created in a toroidal shaped calorimeter in the form of persistent current. 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.

Significance:
The results will be compared to the dynamic renormalization group theory, which have recently been applied to calculate the expected results. The theory, which involves three adjustable parameters, is remarkably successful in explaining the thermal conductivity and the second sound damping near the lambda transition. The proposed experiment will give a much more stringent test of the theory because this new experimental situation allows the theory to make precise predictions without any additional adjustable parameters.

Progress During FY 1993:
We have fabricated a low temperature probe and the internal platform to house the experiment. We tested it at helium temperature. We were able to get a Quantum Design RF SQUID to operate at its lowest noise level inside the vacuum can using an RF line and a SQUID housing designed by us for vacuum operation. The probe leaked initially. After some effort, we have repaired all the leaks. The probe is currently capable of maintaining temperatures below 2K. The computer interface to the Germanium resistance thermometer and the SQUID controller is completed and is currently under test. Parts for the high resolution thermometer have arrived. The growing of the salt pills for the high resolution thermometer has started. Complete assembly and test of the high resolution thermometer is scheduled at the first quarter of FY '94.

Students Funded Under Research:
BS Students: 1

Project Identification: 674-24-04-08

Bibliographic Citations for FY 1993:

Journals:
II. MSAD Program Tasks — Ground-based Research

Discipline: Benchmark Science

Determination of the Correlation Length in Helium II in a Microgravity Environment

PRINCIPAL INVESTIGATOR: Prof. Russell J. Donnelly
University of Oregon

CO-INVESTIGATORS:
No Co-I's Assigned to this Task

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

OBJECTIVE:
The objective of this research is to measure finite size effects in the isobaric expansion coefficient near the lambda transition in liquid helium. We will measure the thermal expansion coefficient for liquid helium confined between parallel plates for a range of temperatures very near the lambda transition temperature (both above and below), a range of pressures from SVP to about 25 bar, and a range of plate separation distances.

DESCRIPTION:
We will measure the dielectric constant of helium confined between parallel plates as a function of temperature at constant pressure. Using the Clausius-Mossotti relation, the density and thus the expansion coefficient of liquid helium will be calculated. The experimental method involves two measurements (at a given temperature) of the balancing ratio of an audio-frequency ratio-transformer capacitance bridge, one with the sample capacitor empty and then one with it filled with liquid helium. Appropriate division of these ratios then yields directly the dielectric constant at that temperature.

The capacitor used to measure the dielectric constant is a parallel-plate design operated as a three-terminal device in a 1-kHz ratio-transformer bridge. The spacing between the electrodes is determined by a precision shim which can be easily changed. An identical capacitor is also mounted on the experimental platform and is operated empty as a reference capacitor. We expect to vary the thickness of the shims between 5 microns and 50 microns.

Initially germanium thermometry will be used for temperature control and measurement. This will allow us to easily cover a wide range of temperature and to gain familiarity with the experiment. At this stage we will want to reconcile our results with older, published data. High-resolution measurements will be made after installing a paramagnetic salt thermometer identical to that used by John Lipa in his lambda-point heat capacity experiment (LPE), which successfully flew on STS-52 in October of 1992.

SIGNIFICANCE:
Finite size effects are manifested as a rounding of the divergence in thermodynamic functions near a critical point as the correlation length increases toward the system size. We can thus test renormalization group theory predictions, universality assumptions, and boundary conditions.

PROGRESS DURING FY 1993:
We have measured the dielectric constant contained within a 500 micron gap over a wide range of temperatures 1.1-4.9 Kelvin. To achieve temperatures near the density minimum we installed an external shutoff valve to the refrigerator input capillary so that it could be run in a "one-shot" mode. This reduction of heat load allowed us to operate the refrigerator near 1.0K for a period of two hours between fills. By comparing the measured dielectric constant obtained in the 50 and 500 micron samples we were able to deduce accurately the pressure coefficient of the capacitor cell at the temperature of the measurements. This is the first such determination of one of the most significant systematic errors in this type of measurement and a necessary step toward being able to accurately deduce finite size effects.
At the density minimum our measured dielectric constant values agree to within 2 ppm absolutely with those determined by Harris-Lowe and Smee. This is important since the known systematic errors are negligible at these temperatures, except for stray capacity. The temperature dependence of Kierstead's data is in excellent agreement with our measurements and indicates that Van Degrifts's use of the older, high temperature data of Onnes to complete tabulation of the temperature dependence of the density is most likely incorrect.

We have used Landau theory to determine the excess density below 1.1 K, using only calculated helium parameters obtained from the tabulated values of Brooks, so that we now have a complete tabulation of the dielectric constant, density, and expansion coefficient from 0K to near the liquid-gas critical point.

The capacitor plates have been re-machined and 5 micron spacers have been installed. We are currently readying the cryostat for a set of measurements at this plate spacing.

A manuscript detailing the bulk measurements is being submitted to JLTP.

**BIBLIOGRAPHIC CITATIONS FOR FY 1993:**

**Journals:**

II. MSAD Program Tasks — Ground-based Research

Discipline: Benchmark Science

Nucleation of Quantized Vortices from Rotating Superfluid Drops

PRINCIPAL INVESTIGATOR: Prof. Russell J. Donnelly
University of Oregon

CO-INVESTIGATORS:
J. Niemela
University of Oregon
W-K Rhim
Jet Propulsion Laboratory (JPL)

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

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.

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.

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 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 1993:

A cryostat designed to produce drops and levitate them electrostatically has been designed and built. It was first cooled down October, 1993. The device consists of two brass capacitor plates, separated by about 1 cm and having a potential difference of approximately 1kV. Helium II drops are formed at the bottom of a slender vessel situated directly above the top plate and which has been filled with helium II via a fountain pump. Film flow of helium II causes drops to form and detach at the bottom of the vessel, to which is attached a sharp tungsten tip. Approximately 2kV is applied to this tip to produce positive helium ions in the drops before they detach. The top plate has a 6mm diameter hole in its center to allow passage of the drop. The cryostat is housed in a glass helium dewar with transparent slits for viewing. The surrounding nitrogen dewar also has unsilvered slits running vertically. A CCD camera connected to a video recorder is used to observe the events. A stroboscope operated at 25,000 rpm is used for illumination. This allows multiple images of the drop on each frame recorded so that the
velocity and charge of the drop can be determined. To date we have successfully deployed drops and observed them, but have not yet produced charge. We are presently working on producing sharper tips so that we don't have to operate near the breakdown voltage of the helium vapor.

**TASK INITIATION: 1/93**  **EXPIRATION: 12/95**
**PROJECT IDENTIFICATION: 674-24-07-12**
**RESPONSIBLE CENTER: JPL**
Kinetic and Thermodynamic Studies of Melting-Freezing of Helium in Microgravity

**Principal Investigator:** Prof. Charles Elbaum

**Co-Investigators:**
J.M. Kosterlitz

**Task Objective, Description & Significance:**

**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.

**Description:**
Special lighting applied to a liquid helium dewar allows viewing of the liquid-solid interface. Rapid image capture equipment allows recording of the response of the interface to pressure or heat pulses.

**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.

**Progress During FY 1993:**
Our experimental activity is focussed on readying the nearly complete apparatus for data collecting activities. The third version of the sample chamber is in design stage and we expect to have it ready in about 10-12 days.

After a period of final "tuning" of the apparatus, we will conduct experiments, initially on growth of helium-four crystals and the evolution of crystal shapes toward equilibrium. Particular attention will be directed to the development of faceted interfaces from rough one (and vice-versa), as a function of temperature and pressure, i.e. growth-melting conditions.

Theoretical studies underway are two-fold: (1) A general theoretical investigation of equations of growth of crystal faces under both driven conditions and relaxation back to equilibrium. Recently, some interesting predictions have emerged, such as the time-dependence of the healing of a rough surface below the roughening temperature. We are attempting to estimate if such an effect is observable in our experimental setup. (2) A particular theoretical investigation of the growth of helium-four crystals, under the conditions of our experimental setup here. This involves a study of the time scales involved for growth by homogeneous nucleation and on rough surfaces depending on whether the temperature is above or below the roughening temperature of the particular facet.
II. MSAD Program Tasks — Ground-based Research

Discipline: Benchmark Science

STUDENTS FUNDED UNDER RESEARCH: PhD Students: 1

TASK INITIATION: 1/93 EXPIRATION: 12/95

PROJECT IDENTIFICATION: 674-24-07-13

RESPONSIBLE CENTER: JPL
Satellite Test of the Equivalence Principle (STEP)

**Principal Investigator:** Prof. C.W. Francis Everitt
**Co-Investigators:**
P. Worden

**Task Objective, Description & Significance:**

**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.

**Description:**
The experiment is one of four candidates for the next European Space Agency (ESA) medium-sized mission, to start around 1995. It is at the end a phase-A study being completed jointly by NASA and ESA. The mission, if selected, will be carried out jointly by the two agencies with an expected launch in the year 2000.

**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 1993:**
The Satellite Test of the Equivalence Principle (STEP) was studied as a joint ESA/NASA experiment during 1992-93. It was one of four medium sized (M2) missions in competition for an ESA new start in 1997. In April 1993, Integral was selected for the M2 mission. However, STEP received such good scientific support that NASA felt it should continue if the basic cost could be reduced to around $100 million.

Stanford University and the Jet Propulsion Laboratory redefined the STEP mission and, indeed, were able to reduce the costs sharply. The new mission is informally called Quick STEP. Significant costs savings were achieved by adopting the philosophy of using an existing, off-the-shelf "lightsat" spacecraft and a Taurus class launch vehicle. These two major components can be purchased for under $50 million. The payload would be sized to fit their capabilities. If the payload could not be made to fit, then it would be descoped until it did fit.

This philosophy resulted in no compromise in the basic $10^{17}$ Equivalence Principle experiment or the Geodesy experiment. The $1/R^2$, $G$ and axion experiments from the M2 mission were given up.

The Quick STEP mission is in phase-A in FY '94, has a planned project start in FY '96 and a Fall 1999 launch. A European partner is presently being sought for both science and hardware collaboration.

**Students Funded Under Research:**
BS Students: 1
PhD Students: 5
PhD Degrees: 1

**Task Initiation:** 10/84  **Expiration:** 9/94
**Project Identification:** 674-24-07-10
**Responsible Center:** JPL
BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:


Proceedings:

Presentations:
Condensate Fraction in Superfluid Helium Droplets

PRINCIPAL INVESTIGATOR: J. Woods Halley

University of Minnesota

CO-INVESTIGATORS:

C. Giese
C. Campbell
K. Goetz

University of Minnesota

Task Objective, Description & Significance:

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.

Description:
During the first year, we are performing theoretical calculations to test the feasibility of this idea. During the second and third years, an increasing portion of the effort will be devoted to experimental development. The main aspects of experimental development are 1) droplet levitation, 2) pulse creation and 3) pulse detection. With respect to aspects 2) and 3) a collaboration has been initiated with I. Silvera of Harvard University to perform a preliminary ground based experiment using detecting and pulsing bolometers developed by his group. With respect to terrestrial levitation, we are exploring the possibility of using facilities at the new Florida State national high magnetic field facility.

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 1993:
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 long range quantum coherence (condensate) in superfluid helium four is detected. Pulses of gaseous helium will be fired at a suspended droplet of superfluid helium four and the resulting emission of helium atoms will be detected.

Our original theoretical analysis of the proposed experiment, based on a simple tunneling Hamiltonian model was published in Physical Review Letters on October 15, 1993. A somewhat more sophisticated analysis, still based on perturbation theory and a Hartree approximation, will be published in the December, 1993 issue of Journal of Low Temperature Physics. In that same issue another paper (by C.E. Campbell) reports relevant new results on the nature of quantum coherence in finite systems. A preliminary quantitative calculation with T. Boinske of the
matrix element appearing in the tunneling Hamiltonian has been performed. The result is so large that it suggests that the low order perturbation theory used in the earlier work may be inadequate. This suggests a large effect but indicates that nonperturbative approaches may be required. In a collaboration with Siu Chin of Texas A & M University we are including the center of mass motion of the droplet in our analysis and are making a more complete many body analysis. An analysis of expected time delays in the emission process has been undertaken with T. Boinske. M. Williams made a calculation of the possibility of producing parallel helium interfaces in a terrestrial experiment by coating the sides of small holes in a container of liquid helium with cesium (which is not wet by superfluid helium). The result is that substantial hole sizes (of order 1mm) could be used, but a more detailed study of pressure fluctuations is required.

A first ground based experiment has been undertaken in collaboration with I. Silvera of Howard University. In this experiment, pulses of helium are fired at the horizontal surface of liquid helium at one end of a U-shaped copper tube about 3 inches in length and 0.25 inches in diameter. A detector at the horizontal liquid helium surface at the other end of the tube can detect re-emitted helium atoms. Mark Williams of this group spent a month in Cambridge in September working on this experiment and will return there for another month in December.

Invited talks on this subject have been given at LT20 and a satellite conference, various NASA meetings, a many-body meeting in Pakistan and at Kansas State University. Invitations for further invited talks have been received from the University of Minnesota, a many-body meeting at Texas A & M University, the Washington meeting of the American Physical Society in April, the AIAA meeting in Nevada in January, and a meeting on Thermophysics sponsored by NIST in June in Colorado.

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**BIBLIOGRAPHIC CITATIONS FOR FY 1993:**

**Journals:**


**Proceedings:**
Boinske, T. Theoretical calculations on the role of the condensate and vorticity in dense Bose systems. Conference on the Role of Condensate and Vorticity in Dense Bose Systems, (poster presentation at conference, to be published in J. Low Temp. Phys.).

II. MSAD Program Tasks — Ground-based Research

 Discipline: Benchmark Science

Presentations:

Giese, C. Experimental aspects of an experiment to measure the condensate fraction in superfluid helium four (4He), Conference: LT-20 (poster presentation at LT-20, to be published in Physica B).

Giese, C. Experimental aspects on the experiment to measure the condensate fraction in superfluid helium four (4He), Conference on the Role of the Condensate and Vorticity in Dense Bose Systems (poster presentation at the conference, to be published in J. Low Temp. Phys.).
II. MSAD Program Tasks — Ground-based Research

Dynamic Measurement Near the Lambda-Point in a Low-g Simulator on the Ground

PRINCIPAL INVESTIGATOR: Dr. Ulf E. Isröëlsson
Jet Propulsion Laboratory (JPL)

CO-INVESTIGATORS:
R.V. Duncan
Sandia National Laboratory

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

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.

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.

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 1993:
The vibration isolated magnetically shielded dewar has been assembled and verified functional at helium temperatures. The thermal platform, less the experimental cell, has been assembled, cooled down, and operated...
successfully at temperatures below 2 kelvin. A prototype melting curve thermometer has been constructed and tested for shorting capacitance at room temperature. Following the successful shorting pressure test which verifies the design, the remaining melting curve thermometers were submitted to the machine shop for fabrication. A magnet design was successfully developed in collaboration with a magnet winding company and an order for the magnet was placed. The design will reduce the effective gravity environment seen in a small size helium cell to below 0.01 g, thereby enabling a two orders of magnitude improvement in studies of gravity smeared properties in a ground based laboratory. The remainder of the experimental cell layout has been designed and drawings are being prepared for submission to machine shops for fabrication. Progress has also been made on modeling the experimental geometry of a cylindrical helium cell with a heat current down its axis and with side wall mounted thermometers in a reduced gravity environment.

**STUDENTS FUNDED UNDER RESEARCH:**

PhD Students: 1  

**TASK INITIATION:** 12/92  
**EXPIRATION:** 11/95  
**PROJECT IDENTIFICATION:** 674-24-04-09  
**RESponsible CENTER:** JPL

**BIBLIOGRAPHIC CITATIONS FOR FY 1993:**

**Journals:**


**Presentations:**

Theoretical Studies of the Lambda Transition of Liquid $^4$He

Principal Investigator: Efstratios Manousakis Florida State University

Co-Investigators:

No Co-I's Assigned to this Task

Task Objective, Description & Significance:

Objective:
We study the critical properties of liquid helium near the superfluid transition temperature $T_\lambda$ 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.

Description:
A recently developed updating technique called cluster Monte Carlo, which eliminates the long-standing problem of critical-slowing-down will allow 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 $T_\lambda(h)$ 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.

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 1993:
Since the beginning of our funding (January of 1993) from NASA’s microgravity program, we have made significant progress towards understanding the role of finite-size effects in two-dimensional superfluids.

Using the $x$ - $y$ model and a non-local updating scheme called cluster Monte Carlo, we calculated the superfluid density and specific heat of a superfluid on a finite lattice of size $L \times L \times H$ (where $L \gg H$). We are interested in the finite-size scaling with respect to $H$ of these two observables in the limit $L \rightarrow \infty$. However, there are very strong finite-size effects with respect to finite $L$ which need to be understood and to be taken into account when the critical temperature is approached. Thus, first, we have studied these finite-size effects in order to be able to extrapolate to the infinite $L$ limit before we consider the role of the finite $H$. 
II. MSAD Program Tasks — Ground-based Research

We have completed our studies of pure two dimensional superfluid on large-size square lattices $L \times L$ up to $400 \times 400$ using the above mentioned technique. This technique allows us to approach temperatures close to the critical point, and by studying a wide range of $L$ values and applying finite-size scaling theory we were able to extract the critical properties of the system. We calculated the superfluid density and from that we extracted the renormalization group beta function. We derived finite-size scaling expressions using the Kosterlitz-Thouless-Nelson Renormalization Group equations and showed that they are in very good agreement with our numerical results. This allowed us to extrapolate our results to the infinite $L$ limit. We also find that the universal discontinuity of the superfluid density at the critical temperature is in very good agreement with the Kosterlitz-Thouless-Nelson calculation and experiments.

These calculations 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. Some of the results of these calculations have been accepted for publication in the proceeding of the LT-20 conference\textsuperscript{1}. A complete paper containing all our results on the finite-size calcing of two-dimensional helium films has been submitted for publications\textsuperscript{2} in Physical Review B.

Now we are in the process of completing our calculation on helium films of finite-thickness $H$ and we study the finite-size scaling of our results with $H$. The results of these calculations will be relevant to the planned confined helium experiment (CHeX) as well as other earth-bound experiments.

\begin{tabular}{ll}
\textbf{Students Funded Under Research:} & \textbf{Task Initiation:} 01/93 \textbf{Expiration:} 12/95 \\
PhD Students: 1 & \textbf{Project Identification:} 674-24-07-15 \\
& \textbf{Responsible Center:} JPL
\end{tabular}

\textbf{Bibliographic Citations for FY 1993:}

\textbf{Journals:}


II-161
II. MSAD Program Tasks — Ground-based Research

Discipline: Benchmark Science

Dynamics and Morphology of Superfluid Helium Drops in a Microgravity Environment

PRINCIPAL INVESTIGATOR: Prof. Humphrey J. Maris

Brown University

CO-INVESTIGATORS:

G. Seidel

Brown University

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

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.

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.

SIGNIFICANCE:
The goal is to achieve data and experience critical for the design of experiments in space.

PROGRESS DURING FY 1993:

During this year we have worked on two projects:

1) Magnetic Levitation:
Helium is diamagnetic and is consequently repelled from a magnetic field. To magnetically levitate liquid helium in earth gravity it is necessary to use a magnetic field which satisfies the condition

$$B(dB/dz) > 22 \text{ Tesla}^2 \text{ cm}^{-1}$$

where $z$ indicates the vertical direction. In addition the lateral variation of the magnetic field has to be such that the value of $B^2$ increases in all horizontal directions away from the drop. The only magnet satisfying these conditions was a Bitter solenoid at the National Magnet Laboratory at MIT. We were aware when starting this project that this magnet had the disadvantage that the field fluctuated with time because of ripple on the power supply. During the past year incremental developments in the performance of superconducting magnet wire has made it possible to construct superconducting magnets that satisfy condition (1). These magnets have the great advantage that the field instability is eliminated. Two companies (Oxford and Cryomagnetics) have proposed specific designs. As a consequence of this development we have decided to purchase a magnet of this type and to perform all experiments in our laboratory at Brown. We were granted a supplement to our NASA funding to make this purchase possible, and Brown University also made a significant contribution to the cost. To work with this magnet we have had to substantially change the design of the optical cryostat and other low temperature components of the experiment. We hope to place the formal order with Oxford Instruments within the next few days.
2) Optical Levitation:
We have constructed an apparatus in which we will be able to optically levitate small (20 μm) drops of helium. The light source is a Neodymium YAG laser operating at 1.06 micron wavelength with a power of approximately 10 watts. The laser output is divided into two counterpropagating horizontal beams of equal power. Two lenses are used to bring each of these beams to a focus at the same point in space. With this apparatuses we have already been able to levitate 20 μm glass spheres and to observe these spheres with a high power microscope and video camera. We are currently trying to achieve helium levitation with this with this optical system. We have been able to produce helium droplets through the use of a hemispherical ultrasonic transducer immersed in the liquid at a depth such that the acoustic focus lies at the liquid surface.

<table>
<thead>
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<th>STUDENTS FUNDED UNDER RESEARCH:</th>
<th>TASK INITIATION: 1/93  EXPIRATION: 12/95</th>
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<tr>
<td>PhD Students: 1</td>
<td>PROJECT IDENTIFICATION: 674-24-07-16</td>
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<td>RESPONSIBLE CENTER: JPL</td>
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Critical Transport Phenomena in Fluid Helium Under Low Gravity

PRINCIPAL INVESTIGATOR: Prof. Horst Meyer  
Duke University

CO-INVESTIGATORS:

F. Zhong  
Duke University

**TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:**

**OBJECTIVE:**
Ground-based experiments will be carried out to study the temperature and density equilibration processes at constant average density \( \rho \) in a pure fluid \((^3\text{He})\) near its liquid-vapor critical point \((T_c, \rho_c)\). Measurements are to be carried out for both the region above \( T_c \) (one phase) and below \( T_c \) (coexisting phases).

**DESCRIPTION:**
Two types of cells are used. In the first one, the density stratification in the Earth's gravity field over a fluid layer height of 2 mm is measured via two thin horizontal superposed semitransparent capacitors that record the dielectric constant. The vertical density gradient is then derived via the Clausius-Mossotti relation. Stratification is recorded after a temperature change, and the resulting diffusion coefficient is determined as a function of temperature and average density. In a cell of the second type, the temperature equilibration in the middle of a fluid layer in the absence of convection will be measured after a rapid change in the cell temperature.

**SIGNIFICANCE:**
Such studies are very relevant to experiments on fluids under mg 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 come into thermodynamic equilibrium, and what are the basic mechanisms that control the equilibrium process. Later the measurements are to be extended to binary \((^3\text{He}-^4\text{He})\) mixtures.

**PROGRESS DURING FY 1993:**

During spring 1992 the existing cryostat for work over a temperature range 1K–5K (previously used in the NASA-sponsored viscosity measurements) was modified to incorporate a temperature-regulated platform supporting the density equilibration cell. Electronic circuitry was installed for high-resolution stable temperature and dielectric constant measurements. Computer programs for temperature step control and automatic data acquisition and reduction were developed. The apparatus was tested over a period of several months, and data-taking routines were developed and perfected. Initial density stratification data were obtained in the one-phase regime along the critical isochore and also in the two-phase regime.

Temperature steps of different sizes and sign were used and the stratification data were analyzed in a preliminary fashion. The stratification time was found to diverge as the critical point was approached from both the single-phase and the two-phase regimes. At a given temperature, it was found to be the same coming from either the colder or the warmer side. We expect to continue with data-taking along other isochores and along isotherms.

**STUDENTS FUNDED UNDER RESEARCH:**

PhD Students: 1

**TASK INITIATION:** 2/92  
**EXPIRATION:** 1/93

**PROJECT IDENTIFICATION:** 674-24-02-01

**RESPONSIBLE CENTER:** JPL

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BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:

**II. MSAD Program Tasks — Ground-based Research**

**Discipline: Benchmark Science**

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**Equilibration in Density and Temperature near the Liquid-Vapor Critical Point**

**PRINCIPAL INVESTIGATOR:** Prof. Horst Meyer  
Duke University

**CO-INVESTIGATORS:**  
F. Zhong  
Duke University

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**TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:**

**OBJECTIVE:**  
Ground-based experiments will be carried out to study the temperature and density equilibration processes at constant average density $\rho$ in a pure fluid ($^\text{3}$He) near its liquid-vapor critical point ($T_c, P_c$). Measurements are to be carried out for both the region above $T_c$ (one phase) and below $T_c$ (coexisting phases).

**DESCRIPTION:**  
Two types of cells are used. In the first one, the density stratification in the Earth's gravity field over a fluid layer height of 2 mm is measured via two thin horizontal superposed semitransparent capacitors that record the dielectric constant. The vertical density gradient is then derived via the Clausius-Mossotti relation. Stratification is recorded after a temperature change, and the resulting diffusion coefficient is determined as a function of temperature and average density. In a cell of the second type, the temperature equilibration in the middle of a fluid layer in the absence of convection will be measured after a rapid change in the cell temperature.

**SIGNIFICANCE:**  
Such studies are very relevant to experiments on fluids under microgravity 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 come into thermodynamic equilibrium, and what are the basic mechanisms that control the equilibrium process. Later the measurements are to be extended to binary ($^\text{3}$He-$^4$He) mixtures.

**PROGRESS DURING FY 1993:**

Systematic measurements of the density equilibration in $^3$He after a change in temperature have been carried out along the critical isochore and several near-critical isochores and isotherms above and below the critical temperature $T_c$. The equilibration process was followed as a function of time $t$ with sensors for the density $\rho$ near the top and the bottom of a flat horizontal cell. The relaxation time of one mode was found to diverge and then tend to a constant value as $T_c$ is approached from both sides. Below $T_c$, the equilibration in the liquid and the vapor phases was found to proceed with different profiles $\rho\text{_{Liq}}(t)$ and $\rho\text{_{Vap}}(t)$. While one mode slows down as $T_c$ is approached, the other one speeds up.

Starting from the differential entropy transport equation for a fluid at constant average density, numerical calculations were carried out of the density equilibration in a fluid layer of height $h$ after a change in temperature of the walls. The calculations were for the single phase above $T_c$. A one-dimensional geometry was used for simplicity, where the fluid layer is bounded by flat horizontal walls of high thermal conductivity, separated by a height $h$. It was found that the density equilibration is dependent on the vertical position. Only asymptotically is there a relaxation time $\tau$ of the exponential transient decay that is the same throughout the cell. This relaxation time is then the same as that for the temperature equilibration. This asymptotic regime occurs only at very long times, when the density transient has decayed to nearly zero, making a measurement of this time $\tau$ very difficult. Hence only "effective" relaxation times at shorter times $t$ will be recorded. Our measured density equilibration curves above $T_c$ have been compared with these numerical calculations without adjustable parameters, and fair
agreement was obtained. As $T_c$ is approached, and the gravity effects become important, the calculations are no longer sufficient.

The influence of gravity is very clearly seen in the equilibration curves as the critical point is approached from above. A hydrodynamic theory is being developed at present where the velocity for the mass transport due to the gravity field is being taken into account.

A large amount of data along the critical isochore and along isotherms, taken over the past 8 months, needs to be analyzed. At present, data along isotherms in the region below $T_c$ are being taken, hopefully to be completed by December '93.

Design studies for the cell of temperature equilibration measurements have been made. It is hoped that this cell will be constructed in late fall or early 1994.

**STUDENTS FUNDED UNDER RESEARCH:**

PhD Students: 1

**TASK INITIATION:** 1/93  **EXPIRATION:** 12/95

**PROJECT IDENTIFICATION:** 674-24-07-20

**RESPONSIBLE CENTER:** JPL

**BIBLIOGRAPHIC CITATIONS FOR FY 1993:**

**Journals:**

Evaluation of Ovarian Tumor Cell Growth and Gene Expression

PRINCIPAL INVESTIGATOR: Jeanne L. Becker, Ph.D.
University of South Florida

CO-INVESTIGATORS:
G.F. Spaulding
NASA Johnson Space Center (JSC)
R.H. Wideu
University of South Florida (Tampa Gen. Hospital)

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

OBJECTIVE:
Optimal growth conditions for ovarian tumor cells in rotating wall vessel.

DESCRIPTION:
Develop a 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 three dimensional aggregates.

SIGNIFICANCE:
Currently, there exists no reproducible model for long term culture of ovarian tumors which facilitates complex tissue-culture like arrangement in vitro, as occurs in vivo. We have accomplished this goal using the NASA-JSC Rotating-Wall Vessel.

PROGRESS DURING FY 1993:
Optimal conditions for growth of the LN1 ovarian tumor cell line in Rotating Wall Vessels have been accomplished. We have successfully evaluated cell cycle kinetics in tumor cells grown three dimensionally under these conditions, in which we observe increases in percentages of proliferating cells with time in three dimensional culture. We have also noted striking cellular morphological changes in LN1 cells during RWV culture.

The data obtained indicates that LN1 cells may differentiate into multiple cell populations exhibiting different phenotypes. This is interesting in view of the fact that the tumor from which LN1 was derived was comprised of multiple malignant cell populations, therefore suggesting that culture under three dimensional conditions in the RWV allows LN1 cells to regenerate some of the characteristics which were expressed in vivo. An abstract of the LN1 model was presented at the Annual Meeting of the American for Cell Biology, and a manuscript on phenotypic changes occurring during RWV culture was published this year in the Journal of Cellular Biochemistry.

For future studies, we plan to evaluate oncogene expression of several relevant oncogenes pertinent to the growth and metastasis of ovarian tumors in vivo. Preliminary studies indicate that during three dimensional growth in the RWV, LN1 exhibits changes in the expression patterns of several of these oncogenes, as a function of the frequency of cell feeding. This will be investigated in greater detail in upcoming experiments.

STUDENTS FUNDED UNDER RESEARCH:

<table>
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PROJECT IDENTIFICATION: 674-23-01-15
NASA CONTRACT NO.: NAG-648
RESPONSIBLE CENTER: JSC
BIBLIOGRAPHIC CITATIONS FOR FY 1993:

**Journals:**
Biosynthesis of Cellulose under Microgravity Conditions

PRINCIPAL INVESTIGATOR: Prof. R. Malcolm Brown
University of Texas, Austin

CO-INVESTIGATORS:
No Co-I's Assigned to this Task

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

OBJECTIVE:
Cellulose biosynthesis is an enzymatically controlled metabolic process involving two steps: (a) crystallization of the glucan chains to form a crystalline microfibril. Because plants contain cellulose walls and respond to gravity, and because cellulose biosynthesis involves a biologically-driven biocrystallization, we have been interested to learn about the effects of microgravity on this process.

DESCRIPTION:
Research Summary — In conjunction with NAG9-397, we have conducted experiments on six separate flight on board NASA's KC-135 airplane. Two platforms were constructed for these experiments. (a) shock mounted temperature controlled pallet on which were placed plastic Petri dishes containing colonies of bacterial for cellulose initiation and termination during flight, and (b) a compound microscope and video camera for observing the motion of cellulose-synthesizing bacteria during flight thereby allowing an assessment of possible altered cellulose assembly during microgravity phases.

SIGNIFICANCE:
An ideal system to study cellulose assembly is the gram-negative bacterium, Acetobacter xylinum. This system was particularly useful for short term microgravity experiments, in that the cellulose is rapidly synthesized and was amenable to microscopic analysis.

PROGRESS DURING FY 1993:
The major experimental data resulted in a publication. These studies have paved the way for planning of long-term microgravity experiments which hopefully will be conducted on board NASA's shuttle using a middeck locker and the BRIC.

STUDENTS FUNDED UNDER RESEARCH:
Total Students: 4

TASK INITIATION: 12/89  EXPIRATION: 10/92
PROJECT IDENTIFICATION: 674-23-01-04
NASA CONTRACT NO.: NAG-397
RESPONSIBLE CENTER: JSC
II. MSAD Program Tasks — Ground-based Research

Discipline: Biotechnology

Crystal Growth and Structure

**PRINCIPAL INVESTIGATOR:** Dr. Daniel C. Carter
NASA Marshall Space Flight Center (MSFC)

**CO-INVESTIGATORS:**
X. He
Universities Space Research Association (USRA)

**TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:**

**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.

**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.

Research Approach — 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 Rucker 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. Rucker 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.

**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 1993:**
The complete atomic structures of human serum albumin and HIV antibody 3D6 were refined to approximately 2.8-Å resolution and the results were published in *Nature* and *Proceedings of the National Academy of Science.*
II. MSAD Program Tasks — Ground-based Research

**Task Initiation:** 01/93  **Expiration:** 01/96

**Project Identification:** 674-23-08-17

**NASA Contract No.:** In-house

**Responsible Center:** MSFC
**Microgravity Studies of Cell-Polymer Cartilage Implants**

**Principal Investigator:** Lisa E. Freed, M.D., Ph.D.  
**Massachusetts Institute of Technology (MIT)**

**Co-Investigators:**
- R. Langer  
  **Massachusetts Institute of Technology (MIT)**
- G.F. Spaulding  
  **NASA Johnson Space Center (JSC)**
- T.J. Goodwin  
  **NASA Johnson Space Center (JSC)**
- D. Ingber  
  **Harvard University**

**Task Objective, Description & Significance:**

**Objective:** The goals of our work are: (1) to culture chondrocyte cells on biodegradable polymer scaffolds in order to create cartilage implants with clinically useful dimensions, and (2) to use this as a model system to study the effects of culture conditions, including microgravity, on tissue regeneration.

**Description:**
Cells are isolated from a small cartilage biopsy specimen, seeded throughout a polymer scaffold, and cultured in vitro. If the environment is favorable, as during normal chondrogenesis (cartilage formation), the cells proliferate and generate a dense cartilaginous matrix consisting of glycosaminoglycan (GAG) and collagen. The polymer (polyglycolic) serves as a temporary scaffold which provides a three dimensional structure for cell seeding and biodegrades in parallel to tissue regeneration. Once the cell-polymer construct has achieved sufficient mechanical integrity, it can be implanted where needed to repair a cartilage defect in vivo. The proposed therapy represents a relatively simple procedure to make cartilage implants with desired characteristics without any limitations in terms of availability of donor tissue or final dimensions.

Our tasks include: (1) fundamental studies to relate cartilage regeneration to the in vitro tissue culture environment, and (2) practical studies to determine optimal conditions to produce clinically sized, functional cartilage implants. Tissue culture bioreactors are being used because we have shown that although it is feasible to regenerate cartilage from isolated chondrocytes and polymer scaffolds, the size and quality of the final product are limited under conventional culture conditions (i.e., static petri dishes). Rotating bioreactors are being used in ground-based research because NASA studies have demonstrated enhanced growth and functional differentiation of cells cultured under "microgravity" conditions. Actual gravity reduction (i.e., space) would further extend the operating limits of the rotating bioreactors; pilot studies will thus be done to evaluate cartilage regeneration on board a shuttle flight. These same approaches and methods will also be extended to other cell-polymer model systems, and are expected to provide new information applicable to basic biological sciences and biomedical engineering.

**Significance:**
Each year, about one million Americans undergo attempted surgical repair of cartilage damaged by arthritis or trauma. Current therapy relies on the use of artificial joint prostheses, but these are limited by loosening at host/device interface, and by inflammation due to inorganic debris. Cartilage transplants are rarely done, due to the limited availability of donor tissue, and the inability to carve harvested tissue into the required shapes. Specifically designed, functional implants could form the basis of new cartilage repair procedures in reconstructive orthopedic or plastic surgery.
II. MSAD Program Tasks — Ground-based Research  
Discipline: Biotechnology

PROGRESS DURING FY 1993:

Specific Aim (1):
Establish methods to culture chondrocytes on biodegradable polymer scaffolds to regenerate cartilage tissue with clinically useful dimensions.

Chondrocytes were cultured on polymer scaffolds using static or mixed petri dishes, stirred-flasked bioreactors, and microgravity bioreactors. Under static conditions, gravity caused the cells to settle to the base of the polymer scaffolds, resulting in implants that were not uniformly cartilaginous. The effects of gravity were even more detrimental when implant thickness was increased. In contrast when cells were seeded and cultured on polymers in mixed petri dishes or stirred flasks, a spatially uniform distribution of well differentiated chondrocytes exhibiting perivascular staining and a hyaline, GAG-rich matrix were observed.

Implants grown in well-mixed cultures contained more collagen in the form of a fibrous capsule, as compared to those grown statically. This finding might be attributed either to a better supply of biochemical factors which stimulate cell proliferation and/or collagen gene expression (e.g., ascorbic acid), or to external shear forces which could stimulate the secretion of collagen which normally provides cartilage with shear stiffness. Most recently, we began to culture cells on polymer scaffolds under low-shear, microgravity conditions using two STLV's (110 m/s) modified to permit seeding and sampling of clinically-sized implants (9 per vessel). At the present time, these cultures are 2 weeks old, and cartilaginous tissue, 1 cm in diameter x 0.5 cm thick, can be seen.

Specific Aim (2):
Amplify chondrocytes isolated from the original cartilage biopsy specimen to obtain the cell mass required to seed clinically sized polymer scaffolds. Cell amplification was achieved by serially passaging cell monolayers in petri dishes, and by growing cells on cytodex beads and as cell-spheroids in stirred-flask bioreactors. We recently began to study chondrocyte amplification under microgravity conditions using a HARV (50 ml). Assessment of whether bioreactors can be effectively used for cell amplification while preventing the redifferentiation of chondrocytes seen with serial passage are currently in progress. High implant GAG contents were observed only when polymers were seeded at a high cell density at time-zero.

Specific Aim (3):
Optimize bioreactor systems to obtain clinically useful cartilage implants. Assessment of whether bioreactors can be used to create high quality cartilage with clinically useful dimensions are currently underway. Cell-polymer cartilage implants have been grown in microgravity and stirred flask bioreactors for 2 and 6 weeks, respectively. Culture medium composition and replacement rate and the effects of factors which stimulate cartilage growth are being evaluated. Mixing in the various bioreactor designs is also being studied with respect to the dependence of cartilage tissue regeneration on mass-transfer rates and shear stresses at the implant surface. The biochemical, biomechanical, and histological characteristics of cell-polymer samples will be compared to those of normal cartilage.

Collaborations:

1) In July and November 1993 bovine cartilage tissue and protocols to isolate and culture chondrocytes were sent from M.I.T. to NASA/JSC where the cells were seeded onto cytodex beads in a STLV. Histological assessment of cell-bead aggregates showed viable but undifferentiated cells and an extracellular matrix containing collagen after 6 weeks of growth.

2) At the NASA investigator's meeting at JSC in October 1993, Dr. Mary Pat Moyer and I made a plan to culture human chondrocytes on polymer scaffolds, and Dr. Charles Hartzell and I discussed a study to culture muscle cells on polymer scaffolds using microgravity bioreactors.
II. MSAD Program Tasks — Ground-based Research
Discipline: Biotechnology

STUDENTS FUNDED UNDER RESEARCH:
BS Students: 1

TASK INITIATION: 2/93 EXPIRATION: 12/93
PROJECT IDENTIFICATION: 674-23-01-11
NASA CONTRACT NO.: NAG-655
RESPONSIBLE CENTER: JSC

BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:


Presentations:


Excitable Cells and Growth Factors under Microgravity Conditions

**Principal Investigator:** Charles R. Hartzell, Ph.D.  
**Co-Investigators:**  
N. Schroedl  
S. Gonda

**Discipline:** Biotechnology

**Task Objective, Description & Significance:**

**Objective:**

Peptide growth factors are intimately involved in the regulation of normal muscle growth and differentiation. Invaluable groundwork 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.

**Description:**

Using the NASA bioreactor, we will examine the effects of a three-dimensional architecture on the growth and differentiation of neonatal cat heart cells and your adult rat 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 coculture of heart and 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-β will be evaluated.

**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 invitro 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 1993:**

1. Rat muscle Satellite Cells Cultured in the HARV Bioreactor

   a. Satellite Cell Enrichment:

   We have investigated the enrichment of the satellite cell preparations from the young adult rat. with Percoll fractionation, we have enriched the satellite cell population from about 10% to 50%. This effort has permitted us to reduce the inoculum of cells from 20 cells per bead to 4 cells per bead in the bioreactor. The real effect of this process is that we now have a more pure preparation of satellite cells that will undergo differentiation to myofibers. The effects of growth factors on the proliferative and differentiation processes can now be clearly distinguished in the satellite cell preparations. Desmin staining procedures have demonstrated that up to 70% of the cells are seen attached to the beads in the HARV are in fact myogenic in origin.

   b. Carrier Bead Comparisons:

   We have compared two bead types, Cytodex-3 and Nunclon. The Cytodex-3 bead is a cross-linked dextran bead coated with pig-skin collagen and has been the standard bead-type utilized in the bioreactor studies to date. However,
we have found that the Nunclon polystyrene bead when coated with 1 to 200 diluted matri-gel or normally coated pig skin collagen serves as a viable surface for cellular attachment and proliferation. The matri-gel surface is an identical matrix surface as the polystyrene petri dishes we normally use in our cell culture procedures. The overall characteristics of these beads in reference to surface area and density compare favorably with the Cytodex-3 beads.

c. Cellular Attachment, Proliferation, Myotube Formation and Bead Aggregation:
Plating efficiency is very high for either surface-coated bead. for the Matri-gel coated Nunclon bead we find greater than 100% efficiency. Since the proliferation process begins almost immediately in our satellite cell preparations, it is very difficult to separate the plating efficiency from the proliferative process. Less than 1% of the cells found free in the medium after 24 hours in the HARV bioreactors. Proliferation begins immediately and proceeds through 144 hours where about 400-300 cells are found per bead when the initial seeding density was 4 cells per bead. during the 5-6th day in culture the cells begin to fuse into myotubes and continues for about 3 more days where the vast majority of all cells on the bead surfaces are incorporated into myotubes. We have characterized these processes by scanning electron microscopy. We have found bead aggregation to be a minor problem where aggregates of 2-3 beads begin to form within 18 hours after inoculation of cells into the HARV. Aggregation of beads continues until we see bead clusters of about 8-10 beads formed after 8-10 days in culture with complete myotube formation.

2. Expression Patterns of Myogenic Genes from Satellite Cells

Four myogenic genes in skeletal muscle are MyoD, MRP4, Myf5, and myogenin. These genes are expressed during the various phases of myogenic cell proliferation and fusion into myotubes and encode nuclear binding proteins which function as transcription factors to control muscle specific genes such as MCK (muscle creatine kinase), MLC (myosin light chain), and desmin that are expressed during muscle differentiation. We have developed the technology to measure these genes during the entire cell culture process. By using RT-PCR of RNA samples collected from cells in the various phases of proliferation and fusion, we have measured the expression of each of these genes.

TASK INITIATION: 11/92  EXPIRATION: 10/96
PROJECT IDENTIFICATION: 674-23-01-07
NASA CONTRACT NO.: NAG-656
RESPONSIBLE CENTER: JSC
II. MSAD Program Tasks — Ground-based Research

Sensitized Lymphocytes for Tumor Therapy Grown in Microgravity

PRINCIPAL INVESTIGATOR: Marylou Ingram, M.D. Huntington Medical Research Institutes

CO-INVESTIGATORS:
No Co-I's Assigned to this Task

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

OBJECTIVE:
The unique capabilities of NASA's High Aspect Ratio Vessel (HARV) are utilized to prepare stimulated immune cells that are specifically sensitized to a patient's own tumor. These are tumor-sensitized lymphocytes (TSL). TSL will be used in a small-scale, in-depth clinical trial of local immunotherapy for malignant tumors of the urinary bladder.

DESCRIPTION:
This is a small scale, in-depth pilot study in which non-specifically stimulated autologous lymphocytes (ASL) and tumor sensitized (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. For bladder concern the urological surgeon will inject cell 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 individual sites at various stages of clinical response can be expected to improve our insight into the basic biology of these tumors. Analysis of cells 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. 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.

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 use 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. TSL can be expected to show increased tumor cell killing, as do tumor-infiltrating lymphocytes (TIL) and they also hold forth the promised 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.

We initially proposed developing the TSL immunotherapy protocol to treat recurrent malignant glioma and obtained as IND from the U.S. Food and Drug Administration for such a trial. Because of funding problems and other operational considerations that trial has been deferred. We were especially pleased when the opportunity arose to develop a TSL-immunotherapy protocol for bladder cancer because that clinical problem 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 1993:

A. Interactions with FDA
It was necessary to apply for a new IND for the bladder cancer immunotherapy phase I trial to demonstrated that the proposed clinical trial is justified, that we are qualified to conduct it and that all aspects of the proposed laboratory and clinical methods and procedures are in compliance with the FDA Investigational New Drug Antibiotic and Biologic Drug product Regulations, CFR parts 312 and 314. Since preparation of the stimulated lymphocytes requires Interleukin-2(IL-2) and we plan to use AMGEN recombinant human IL-2, it was also necessary to have the proposed protocol reviewed and approved AMGEN's internal review board and to persuade AMGEN to provide the IL-2 for the study. Since AMGEN has discontinued almost all clinical trials of IL-2 for the time being, obtaining their approval was by no means a trivial undertaking. It was also necessary to have the clinical protocol and the informed consent form approved by the Institutional Review Board of the Huntington Memorial Hospital. The NASA bioreactor was described in the application to the FDA. They had a number of questions about it during various telephone conversations but so far seen to accept it. (They always have the option of raising additional questions and requiring additional information.) We now have permission to conduct the trial under BB IND 5163 and hope to enter the first patient in January 1994.

B. Laboratory Studies Using the HARV Bioreactor
During FY93 we have conducted extensive cell culture studies using the HARV bioreactor. Our collaborating scientific colleagues at JSC, Dr. Glenn Spaulding, and Thomas Goodwin, have provided invaluable guidance expert advice and support in this research. These exploratory studies have been invaluable in that they have allowed us to evaluate some of the major biological and methodological variables that must be considered as we learn how best to exploit the new bioreactor technology to achieve improved immunotherapy for malignant disease.

Cell Cultured — A number of human malignant glioma cell lines established at HMRI were studied in multiple cultures. As a rule parallel conventional monolayer cultures were established for comparison. Many of these were also cultured encapsulated in alginate. Other HMRI cell lines cultured in parallel bioreactor and monolayer cultures; human tumor metastatic to brain, the widely studied prostate cancer cell lines, PC3, LNCap, and DU. Several surgical biopsy specimens of known bladder cancer have been obtained so that we might evaluate the special problems to be anticipated when establishing primary cultures in the bioreactor. Establishing primary cultures is,
II. MSAD Program Tasks — Ground-based Research  Discipline: Biotechnology

of course, a particularly demanding tissue culture task and it is unrealistic to expect that cell lines can be established from all tumor biopsy specimens. Whenever possible this will be done but in some cases it will be necessary to use tumor mince specimens to sensitize lymphocytes. Since serial biopsies will be obtained from each bladder cancer, the chances of establishing a cell line from at least one if substantially greater.

Other aspects of bioreactor culture methodology that have been evaluated include 1) number of cells required to initiate culture, 2) requirement for fetal bovine serum, 3) time to first appearance of "spheroids," frequency of media change, size of spheroids formed under various conditions, effect of epidermal growth factor and of I fn- on cell proliferation and spheroid production. In general, the major variable has been the cell type per se. Cells that grow will in monolayer culture test to do well in the bioreactor and vice versa. We have not use carrier beads because we hope to avoid the effects of anchorage. Human glioma cell lines grow well in the bioreactor and produce spheroids within hours in cultures seeded with a minimum of 2x10^5 cell/ml. Spheroid size tends to vary from one cell line to another and to be reasonably reproducible for a given cell line. Culture have been maintained in bioreactor for periods of a few days to more than a month. When large spheroids are formed in older cultures, they are usually found to be made up of smaller, merged spheroids. The individual spheroid size still corresponds to the size that is characteristic of the cell line. Requirement for fetal bovine serum is usually approximately 10% although some lines that have previously been "weaned" do well with only 3% FBS. So far all bladder cancer cultures require epidermal growth factor.

Initially, we attempted to quantitate the yield of cells from bioreactor cultures but this proved to be unrewarding. Varying proportions of the cell populations attach to the bioreactor walls and some cells growing in spheroids are damaged or destroyed when more aggressive dispersal technique are required. Since our main interest is comparing "markers" in spheroid vs. monolayers cells we elected to concentrate on fixed, stained sections of spheroids. a number of fixing agents and protocols have been evaluated. The major "markers evaluated in these exploratory studies are 1) proliferating cell nuclear antigen, 2) EGFR, 3) P53 tumor suppressor gene and 4) c-erB-2. WE are currently preparing spheroid and monolayer cultures that will be evaluated in terms of expression of cell adhesion molecules. So far, whatever the "marker of interest, the spheroids show a characteristic gradation of expression that is greatest at the surface and least in the center. For this reason, when preparing spheroids as "targets" for sensitizing lymphocytes we will attempt to work with relatively small spheroids that have a larger percentage of their cells in the high-expression, more superficial area. For bladder cancer immunotherapy, lymphocytes to be sensitized will be exposed to tumor mince, monolayer tumor cell cultures, and to tumor spheroids in parallel whenever possible so that we may acquire definitive comparative data about the relative "antigenicity" of the three tumor presentation methods. Ultimately we hope to use the lymphocytes as probes to identify the important tumor antigens and refine the immunotherapy through isolation and cloning of the relevant antigens using the technology of modern molecular biology.

TASK INITIATION: 11/92  EXPIRATION: 11/95
PROJECT IDENTIFICATION: 674-23-01-14
NASA CONTRACT NO.: NAG-649
RESPONSIBLE CENTER: JSC
Three-Dimensional Modeling of Human Colon Tissues

PRINCIPAL INVESTIGATOR: J. Milburn Jessup, M.D. New England Deaconess Hospital

CO-INVESTIGATORS:
P. Thomas Harvard Medical School, Deaconess Hospital

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

OBJECTIVE:
The objectives of this project are to determine whether 1) microgravity permits unique, three-dimensional cultures of neoplastic human colon tissue and 2) this culture interaction produces novel intestinal growth and differentiation factors.

DESCRIPTION:
The initial phase of this project will test the feasibility of microgravity for the cultivation and differentiation of human colon carcinoma. We propose to do this in rotating wall vessels (RWVs) which provide a low shear stress environment in unit gravity. In this environment, early experiments have demonstrated normal human colon fibroblasts stimulate the differentiation of certain human colon carcinoma cell lines so that they produce three-dimensional tissue masses that are similar to neoplasms in patients or in xenografts in athymic nude mice.

SIGNIFICANCE:
The important question is whether this differentiation induced by fibroblasts is due to the low shear stress environment of the RWV or whether suspension cultures in the RWVs to similar cultures in standard culture systems in unit gravity. Should the low shear stress environment of the RWV be superior to that of conventional culture systems, then the co-culture experiments should be attempted in an actual microgravity environment.

PROGRESS DURING FY 1993:
The intent of this grant was to determine whether normal stromal cells cultivated with epithelial cells would form three-dimensional structure that were similar to tissues that developed in mice or humans. We started with adenocarcinomas of the colon because the laboratory had expertise with that and because adenocarcinomas were relatively easy to cultivate and could be examined under different culture conditions. In addition, we wanted to study normal colonic epithelium because the rotating wall vessel (RWV) may provide a unique opportunity for cultivating normal colonic epithelium.

To date, we have developed a procedure for isolating colonic epithelium that does not have associated stromal cells. As others have found, incubating intact crypts on various gel substrates, such as type IV collagen, laminin and Matrigel did not improve the survival of colonic crypt cells. The coloncytes were essentially all dead within 24 hours with dissolution of the crypt architecture. This occurred even when crypts were embedded in a semisolid matrix that supports the weight of the crypts. We then proceeded to dissociate the crypts into single cells to study their adhesion.

We discovered that isolate colonocytes bind best to carcinoembryonic antigen (CEA)-coated surfaces. The second most common substrate was type IV collagen which is a constituent of normal basement membranes. Normal colonocytes did not bind to laminin, a constituent of basement membranes to which essentially all human colorectal
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Discipline: Biotechnology

carcinomas bind. This is an important observation because it indicates that the development of a colorectal carcinoma is associated with the development of the capacity to bind to laminin. Many molecules are involved in this and our laboratory has recently shown that CD44 will bind to laminin and that CD44 expression is up-regulated in carcinoma cells compared to normal coloncytes.

As a precursor to RWV cultures, we have attempted to cultivate normal coloncytes on CEA attached to a solid phase, as well as type IV collagen. Although normal coloncytes adhere to CEA, they remain rounded and these cells in suspension die within 48 hours. In contrast, normal coloncytes cultivated on type IV collagen spread and assume a more epithelioid shape. However, these cells also die within 48 hours. We are preparing to do cultures in the RWV, but have found that the culture volumes are too large to incubate either isolated crypts or crypt cell suspension that we prepare. We anticipate that we will have access to smaller volume RWVs and will be able to examine normal coloncyte growth in the near future. Thus our progress in this application has been to establish the conditions for isolating normal coloncytes and to evaluate the adhesion requirements to optimize coloncyte attachment.

An interesting recent finding by Kerbel and his colleagues in Toronto is that a mammary adenocarcinoma of BALB/c mice that is resistant to chemotherapy when passages in vivo is also resistant when grown as three-dimensional spheroids but not as a monolayer. We attempted to confirm this, but were not able to do so. An investigator at the Dana-Farber Cancer Institute established the resistant cell lines by initiating tumors in female adult mice and then treating when with selected chemotherapeutic agents. Then, several days after the treatment but before the tumors could regress, the tumors were transferred to a naive adult female mouse and allowed to grow and then retreated with the same agent. Ultimately sublines of the tumor were developed that were resistant to the chemotherapeutic agent. Interestingly, these sublines of EMT-6 mouse mammary carcinoma cells were sensitive to the agent when grown in in vitro monolayer cultures.

Kerbel and colleagues found that the cells that were resistant in vivo were resistant to the appropriate chemotherapeutic agent when grown in three-dimensional tumor spheroids. We tried to duplicate this by growing EMT-6 cells resistant to cisplatin on microcarrier beads in the RWV and found that these cells were just as sensitive to the chemotherapeutic agent in the three-dimensional culture system as in monolayer culture. The difference between the Kerbel data and ours was that we could not develop culture masses that were thicker than the spheroids that Carryable created. This is important because part of the resistance could be due to simple diffusion barriers. Another difference between the RWV and Kerbel experiment was that because of the structure of the RWV, the drug was left in the system continuously, whereas the Kerbel experiment relied on a short incubation before plating. Nonetheless, the studies suggest that continuous exposure may overcome the differences that Kerbel had seen with his spheroids.

Finally, as we prepare for a potential flight experiment, we have investigated the possibility of culturing cells other than HT-29KM in actual microgravity. We have cultured MIP-101, KM-12c, CCL-188, as well as HT-29KM. We have examined cultures for growth as well as CEA production. All three cell lines grow progressively are quite similar. We have not identified spontaneous morphologic differentiation. We are examining the possible co-culture of these cell lines with murine embryonic fibroblasts. These fibroblasts would be similar to the fibroblasts that line the mouse peritoneal cavity that may be important for the MIP-101 cultures. The difficulty with the 3T3 cells is that MIP-101 cells do not grow well with 3T3 fibroblasts. In fact, the cells go into suspension in the presence of 3T3 cells even faster than they did when cultured by themselves. Cultures of KM-12 and CCL-188 with 3T3 fibroblasts are planned in the near future.
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BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:


Presentations:


Three-Dimensional Tissue Interactions in Colorectal Cancer Metastasis

**Principal Investigator:** J. Milburn Jessup, M.D. New England Deaconess Hospital

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

**Task Objective, Description & Significance:**

**Objective:**
The objective of this project is to test the fidelity with which microgravity models three-dimensional tissues by assessing how well microgravity affects the molecular and biological function of MIP-101, a human colorectal carcinoma.

**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.

**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 in 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. Further, 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 micro-environment (the three-dimensional environment) of the abdominal cavity induces MIP-101 cells first to produce CEA and then to develop blood-born 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.
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Discipline: Biotechnology

PROGRESS DURING FY 1993:

After our initial findings, our progress has been hampered during this first year of the grant by an inability to perform long term cultures with MIP-101 cells. We have found that MIP-101 cells cultured on microcarrier beads cover the beads and form aggregates. MIP-101 cells then will raft the beads together to produce masses that are 3-4 mm in diameter after 4-5 days. However, the MIP-101 cells remain at that size for the remainder of the culture, and, after about 2 weeks, many of the culture runs have had cells come off the beads and die. The MIP-101 cells also increase in size and CEA production increases as the culture terminally differentiates. Thus, our data suggest that the MIP-101 cell line may be terminally differentiating in the RWV and express CEA because of that terminal differentiation. As these cells undergo terminal differentiation, they lose their ability to adhere to the collagen-coated microcarrier beads and go into suspension. The cell nuclei increase in size, and fragment as their viability in the culture decreases. Since CEA is produced by cells that are terminally differentiating in normal colonic epithelium, the RWV may be inducing the gene expression that is appropriate for the culture environment. Our immediate goal is to determine why the cells are terminally differentiating or undergoing apoptosis in the RWV.

To understand the loss in cell viability, we have first examined the use of glucose. We have found that the cells are extremely metabolically active in the first two weeks and as they begin to differentiate after 2 weeks in culture, the cell cycle elongates and glucose utilization decreases when calculated on a per cell basis. CEA production appears to increase when the number of cells plateaus at 4 weeks of culture. We are currently investigating whether glucose depletion occurs causing irreversible cell damage. We have performed several experiments in which the number of cells is decreased so as to prevent total glucose depletion. When we do this, we still find that the MIP-101 cells terminally differentiate and become less viable. An alternative hypothesis is that some other substance is rate limiting in the RWV and ultimately inhibits cell growth. We suspect that there may be a low oxygen tension in the medium. As a result, we are initiating experiments to measure the rate of consumption of oxygen in our system. Oxygen tension measured in millimeters of mercury of dissolved oxygen may be low in actively metabolizing cultures. No oxygen carrying protein is present in the medium that would permit the easy diffusion of oxygen from the silicone covered spindle. If we find that the oxygen capacity of the culture medium is too low for the MIP-101 cells, then we will increase the oxygen tension in the incubator by making the incubator environment hyperoxic and consider adding stroma free hemoglobin or some other oxygen carrier.

The role of oxygen may be extremely important because the amount of oxygen present may control glucose consumption and oxidative phosphorylation. Essentially all RWV cultures of eukaryotic cells decrease glucose utilization in the terminal phases of the cultures. It has been thought that this was due to decreased diffusion of glucose across and through masses of tissue as the bead aggregates increased in size. However, in the current cultures, it is unlikely that diffusion inhibits glucose uptake, because the MIP-101 cultures are sufficiently loosely aggregated that physical diffusion should not be a barrier. Diffusion from the semi-permeable membrane in the center of the STLV chamber may be a mass transfer problem that will need reconfiguration in the future. The high aspect ratio vessel (HARV) may have better oxygen diffusion characteristics. Some cell lines appear to grow better in the HARV than they do in the RWV. These are important concerns and should be addressed in a simple system such as the MIP-101 cells because the optimization of oxygen and glucose delivery is important for successful cultivation of all eukaryotic and prokaryotic cells.

The evaluation of CEA production in the MIP-101 cultures has been extremely interesting. In the RWV, the percentage of cells expressing CEA determined by immunoperoxidase of fixed histologic sections of MIP-101 bead aggregates demonstrate that as many as 10-15% of the MIP-101 cells may produce CEA. In contrast, only 1% of MIP-101 cells grown in the peritoneal cavity of nude mice produced CEA. Similarly, when sodium butyrate was used to stimulate CEA production, the MIP-101 cells did not release CEA into the medium, but did differentiate and produce CEA in nearly 30% of cultured and stimulated cells. This suggests that not only do cells have to be

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Discipline: Biotechnology

stimulated to produce CEA, but also to induce a second enzyme that releases the CEA from the cell. These observations are important because they may help us to understand the process of metastasis better. It may be that the CEA-producing MIP-101 cells are actually the metastatic progenitor cells that normally are present in only 0.2% of cells in conventional monolayer culture and in only 1 in 10,000 cells grown in subcutaneous tissue of nude mice. If this is the case, then attempts to selectively grow MIP-101 cells may enrich the population for metastatic precursor cells. Thus the RWV may stimulate selective outgrowth of metastatic progenitor cells even at the same time as it is inducing another population of MIP-101 cells to terminally differentiate. We are now isolating cells by surface expression of CEA to assess their biological characteristics and determine whether their growth in the RWV is different from that of the unselected MIP-101 cell population.

We have shown that cells grown for 5-7 days in the RWV have normal adhesion characteristics. In other work, we have demonstrated that CEA is necessary for binding to CEA, i.e. that CEA is a homophilic binding protein that mediates homotypic cell adhesion. We have also recently determined that another CEA family member, non-specific cross-reacting antigen (NCA), also participates in adhesion to CEA. We have further identified another cell adhesion molecule, CD44. CD44 was originally defined as a lymphocyte homing receptor that may be involved in adhesion to, not only CEA, but also laminin and type IV collagen and has alternately spliced variants that are expressed.

Using image analysis that was made possible through the NASA research, we not only have performed the immunoperoxidase analysis of the RWV cultures, but have also examined the distribution of antigen on the cell membrane in MIP-101 cells as well as in other colorectal carcinoma cells that express both CD44 and CEA. We are currently using this technology to map the distribution of antigen to identify what antigens are co-localized and whether growth in the RWV alters co-localization. Cultures of MIP-101 cells that are longer than 7 days in the RWV have lost their adhesion to the collagen coated microcarrier beads. We will analyze the distribution of cell surface CD44 and CEA as well as the gene expression of other CEA family members. Finally, in other work, we have shown that CEA mediated adhesion is an active process requiring ATP production and an intact microtubule system. We are currently evaluating whether the cell surface distribution is altered in the RWV. CEA is a glycosylphosphatidylinositol(GPI)-linked molecule that may be carried by microtubules since inhibitors of microtubule function reversibly block CEA-mediated adhesion. This is important because CEA should be clustered in recently identified membrane receptacles called caveolae that concentrate GPI-linked molecules in membrane pouches.

NASA support for the RWV cultures has been important to this effort because the identification of the CEA production by MIP-101 has allowed us insights into both imaging and analyzing the adhesion of cells to CEA. It also has lead to hypotheses that the loss of adhesion and terminal differentiation seen in the majority of MIP-101 cells is due to defects in signal transduction. Work is in progress to determine whether this is merely a mass transfer problem that prevents the distribution of nutrients and the elimination of waste or whether this a fundamental problem for this particular cell line in a suspension culture system. However, these experiments in CEA production, apoptosis, and metastasis progress, the information should be essential to the design of more efficient space-based bioreactors in the future.
BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:

Applications of Atomic Force Microscopy to Investigate Mechanisms of Protein Crystal Growth

**PRINCIPAL INVESTIGATOR:** Dr. John H. Konnert  
**Naval Research Laboratory**

**CO-INVESTIGATORS:**  
K. Ward  
P. D'Antonio  
**Naval Research Laboratory (NRL)**

**TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:**

**OBJECTIVE:**  
The research objective is to use Atomic Force Microscopy 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.

**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).

**Research Approach** — 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.

**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 1993:**
The major practical problems of protein crystal growth which all studies ultimately wish to address are:

- What crystallization parameters determine the diffraction quality (i.e., the limit of resolution of diffraction data) of protein crystals? Is the rate of crystal growth important?

- What parameters affect the ultimate size of crystals of a given protein? What is responsible for the frequent observation that, for certain proteins, crystal growth ceases after a certain size?

It is generally believed that both of these questions are related to the relative number of growth defects introduced into the crystalline lattice during the growth process. However, there is scant data to support this idea because, until recently, there was no convenient way to study these defects.

**BIBLIOGRAPHIC CITATIONS FOR FY 1993:**

**Journals:**

**Proceedings:**

Neuro-endocrine Organoid Assembly in Vitro

principal investigator: Peter I. Lelkes, Ph.D. University of Wisconsin

Co-investigators:
B.R. Unsworth Marquette University

Task Objective, Description & Significance:
Objective:
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 (immunocytochemistry), c) functional maturation and its hormonal control by corticosteroids, and 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).

Description:
Our long-term research goal is to understand fundamental mechanisms of neuro-endocrine gland assembly and differentiation. In our particular model system, the adrenal medulla, neural crest-derived cells of the sympathetic-adrenal lineage differentiate into neuro-endocrine chromaffin cells. We are particularly interested in the role of the microenvironment in this process. We have previously presented evidence that vascular endothelial cells might provide certain organ-specific, differentiative, cues. We hypothesize that during assembly of neuro-endocrine organs, in general 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". In this proposal, we will extend our ongoing studies on the mechanisms of organ-specific differentiation, using alternate methods of coculture. We will exploit 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 controlled microgravity environment of the NASA vessels will exclude detrimental shear forces during organ-assembly in vitro, and possibly, accelerate differentiative, heterotypic cell-cell contacts.

Significance:
By using the NASA vessel as a novel, alternate approach to cell culture, we will participate in NASA's assessment of low-gravity conditions for organ-specific culture and cellular differentiation. The major advantage of using the NASA devices in enhanced cell viability, and tissue differentiation under conditions of microgravity and minimized shear stress. The unique conditions in the NASA vessels might differentially accelerate and/or facilitate these processes. A critical question to be answered by our studies, is whether the spatial arrangement of a functional organ is impaired by gravitational forces/vectors. In other words, if we eliminate gravitational vectors, such as those present in static two-dimensional monolayers, or in suspension cultures, will a more realistic representation of tissue assembly, as seen in vivo, be obtained? The enhanced cellular viability and differentiation, reportedly produced by microgravity cell culture environments, may prove to be of general advantage for tissue culture, particularly when culturing and/or co-culturing fragile cell types, isolated at early stages of embryonic development. The long-term goal of our studies, beyond the scope of the present proposal, is to offer NASA an appropriate cellular model system for organogenesis and differentiation, suitable for in-flight (middeck) testing.
II. MSAD Program Tasks — Ground-based Research

Discipline: Biotechnology

PROGRESS DURING FY1993:

1. As outlined in the proposal we have used the first year to assemble the necessary knowledge to carry out the proposed experiments. We hired a molecular biologist (Dr. J. Liu), and have synthesized all the necessary molecular biological probes to test for differential gene activation during neuroendocrine development of adrenal medullary organoids. We have also begun the control experiments co-culturing PC12 cells and endothelial cells in conventional spinner cultures, and observed the in vitro formation of organoids (Lelkes et al., Molec. Biol. Cell, Vol. 3, Supp:290a).

2. All the experimental techniques to be used in our studies, including the above mentioned molecular probes were utilized during our investigation of the effects of microgravity on adrenal medullar in rats that had been flown in space during Space Shuttle Mission STS-51. The results of this investigation be presented during then upcoming ASCB meeting (Lelkes et al. Molec. Biol. Cell, vol 4 Suppl:109a). A manuscript of a paper per titled "Space flight decreases rat adrenal medullary tyrosine hydroxylase expression" is currently under review in the journal Science.

The main experimental results and conclusions from this study are as follows:

In summary, tyrosine hydroxylase (TH), the rate limiting step in catecholamine synthesis was specifically affected by microgravity. By contrast, phenylethanolamine-N-methyl-transferase (PNMT), the adrenal medullary chromaffin cell-specific enzyme with converts norepinephrine to epinephrine was not affected. The specific activity, immunoreactivity and gene expression (mRNA level) were all depresses about 30% in the space flight adrenals, compared with land-based controls. There was also a decrease in total catecholamine contents in the space flight adrenal medullae concomitant with a specific decrease in the ratio of tissue epinephrine to norepinephrine in the flight samples. We hypothesized that the these observed effects of space flight were not due to stress response during reentry, because of the relatively long time course (about 12 hours) of TH depression and lowered catecholamine contents. Furthermore, based on evidence in the literature we speculated that microgravity may be exerting its effect on catecholamine synthesis by acting on signal transduction pathways, since intracellular signaling pathways appear to be sensitive to the microgravitational forces found aboard the Space Shuttle.

3. In preparation of using the NASA bioreactors we tested the compatibility of a novel cell culture medium, GTSF, recently developed at NASA JSC, with our cells. Our data confirm that this medium, with slight modifications will sustain the growth of both PC12 and of adrenal medullary endothelial cells (Lelkes et al., manuscript in preparation).

4. Since the beginning of November, the first exploratory growth experiments with our cells in NASA reactors are being conducted at JSC. Preliminary data indicate that both cell types and the co-cultures do much better in the STLV than in the HARV.

Preview of FY94: We will substantiate the initial cell culture studies using HARV's. Samples will be removed periodically an asses for organ-specific differentiation by microscopic, biochemical, and molecular biological techniques. Early next year, a predoctoral student involved in the program will be in serviced at JSC and then we will continue with the experiments in close collaboration between our lab and JSC, according to the original plans.

STUDENTS FUNDED UNDER RESEARCH:

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TASK INITIATION: 11/92  EXPIRATION: 11/95

PROJECT IDENTIFICATION: 674-23-01-12

NASA CONTRACT NO.: NAG-651

RESPONSIBLE CENTER: JSC
II. MSAD Program Tasks — Ground-based Research

Discipline: Biotechnology

BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:


Crystallization Studies in Microgravity of an Integral Membrane Protein: The Photosynthetic Reaction Center

**Principal Investigator:** Dr. James R. Norris

**Co-Investigators:**
- J. Deisenhofer
- M. Thurnauer
- D. Tiete
- P. Thiyagarajan
- M. Schiffer
- R. Furrer

**Argonne National Laboratory**

**Howard Hughes Medical Institute**

**Free University Berlin**

**Task Objective, Description & Significance:**

**Objective:**
The objective of this project is to determine the effects of microgravity on the crystallization of the integral membrane proteins.

**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. Previous crystallization experiments in microgravity have suggested that the elimination of density-driven convection and sedimentation may generally lead to larger, better-ordered crystals for water-soluble proteins. The potential beneficial effects of microgravity on the crystallization of integral membrane proteins have not yet been tested. The necessity of maintaining membrane proteins in detergent micelles during the crystallization process suggests that the deleterious effects of gravity-driven convection may be more significant during the crystallization of membrane proteins than water soluble proteins.

Research Approach — The effects of microgravity on crystallization of membrane proteins will be tested using the *Rhodopseudomonas viridis* and *Rhodobacter sphaeroides* photosynthetic reaction centers as test proteins. The two reaction centers differ in the proportion of the accessible surface area that is buried in the micelle environment. The comparison of *Rps. viridis* and *Rb. sphaeroides* crystallization will provide a useful indicator of whether the effects of microgravity can be linked to differences in volume fraction of detergent in the detergent-protein complexes. The spaceflight and ground control crystallization experiments will be analyzed by three techniques: optical microscopy, x-ray diffraction, and small angle neutron (SANS) and light scattering. The optical microscopy and x-ray diffraction of crystals will determine the effects of microgravity on crystal growth and molecular ordering. SANS and light scattering studies on reaction center solutions at various extents of completion of crystallization will be used to determine the effects of microgravity on the nucleation events that precede and then continue during the crystallization process. The results of reaction center nucleation and crystallization studies in microgravity will be relevant to the crystallization of integral membrane proteins in general.

Since 1988, despite a great deal of worldwide effort, relatively few other membrane proteins have been crystallized in a form suitable for high-resolution structural studies. To date, only the reaction center structures have been solved. Many other membrane proteins have been crystallized, but the generally small crystal size and poor diffraction quality has prevented structural determination. A complicating factor is the presence of the detergent micelle. The effect of the detergents on the crystallization process is unknown. However, the effects of small amphiphiles on crystallization suggest that micelle size or radius may play an important role in determining
whether protein-protein contacts predominate to produce a crystalline structure, or whether non-specific micelle-micelle contacts predominate to yield a less ordered array. Crystallization frequently occurs at or near the phase separation of the detergent. This correlation between crystallization and detergent phase separation has led to the investigation of the micelle-micelle attraction, and the role this will play in the early nucleation process. Ultimately, the micelle aggregation must be balanced by protein-protein interactions to result in crystal formation.

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.

PROGRESS DURING FY 1993:
In designed space growth experiments involving doped and undoped BSO and BTO, it is considered possible to establish controlled growth perturbations and, through their analysis, to gain insight into cause and effect relationships for defect generation during growth on Earth. Space experiments are similarly to be used to obtain for these opto-electronic materials small quantities as reference standards for a data base of physical properties.

Discussions of the design, conduct and analysis of the proposed space growth experiments with members of the opto-electronic industrial research community have been productive and indicated broad-based interest as well as the desire to contribute actively to the success of the proposed research undertaking.
Shear Sensitivities of Human Bone Marrow Cultures

PRINCIPAL INVESTIGATOR: Bernhard O. Palsson, Ph.D.  
University of Michigan

CO-INVESTIGATORS:  
No Co-I's Assigned to this Task

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

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.

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.

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 1993:

1) We can now expand human bone marrow cells from whole bone marrow, density separated mono-nuclear cells (MNC), or highly enriched progenitor populations over a 2 week period without replating the cells. When we grow MNC in the presence of the stem cell factor (SCF), erythropoietin (Epo), granulocyte/macrophage stimulating factor (GM-CSF), and interleukin 3 (IL-3), we can expand the population by 10- to 15-fold over a two week period. Most of the growth is observed during the second week of culture. Epo and GM-CSF are particularly effective in recruiting cells into differentiation (SCP synergises strongly with Epo) while SCF and IL-3 under perfusion conditions allow the progenitor cell population not only to be maintained but expanded by a factor of 10-20 over the two week period. During the coming year we will look more closely at the role of the specific growth factors and their influence on lineage development.

2) We plan to address this objective during the second year of this program. We are in the process of identifying a postdoctoral student to undertake this work.

3) We have a frozen stock of bone marrow cells from allogeneic donors that is frozen down in 100 million cell aliquots. This stock gives the advantage of being able to start experiments from a consistent cell source. Half of this stock has been shipped to the Biotechnology groups at Johnson Space Center. They have implemented our thawing and growth protocols for bone marrow successfully and have the ability to produce cells over an extended period of time. Further they have implemented these conditions in a rotating wall vessel and obtained preliminary results. Once the JSC group has identified approximate operating conditions, we will received RWV's in Ann
Arbor and carry out the proposed studies under goal 3. The post doctoral fellow who is being recruited will be responsible for these studies.
Microgravity Crystallization of Avian Egg White Ovostatin

**Task Objective, Description & Significance:**

**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 \( \alpha_2M \) 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.

**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 6 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 \( \alpha_2M \). Currently, the only structural information extant is from electron microscopy studies of isolated molecules.

**Research Approach —**

Task 1: Preparation of ovostatin to 1/94 for protein to be used in flight experiment(s). Large scale protein purification will be continuous for the duration of the project. However, an accumulated supply (\( \geq 10 \) gm) of purified protein will be the sole source of material, starting in October 1993, for the flight and final ground-based experiments. This material will be used to establish the reference ground-based crystal quality against which flight crystals will be measured.

Task 2: Screening of crystallization condition to 7/93. The best crystallization conditions determined at this time will be used for the 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 to flight + 4 months. Multiple data sets will be acquired using the x-ray diffractometer. These will be used to establish the baseline ground-grown crystal quality which the flight crystal(s) will be measured against. Subsequent runs will be done after the flight, using the same time-lines, conditions, and solutions as the flight experiments to further define this baseline.

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.
II. MSAD Program Tasks — Ground-based Research

SIGNIFICANCE:
Any knowledge gained about ovostatin based on crystal structure analysis would advance the overall knowledge of this family of proteins.

PROGRESS DURING FY 1993:
Previous microgravity protein crystal growth experiments have used vapor diffusion, free interface diffusion, and dialysis techniques with most being done using vapor diffusion. Litke 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.

BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:


Study of Crystallization and Solution Properties of Redesigned Protein Surfaces

PRINCIPAL INVESTIGATOR: Prof. David C. Richardson
Duke University Medical Center

CO-INVESTIGATORS:
M. Hecht
K. Gernert
J. Richardson
Princeton University
Duke University Medical Center

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

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.

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.

Research Approach — 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.

SIGNIFICANCE:
The research aims are 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. A detailed study of protein crystal contacts and their specific effects may also help in the future to sort out 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.

**Progress During FY 1993:**

Progress on the wild-type protein includes purification of approximately 200 mg, crystallization, and packing of solubility columns. Crystals of wild-type B562 have been grown under conditions used for structure determination, 3.5 M PO4, pH 6.8. It was under these conditions that a crystal slurry of wild-type protein was grown and packed into four solubility columns which are now being used. These columns will be used to collect solubility data of the wild-type crystals under a variety of buffer conditions and additives, and a range of temperatures.

**Students Funded Under Research:**

PhD Students: 1

**Bibliographic Citations for FY 1993:**

*Proceedings:*

II. MSAD Program Tasks — Ground-based Research

Discipline: Biotechnology

Convective Flow Effects on Protein Crystal Growth and Diffraction Resolution

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

**Co-Investigators:**  
L. Monaco  
University of Alabama, Huntsville

**Task Objective, Description & Significance:**

**Objective:**  
The overall objective of this research is to establish a correlation between well-defined solution flow and supersaturation conditions about protein crystals, their growth kinetics, and resulting X-ray diffraction resolution. Towards this end, we will study the morphology and kinetics of selected proteins during growth in a temperature-controlled flow-through cell, with *in situ* high-resolution microscopy. The proteins used in these studies will be carefully purified and characterized by gel electrophoresis and column chromatography. Precipitant compositions and concentrations that have been shown to lead to specific molecular aggregation behavior in the solutions will be used.

**Description:**  
The long term objectives of the proposed research are an advancement in the understanding of the fundamental mechanism by which protein crystals grown, and the origin and possible reduction of the limitations of the diffraction resolution of protein crystals. Attention will be focused on limitations in crystal homogeneity possibly resulting from nonsteady repartitioning kinetics of the precipitant and the couple attachment kinetics of the protein.

Research Approach — For the reversible control of the supersaturation, we will utilize the temperature dependence of the solubility of the proteins, which, in turn, will be determined by a miniaturized optical scintillation technique recently developed in our group. In addition, we will monitor the repartitioning of the precipitant and buffer components during the crystallization by electrochemical techniques and atomic absorption spectroscopy.

The characterizations of the flow and concentration distributions will be aided by numerical modeling. In order to facilitate realistic modeling, we will measure the protein diffusivities and the solution viscosities at the growth conditions utilizing novel interferometric and viscometric techniques, respectively.

The structural quality of selected crystals, for which variations in transport and supersaturation conditions are found to significantly influence growth kinetics, will be characterized through three dimensional x-ray diffraction data set determinations.

**Significance:**  
In spite of significant progress over the last few years, the growth of single protein crystals with high structural quality remains more an art than a science. Most workers have taken a pragmatic approach. Often protein crystallization experiments are designed to statistically vary the large number of parameters thought important. Relatively little attention has been given to studying the actual nucleation and growth mechanisms, the understanding of which could lead to an advantageous choice of crystal growth conditions. It appears that protein crystal growers have only recently become aware of the fact that many of the problems encountered in current crystallization practices reflect a lack of control in purity and of process parameters, rather than intrinsic features of protein crystallization. In particular, none of the current protein crystallization techniques allow for the establishment of conditions that are conducive to the growth of a few, large crystals. Most techniques retain the
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Discipline: Biotechnology

high supersaturations (200-300%) required for protein nucleation for long periods after nucleation has occurred. As a consequence, nucleation tends to proceed after the initial nuclei have formed and begun to grow. This leads to the typical large number of crystals, which often limits their size and, through mechanical interference, results in poor crystal quality.

**PROGRESS DURING FY 1993:**

Through NASA Grants NAG8-711, 824, and 868 in effect from 1987 through 1992, we have been able to:

- Thoroughly familiarize ourselves with the key problems in protein purification, characterization, and crystallization;
- Acquire state-of-the-art preparation and research equipment;
- Develop miniaturized techniques for solubility studies and temperature control of growth processes; and
- Develop instrumentation for and begin in situ high-resolution microscopic studies of growth and etching morphologies.

The involvement in protein crystallization, made possible by NASA's Microgravity Science and Applications Division through the above cited grants, will greatly benefit the work being performed under this task.

**STUDENTS FUNDED UNDER RESEARCH:**

PhD Students: 2

**BIBLIOGRAPHIC CITATIONS FOR FY 1993:**

**Journals:**


Enhancement of Cell Function in Culture by Controlled Aggregation

PRINCIPAL INVESTIGATOR: W. Mark Saltzman
Johns Hopkins University

CO-INVESTIGATORS:
No Co-I's Assigned to this Task

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

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 objective of this program are: 1) development of methods for controlling cell aggregation using bioactive polymers and polymer microspheres and 2) systematic study of the function and behavior of suitably aggregated cells in culture. The discovery of new methods for improving cell growth and function is critical to the development of hybrid artificial internal organs and mammalian cell bioreactors.

DESCRIPTION:
To achieve the main objective, we have formulated the following specific tasks:

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 microgravity). 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 in 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 and the PC12 cell line.

Objective 4: Measurement of Cell Function Under Different Aggregation Conditions in Gel Culture
We have already developed methods for monitoring cell growth and metabolism in the 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.
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.

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.

PROGRESS DURING FY 1993:

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 grafted to the termini. This work is being prepared for publication.

Objective 2:
We have synthesized polystyrene polymers modified with carbohydrates at the surface.

The abstract of a paper that was recently accepted for publication is appended: (Gutsche, A.T., Parsons-Wingerter, P., Chand, D., Saltzman, W.M., and Leong, K.W. N-acetylglucosamine and adenosine derivatized surfaces for cell culture: 3T3 fibroblast and chicken hepatocyte response, Biotech. & Bioeng., in press).

Objectives 3 and 4:
We have developed methods for culturing and forming aggregates under unit gravity.

The abstract of a paper that was recently accepted for publication is appended: (Krewson C.E., Chung, S.W., Dai, W., and Saltzman, W.M. Cell aggregation and neurite growth in gels of extracellular matrix molecules, Biotech. & Bioeng., in press).

Objective 5:
Only applicable in the third project year.
II. MSAD Program Tasks — Ground-based Research  

Discipline: Biotechnology

Culture of Porcine Islet Tissue: Evaluation of Microgravity Conditions

**Principal Investigator:** David W. Scharp, M.D.  
Washington University School of Medicine

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

**Task Objective, Description & Significance:**

**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.

**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.
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 1993:
Porcine islet tissue, even with its inherent problems of retrieval, rapidly becoming the species of choice in islet cell transplantation and research. Although islet numbers and culture survival produce challenges now for the investigator, the similarity of porcine insulin to that produced by humans, may alleviate future transplant problem for the recipient.

As equipment was being gathered and readied at KRUG Life Sciences for our work, we began preliminary studies isolating adult pig islets to use as the first controls in the culturing systems. In acquiring a new potent collagenase from Sigma Chemical Company, St. Louis, MO, we established in improved method of pancreas digestion yielding an average of 3673 islet equivalents per gram of tissue. Improvements in the purification of these pig islets by using hypaque focoi! in a continuous gradient in the Cobe Cell Processor has increased recovery to an average of 100% and purity to 95% while cutting our expenses by 70% and the time for processing by 75%.

With the first few shipments we discovered a contamination problem of a budding yeast that would manifest itself in large quantities after about a week in the MGT devices. Although static controls left in St. Louis did not show the yeast growth, assumptions were made that the extraordinary culture conditions and high oxygen content allowed a small contamination to take hold. Precautions were taken at both facilities to eliminate any possible technical problems that may have initiated the contamination as well as the additional of bacteriostatic and fungistatic agents to all solutions used in processing and culturing. Because the most likely source of the yeast was the donor pig, more stringent measures were taken during procurement. In addition to the usual cleansing of the pancreas with a Betadine solution, followed by serial rinses of Hanks Buffered Solution, and EuroCollins, bacteriostatic and fungicidal agents were used at each step. Routine monitoring of the preparation at various points in the process for fungal and bacterial growth are being done in ST. Louis and samples are also being processed at KRUG on the final preparation. At this point, the problem seems to have been solved.

The initial time has been beneficial to the crew at KRUG Life Sciences also. Never having worked with islets before, they were challenged with the problems or working with large cell clusters instead of the usual cell lines they were familiar with from past experiences. Methods had to be established to determine proper cell concentration, seeding techniques, familiarity with the morphology of the islets.

These initial studies involved the use of both the STLV and HARV culturing systems as well as status T-flask controls. Microcarriers were also utilized to help reduce the rotational sheer to negligible levels. Approximately 200 collagen microcarriers, with an internal volume of 25 microliters, were initiated into a STLV containing CMRL 1066 with 10% Fetal Bovine Serum, antibiotics, and supplements. Cytodex 3 beads were used in both the STLV and HARV systems at 5mg/ml of cell suspension. Cell concentrations have been varied to establish the optimum density for each system and each carrier. Daily readings for pH, CO2, O2 and glucose consumption rate were taken as well as samples assayed for insulin content.

Further studies have begun on suspension culture in a status system. Hydrogel matrices of agarose and alginate were tested using both CMRL and Minimal Essential Medium (MEM) as base media. Islet viability was established with Fluorescein Diacetate staining and glucose challenges in static incubations. Preliminary results show promise with both matrices.
II. MSAD Program Tasks — Ground-based Research

Discipline: Biotechnology

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**Task Initiation:** 1/93  **Expiration:** 1/95

**Project Identification:** 674-23-01-08

**NASA Contract No.:** NAG-653

**Responsible Center:** JSC
Role of Solvation Forces in Protein Crystallization in Microgravity

PRINCIPAL INVESTIGATOR: Dr. Frank B. von Swol
University of Illinois, Urbana-Champaign

CO-INVESTIGATORS:
C. F. Zukoski
University of Illinois, Urbana-Champaign

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

OBJECTIVE:
The research study is a comprehensive investigation of fundamental problems in the thermodynamics and growth of macromolecular crystals. The research builds on recent theoretical advances in describing the role of solvation interactions in ordering processes observed in such diverse systems as lipid bilayers, DNA strands, protein crystals and clays.

DESCRIPTION:
A comprehensive study of the role of solvation forces in protein crystallization will be undertaken. This program combines new approaches for describing the statistical mechanical properties of suspensions containing hydrophilic particles with an experimental program where models can be verified and extended. The goals of the efforts are to:

- Develop a fundamental understanding of how solvent chemical potential modifies protein interactions and phase behavior; and

- Exploit solvation interactions in the development of methods for preparing protein crystals with substantially lower degrees of hydration, enhanced crystal strength, and higher degrees of order to increase resolution of x-ray structural determinations.

Research Approach — The ability of solvation forces to influence protein crystallization will be investigated through the study of model macromolecular compounds chosen for their availability, crystallizability and degree of solvation in the crystalline phase. These materials will be used in an extensive study of the influence of solvent chemical potential on crystal structure, nucleation rates and crystal growth kinetics.

SIGNIFICANCE:
The research study is designed to develop further insight on how to control protein crystal nucleation and growth. The investigations are expected to shed light on the role of hydrodynamic disturbances on the degree of crystal order. New methods of preparing protein crystals with higher packing densities and fewer packing defects will be explored.

PROGRESS DURING FY 1993:
The von Swol group has worked on interfacial modeling using statistical mechanics and simulation for a number of years. Previous work relevant to this research has focused on development of new simulation techniques for interfacial phenomena including constant pressure simulations and grand canonical molecular dynamics. In addition, a new ensemble has been developed to describe the Surface Forces Apparatus (SFA). The work on the SFA has been crucial in developing our current views on solvation forces.
II. MSAD Program Tasks — Ground-based Research

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Discipline: Biotechnology
Automation of Protein Crystallization Experiments: Crystallization by Dynamic Control of Temperature

**PRINCIPAL INVESTIGATOR:** Dr. Keith B. Ward  
**Naval Research Laboratory**

**CO-INVESTIGATORS:**  
W.M. Zuk  
M.A. Perozzo  
Geocenters, Inc./Naval Research Laboratory (NRL)  
Naval Research Laboratory (NRL)

**TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:**

**OBJECTIVE:**  
The goal of this research program is to develop a dynamically-controlled crystallization cell (DCCS), in which protein supersaturation is controlled by varying the temperature while crystallization is monitored by optical means. This device is also capable of being controlled telerobotically. The program intends to extend its accomplishments in this area by enhancing the capabilities of the DCCS by miniaturizing it more, decreasing the sample volumes required for tests, 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.

**DESCRIPTION:**  
A study of a representative sample of 50 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.

Research Approach — The proposed methods of research include modifying the current design to allow the use of solutions as small as 100 microliters, to incorporate video monitoring to provide 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 studies 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 experiments on space platforms in microgravity. Collection of protein temperature solubility data will be enhanced by the development of automated software algorithms. The temperature will slowly be changed, step-wise, until the level of the scintillation signal indicates that the crystallization phase boundary has been crossed. The temperature will then be recycled, and the point obtained again using a finer temperature step size, until the ultimate precision of this method will be determined.

**SIGNIFICANCE:**  
This research is important in continuing the development of the DCCS at the Naval Research Laboratory as part of the current team effort of NASA-funded Principle Investigators to develop a dynamically-controlled system for the Advanced Protein Crystal Growth apparatus. This system will be used, while it is being developed, to explore whether temperature control of supersaturation is a technique that can have wide applicability in protein crystallization.

**PROGRESS DURING FY 1993:**  
NASA-supported research in the field of protein crystallization has led to an increased understanding of the important variables that control nucleation and crystal growth mechanisms. One outstanding result of these studies is that
II. MSAD Program Tasks — Ground-based Research

Discipline: Biotechnology

Levels of solute supersaturation optimal for nucleation are not the same as those required for optimal crystal growth. It has also become clear that the temperature is a convenient variable for controlling protein supersaturation, although it is not routinely used by protein crystallographers. The continued development of automation systems will make use of these findings. This work will be conducted as part of a continuing collaborative effort of currently-funded NASA investigators to develop an improved generation of protein crystallization apparatuses for use on space platforms.

BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:


Proceedings:

Presentations:
II. MSAD Program Tasks — Ground-based Research

**Thermal Optimization of Growth and Quality of Protein Crystals**

**Principal Investigator:** Dr. John M. Wiencek          Rutgers University

**Co-Investigators:**
E. Arnold          Rutgers University

**Task Objective, Description & Significance:**

**Objective:**
The specific objectives of this research include:

- Development and utilization of quantitative temperature control strategies that maintain constant growth rates during crystal growth, and measure growth rates by time-lapse video microscopy;

- Utilization of titration calorimetry to determine temperature dependence of protein solubility, and investigation of the suitability for measuring crystal growth rates; and

- Assessment of protein crystal quality under varying rates of growth.

**Description:**

Three protein systems have been chosen for study: lysozyme, equine serum albumin (ESA), and reverse transcriptase (RT). Each of these proteins have unique features that make them interesting. Lysozyme and ESA represent fairly inexpensive and readily available proteins (available from Sigma Chemical Company, for example) and will be model systems for investigations elucidating the effects of growth rates on crystal quality. Once strategies that are optimal are available, these strategies will be applied to the RT system as a realistic test case.

All proteins systems will be assessed for purity by isoelectric focusing and SDS-PAGE electrophoresis. If necessary, the proteins can be purified by recrystallization as described for lysozyme by Pusey and Genert (1988). Our experience to date has shown that lysozyme received from Sigma is sufficiently pure for direct use.

Research Approach — Experimental evidence exists which indicates that larger and higher quality crystals can be attained in the microgravity environment of space. Fundamental studies have attempted to elucidate 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 outlines a ground-based effort to measure protein size and quality as a function of growth rate. Growth rates will be maintained at a constant value by controlling the supersaturation via temperature. Quality assessment will utilize standard techniques which assess the x-ray diffraction quality. The results of this study will indicate whether crystals grown at slow rates (as may happen in microgravity due to the absence of convection) are linked to improved quality. In addition, processing strategies will be developed which x-ray crystallographers can use to grow larger, high-quality crystals once an experimental condition producing microcrystals is attained. During the course of the investigation, information will be accumulated on the growth rates and solubility of lysozyme and equine serum albumin via time-lapse video microscopy and titration calorimetry. Once the effects of growth rates on crystal quality are understood, plans are to apply what has been learned to an optimization of crystal quality of a protein of current interest, namely, HIV-1 reverse transcriptase.
II. MSAD Program Tasks — Ground-based Research

SIGNIFICANCE:
Development of a systematic method of protein crystallization may lead to crystallization of previously uncrystallizable proteins and add to the current knowledge of protein structure/function relationships. Knowledge of detailed protein structure is absolutely essential for rational approach to protein engineering and has important implications for design of improved small molecule pharmaceuticals.

PROGRESS DURING FY 1993:
Interest developed in the possibility of utilizing pressure to control rates of nucleation and growth. In studies of hen egg-white lysozyme crystallization under high pressure (1 to 2 kbar), there was no acceleration of crystallization for samples exposed to rapid letdown of pressure from 1 or 2 kbar as measured by concentration of lysozyme in solution. There was an apparent arrest of the crystallization process in samples held under high pressure for 48 hours in both seeded and unseeded batches (Brodeck et al., in preparation). Crystallization resumed at a normal rate upon restoration of atmospheric conditions. Lack of crystallization at high pressure does not appear to be due to a change in the solute/crystal equilibrium at high pressures. Samples containing lysozyme crystals in equilibrium with mother liquor were placed under pressure (1.0 kbar) for 48 hours and showed no evidence of dissolution as measured by lysozyme concentration in solution.

STUDENTS FUNDED UNDER RESEARCH:

PhD Students: 2


Project Identification: 674-23-08-28

NASA Contract No.: NAG8-975

Responsible Center: MSFC
Search for Dilute Solution Property to Predict Protein Crystallization

PRINCIPAL INVESTIGATOR: Dr. W. William Wilson Mississippi State University

CO-INVESTIGATORS: No Co-I's Assigned to this Task

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

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.

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, PEGs and other organics). The laser scattering solution parameters will be measured in the dilute protein concentration regime, often 10-20 times below protein saturation.

Research Approach — 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 man-power 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.

SIGNIFICANCE:
It is anticipated that a particular solution parameter (or combination of parameters) will be discovered that has quantitative values within a reasonable 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 1993:**
Since the task initiation date, the appropriate personnel have been started on the project. There are one postdoctoral, Dr. Abraham George, and two graduate students funded by this task. Initial experiments accomplishing the objectives have begun.

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Radiative Extinction of Diffusion Flames

**Principal Investigator:** Prof. Arvind Atreya
*University of Michigan*

**Co-Investigators:**
Prof. I.S. Wichman
*Michigan State University*

**Task Objective, Description & Significance:**

**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.

**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 understand 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 inert gases into the fuel-flow stream. In these tests measurements of flame temperatures and radiated flux will be used for comparisons with theory.

A numerical model will be developed to simulate the reduced-gravity experimental configuration, and will include chemical kinetic modeling and modeling for the production and consumption of soot particulates. A model for the radiant emissions from the flame associated with the particulates will be developed.

**Significance:**
The microgravity flames will demonstrate the concept of radiative extinction in stabilized flames, distinct from the case of spreading diffusion flames.

**Progress During FY 1993:**
Soot volume fractions, species concentrations, and temperature distributions inside a normal-gravity, counterflow diffusion flame have been measured.

A 2.2 second drop tower apparatus has been designed, built and tested. Diffusion flames using both methane and ethylene were stabilized upon spherical burners in microgravity; in each case over a range of fuel flowrates. These tests were all conducted in air at normal atmospheric pressure. Flames ignited in normal gravity were found to approach a microgravity configuration more rapidly than flames ignited in microgravity because of a lengthy ignition transient. Extinction of flames was observed during the transition from normal-gravity stabilization to a microgravity environment, in which the flames approached a quasi-spherical shape.
II. MSAD Program Tasks — Ground-based Research

Discipline: Combustion Science

A more complete set of 2.2 second drop tower experiments are planned using the spherical burner, extending the experiments to a higher pressure regime where experiments may progress further in the available test time.

STUDENTS FUNDED UNDER RESEARCH:

MS Students: 2

TASK INITIATION: 4/91  EXPIRATION: 12/94

PROJECT IDENTIFICATION: 674-22-05-29

RESPONSIBLE CENTER: LeRC

BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:


Presentations:
II. MSAD Program Tasks — Ground-based Research

Discipline: Combustion Science

Ignition and Combustion of Bulk Metals in Microgravity

Principal Investigator: Prof. Melvyn C. Branch
University of Colorado

Co-Investigators:

J.W. Daily
University of Colorado, Boulder

G. J. Fiechtner
University of Colorado, Boulder

A. Abbud-Madrid
University of Colorado, Boulder

Task Objective, Description & Significance:

Objective:
Most structural metals, when exposed to a threshold oxygen partial pressure and a sufficiently high temperature, will ignite and burn vigorously. This project is an analytical and experimental investigation of the mechanisms of ignition and combustion characteristics of metals in bulk pellet configurations, rather than conventional powders, under low-pressure oxygen atmospheres in a range of gravity fields. Other dedicated experimental features are found in the diagnostics for determination of fuel mass loss, flame characteristics, surface structure, and temperatures of each phase.

Description:
The experiments were conducted initially under normal gravity to establish the utility of the continuous radiant-energy ignition and to determine reference data on metal-surface morphology, ignition criteria, flame characteristics, and heat and mass transfer mechanisms. Experiments followed at elevated gravity, conducted in the unique University of Colorado Geotechnical Centrifuge, to establish the sensitivity to convective flows and to calibrate systems for mass-loss measurements. The eventual low-gravity experiments are under design for installation in the NASA Low-gravity Airplane Laboratory (KC-135). Concurrent numerical modeling calculations guide the experimental research.

Significance:
This study will explore the entire process of metal combustion, starting from the heat-up and ignition phases. The microgravity experiments will simplify the isolation of the individual phases of the phenomena by reducing the interactions of convective flows. Metal combustion in microgravity also differs strongly from that in normal gravity because the reduced mobility of the liquid phase changes the surface exposure of the burning pellets. The study has practical significance in its potential application to fire safety for spacecraft oxygen storage and handling systems. An additional benefit of the study is in the development of new experimental capabilities with wide-ranging applications. The major innovation is in a high-heat-flux radiant light source for ignition, a method that eliminates the contamination of metal fuels associated with the usual promoted igniters.

Progress During FY 1993:
The normal-gravity experimental study has been completed. The novel application of isolating the ignition process through use of a non-intrusive radiation source was successful. For this purpose, a 100-W Xe arc lamp was used lamp to irradiate cylindrical metal specimens, 5-mm in diameter and 5-mm high. The experimental results provided a qualitative visual record of the heat-up, ignition, and combustion processes and corresponding measurements of the surface temperature histories. Metal specimens of iron, titanium, zirconium, magnesium, zinc, tin, and copper all ignited and sustained combustion under a quiescent, pure-oxygen atmosphere at a pressure of 0.1 MPa. Only aluminum, in this set of common structural metals, failed to ignite under these conditions. On the other hand, copper, which previous studies had classified as nonflammable, was found to burn readily under the downward...
propagation and non-intrusive radiant-flux conditions of the experiment. Ignition temperatures for all the metals were found to be below, above, or in the range of the metal melting point.

Interpretation of the normal-gravity ignition and combustion-test data characterized the distinct stages of heat-up, ignition, and combustion through well-defined surface structure changes, surface temperature behavior, and flame-zone luminosity. The unexpected flammability of copper confirmed the strong influence of sample geometry, experimental configuration, and ignition source on the ignitionability of metals in these small-scale tests.

The experimental apparatus is now being adapted for mounting and testing on the University of Colorado Geotechnical Centrifuge. Experiments corresponding to those under normal gravity are being conducted at levels of 2 to 5 g (g=9.8 m/s²). The data-acquisition system under development permits the transmission of information from the spinning apparatus in real time. A high-speed camera setup will monitor the observations of the surface and flame zones.

The concurrent analysis has provided a computational model of the experimental conditions of the heat-up, ignition, and combustion stages. Application of representative normal-gravity data has verified the accuracy of the modeling. The complete analysis computes the specimen temperature and phase response for a constant-input heat flux over a range of gravity from 0 to 10g for 100%-oxygen atmosphere with pressures from 0.01 MPa to 1.0 MPa. Several interesting conclusions are provided by the analysis. As expected, convective heat transfer increases with increasing gravity, and consequently specimen temperature decreases with gravity. Specimen temperature also decreases with pressure increases. Both the gravity and pressure effects are strongest at lower values, i.e., most of the gravity influence is over the range of microgravity to one g. The analysis implies that the bulk metals may be easier to ignite in the microgravity environment because of the reduced rate of convective heat transfer.

Preliminary design and test condition selection are now underway for the final phase of the study, the experiments under low gravity. The current centrifuge design is adaptable in principle, but it will require refinements for a modest reduction in volume to meet the restraints of the NASA KC-135 Airplane Laboratory.

STUDENTS FUNDED UNDER RESEARCH:

Total Students: 2

TASK INITIATION: 4/91 EXPIRATION: 11/94

PROJECT IDENTIFICATION: 674-22-05-30

RESPONSIBLE CENTER: LeRC

BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Proceedings:


II. MSAD Program Tasks — Ground-based Research

Discipline: Combustion Science

Modeling of Microgravity Combustion Experiments

PRINCIPAL INVESTIGATOR: Prof. John C. Buckmaster
University of Illinois, Urbana-Champaign

CO-INVESTIGATORS:
No Co-I's Assigned to this Task

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

OBJECTIVE:
The objective of this program is to improve our understanding of basic combustion phenomena through the analytical and numerical models developed from first principles for various systems/configurations undergoing experimental study in NASA's microgravity combustion program.

DESCRIPTION:
Experimental results amenable to modeling are identified and modeling is carried out. This is done in close collaboration with the experimentalists when possible.

SIGNIFICANCE:
Modeling provides invaluable physical insights into the experimentally observed behavior.

PROGRESS DURING FY 1993:

An analytical/numerical model for flame-balls in hydrogen-air mixtures including radiative heat loss effects is completed and the results are published in peer reviewed journals.

In another study dealing with the "absolute" flammability limits they examine optically thick flame-balls and determine that strong absorption precludes the existence of stable solutions. For sufficiently small ignition sources an intrinsic (apparatus independent) flammability limit is defined by the generally multivalued steady solution. Beyond the flammability limit a steady solution exists for arbitrarily weak mixtures, but is unrealizable since it is unstable to one-dimensional perturbations. An unsteady propagating flame solution is also possible for arbitrarily weak mixtures, but can only be generated by a large ignition source.

Intrinsic and acoustic instabilities in flames fueled by multiphase mixtures (gas and droplets/particles) are described for confined or unconfined flames, such as for propagation in a tube with one end open and the other end closed or spherical flames. The implications of findings for gas turbine acoustic instabilities, where the condensed phase is injected at a finite point (rather than dispersed throughout the gas phase), are pointed out.

Another numerical model is developed which accounts for the effects of self-absorbed radiation in gas mixtures with varying optical thicknesses. The analysis was successful for partially explaining the experiments of Paul Ronney from Princeton where he observed flame-strings in SF₆ diluted mixtures.

A modeling effort on metal burning has recently been initiated.

STUDENTS FUNDED UNDER RESEARCH:
Total Students: 4 Total Degrees: 1

TASK INITIATION: 11/91 EXPIRATION: 10/93
PROJECT IDENTIFICATION: 674-22-05-31
RESPONSIBLE CENTER: LeRC
BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:


Proceedings:


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, Description & Significance:

Objective:
The objective of the experimental program is to investigate low-Reynolds-number, premixed turbulent flames in microgravity to gain a fundamental understanding of the gravitational effects on flame-turbulence interaction processes.

Description:
The experimental work is focused on developing the methodology, experimental apparatus, and laser diagnostics for application in the NASA Lewis Research Center 2.2 drop tower. Flow visualization techniques coupled with computer-controlled image processing are used to characterize the behavior of the aerodynamic flowfield and mean flame properties under microgravity.

To provide the necessary scientific background for the microgravity experiments, a parallel study of laminar and turbulent flames subjected to +g (upwards) and -g (downward) forces is also conducted. These +g and -g laboratory flames are investigated with the use of more sophisticated laser diagnostics such as laser Doppler anemometry to obtain statistical data.

Significance:
This aspect of turbulent flame propagation is not well understood and has yet to be considered in current turbulent combustion theories. Our experimental results are expected to provide guidance and validation for the development of turbulent combustion models to include the effects of gravity.

Progress During FY 1993:
A 2.2-second drop tower rig was constructed and tested. The behavior of laminar and turbulent Bunsen type premixed flames under microgravity has been observed by the use of laser schlieren. This is the first successful application of the laser schlieren technique in the 2.2-second drop tower. The schlieren images are recorded on videotape and are analyzed by computer-controlled image processing. Comparison of the microgravity results with those observed under normal gravity shows that gravity affects flame propagation through the coupling of the flame dynamics and the surrounding flowfield. Under normal gravity, buoyancy-driven flow instabilities are shown to induce flame flickering.

In microgravity, the laminar flames still exhibit some flame instability which is small compared to the normal gravity flame flickering. In the turbulent flames, due to the random nature of the wrinkled flame fronts, differences are more difficult to identify and quantify. The differences are being investigated using the appropriate image analysis and display software for characterizing the flame wrinkle scales and their evolution. A single beam deflection method has been tested and applied to measure the flame fluctuation spectra at higher temporal resolution than the schlieren images can provide.
Laboratory measurements of the mean flow and turbulence characteristics of the flames chosen for microgravity experiments were made using laser Doppler velocimetry in +g and -g. The results show that buoyancy-driven flow instabilities induce turbulence fluctuations in the reactants upstream of the flame zone which have the same frequency as the flame flickering observed by the Schlieren. Far-field influences affect the intensity of the fluctuations. This discovery suggests that a better understanding of their coupling with the flame front dynamics is necessary to isolate the effects of microgravity on flame dynamics. It was also found that the natural acoustic frequency of the burner matched the 180 Hz fluctuation frequency of the small flame wrinkles. By filling the burner cavity with fiber glass wool the acoustical instability is reduced.

BIBLIOGRAPHIC CITATIONS FOR FY 1993:

**Proceedings:**
Combustion of Interacting Droplet Arrays in a Microgravity Environment

P R I N C I P A L  I N V E S T I G A T O R :  D r .  D a n i e l  D i e t r i c h  
S v e r d r u p  T e c h n o l o g y  I n c ,  L e R C  G r o u p

C O - I N V E S T I G A T O R S :
No Co-I's Assigned to this Task

T A S K  O B J E C T I V E ,  D E S C R I P T I O N  &  S I G N I F I C A N C E :

O B J E C T I V E :
The research program involves the study of one-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.

D E S C R I P T I O N :
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.

S I G N I F I C A N C E :
The eventual goal being to use the results of this work as inputs to 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).

P R O G R E S S  D U R I N G  F Y  1 9 9 3 :

A normal gravity gravity droplet combustion apparatus was built. The apparatus consists of a 551 combustion chamber with optical access from four orthogonal windows. A special quartz window was purchased to allow imaging, with a CCD intensified array camera, of the OH radiation from the flame. The chamber is mounted on a cart with an integrated gas mixing system so that any ambient environment (He-O₂, N₂-O₂, etc.) with an ambient pressure from between 0 and 1 atm can be accommodated. Droplets are supported on 0.125 mm quartz optical fibers with a 0.250 mm bead on the end. The droplets are currently ignited by a hot-wire.

The apparatus was used to burn single droplets at atmospheric to sub-atmospheric pressures and enriched ambient environments. The purpose was to duplicate previous work by other investigators and establish reasonable operation of the hardware. The apparatus has also been used to examine the ability of an intensified array camera to image the OH emission (310 nm) from a burning droplet, because OH is generally considered a better indicator of flame position. Flame images from an intensified array camera with a 310 nm bandpass filter were compared with flame images from taken with a standard color CCD camera. The images resulting from the two cameras were clearly different, with flame dimensions dependent on the camera used. Finally, the apparatus was modified to burn linear arrays of droplets. Work has begun on the testing of two droplets at atmospheric and sub-atmospheric pressures.
In reduced gravity work, droplets were successfully suspended and burned on 15 μm SiC fibers (Dietrich, 1992). A reduced gravity apparatus was designed and built to study the combustion of one- and two-dimensional arrays of droplets in atmospheric and sub-atmospheric pressures. The design can accommodate any one or two dimensional geometry with a maximum of nine droplets. The droplets are ignited by hot wires. The data consists of a backlit view of the droplets with a cine camera to obtain the droplet size as a function of time and an orthogonal view with a black and white CCD camera to obtain the flame dynamics as a function of time. The apparatus is designed to support the droplets on 15 μm SiC fibers, but thus far testing has only been conducted on 0.125 mm quartz optical fibers (the same as the normal gravity apparatus).

This experiment was originally built for the 2.2 second drop tower at the NASA LeRC, and has been modified to be used in the 10 second drop tower at the Japan Microgravity Center (JAMIC). The first series of experiments were conducted at JAMIC while the principal investigator was a visiting scientist for six months at the GIDLH. The experiments conducted were of approximately 4 mm n-decane droplets burning in air at 1 atm pressure.

The results showed unusual burning behavior at these large droplet sizes. Immediately after ignition the droplet flame was expected, bright yellow, sooty and spherical (except for a small non-uniformity due to a small convective flow). As the burning progressed, the luminosity of the flame decreased, and the flame standoff ratio (the ratio of the flame diameter to the droplet diameter) was much smaller than expected; approximately 5 compared to the typical 7-8 for this condition. By the end of the drop, the droplet had not yet burned to completion, and the flame was nearly invisible to the CCD camera. Further analysis is required, before any conclusions from this preliminary data. It is interesting to note that a much smaller (1-2 mm initial) n-decane droplet burned to completion as expected, with a bright yellow, sooty, spherically symmetric flame.

BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Presentations:
Combustion of Electrostatic Sprays of Liquid Fuels in Laminar and Turbulent Regimes

**Principal Investigator:** Prof. Alessandro Gomez

**Co-Investigators:**
M.B. Long  
M.D. Smooke

**Task Objective, Description & Significance:**

**Objective:**
This research is a combination of theoretical and computational work by Professors Alessandro Gomez (PI, experimental), Marshall B. Long (Co-I, diagnostics) and Mitchell D. Smooke (Co-I, computational), all from Yale University. It involves studying the formation and burning of electrospays of liquid fuels at both normal and reduced gravity.

**Description:**
Normal-gravity testing of electrostatic sprays is conducted with cold-flows (i.e., non-buoyant) and in counterflow diffusion flames. Microgravity tests of the counterflow diffusion flame are conducted in the 2.2-second Drop Tower. A numerical model of a gas-phase counterflow diffusion flame is being extended to account for the droplet spray, for comparison of predictions with experimental results.

**Significance:**
Electrospays are being studied because they offer several advantages over conventional fuel sprays, such as (a) generation of a relatively narrow droplet-size distribution; (b) elimination of droplet coalescence and the production of a relatively uniform spray due to the self-dispersion property of the spray; and (c) the possibility of manipulating droplet trajectories.

**Progress During FY 1993:**

**Experimental: Normal Gravity**
The work has been focused on (1) experiments in cold flows to understand electrostatic spray synthesis, i.e., how to produce a spray with the desired properties, and (2) combustion experiments with well-controlled spray flames. The cold-flow experiments are coming to end, with the completion of two journal articles, one on the Coulombic disruption of charged droplets and the other on the structure of the electrostatic sprays. The effect of the Coulombic droplet explosion on the flame has been investigated, resulting in another journal paper. The individual effects of the initial droplet size, velocity, and the strain rate on the structure of the flame has been investigated. It was concluded that similar structural changes can be made by changes in either the initial droplet size or the velocity, whereas the strain rate has a weaker influence.

Work is underway to improve the droplet size measurement from 4% to at least 1%, via use of scattering measurements. Upon accomplishing this, the work will be focused on making the measurements sufficiently quickly that the droplet size distribution can be determined.

**Experimental: Microgravity**
Preliminary tests were conducted in April 1993 in the 2.2-second Drop Tower. The counter-flow, spray, diffusion flame burner was adapted to an existing drop rig. The flame was ignited in normal gravity, prior to the drop. The video of the flame revealed that the flat normal-gravity flames became curved in microgravity (complicating analysis...
and modeling), indicating that the flatness was apparently due to buoyant effects. Subsequent normal-gravity testing with a seeded flow revealed that the cold-flow stagnation plane was curved, consistent with the microgravity observations. The flatness has been considerably improved by changing the spray coflow from nitrogen to carbon dioxide, and thus increasing the Reynolds number at a constant strain rate. Further microgravity tests have not been conducted due to the Drop Tower shutdown.

Modeling
A gas-phase counterflow model is being extended to account for the effects of the electrospray. The overall system could be solved with a coupled initial-boundary value problem, if the transport rates between the droplet and gas phase are independent of the droplet position. Instead, the gas phase system is solved by Newton's method and these results are used to solve the spray equations (in a Runge Kutta approach). Preliminary results have been obtained for experimental comparison for a typical spray flame under conditions where there is no direct interaction between droplets and the flame; the droplets simply act as a source of fuel vapor which is convected or diffuses toward the flame. The sensitivity of the temperature profile to the chemical kinetic mechanism is now under investigation.

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BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:


Presentations:


Time-Dependent Computational Studies of Pre-Mixed Flames

**Principal Investigator:** Dr. K. Kailasanath
**Co-Investigators:**
- Dr. G. Patnaik

**Task Objective, Description & Significance:**

**Objective:**
The objective of this work is to predict, via numerical modeling, the effects of gravity on specific fundamental phenomena (such as propagation, extinction, and stability) in lean-limit combustion of premixed gases.

**Description:**
The structure and dynamics of premixed flames are studied by performing detailed numerical simulations using time-dependent, one- and two-dimensional numerical models. These models solve the multispecies coupled partial differential reactive-flow equations.

The models include fluid thermodynamic and transport properties, chemical kinetics, radiative heat transfer, buoyancy and boundary heat loss effects. Reduced chemistry models are intended to be incorporated, where applicable.

**Progress During FY 1993:**
NRL has successfully modified the multidimensional flame code, FLIC2D, for hydrogen- and methane-air chemistries, diffusive transport properties and simple radiative loss mechanisms. They tested the modified code against the existing FLAME1D code, where applicable.

For hydrogen flames, they determine that heat losses to the burner can stabilize flames (i.e. suppress formation of cellular structures) with even more than 11% hydrogen in air for a range of inflow velocities. They conclude that gravity (at least up to earth gravity) plays a secondary role for stabilizing flames compared to heat losses to the burner.

For methane flames, the results to date indicate that a 6% methane-air mixture exhibit a multidimensional structure that appears different from those observed in lean hydrogen-air mixtures.

For most of the hydrogen and methane air flames the contribution of radiation is found to be small. However, in very lean (e.g. 4.5%) methane-air flames, inclusion of radiative losses results in extinguishment, whereas neglecting radiation gives finite planar flame burning velocities. Likewise, for rich (81%) hydrogen-air flames, including radiative losses leads to oscillations with larger amplitudes and eventual extinguishment, as compared to undamped oscillatory flames if radiation is neglected.

**Bibliographic Citations for FY 1993:**

**Task Initiation:** 2/92  **Expiration:** 2/94
**Project Identification:** 674-22-05-22
**Responsible Center:** LeRC

II-228
Journals:


Presentations:

II. MSAD Program Tasks — Ground-based Research

Discipline: Combustion Science

Radiative Ignition and Transition to Flame Spread in Microgravity

**Principal Investigator:** Dr. Takashi Kashiwagi  
National Institute of Standards and Technology (NIST)

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

**Task Objective, Description & Significance:**

**Objective:**
This project models and solves numerically, with experimental verifications, the two- and three-dimensional, time-dependent relationships of ignition and flame spread for paper ignited by radiant energy in a microgravity environment. Processes to be modeled include the endothermic surface pyrolysis, the exothermic surface (char) oxidation, and the exothermic gas-phase combustion. Variations to model realistic situations include thermally thin and thick fuels and quiescent and air-flow conditions.

**Description:**
Numerical models are developed, with experimentally determined properties and reaction rates, to predict the time-dependent radial contours of temperature, density, velocity, and chemical species in the gas phase during the steps of ignition, pyrolysis, and subsequent flame spread. Verifying experiments are defined for ground-based and flight microgravity facilities.

**Progress During FY 1993:**
During the past year, a parametric study of auto-ignition and subsequent flame spread in a quiescent axisymmetric configuration has been completed. The three-dimensional code was streamlined to improve its computational speed and accuracy. Some cases have been run which consider the effect of a weak flow on the auto-ignition and subsequent flame spread. Additionally, the ability to animate the computed results was developed.

The computations for three dimensional flame ignition and spread indicate that the ignition process is very sensitive to both radiant flux level and irradiated area. If either of these is too small, the fuel will be pyrolyzed, but a gas-phase ignition process will never occur. If a hot spot is added to the gas phase (similar to an ignitor wire in a physical experiment), then ignition occurs. If the levels of irradiation and area are sufficiently large, then gas-phase auto-ignition will occur without the need for a "pilot".

Two types of flame spread are calculated depending on the conditions; one is a result of the rapidly outward expansion of excess fuel-rich flow caused by a strong ignition and the flame slides over the sample surface because the coupling between flame and sample is weak. The other is the more conventional flame spread in which a traveling flame is supported by the supply of fuel gases from the degradation of the sample near/beneath the flame front.

Under quiescent conditions, the axisymmetric flame remains symmetric, with the post-ignition, semi-spherical flame cap changing to a donut-shaped flame with the central portion extinguishing due to burnout of the fuel. However, in the presence of a weak flow, as the flame propagates further, the downstream flame extinguishes due to the local relative depletion of oxidizer caused by the upstream flame, and the flame opens into more of a horseshoe shape.
II. MSAD Program Tasks — Ground-based Research

Discipline: Combustion Science

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BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:


Presentations:

Kashiwagi, T. Transition from localized ignition to flame spread over a thin cellulosic material in microgravity, Presented at the 1993 Fall Technical Meeting, Eastern Section of the Combustion Institute, October 1993, Princeton University.
II. MSAD Program Tasks — Ground-based Research

Discipline: Combustion Science

Sooting Turbulent Jet Diffusion Flames

PRINCIPAL INVESTIGATOR: Prof. Jerry C. Ku
Wayne State University

CO-INVESTIGATORS:
P.S. Greenberg
NASA Lewis Research Center (LeRC)

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

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.

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).

SIGNIFICANCE:
Microgravity combustion is not only relevant to fire safety on board a spacecraft but also provides a unique condition for better understanding of the combustion fundamentals.

PROGRESS DURING FY 1993:

Numerical aspects of modeling for soot formation and radiation heat transfer have been completed. Soot transport equations, the energy equation, and solutions for the radiative transport equation (RTE) have been incorporated into a parabolic solver code for nonpremixed turbulent free-shear layer flows. The RTE can be solved by the YIX method of spherical harmonics (P_n). The former is considerably more accurate and suitable for inhomogeneous media. It is disadvantageous, however, due to the requirement for long computation times. The latter is computationally more expedient, but has been observed to exhibit numerical instabilities in the optically thin limit. Iterations are then required between the energy equation and these solutions. Reasonable agreement has been found between predicted and measured soot volume fraction and temperature data. The final part of modeling to be completed is an accurate and efficient way for spectrally integrating the spectral radiative fluxes and properties.

Thermophoretic probe sampling measurements and subsequent TEM analysis for soot particle size and morphology have been completed in the laboratory on propane and ethylene diffusion flames. Full-field laser-light extinction measurements have also been successfully demonstrated. The tomographic inversion of the data to provide spatially resolved mass fractions has also been demonstrated. These results are presently being assembled for publication. A package has been constructed to perform both of these measurements in the LeRC 2.2 sec. drop tower. Data elucidating primary particle size distributions for both propane and ethylene diffusion flames under both normal and...
reduced gravity conditions has been successfully obtained and is being submitted for publication. Reduced gravity measurements of soot volume fraction were not, however, completed before the shut-down of the 2.2 second tower and hence will resume pending the availability of this facility. In the interim, other methods are being pursued in the laboratory, including interlacing fine, rapid response thermocouples between the probe grids, simultaneous absorption/pyrometry measurements, and possibly flow-field mapping via laser doppler velocimetry.

**STUDENTS FUNDED UNDER RESEARCH:**

| Total Students: 3 |

**BIBLIOGRAPHIC CITATIONS FOR FY 1993:**

**Journals:**


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, Description & Significance:**

**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 nonpremixed laminar flames.

**Description:**
This program comprises three 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 test apparatus is to be used to observe premixed laminar flames stabilized about a cylindrical and then a spherical porous burner to distinguish heat loss and flow divergence influences upon the flame stabilization and flamefront stability; and

3) A drop-tower apparatus is to then be used to observe nonpremixed laminar flames stabilized about the cylindrical and spherical burners to study soot formation, gas-phase unsteadiness, kinetic extinction, and radiative extinction.

**Significance:**
This work takes advantage of the microgravity environment to obtain nonplanar, one-dimensional flames that are stabilized around curved burners in the absence of buoyant or externally imposed flows. This flame configuration is a more fundamental flame paradigm than can be established in a normal-gravity environment, providing stabilized flames with minimal nonadiabaticities and without aerodynamic straining.

**Progress During FY 1993:**
During 1993 a redesigned 2.2 second drop tower apparatus was completed to perform stabilized premixed flame tests in microgravity around a cylindrical burner. A series of tests in mixtures of methane, oxygen, nitrogen, and helium were completed in the drop tower over a small range of fuel/air velocities. Helium was included to: (a) reduce the flame temperature, thereby suppressing hydrodynamic flamefront instability; (b) increase the Lewis number of the flame, thereby suppressing thermo-diffusive flamefront instability, and (c) reduce the laminar flame speed in order to stabilize the flame at larger standoff distances from the burner, thereby reducing heat losses to the burner.

Substantial improvements in flame uniformity were observed compared to the first cylindrical burner used during the previous year. The Sandia flame code was modified for use with the cylindrical geometry and was used to compare the numerically calculated flame response with the microgravity flames observed experimentally. Although trends with flow velocities and mixture ratios are the same, the computed flame location is larger than observed experimentally. Separately, an asymptotic analysis of cylindrical flames has been developed.
II. MSAD Program Tasks — Ground-based Research

Discipline: Combustion Science

An extension of the experimental effort into a free-floated experiment aboard the NASA reduced-gravity aircraft is being designed and implemented.

**Students Funded Under Research:**

PhD Students: 1

**Task Initiation:** 5/90  **Expiration:** 11/94

**Project Identification:** 674-22-05-37

**Responsible Center:** LeRC

**Bibliographic Citations for FY 1993:**

**Journals:**

**Proceedings:**

**Presentations:**
A Fundamental Study of the Combustion Syntheses of Ceramic-Metal Composite Materials Under Microgravity Conditions

PRINCIPAL INVESTIGATOR: Prof. John J. Moore
Colorado School of Mines

CO-INVESTIGATORS:
No Co-I's Assigned to this Task

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

OBJECTIVE:
The objective of this research program is to get a better scientific understanding of the effect of gravity on the combustion synthesis of ceramic matrix-metal infiltrated composites.

DESCRIPTION:
Novel and innovative exothermic combustion synthesis reactions are conducted to generate porous ceramic matrices in the presence of 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 also studied under different levels of gravity.

PROGRESS DURING FY 1993:
The combustion synthesis reactions for five model systems were carried out on the ground with respect to the effect of gravity, i.e. vertically upward and downward propagating and horizontally propagating reactions. NASA-LeRC Learjet flight training is completed, and a safety report is prepared and a permit is obtained.

In order to further investigate the established effect of gravity on the properties of combustion synthesis produced composites they conducted selected experiments under both low and high gravity conditions using the Learjet. They flew ten successful missions (six trajectories each) for the TiC, ZrB₂, B₄C, systems at various levels of excess Al (and also Ti for TiC) including no excess.

They characterized the microstructure, morphology and chemical composition of the samples, other than the usual combustion velocity and temperature profile measurements, by using scanning electron microscopy (SEM) and X-ray diffraction (XRD). They observed whiskers in µg processed materials which were not observed in any of the earth gravity processed materials. Two of the cases processed under both low-g and 2-g (flown on the Learjet) showed the largest difference from the normal-g counterparts. One of these cases involved the generation of gases at the combustion front (i.e. B₂O₃, C + (4+x)Al = B₄C + Al₂O₃ + xAl system) and resulted in a larger expansion of the porous foaming ceramic matrix structure under low-g. The other case involved the creation of a liquid metal at the reaction front (i.e. liquid Al in the 3TiO₂ + 3C + (4+x)Al = 3TiC + 2Al₂O₃ + xAl system) and resulted in a much more uniform infiltration of the metal into the porous ceramic matrix forming a continuous metal network.

The TiC/Al₂O₃/Al system is also being processed by using a high temperature hot press to investigate if the same level of densification and uniformity of the metal network could be attained under normal-g conditions.

One of the two graduate students supported by this NRA has successfully defended his Masters Thesis on July 28th. A new graduate student will pursue the more in depth analysis of the B₄C/Al₂O₃/Al system.
II. MSAD Program Tasks — Ground-based Research  

Discipline: Combustion Science  

Students Funded Under Research:  
MS Students: 1  

Task Initiation: 8/91  Expiration: 7/95  

Project Identification: 674-22-05-38  

Responsible Center: LeRC  

Bibliographic Citations for FY 1993:  

Journals:  

Presentations:  


Combustion Research

Principal Investigator: Dr. Howard Ross

Co-Investigators:

No Co-I's Assigned to this Task

Task Objective, Description & Significance:

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.

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 SN-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.

Progress During FY 1993:

During FY93, Dr. D. Dietrich went to Hokkeido, Japan, to begin a 6 month residency as a visiting scientist at the Hokkeido 10-sec. drop tower. We completed final assembly of the hardware and shipped the 1g rig and the drop tower rig for his droplet arrays study; these international shipments required numerous memos (re technology transfer, commercial parts, custom parts, dollar value). Upon arriving, it was learned that the drop rig required several, minor mechanical modifications. His 1g rig was working well, after several electrical problems were eliminated; in addition new igniters were fabricated in rapid fashion by Lewis Research Center outside fab shop. These new igniters showed that the old system may vaporize too much of the droplet prior to ignition. Several rolls of 16 mm film were exposed to establish camera settings. There remained some impediments to the rigs being completely functional in the Japanese laboratory; small items like English to metric couplings, lower frequency electrical service, etc., tended to make life challenging for the researcher. After some startup problems, five of the final seven drop tests in the Hokkeido tower were performed successfully. All of the successful drops were of n-decane droplets with an initial diameter or about 3 mm supported on a 125 micron optical fiber burning in air at 1 atm. The last two drops (both successful) were demonstration drops for a major Japanese economic committee. In the same drop tests, candle experiments were performed successfully. The data from these tests has not yet been analyzed, though it can be reported that they did not burn to completion in the available microgravity time. Testing in normal gravity of single and two interacting drops was performed at atmospheric and subatmospheric pressure.

Dr. Dietrich returned to the U.S. in early August. Plans were made to continue the collaboration with the Japanese pending successful renewal via the NRA for the droplet arrays work.

During FY93 Dr. M. Vedha-Nayagam was involved in a number of in-house and external collaborative research efforts. Highlights of some those activities are summarized below.
II. MSAD Program Tasks — Ground-based Research  
Discipline: Combustion Science

1. Surface Tension Induced Flows:
Surface tension induced flows play a critical role in a number of areas, including weld pool geometry, flame spread over liquid fuels, fluid management devices in low gravity, and materials processing. A theoretical study was carried out to investigate the combined effects of buoyancy, rotation, and surface tension forces in a centrally heated liquid pool. It is shown that an exact solution to the Navier-Stokes equations is possible when the imposed heat flux varies quadratically with the radial co-ordinate. The results of this study were presented at the National Heat Transfer Conference (This work was done in collaboration with R. Balasubramaniam, NASA/LeRC). Work continues in this area to include a shear flow in the gas phase. This configuration models the flow conditions encountered during flame spread over liquid pools.

2. Soot studies:
Radiative emission from soot particles can constitute a large fraction of total heat release from hydrocarbon fuels. Because the soot particles act, through radiative emission, as a 'sink' through which sensible enthalpy is removed from the flame gases, the soot concentration field is obviously an important factor in the flame temperature field. We theoretically and numerically examined the coupled effects of radiation and thermophoresis on the concentration dynamics of emitting particles in a high-temperature gas. The combined effect of radiation and thermophoresis leads to a 'compression' of the particle field, in that the particles are transported into regions of increasing particle concentration. (This work was carried out in collaboration with Dan Mackowski, Auburn University).

Recently sooting characteristics of liquid hydrocarbon fuels were measured using fiber supported droplets in normal gravity in order to obtain global kinetic parameters for soot formation and oxidation processes. We plan to use this information in spherically symmetric droplet combustion with soot shell formation.

3. Rotating Disk Combustion
Flames embedded in the boundary layer generated by a rotating disk provides an excellent opportunity to investigate a number of fundamental combustion phenomena with experimental as well as theoretical ease. Earlier, in collaboration with Prof. Midkiff (University of Alabama, Tuscaloosa) we have developed a model for the boundary layer combustion of a rotating solid fuel disk. Experiments were conducted using PMMA fuel disks and shown to compare favorably with the theoretical predictions for the burning rate.

Recently, we have extended the previous analysis using activation energy asymptotics to predict extinction conditions.

A problem of unsteady behavior of opposed-jet diffusion flame also has been analyzed with Dr. U. Hegde. During FY93, studies were conducted by Dr. U. Hegde in primarily three areas:

1. G-jitter effects on combustion phenomena in microgravity
The effects of g-jitter on microgravity combustion phenomena has not received much attention to date. However, effects such as flame oscillations have been observed in combustion experiments conducted during low-gravity trajectories on aircraft. These effects can, potentially, interfere with interpretation of results. A presentation on g-jitter effects on diffusion flames was made at the International Workshop on G-jitter held in Potsdam, NY.

2. Heat release effects on shear-layer instabilities
The effects on unsteady heat addition on instabilities of parallel shear layers in the absence of gravity was analyzed. It was shown that, in the linear regime, the influence of heat addition is felt only through the time-averaged velocity and temperature fields, that is, the unsteady component of the heat addition plays no active role. The analysis was submitted and accepted for publication in AIAA Journal. The analysis has application to both premixed and diffusion flames.
3. Unsteady behavior of opposed-jet diffusion flames

In collaboration with Dr. M. Vedha-Nayagam, analysis of the oscillatory behavior of opposed-jet diffusion flames was initiated. This is an area where microgravity environment may be of use in studying the effects of turbulence on flames. The effects of velocity fluctuations in the oxidizer stream on flame location was investigated. It was found, for cases considered, that the velocity oscillations caused a time-averaged shift of the flame toward the fuel side. This shift increases the heat transfer from the flame to the fuel stream and could result in increased combustion rates in many situations, for example when the fuel stream is formed by a pyrolizing fuel sample. Consideration of non-linear second-order terms indicated that the flame shift could be understood in terms of an oscillation-enhanced oxidizer diffusivity. It is planned to submit the results to date for presentation and/or publication.

A research program was initiated by Dr. David Urban in FY93 to study the interactions between flames which are spreading or established over parallel solid surfaces. Preliminary low gravity testing has been conducted in the 2.2-second drop tower using a sealed, quiescent chamber, various oxygen/diluent combinations, and parallel fuel sheets of Kimwipe™ (selected as the fuel due to its very low mass/area) at several different separation distances. As expected, as the sheets are moved together from infinite separation, the flames interact and then merge. However, at a critical separation, which depends upon the oxidizer gas properties, the internal flame (between the sheets) becomes unstable and then extinguishes.

A proposal was written to the combustion NRA proposing that these tests be continued, and that another series of tests be conducted under opposed and concurrent flow conditions using an existing low-gravity rig a.k.a. the Spacecraft Fire Safety Facility (SFF) (designed by the Principal Investigator) on NASA 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, onflammability and spread and burning rates.

The overall goal of the research by D. Stocker is to determine the effect of buoyancy on the Burke-Schumann diffusion flame. The specific objectives are (1) to verify the zero-gravity Burke-Schumann analysis as well as the gravity-dependent Hegde-Bahadori extension of that analysis, and (2) investigate the influence of the buoyancy-dependent flow-field on the flame, particularly as a function of the coflow. A secondary goal is to investigate the effect of buoyancy on the flame-vortex interactions.

Microgravity tests are conducted in the 2.2-second Drop Tower and compared with normal-gravity tests. The experimental results are compared with analytical and numerical predictions to aid in analysis of the phenomena.

This work is a continuation of that conducted for Stocker's M.S. thesis on "The Effect of Reduced Gravity on the Laminar Burke-Schumann Diffusion Flame."

The significant accomplishments in FY93 include:
(a) A flow-visualization scheme was demonstrated in the existing Burke-Schumann burner in a cold air flow, although additional work is necessary. The flow was visualized by Mie scattering from preformed particles (added to the air flow) as they passed through a light sheet generated by a 5-mW laser diode. The goal of the work is to perform particle streak velocimetry during the drop tests, using a 35-mm still photography.
II. MSAD Program Tasks — Ground-based Research

Discipline: Combustion Science

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(b) Approximately fifty drop tests were conducted in the Drop Tower, prior to the facility shutdown. The tests were limited to the spring of 1993, since the Burke-Schumann rig was lent to L. Zhou in support of Bahadori's project. These tests were focused on non-luminous or weakly luminous flames, to allow for a more direct comparison of the experimental results with the theoretical predictions, which are typically do not account for soot. The sooting was inhibited by diluting the fuel with an inert; a 50/50 mixture of methane and nitrogen was used in most of the tests. A narrow-bandpass filter was also found to be successful in eliminating a weak flame-tip luminosity from the photographed image. In general, the flames appear to be in agreement with the theoretical predictions and previous results. However, in some cases the fuel dilution led to a lifting of the flame in normal gravity, so other fuels will be considered in future testing.

(c) A few exploratory tests were conducted where the fuel flow was pulsed at a low frequency, e.g., 10 hz, to allow for an investigation of the flame-vortex interactions. With appropriate flow control, the flame was found to oscillate strongly in height, sometimes with tip cutting. A subharmonic response in flame shape was often observed, where the response was a function of both the gravity level and fuel flow. Supporting modeling by L.-D. Chen (University of Iowa) and a student, J. C. Sheu, has shown a harmonic response to the flow pulsations, including the observed tip cutting, but have not yet predicted the subharmonic response. Based on this work, the three researchers will present their "Preliminary Observations of the Effect of Microgravity on a Pulsed Jet Diffusion Flame" at the fall meeting of the Western States Section of The Combustion Institute.

(d) L.-D. Chen (University of Iowa) and D. Stocker submitted a joint NRA proposal in September 1993 for support of this project, with Chen as the Principal Investigator and Stocker as the Co-Investigator.

(e) The drop rig was completely rewired in preparation for the planned tests. The power and DDAC (Droppable Data Acquisition and Control) systems were both replaced with upgraded versions.

(f) Two Worcester Polytech undergraduate students will be working full-time at Lewis Research Center, from October to December (at no cost to NASA), on the design of new Burke-Schumann burners. The burners will be designed and fabricated to allow for use of various diagnostics (e.g., thin filament pyrometry, absorption spectroscopy, particle seeding, schlieren, etc.) to allow for a through comparison with the theoretical predictions.

Buoyant Low Stretch Diffusion Flames
Co-Investigators (Numerical Work) Jennifer Rhattigan (LeRC) and Prof. James S. Tien (CWRU)

* The purpose of this work is to study the diffusion flame structure and extinction characteristics at very low stretch rate which is representative of a reduced gravity environment. This program uses an innovative approach to simulate a low gravity (spacecraft) diffusion flame in normal gravity through the application of scaling laws and similarity between buoyant and forced flow.

The flame configuration chosen is that of the forward stagnation-point region of a cylindrical fuel sample. A parallel experimental/numerical modeling effort has been underway for two years.

In modeling, the code was written in the first year, incorporating gas phase as well as solid phase radiation. During the second year, parametric studies were conducted to determine the importance of radiant heat transfer from both the solid and gas phases. The predictions demonstrate that the interaction of gas-phase radiation with surface properties can be important in the burning of condensed fuels. The radiative heat flux from the gas-phase to the solid is larger than expected. Absorption and reflection by the surface are both significant factors in the heat balance at the surface. The interaction between gas phase radiation and the surface absorption and emission is found to have a very strong effect on the flame at low stretch.
In the experiments, the lab and burner/cage assembly was developed and a safety permit issued for the lab during the first year, and during the second year experiments have been conducted which yielded the lowest burning rates obtained on earth to date. The trend with burning rate is consistent with previous buoyant stagnation data at smaller scales. Two radii of curvature have been tested to date: 20 cm and 50 cm. Temperature profiles for the two radii have been obtained, and are used to estimate the stretch rates for the two radii. Problems with solid phase response times, dripping material, and room exhaust were encountered, which hindered progress in the experiments. Flame structure probing is continuing into a third year.

Qualitative comparisons between experiment and theory show promise in that appropriate trends are predicted in both burning rate and temperature profiles. A third year funding request has been approved to continue the work.

* The objective of this subtask [Phase I, Ohio Aerospace Institute (OAI) Core Research Project] is to develop and verify chemical reaction models to predict Chemical Vapor Deposition (CVD) rates of carbon from methane under conditions relevant to the densification processing of carbon/carbon materials by Chemical Vapor Infiltration (CVI).

This is a one-year collaborative effort between Dr. Suleyman Gokoglu of NASA, BF Goodrich Aerospace Company, Akron University and OAI. The combined modeling and experimental approach involved: a) complete characterization of the flow and thermal fields of the BFG experimental reactor by using the computational fluid dynamics package FIDAP, b) modification of the BFG reactor and substrate geometry/configuration based on FIDAP modeling to obtain simpler flow and thermal fields for a better study of the chemical reaction mechanisms, c) running carbon deposition experiments using different flow rates, (i.e. concentrations and reactor residence times) of a methane source gas and various diluent gases (Ar, N₂, and H₂) under different temperatures (900-1200°C) and pressures (5-50 torrs), d) initially postulating and developing reduced chemical mechanisms (not to exceed three gas phase and surface reactions each) for predicting the experimental deposition rate trends using simpler (well-mixed) reactor models, and e) testing the developed reduced chemical mechanism by a full FIDAP model of the real reactor for further modification.

The modifications of the original BFG experimental reactor based on the flow and thermal field analysis done by FIDAP modeling are complete.

We extensively reviewed the literature for the unimolecular dissociation kinetics of methane and the relevant gas and surface reactions expected to be present for carbon deposition. Based on our findings, we postulated an initial reduced chemical mechanism and tested it first in a simpler well-mixed reactor model. The mechanism accounts for the differences in bath gases and low pressure fall-off effects. After a few iterations of mechanism modifications, the model showed the basic deposition rate trends observed in our initial experiments.

Then, we designed a set of experiments to provide a better test of our reduced mechanism with respect to relative and total gas flow rates, temperatures, pressures, and diluent gases. After we incorporated gas phase and surface kinetics into FIDAP, along with temperature dependent mixture transport properties, we tested the reduced mechanism with FIDAP. Though the initial results look encouraging, they require more experimental confirmations under various conditions.

Further experiments are currently hampered by oxygen leaks and micro-balance stability at low flow rates and pressures. However, there is no reason, so far, to suspect the validity of our earlier runs, which the modeling work has relied on.

Funds were also expended on miscellaneous equipment such as film development, personal computers, A/D and I/O boards, thermocouples, thermistors, transducers, mass flow controllers, video cameras, bottled gases regulators, translation stages, optical breadboards, etc, in support of several 1-g and drop-tower investigations.
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Additionally in-house support was provided to other visiting researchers (Premixed Gas Combustion by Princeton, High Pressure Drop. Combustion by Japanese/UCSD, Turbulent Premixed Gas Studies by R. Cheng/LBL; Flame Curvature Effects by C. K. Law/Princeton, etc.) and those on no-cost time extensions; this support consists typically of purchasing of bottled gases, regulators, portable PCs, fabrication services, etc. Finally, in-house support of the Combustion Science Discipline Working Group (DWG) and review committees will be provided.

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BIBLIOGRAPHIC CITATIONS FOR FY 1993:

**Journals:**


**Presentations:**


Combustion of Solid Fuel

**PRINCIPAL INVESTIGATOR:** Kurt R. Sacksteder

**NASA Lewis Research Center (LeRC)**

**CO-INVESTIGATORS:**

No Co-I's Assigned to this Task

**TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:**

**OBJECTIVE:**
The objective of this program is to obtain experimental experience and data to improve the understanding of the mechanisms that control the spreading of flames and the flammability of materials in low-speed oxidizing flows, including buoyantly-driven and forced concurrent flows and buoyantly-driven opposed flows.

**DESCRIPTION:**
New and unique experimental apparatus are to be developed to obtain flame spreading observations in partial gravity and in forced flows in microgravity. Partial gravity tests are to be conducted aboard NASA research aircraft flying parabolic trajectories, altered from the traditional condition of near free fall, to obtain the desired reduced levels of acceleration. Low-speed forced flows are to be obtained in microgravity drop towers using mechanical devices to establish purely-forced (non-buoyant) flows. In each instance, detailed measurements of flame ignition, spreading, and limiting behavior are to be made.

**SIGNIFICANCE:**
This work attempts to provide a fundamental understanding of the practical flame-spreading environment of low speed flows including: purely buoyant flows in partial gravity (between microgravity and normal gravity), and purely forced flows only possible in microgravity. In normal gravity buoyancy forces induce flows in spreading flames having velocities of no less than 20-30 cm/sec. Interactions between flames and lower speed flows, therefore, cannot be observed in normal gravity. In the low-speed flow regime, flow velocities approaching the spreading velocity of the flames can be examined. These flames are expected to demonstrate entirely new spread-rate limiting mechanisms, different than the normal-gravity counterpart. These tests are also expected to demonstrate lower flammability limits of solid fuels than any observed in normal gravity. Additionally, this work will attempt to distinguish between influences of buoyantly induced flows and flows of similar intensity that are externally imposed.

**PROGRESS DURING FY 1993:**

A second series of 2.2-second drop tower tests have been completed using a combustion tunnel modified for concurrent flow testing. Significant improvements in the system photography, for visualizing the growth of igniting flames and the progress of the pyrolysis front on the fuel surface, were introduced. Additional improvements were made in controls over the atmospheric content of oxidizer and diluent. An improved ignition scheme was implemented. Generally, while providing high-fidelity flame growth data, the results show that drop tower experiments may be too brief for flame development beyond the ignition transient, in contrast to the opposed flow case. Comparisons of the forced flow results with the theoretical calculations show good agreement at the point of flame stabilization, which equilibrates quickly. Downstream the observed flame tips are still growing and do not reflect the predicted steady shapes.

A complex automated testing apparatus has been developed for use in the NASA KC-135 aircraft. The apparatus includes a color schlieren system, for visualizing near-limit flames and to provide measurements of the density field
about the flame, and a duplicate of the SAMS accelerometer system for precise high frequency measurements of local accelerations to correlate with the flame spreading observations. Aircraft experiments have been conducted in which a complete flammability map was established for downward burning cellulosic fuel over a range of gravity levels from 0.02g to 0.6g. A significant increase in flammability compared to both the microgravity and normal gravity cases was demonstrated. A detailed comparison between flames spreading in purely buoyant and purely forced flows has been completed. An incidental discovery that materials will be more flammable in Lunar and Martian habitations than they are on Earth follows from these tests.

A matrix of upward burning tests in partial gravity has also been completed. In these tests, regions in which the flames are stabilized are formed at the bottom of the fuel. The downstream flame and plume become hydrodynamically unstable; vortex rollup and shedding are visible in the schlieren images. The limiting oxygen concentrations which supports upward spreading are always lower than in the downward spreading case, as has been observed in normal gravity.

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**STUDENTS FUNDED UNDER RESEARCH:**

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**TASK INITIATION:** 2/91  **EXPIRATION:** 12/94  **PROJECT IDENTIFICATION:** 674-22-05-28  **RESPONSIBLE CENTER:** LeRC

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**BIBLIOGRAPHIC CITATIONS FOR FY 1993:**

**Journals:**


**Presentations:**


Reduced Gravity with 2-Component Miscible Droplets

**Principal Investigator:** Prof. Benjamin Shaw
**University of California, Davis**

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

**Task Objective, Description & Significance:**

**Objective:**
The objective of this investigation is to study the combustion characteristics of miscible, binary droplets initially in the millimeter-size range, in a low-gravity environment.

**Description:**
Binary, miscible fuel droplets will be deployed in a low-gravity environment and ignited. High-speed films of both the droplet size as well as the flame position will be obtained. These high-speed films will be analyzed frame by frame using digital image analysis techniques to obtain droplet burning rates, and flame positions as a function of time. Any other phenomenon, such as microexplosion or soot particle transport, will also be recorded for analysis and interpretation. All the experiments will be performed using an experimental rig similar to the Droplet Combustion Experiment rig at NASA Lewis Research Center in the 2.2-second drop tower.

**Significance:**
The fuel mixture components are selected to provide significant variations in component volatilities (e.g., heptane and hexadecane). Specifically, experimental data on transient droplet diameters (including two-stage combustion and droplet disruption), transient flame behavior (including sudden flame contraction and extinction), and transport of soot particles will be obtained using ground-based low-gravity facilities.

**Progress During FY 1993:**
Over the past summer, 1-g experiments were performed using the drop rig constructed for reduced-gravity experiments in the NASA Lewis 2.2 sec drop tower. The focus of these experiments was to determine the extent and strength of droplet internal circulation induced by the droplet formation and deployment processes and subsequent ignition and combustion. Experiments consisted of forming, deploying and sometimes igniting droplets in a closed chamber that sealed off ambient air currents. Droplets were deployed onto a support fiber (~120 μm in diameter with a flared end of ~200 μm). Droplet environment conditions were air at 300 K and one atm. Droplets were typically composed of 90% n-heptane and 10% n-hexadecane by mass, and were initially about 1 mm in diameter. Internal flow patterns were visualized by seeding the droplets with aluminum oxide particles.

The results show that the internal circulation from formation of 1 mm droplets is quite intense, with characteristics internal liquid velocities measured to be as large as 30-40 mm/s. Droplet internal circulation from formation of 1mm n-heptane/n-hexadecane droplets in air at 1-g decays to negligible levels over time scales as long as 30-40 seconds. Droplet internal circulation is considered negligible when characteristics liquid circulation times, defined by the ratio of the droplet diameter to characteristic droplet internal velocity, are large relative to characteristic droplet-burning lifetimes. For 1 mm droplets burning in air, circulation velocities are negligible when they are significantly less than about 1 mm/s. The most important aspect of this work is the finding that internal circulation resulting from droplet formation has such a long decay times in 1-g.

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During this reporting period numerical calculations have been performed to predict the effects of thermocapillary forces on single-component droplet vaporization in a hot environment. Calculations have shown that surface-tension gradients can dramatically influence internal circulation behaviors.

**Presentations:**
Niazmand, H., Shaw, B.D., and Dwyer, H.A. Effects of marangoni convection on transient droplet evaporation in reduced gravity, Fall Meeting of the American Chemical Society, August 22-27, 1993.
Combustion of Solid Fuel in Very Low Speed Oxygen Streams

PRINCIPAL INVESTIGATOR: Prof. James S. T'ien  
Case Western Reserve University

CO-INVESTIGATORS:  
No Co-I's Assigned to this Task

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

OBJECTIVE:  
The objective of this program is to develop a theoretical model to improve the understanding of the mechanisms that control the spreading of flames over solid fuels and the flammability of materials in low-speed oxidizing flows, including buoyantly driven concurrent and opposed flows and forced-concurrent flows.

DESCRIPTION:  
A numerical simulation of the concurrent-flow flame spreading process is to be developed that accounts for both the stabilization of the flame at the leading edge facing the flow and the downstream tip of the flame where the flame spreading occurs. The required computational domain must therefore be large in comparison to the opposed-flow case. A steady solution will be formulated and solved first, followed by a transient version. The model is to accommodate first thin and then thick fuels.

SIGNIFICANCE:  
The work attempts to provide a fundamental understanding of the practical flame-spreading environment of low speed flows including: purely buoyant flows in partial gravity (between microgravity and normal gravity), and purely forced flows only possible in microgravity. In normal gravity buoyancy forces induce flows in spreading flames having velocities of no less than 20-30 cm/sec. Interactions between flames and lower speed flows, therefore, cannot be observed in normal gravity. In the low speed flow regime, flow velocities approaching the spreading velocity of the flames can be examined. These flames are expected to demonstrate entirely new spread-rate limiting mechanisms, different than the normal-gravity counterpart. These tests are also expected to demonstrate lower flammability limits of solid fuels than any observed in normal gravity. Additionally, this work will attempt to distinguish between influences of buoyantly induced flows and flow of similar intensity that are externally imposed.

PROGRESS DURING FY 1993:

The steady numerical model of concurrent-flow flame spreading has been completed and was exercised to study both strictly forced-flow cases and mixed-convection (combined forced and buoyant flows) cases. The model includes an elliptical formulation of the fluid mechanics model (the full steady Navier Stokes equations) which are required in order to accurately describe flame stabilization in the concurrent flow case and also in order to accurately predict flame behavior in buoyant flows.

The model has predicted the existence of a flammability boundary in low speed flows that includes two distinct limiting mechanisms: blowoff extinction at the comparatively larger relative velocities and quenching due to radiative losses in the lowest velocity range. These complex flammability boundary structure had not been anticipated for concurrent flows.
II. MSAD Program Tasks — Ground-based Research

Discipline: Combustion Science

In parallel efforts a transient capability and a gas-phase radiative loss mechanism are being added to the computation. These elements will be required for and will be followed by calculations of flame spreading over thick fuels in the same low-speed flows.

STUDENTS FUNDED UNDER RESEARCH:

PhD Students: 1 PhD Degrees: 1

TASK INITIATION: 2/91 EXPIRATION: 12/94

PROJECT IDENTIFICATION: 674-22-05-25

RESPONSIBLE CENTER: LeRC

BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:

Presentations:

II. MSAD Program Tasks — Ground-based Research

High Pressure Droplet Combustion Studies

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

CO-INVESTIGATORS:
M. Kono
University of Tokyo, Japan
T. Nioka
Tokoku University, Japan

Task Objective, Description & Significance:

Objective:
The focus of this international cooperative flight 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 California, San Diego, and the NASA Lewis Research Center. 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.

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.

Significance:
The general purpose is to study near-critical and super-critical combustion of the droplets and to see whether three-stage burning, observed at normal gravity, persists at high pressures in microgravity.

Progress During FY 1993:
The objective of this research is to study near-critical and super-critical combustion of droplets consisting of binary fuel mixtures. Experimental results were obtained on the burning of fiber-supported droplets of mixtures of n-heptane and n-hexadecane, initially about 1 mm in diameter, under free-fall microgravity conditions. The ambient pressures range up to 3.0 MPa, extending above the critical pressure of both fuels, in room-temperature nitrogen-oxygen atmospheres having oxygen mole fractions of 0.12 and 0.13. Three-stage burning of the binary fuel droplets is observed, and the onset time of the second stage is compared with the predictions of an existing theory. Experimental evidence of thermo-capillary and/or diffuso-capillary convection during the droplet burning is obtained. The results contribute to improving understanding of binary-fuel droplet-combustion processes at high pressures. This work was published in Combustion Science and Technology (1993, Vol. 90, pp. 111-123).

Recently, the modification of the apparatus to burn linear arrays of binary fuel droplets at high pressures was completed. Preliminary experiments of linear arrays of up to three droplets were started.

Task Initiation: 10/89
Expiration: 12/94
Project Identification: 674-22-05-41
Responsible Center: LeRC
II. MSAD Program Tasks — Ground-based Research

Discipline: Fluid Physics

Study of Two-Phase Flow and Heat Transfer in Reduced Gravities

Principal Investigator: Dr. Davood Abdollahian

S. Levy Incorporated

Co-Investigators:

No Co-I's Assigned to this Task

Task Objective, Description & Significance:

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.

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.

Progress During FY 1993:

Freon 114 was selected as the test fluid and the test section inner diameter was selected to be 6.0 mm. Design of the test section, flow loop, and electrical power, control and data acquisition systems has been initiated. Parts selection has also been initiated.

Students Funded Under Research:

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Task Initiation: 3/93  Expiration: 3/96

Project Identification: 674-24-05-50

Responsible Center: LeRC
Stability Limits and Dynamics of Nonaxisymmetric Liquid Bridges

PRINCIPAL INVESTIGATOR: Dr. Iwan D. Alexander
University of Alabama, Huntsville

CO-INVESTIGATORS:
Dr. J.M. Perales
Universidad Politecnica de Madrid
Dr. J. Meseguer
Universidad Politecnica de Madrid

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

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.

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.

SIGNIFICANCE:
Liquid bridge stability is an important factor in determining the stability of molten liquid zones associated with floating zone crystal growth experiments. Such understanding can aid design of in-space materials processing experiments using liquid bridge configurations.

PROGRESS DURING FY 1993:
The experiment apparatus has been designed, built and assembled at UAH. Simple experiments were performed with the translation tables and motors to test the control software and to use indicator gauges to verify the degree of motion and the responsiveness of them.

TASK INITIATION: 12/92
EXPIRATION: 12/95
PROJECT IDENTIFICATION: 674-24-05-60
RESPONSIBLE CENTER: LeRC
Electrokinetic Transport of Heterogeneous Particles in Suspensions

PRINCIPAL INVESTIGATOR: Prof. John L. Anderson Carnegie Mellon University

CO-INVESTIGATORS:

S. Garoff Carnegie Mellon University

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

OBJECTIVE:
This research addresses Earth-bound experiments designed to study the effects of surface heterogeneity on the electrophoretic motion of colloidal particles.

DESCRIPTION:
Theoretical modeling is also necessary to provide a framework for interpreting the results of these experiments and for suggesting new experimental strategies. The real payoff will come with experiments on supra-micron particles, because the single-particle kinematics, including rotational motion, can be followed by direct optical methods.

Research Approach — During the first year of this research, the focus will be on two aspects of the project: preparation of suspensions of "model" nonuniformly-charged colloids (clays, latex doublets) and experiments with the Mark II Microelectrophoresis Apparatus to measure effects of field strength on electrophoretic mobilities of these particles; and extend the hydrodynamic model for the motion of nonuniformly-charged spheroids to include the effect of thin, double-layer polarization \([kR] \geq 1, (kR)^3 \exp(\zeta^2/2 - 0(1))\). It is hoped to initiate work on an apparatus for determination of nonspherical particles to determine particle alignment. In the second year of this work, the development and testing of the light scattering technique associated with the nonspherical particle apparatus will be continued. In addition, models for the electrophoretic mobility of suspensions when the distribution of particle orientations is biased by hydrodynamic or electrical (ac) fields will be developed.

SIGNIFICANCE:
The development of optical tracking methods to follow the kinematics of supra-micron particles in electric fields is seen as a long-term goal. There are problems associated with sedimentation of large particles, especially given the high density of minerals. It is not possible to make the particles neutrally buoyant by adjusting the density of the aqueous solution because additives to increase the density would probably affect the surface chemistry of these particles.

PROGRESS DURING FY 1993:

Theoretical models have recently been developed to allow for a nonuniform \(\zeta\) on the surface of an axisymmetric particle retaining the equilibrium double layer assumption embodied in \((kR)^3 \exp(\zeta^2/2 - 0(1))\). The translational mobility is no longer isotropic; the mobility parallel to the axis of rotation differs from the perpendicular direction. The parallel and transverse mobilities depend on the even moments of the distribution of zeta potential along the axis of the particle. Unless it is aligned with the electric field, a particle will move in a direction different from that of the field. This prediction has yet to be verified experimentally, primarily because micron-size particles would be needed to simultaneously measure orientation and direction of motion; such particles settle too fast in the Earth's gravitational field to allow accurate measurements.

We are currently using techniques of image analysis to compare theory with experimental data, in the form of videotapes, for the rotation of colloidal doublets in electric fields. As the direction of the field is reversed, the more
positive end of the doublet rotates toward the field. By "grabbing" frames and measuring projection lengths and apparent solid angles versus time for a single doublet, we have obtained experimental results for the rotation coefficient. Comparison with theory indicates the theory is lacking. The next course is to take another video and repeat the image analysis.

**STUDENTS FUNDED UNDER RESEARCH:**

**PhD Students:** 1

**BIBLIOGRAPHIC CITATIONS FOR FY 1993:**

**Journals:**


II. MSAD Program Tasks — Ground-based Research

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: Dr. James C. Baygents
University of Arizona

CO-INVESTIGATORS:
H. Stone
Harvard University

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

OBJECTIVE:
The investigation consists of a comprehensive numerical and theoretical study of the motion, deformation, and interaction of pairs of viscous drops owing to an applied electric field. 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 physicochemical and transport process common to a variety of low-gravity flows. The "leaky" dielectric model and the Stokes equations will be employed to describe the constitutive behavior of the dielectric media. Solutions for the velocity and electric fields will be obtained by boundary integral methods.

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, physicochemical processes (e.g., emulsion breaking and drop coalescence) and bioseparations processes (e.g., aqueous two-phase partitioning).

Research Approach — 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 first part of the research will use the leaky dielectric for the two phases. The numerical calculations will use established integral equation methods for solving this class of time-dependent free boundary problems. With these detailed solutions will come an improved understanding of the response of multiphase systems to externally-applied electric fields.

SIGNIFICANCE:
The systematic study outline will lead to a quantitative description and understanding of electrically-driven, low-gravity fluid motions that are related directly to NASA's research interests and that have been mentioned frequently as being important for space-based materials processing.

PROGRESS DURING FY 1993:

Previous research on the deformation of drops by electric fields has focused, for the most part, on the behavior of isolated drops in uniform applied fields.

STUDENTS FUNDED UNDER RESEARCH:

PhD Students: 1

TASK INITIATION: 2/93  EXPIRATION: 2/96
PROJECT IDENTIFICATION: 674-24-08-13
NASA CONTRACT NO.: NAG8-948
RESPONSIBLE CENTER: MSFC

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BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:
II. MSAD Program Tasks — Ground-based Research  

**Discipline: Fluid Physics**

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**Marangoni Instability Induced Convection in Evaporating Liquid Droplets**

**Principal Investigator:** Dr. An-Ti Chai  
**NASA Lewis Research Center (LeRC)**

**Co-Investigators:**  
Prof. V.R. Arpaci  
**University of Michigan**

**Task Objective, Description & Significance:**

**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.

**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.

**Significance:**
In view of the continued interest of the fluids research community in the Marangoni instability phenomena and associated thermocapillary convections, and the high expectations people hold for space experiments, the investigators will undertake the challenge of investigating the phenomena in a near ideal environment.

**Progress During FY 1993:**
Most feasibility issues have been demonstrated experimentally. An analytical and numerical scheme has been applied to model the quasi-static thin shell configuration. Qualitative agreement has been obtained between this model and dimensional analyses.

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**Task Initiation:** 4/93  
**Expiration:** 4/96  
**Project Identification:** 674-24-05-65  
**Responsible Center:** LeRC
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, DESCRIPTION & SIGNIFICANCE:

OBJECTIVE:
The general objective of the program is to investigate the physics of rewetting of heated, grooved flat plates in a microgravity environment.

The specific objectives of the program are: to add to the fundamental understanding of the physics of the rewetting/dryout front, to better quantify the conditions under which rewetting occurs, and to obtain more accurate predictions of the rewetting velocity for a range of parameters.

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.

SIGNIFICANCE:
Understanding the dryout and rewetting characteristics of grooved surfaces is important in predicting the behavior of monogroove heat pipe designs proposed for Space Station Freedom.

PROGRESS DURING FY 1993:
Preliminary laboratory testing has been undertaken. Several aspects of the apparatus are being changed to improve the suitability for short-duration microgravity experiments:

1. The liquid delivery system is being redesigned to eliminate reliance on gravity to drive the flow.

2. The heater has been redesigned to reduced the response time. Both constant-flux and constant-temperature heaters will be used.

3. Video is being investigated as a means of tracking movement of the wetting front. Problems of condensation on the lens and the difficulty of determining the exact wetting front location need to be overcome.

Theoretical work has been started on predicting the wetting speed on finite, initially-isothermal plates subject to constant heat flux.
II. MSAD Program Tasks — Ground-based Research

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**II. MSAD Program Tasks — Ground-based Research**

**Discipline: Fluid Physics**

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**Marangoni and Double-Diffusive Convection in a Fluid Layer under Microgravity**

**PRINCIPAL INVESTIGATOR:** Prof. Chuan F. Chen  
University of Arizona

**CO-INVESTIGATORS:**

C. Chan  
University of Arizona

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**TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:**

**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.

**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 method 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.

**SIGNIFICANCE:**
The proposed work not only has practical implications for materials processing in space, but is a fundamental phenomenon in fluid mechanics that has not been fully explored.

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**PROGRESS DURING FY 1993:**

A low-frequency large-amplitude shaker has been designed and constructed. It is capable of performing sinusoidal oscillations from 1 to 5 Hz with an amplitude of 10cm. It is currently being tested to delineate its operational characteristics. A Bernard test cell using air up to 4 atm as the test fluid has been fabricated. Preliminary results show that sinusoidal oscillations at 1 Hz with an amplitude of 10cm have no effect on the onset of Bernard convection. This result agrees well with the predictions of the linear stability analysis. The stability characteristics of a binary fluid layer under the influence of surface tension have been studied.

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**BIBLIOGRAPHIC CITATIONS FOR FY 1993:**

**Journals:**
Chen, C.F., and Chen, C.C.  

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**TASK INITIATION:** 12/92  
**EXPIRATION:** 12/95  
**PROJECT IDENTIFICATION:** 674-24-05-51  
**RESPONSIBLE CENTER:** LeRC
II. MSAD Program Tasks — Ground-based Research

Transport Phenomena in Stratified Flow in the Presence and Absence of Gravity

PRINCIPAL INVESTIGATOR: Dr. Norman Chigier Carnegie Mellon University

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

OBJECTIVE:
The strategy of this study is:
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.

DESCRIPTION:
1. Conduct detailed measurements in turbulent mixing layer with velocity and density gradients;
2. Conduct a scooping 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.

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.

PROGRESS DURING FY 1993:
The design of the apparatus is in process. The concept of using divergent nozzles to achieve uniform velocity and temperature distributions at the nozzle exit is being examined. Boundary layers will form along the length of the splitter plate, separating the upper from the lower flows, with separate velocities and temperatures. The splitter plate introduces asymmetry into the flows and creates boundary layers that can influence the free shear layer. Also, heat transfer across the splitter plate will result in the formation of thermal boundary layers, and consequently non-uniform exit velocity and temperature profiles. An alternative approach, suggested by Dr. Nasser Rashidnia of LeRC, is to use no contraction and rely on honeycombs and screens to generate and manage turbulence. The honeycombs and screens could also be used for heating the flow to generate uniform temperatures in the upper and lower flows.
II. MSAD Program Tasks — Ground-based Research

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PROJECT IDENTIFICATION: 674-24-05-71

RESPONSIBLE CENTER: LeRC
II. MSAD Program Tasks — Ground-based Research

**Discipline: Fluid Physics**

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**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

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**Task Objective, Description & Significance:**

**Objective:**
The main objective is to study—in microgravity—the effects of external force fields on nucleation, two-phase bubble dynamics, and boiling transport.

**Description:**
In the experimental portion of the work, visualizations with laser sheet and high-speed photography and heat transfer measurements will be performed in a drop tower at Washington State University and on board a Learjet at NASA Lewis Research Center. The visualization photographs will be analyzed by a digital image analysis system. Also, boiling curves will be developed based on measurements of heat fluxes and surface superheats for various system conditions. Special attention will be focused on the critical heat flux and Leidenfrost point. Specifically, the effects of bulk convective motion will be determined. Quantitative assessment of the effects of impurities and surfactants on the boiling heat transfer rates will also be included. Analytical and numerical modeling will compliment the experiments. Perturbation and asymptotic techniques will be applied for low Reynolds number bubble dynamics, linearized bubble stability analysis, and small oscillation of bubbles in microgravity to account for small disturbances and g-jitter.

**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 or a velocity shear resulting from the relative fluid motion with respect to the surface;

**Progress During FY 1993:**
An old elevator shaft at Washington State University has been transformed into a 2.15-second drop tower. Seven successful drops were conducted in August 1993. This drop tower uses air bags to decelerate the test package. The drop tower is now fully operational.

Both the forced convection microgravity boiling experiment and the electrostatic microgravity boiling experiment have been built and tested in the laboratory. Gold film heaters have been successfully built and tested. To reduce the cost the substrate material is polycarbonate instead of quartz. The heaters use a 400 Angstrom gold surface with a power output of 7W/cm².
## II. MSAD Program Tasks — Ground-based Research

**Discipline:** Fluid Physics

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*PROJECT IDENTIFICATION:* 674-24-05-59  
*RESPONSIBLE CENTER:* LeRC
Reactive Fluids Experiment: Chemical Vapor Deposition

**Principal Investigator:** Dr. Ivan O. Clark
**NASA Langley Research Center (LaRC)**

**Co-Investigators:**
P.V. Hyer
E. J. Johnson

**Task Objective, Description & Significance:**

**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.

**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.

**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 1993:**
The numerical modeling of InP has been extended to three spatial dimensions 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 the 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, has begun. Numerical modeling of this reactor geometry has been expanded to include the effects of thermal gradients on the trajectories of tracer particles used for the LV measurements.
II. MSAD Program Tasks — Ground-based Research

Discipline: Fluid Physics

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PROJECT IDENTIFICATION: 674-24-06-03
RESPONSIBLE CENTER: LaRC

BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Proceedings:

Presentations:


Microgravity Particle Dynamics

Principal Investigator: Dr. Ivan O. Clark

Co-Investigators:

E.J. Johnson
J.F. Meyers
S.O. Kjelgaard

Task Objective, Description & Significance:

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 proposed research also seeks to identify the critical parameters and instrumentation requirements for subsequent microgravity experiments.

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.

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 1993:

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 is being implemented.
II. MSAD Program Tasks — Ground-based Research  

Discipline: Fluid Physics

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.

TASK INITIATION: 11/92  EXPIRATION: 11/95  
PROJECT IDENTIFICATION: 674-24-06-04  
RESPONSIBLE CENTER: LaRC

II-268
Global Instability and Pattern Formation in Binary Alloy Solidification

Principal Investigator: Prof. Julian D. Cole
Rensselaer Polytechnic Institute

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

Task Objective, Description & Significance:

Objective:
Confirmation of a profound instability mechanism, discovered over the past few years, for free dendrite growth systems in terms of the matched asymptotic expansion methods. This instability mechanism is driven by the interaction of interfacial traveling waves near a special simple turning point in the complex plane and the tip of dendrite. It is now called the "global trapped wave" (GTW) mechanism.

Description:
In order to confirm this entirely new mechanism, this work will investigate a simple model equation of 2-D dendrite growth and use a numerical approach to resolve it. Numerical results fully confirm and verify the global instability mechanism, and are in good agreement with the results obtained by using asymptotic methods.

Progress During FY 1993:
This work investigates the global instability mechanisms in binary alloy solidification in terms of a simpler approach. The results obtained in the present project confirm the interfacial wave theory previously developed for dendritic solidification from a pure substance.

Bibliographic Citations for FY 1993:

Journals:
Fluid Interface Behavior Under Low- and Reduced-Gravity Conditions

PRINCIPAL INVESTIGATOR: Prof. Paul Concus
University of California, Berkeley

CO-INVESTIGATORS:
R. Finn
Stanford University

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

OBJECTIVE:
The objective of this study is to develop a more complete understanding of the physical behavior of fluids in partially filled containers where surface forces are dominant.

DESCRIPTION:
The approach for this research task is to pursue parallel theoretical, computational, and experiment studies to examine the fluid surface shape for a variety of vessel/fluid combinations when capillary forces dominate. The theoretical aspects include both formal mathematical analysis and computational studies of individual configurations of special interest. Two fluid-container configurations will be examined: (1) marble wedge and (2) proboscides vessels. Consultation will be provided for experimentation to be conducted at the Lewis Research Center. These experiments will provide verification for the theoretical findings and possibly uncover unforeseen phenomena.

SIGNIFICANCE:
Anticipated Results:
1. Develop a complete understanding of surface phenomena of liquids in containers in a low-gravity environment. In particular, to obtain new information on the significance of contact angles as an intrinsic physical property;
2. To possibly develop new methods of measuring contact angles;
3. Provide new insight to managing fluids in space.

PROGRESS DURING FY 1993:
Efforts in FY93 centered on our mathematical studies of capillary surfaces and on related ground-based and space experiments.

In our study of discontinuous and "nearly-discontinuous" behavior of capillary surfaces, a class of optimally shaped containers has been discovered. These containers are cylinders with sections related to the two-circle sections studied earlier that have the shape of a larger circle with a portion of a smaller circle protruding from it. The containers exhibit a nearly discontinuous transition from existence to non-existence of solution surfaces, in which small changes in geometry or contact angle can result in large movement of the fluid bulk that partly fills the container, with arbitrarily large rise height over a portion of the base. The optimal shapes for the protrusions (proboscides) that have been found have the remarkable property that they can be constructed so that the transition from existence to non-existence at any prescribed contact angle occurs with the unbounded rise height over domains that can be made relatively as large as desired. From a formal mathematical point of view, the proboscides admit entire continua of a particular kind of minimizing externals (and no other minimizing externals) for a subsidiary variational problem, and these appear in a discontinuous way at the prescribed contact angle. The proof of these
assertions was completed (jointly with a student, T. Leise), and the detailed behavior of solution surfaces has been investigated (jointly with a student, F. Zabihi). Along with a movable wedge container, the proboscis containers will form part of a planned experiment accepted for the USML-2 Glovebox. These shapes have possible application to exploiting microgravity for the accurate determination of contact angle. Under the auspices of ESA, a MAXUS sounding rocket experiment is planned for next year, as a preliminary to the USML-2 experiment.

Investigation continued of the problem of the free surface of a liquid partly filling a wedge container in the absence of gravity, when the contact angles on the two sides of the wedge are allowed to have different (constant) values. Our earlier results for the equal-angle case led to a condition that for a wedge section of opening angle $2 \alpha < \pi$ if a solution surface $u(x,y)$ making constant angle $\gamma (0 \leq \gamma \leq \pi/2)$ within the vertical walls of the wedge exists, then $\alpha + \gamma \geq \pi/2$. Under some natural restrictions, the condition is also sufficient. If this condition fails, then no solution can exist. If differing contact angles are prescribed on the two walls, then the same method of proof for the single angle case yields the necessary condition $\cos \gamma_1 + \cos \gamma_2 \leq 2 \sin \alpha$. One can show by example that this condition no longer suffices for existence of a surface, however. Thus, the problem must be considered on another level. We have now largely characterized the kinds of behavior that can occur, some of which is strikingly different from what is possible for the single angle case. In general terms, the new behavior that can occur, over particular ranges of contact angle pairs, is one for which the surface normal is necessarily discontinuous at the wedge vertex $V$. Numerical evidence suggests a wide range of varying behavior is possible for these ranges, depending on the local boundary curvatures on either side of $V$. In one explicit example, the existence of a solution with an infinite jump discontinuity at the vertex is demonstrated. Under other conditions bounded solutions can exist; further, we can show that whenever a bounded solution exists then every solution is bounded. The precise characterization of all admissible behavior patterns is a principal question that is currently be addressed. The above results suggest new possibilities for experimental determination of contact angle and should lead to development of related experiments in microgravity.

As part of the design of containers for our USML-1 experiment, the stability of liquid bridges between parallel plates was studied, for the case of equal contact angles on the two plates. In joint work with T. I. Vogel it was proved that for any contact angle the greatest lower bound on fluid volumes at which such a bridge can be stable always exceeds the value that occurs for a contact angle of 90 degrees. Since in this latter case the critical volume is known explicitly, a working estimate is obtained which is valid for any contact angle. In this way containers can be designed so as to exclude the occurrence of (unwanted) liquid bridges between top and bottom planar surfaces. In work with a student, L.-M. Zhou, the case in which the contact angles on the top and bottom are permitted to differ was studied. The method for the equal-angle case then fails, but she obtained again the same result using different techniques. She also obtained lower bound estimates for the greatest lower bound on fluid volume in terms of the contact angles. In current work she is obtaining results on general stability criteria for liquid bridge configurations, some of which demonstrate that the stability set in a nonsingular parametrization of a solution family can be disconnected. Possible related microgravity experiments suggest themselves.

Analysis of the data from our USML-1 glovebox experiment continues. This experiment concerned axially symmetric "exotic" containers. The containers, which are in the shape of a right circular cylinder with mathematically derived toroidal-like bulge, have the property that they admit in zero gravity an entire continuum of axially symmetric equilibrium interfaces for the prescribed fluid volume and contact angle. However, it was proved that the entire container can admit no symmetric stable interface; thus the energy minimizing interface (which is known to exist) must necessarily be asymmetric. This behavior was verified in the USML-1 experiment; as the fluid enters the bulge in the container, it appears at first as a spherical cap. But in response to a disturbance it immediately adopts an asymmetric shape. Most of the video tapes of our experiment taken on board USML-1 have now been received, and final analysis of the data is underway.
II. MSAD Program Tasks — Ground-based Research
Discipline: Fluid Physics

STUDENTS FUNDED UNDER RESEARCH:
MS Students: 2

TASK INITIATION: 3/90 EXPIRATION: 3/95
PROJECT IDENTIFICATION: 674-24-05-28
RESPONSIBLE CENTER: LeRC

BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:

Proceedings:


Convection and Morphological Stability During Directional Solidification

**PRINCIPAL INVESTIGATOR:** Prof. Sam R. Coriell  
National Institute of Standards and Technology (NIST)

**CO-INVESTIGATORS:**  
G.B. McFadden  
National Institute of Standards and Technology (NIST)  
B.T. Murray  
National Institute of Standards and Technology (NIST)  
J.R. Manning  
National Institute of Standards and Technology (NIST)

**TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:**

**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 that study the evolution of morphological patterns without the interference of fluid flow may be possible. This research will be carried out by a consortium of NIST, US industry, and NASA. The goal is to develop and transfer to all of the consortium members technology that will provide for the rapid design and prototyping of new precision cast parts, enhance product quality, and reduce reject rates.

**DESCRIPTION:**  
The goal will be accomplished by carrying out four integrated tasks dealing with: (1) macro/micro process modeling, (2) sensors for process modeling and on-line process control, (3) thermophysical properties data for three model systems (Ni-based superalloy, Ti alloy, and Al alloy), and (4) model validation and systems calibration/integration.

This ground-based research will also provide theoretical interpretation and guidance for a series of space experiments to be carried out by J.J. Favier and colleagues on tin-bismuth alloys using the MEPHISTO apparatus and by K. Leonartz and colleagues on succinonitrile-acetone alloys.

**SIGNIFICANCE:**  
This research will help improve the processing by fostering and utilizing intelligent processing concepts.

**PROGRESS DURING FY 1993:**

We have considered the directional solidification of a binary alloy during the initial transient period in which the interface velocity, concentration, and temperature gradients are changing with time. We introduce sinusoidal perturbations of the planar crystal-melt interface and numerically calculate the time evolution of these perturbations. The results for morphological instability are in good agreement with the Mullins and Sekerka analysis of the time-independent base state when the instantaneous values of the temperature gradient and solidification velocity are used in the analysis; the agreement improves with decreasing degree in instability. Calculations have been carried out for a tin (0.5% bismuth) alloy, which was used in the MEPHISTO apparatus during the USMP-1 mission.

In collaboration with A. A. Chernov of the Russian Academy of Sciences, the role of anisotropic interface attachment kinetics on morphological stability during solution and melt growth is being treated. This is important for bismuth alloys which will be studied during the USMP-2 mission.

For growth from a supersaturated solution, stability with respect to step bunching of a step train forming a vicinal face is considered taking account of both capillarity and anisotropy of interface kinetics. It is found that the step
motion with respect to a stagnant solution provides stabilization at sufficiently large wavelengths for which the typical diffusion rate is comparable to the step rate. Since capillarity can stabilize the interface against short wavelength perturbations, the combined action of both kinetic anisotropy and capillarity provides complete stability at sufficiently high growth rates.

For growth of a binary alloy at constant velocity, the effect of anisotropic interface kinetics on morphological stability is being calculated. The dependence of the interface kinetic coefficient on crystallographic orientation is based on step motion near a singular crystallographic orientation. Anisotropic kinetics give rise to traveling waves along the crystal-melt interface, and can lead to a significant enhancement of morphological stability. This enhancement increases as the orientation approaches a singular orientation and as the solidification velocity increases. Specific calculations are being carried out for germanium-silicon alloys.

BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:


Proceedings:


II. MSAD Program Tasks — Ground-based Research

Drop Microphysics

PRINCIPAL INVESTIGATOR: Prof. Robert H. Davis

University of Colorado

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

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.

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.

SIGNIFICANCE:
Drop interactions and coalescence play key roles in a variety of phenomena, including liquid-liquid extraction, raindrop growth, multiphase flow, and processing of bimetallic melts within the liquid-phase miscibility gap.

PROGRESS DURING FY 1993:

It has been shown that a spherical drop will come into contact with another spherical drop (or a flat interface) in finite time under a constant driving force, such as gravity. This is in contrast to rigid spheres—they approach each other only asymptotically. However when drop deformations are taken into account, the behavior in the limit is like that of rigid spheres and an attractive force is required to bring the drop interfaces into contact in a limited time.

The evolution of the drop shape has been tracked from an undeformed state until a dimple is formed and long time quasi-steady-state pattern is established. A dimple is always formed and for long times, the film thickness is proportional to inverse powers of time. When Van der Waals forces dominate over gravity, the film ruptures at the nose of the drop prior to the formation of a dimple.
The thermocapillary motion of drops nearly in contact has been studied when two drops are non-conducting or when a non-conducting drop approaches a contact temperature surface or an interface. These results have been incorporated in trajectory analyses of thermocapillary-induced collision rates. When the drops are highly conducting, the relative velocity reverses sign and coalescence is impossible.

Experiments have been performed on the near-contact interaction of two drops due to gravity. It has been observed that (i) deformation retards the drops' motion, more so in the case of approaching drops than receding drops and (ii) small drops deform less and coalesce more easily.

**STUDENTS FUNDED UNDER RESEARCH:**

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**TASK INITIATION:** 5/91  **EXPIRATION:** 5/94

**PROJECT IDENTIFICATION:** 674-24-05-35

**RESPONSIBLE CENTER:** LeRC

**BIBLIOGRAPHIC CITATIONS FOR FY 1993:**

**Journals:**

Phase Segregation Due to Simultaneous Migration and Coalescence

Principal Investigator: Prof. Robert H. Davis
University of Colorado

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

Task Objective, Description & Significance:
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.

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.

Progress During FY 1993:
The population dynamics equations for homogeneous dispersions have been solved for droplet growth due to the separate effects of Brownian, gravitational and thermocapillary motion and coalescence, and due to the combined effects of thermocapillary and gravitational motion and coalescence.

A computer code has been developed for nonhomogeneous dispersions undergoing simultaneous phase separation and motion and coalescence due to gravity. The rate of phase separation initially increases due to coalescence and then decreases due to the larger drops moving out of the suspension.

Experiments to observe drop coalescence and phase segregation due to gravity have been initiated.

Students Funded Under Research: PhD Students: 1

Task Initiation: 12/92 Expiration: 11/95

Project Identification: 674-24-05-39

Responsible Center: LeRC

Bibliographic Citations for FY 1993:

Journals:


Interaction and Aggregation of Colloidal Biological Particles and Droplets in Electrically-Driven Flows

PRINCIPAL INVESTIGATOR: Prof. Robert H. Davis
University of Colorado

CO-INVESTIGATORS:
P. Todd
M. Loewenberg
University of Colorado, Boulder

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

OBJECTIVE:
The aim of this research is to develop a fundamental understanding of aggregation and coalescence processes during electrically-driven migration of particles (or cells) and droplets.

DESCRIPTION:
This research will be accomplished through 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 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

• Experiments conducted to observe electrically-driven aggregation and coalescence by conducting terrestrial experiments to test the theoretical description of electrically-driven particle aggregation, performing laboratory observations of electrically-driven droplet coalescence, and designing a flight-based experiment to gather quantitative observations for testing a theoretical description of electrically-driven droplet coalescence.

Research Approach — The research to be carried out during the first year will be to develop the new detection and analysis techniques and to test their effectiveness using a range of model solutes under conditions which can be studied effectively in a normal gravity environment. The two subsequent years will be devoted to developing an important data base on interfacial transport characteristics and to adapting the entire system to be compatible with whatever constraints are imposed by NASA on the experimental rig to be sent on a space shuttle mission. Concurrent with these developments, the theoretical modeling will be pursued. The research study consists of experimental and (a limited amount of) theoretical work necessary to prepare a Science Requirements Document (SRD) for microgravity experiments. It appears that the need for a microgravity environment can be justified. Nevertheless, the possibility that some of the work can be accomplished in drop towers will be investigated, which may be feasible using systems with short relaxation times. For this purpose, less viscous fluids will be essential. The study will focus on:

• The use of ionic surfactants to control the conductivity of low dielectric constant liquids. This will provide a means to explore the role of fluid conductivity in the deformation of drops and the stability of fluid cylinders.

• The stability of fluid cylinders in longitudinal fields with isopycnic systems. This will furnish experience with this geometry and provide some information to test the leaky dielectric model for the stability of fluid columns. To the extent possible, ionic surfactants to control fluid conductivity will be used.

• The deformation of droplets using fluids whose conductivity has been adjusted using a non-ionic surfactant. Nucleation and chiral symmetry breaking of sodium chlorate under different flows will be studied experimentally.
In the first year, the research will concentrate on measurements of chiral symmetry breaking as a function of nucleation rate. The central issue here is whether the symmetry breaking transition a continuous or an abrupt one. By improving statistics of our measurements, we hope to answer this question quantitatively. In the second year, experiments will be performed in Couette cells, and chiral symmetry breaking will be measured in different flow regimes, laminar, Taylor vortex flow and turbulence, using these cells. The experiment is not perfect at this stage because the nucleation process is still influenced by sedimentation effect. However, one should be able to observe qualitatively different features of nucleation and chiral symmetry breaking associated with various flow regimes. On the third year, the experimental apparatus, consisting of ten identical Couette cells and a temperature-controlled bath, should be ready for the space mission. With gravitational effect greatly reduced, we hope to demonstrate in the clearest way that chiral symmetry breaking is a result of competition between nucleation and convection. This research focuses on the electrically-driven aggregation of particles and coalescence of droplets. The coagulation of biological or other solid particles migrating in an electric field will be investigated first; a subsequent study of droplet coalescence during electrophoresis will be conducted. In each case, this involves the development of a detailed mathematical description of two-body interactions, especially for near-contact. These descriptions must account for hydrodynamic interactions arising from the relative motion of the particles or drops in the solvent, electrokinetic interactions that arise from the convection of fluid near a charged particle surface (or drop interface), and any significant, non-hydrodynamic interactions such as van der Waals attraction and electrostatic double-layer repulsion. Ultimately, collision rates are sought that provide the link between micro- and macro-physics of electrically-driven aggregation. The former is concerned primarily with two-body interactions, while the latter includes particle/drop size distributions and overall, macroscopic phase-separation behavior. Ground-based experiments will be conducted to make observations of electrically-driven particle aggregation and droplet coalescence for comparison with the theory to be developed. It is anticipated that gravitational effects encountered in terrestrial experiments will obscure a quantitative comparison with theory for droplet coalescence.

SIGNIFICANCE:
The fundamental study of particle aggregation in electric fields is expected to have practical application to electrically-controlled cell floculation 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 bi-phasic 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 1993:
During the first six months of support, we have made considerable progress on theory and experiments aimed at understanding electrically-driven aggregation of particles. Calculations to determine the electrically-driven, near-contact motion of two particles are underway. Electrically-driven, relative motion between two spherical particles depends only on their size ratio and relative position (center-to-center separation and orientation with respect to the applied electric field); it is proportional to their $\zeta$-potential difference. Near-contact particle motion is important because it has a large effect on electrically-driven, pairwise collision rates, and because it reveals the essential physics of the electrically-driven aggregation process. The results that we have obtained have a particularly simple, and therefore useful, form: the relative, near-contact velocity depends only on size ratio; it is proportional to the $\zeta$-potential difference and the gap width separating the particle surfaces. A closed-form, analytical solution has been obtained for equisized particles and for the opposite, disparate-sized extreme. Between these complementary regimes, a numerically determined function of size ratio is needed, but an algebraic form may accurately approximate the numerical calculations. The results obtained thus far show that electrically-driven aggregation is much more efficient than its gravity-driven analog.
Calculations for determining electrically-driven, pairwise aggregation rates are in progress; the foregoing results for near-contact, particle motion will be incorporated. In the absence of nonhydrodynamic forces, pairwise, electrically-driven, collision efficiencies depend only on the size ratio of the particles, where the collision efficiency is the collision rate normalized by the hypothetical rate obtained for noninteracting particles that move rectilinearly through a suspension. However, London-van der Waals and electrostatic forces will generally become important as the distance between two, electrically-charged, colloidal particles becomes small compared to their size; in fact, collision rates would vanish without the help of van der Waals attraction because of the viscous, lubrication resistance between suspended particles. Five additional (dimensionless) parameters are thereby introduced: two that describe the strength of van der Waals and electrostatic interparticle forces (at a given interparticle separation) compared to the electrical force that propels a particle through the suspension against its viscous resistance; and three minor parameters that characterize these nonhydrodynamic forces. The minor parameters include the ratios of the particle size to the electric double layer thickness and to the London wavelength, and the ζ-potential ratio of the particles. For non-Brownian systems, the electric double layer thickness (≤ 10 nm) and London wavelength (~ 0.1 μm) are usually small compared to the particle size (~ 1 μm); however, the ζ-potential ratio may vary corresponding to a variety of heterogeneous systems. Since the London wavelength is small compared to the particle size, the retarded van der Waals potential must be incorporated in the calculations. If the double layer is sufficiently thin, van der Waals forces will dominate electrostatic repulsion at all separations. If so, we obtain the surprising result that electrically-driven collision efficiencies depend only on the size ratio and the (retarded) van der Waals forces; they are independent of ζ-potentials. The conditions under which this conjecture holds will be investigated. Generally, the focus is on conditions relevant to those which we can achieve experimentally.

We have obtained and installed a microelectrophoresis device that has 800x magnification capability and built the requisite microelectrophoresis chamber. We possess a computer program that interprets raw migration velocity data, corrects for the electrosmotic background velocity, and yields electrophoretic mobilities. Fixed red blood cells from humans, dogs, and rabbits form approximately spherical particles with electrophoretic mobilities in the ratio ~ 1 : 2 : 3; initially, these will be used in particle aggregation experiments.

We have purchased the image processing equipment described in our proposal, including a 486 personal computer, a basic image-processing software package, and a high-quality frame grabber capable of capturing paused images; the equipment has been installed and is currently being calibrated. Thus, we shall soon be capable of making quantitative comparison between theoretical predictions and observations of electrically-driven, two-particle interactions and collisions.

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**STUDENTS FUNDED UNDER RESEARCH:**

PhD Students: 1

**TASK INITIATION:** 3/93  **EXPIRATION:** 3/96

**PROJECT IDENTIFICATION:** 674-24-08-10

**NASA CONTRACT NO.:** NAG8-945

**RESPONSIBLE CENTER:** MSFC

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**BIBLIOGRAPHIC CITATIONS FOR FY 1993:**

**Journals:**

II. MSAD Program Tasks — Ground-based Research

Theory of Solidification

**Principal Investigator:** Prof. Stephen H. Davis
Northwestern University

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

**Task Objective, Description & Significance:**

**Objective:**
This work concerns the 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.

**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.

**Progress During FY 1993:**
Progress has been made in many areas of solidification including the coupling of a freezing front with Soret convection, linear and nonlinear stability theories of rapid solidification in which thermodynamic non-equilibrium effects at the interface are allowed, the effect of elastic stresses in morphological instability, the dynamics of trijunctions during phase transition, and the emergence of islands of instability in epitaxial growth. A survey article on the interaction of flow and morphology has appeared in the Handbook of Crystal Growth. Davis was general chairman of a NATO Advanced Research Workshop "Interactive Dynamics of Convection and Solidification."

**Students Funded Under Research:**

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**Task Initiation:** 10/87  **Expiration:** 12/93

**Project Identification:** 674-25-05-16

**Responsible Center:** LeRC

**Bibliographic Citations for FY 1993:**

**Journals:**


II. MSAD Program Tasks — Ground-based Research

**Discipline: Fluid Physics**


**Presentations:**


Davis, S.H. Rapid solidification of a binary alloy, University of Akron, Department of Mathematical Sciences, 1992.

Davis, S.H. Microscale coupling of solidification and flow, NATO Advanced Research Workshop, Chamonix (Plenary Lecture) Interactive Dynamics of Convection and Solidification.

Davis, S.H. Strongly-nonlinear oscillations in rapid directional solidification, Department of Mathematics, University of Utah, 1992.


Davis, S.H. Rapid solidification, Department of Chemical Engineering, University of Florida.

Davis, S.H. Rapid solidification of binary mixtures, Physics Department, University of Texas, Austin, 1993.


Studies of Two-Phase Flow Under Microgravity

**Principal Investigator:** Prof. A. E. Dukler, University of Houston

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

**Task Objective, Description & Significance:**

**Objective:**
The objective of this study is to develop and experimentally verify theoretical models that predict two-phase flow regimes and characteristics in reduced gravity.

**Description:**
The approach is to conduct reduced gravity experiments on the Learjet and in the 2.2-second drop tower and concurrently to develop models that predict the hydrodynamic characteristics of the gas-liquid flow as a function of the system operating conditions.

**Significance:**
The significance of the results will have application in space power, fluid and thermal management, and propulsion systems. Furthermore, by developing an understanding of the effect of gravity on flow regime characteristics and flow directions, it will be possible to have applications in the petroleum and nuclear industries.

**Progress During FY 1993:**
As a result of 39 low-gravity aircraft flights, the flow-regime maps were significantly expanded, especially for the water-glycerine, and water-surfactant liquid solutions. These flights recorded data using the new conductivity probe electronics, new differential pressure transducers, and in a SAMS accelerometer. As a result of these tests, the transition criteria for the flow regimes are being examined. The Learjet rig was rewired to accommodate a new data acquisition and control system. Hardware has been procured and fabricated to facilitate using the rig for 1.0 in.- (2.54 cm.) inner-diameter test sections.

**Students Funded Under Research:**

PhD Students: 1

**Task Initiation: 12/91**  **Expiration: 11/92**

**Project Identification:** 674-24-05-18

**Responsible Center:** LeRC
Microgravity foam Structure and Rheology

PRINCIPAL INVESTIGATOR: Prof. Douglas J. Durian
University of California, Los Angeles

CO-INVESTIGATORS:
Dr. D.A. Weitz
University of California, Los Angeles

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

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.

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.

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 1993:
A new optics system has been installed to measure the angular dependence of multiply scattered transmitted light. Instead of imaging a small portion of the surface onto a detector, we now place the sample at the focal point of a large-diameter lens to collect all light emerging from the sample within a small solid angle. This significantly reduces the systematic errors in our measurements. Other systematic errors have been analyzed and, where possible, reduced. We now are very close to taking reliable data from which the extrapolation length ratio may be deduced.

Analysis has begun of DWS data on the short-time dynamics in a commercial shaving foam for which the long-time dynamics are known. He is finding that the liquid-vapor interfacial mean-squared displacement has been found to grow proportionally to time raised to the one third power, such that it would take many days for the motion to be comparable to the wavelength of light. This may be the first observation of the accumulation of local strain in a coarsening foam which leads to the sudden topological changes seen in the long-time dynamics. Preliminary measurements on home-made foams show similar behavior.
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Task Initiation: 12/92  Expiration: 12/95
Project Identification: 674-24-05-73
Responsible Center: LeRC
Theoretical Influence of Microgravity on Critical Fluid Measurements

PRINCIPAL INVESTIGATOR: Prof. Richard A. Ferrell

Unviersity of Maryland

CO-INVESTIGATORS:

No Co-I's Assigned to this Task

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

OBJECTIVE:
This endeavor is tailored to provide theoretical support for these current critical fluid microgravity Space Shuttle experiments: the Fluid Light Scattering Experiment (CFLS), the LeRC Critical Fluid Thermal Equilibration Experiment (CFTE), and several other microgravity experiments now in the definition phase.

DESCRIPTION:
The science activity will look at six areas; three each for classical fluids and for super-fluid Helium. For classical fluids, they are: short and long time scale equilibration driving forces, the anomalous dimension critical exponent of the density fluctuation correlation length, and shear viscosity near the liquid-vapor critical point. For super-fluids, they are: a better prediction of the thermal conductivity temperature scaling, the frequency dependence of the shear viscosity, and theoretical insight into the unexpected temperature dependence of the second-sound velocity of $^4$He near its Lambda transition. This will employ a graduate student, a part-time post-doctoral fellow, and a visiting faculty fellow to assist in this work.

SIGNIFICANCE:
The results of the proposed study will both aid the interpretation of the data and demonstrate the need for the data from the microgravity experiments to confirm science conclusions. The conclusions will greatly improve the cost effectiveness of the science from identified flight experiments.

PROGRESS DURING FY 1993:
The computation of the effect of fluctuating inertial forces indicates that the level of g-jitter generally observed in the space shuttle is not likely to change the rate of relaxation of the density fluctuations, the measurement of which is the goal of the Zeno experiment.

We have found that the adiabatic equilibration of a fluid in a closed container is strongly affected by a "thermal bottleneck" resulting from the finite thermal conductivity of the container walls. We expect to be able to put our theoretical findings to an experimental test in Adiabatic Fast Equilibration (AFEQ).

A hitherto unnoticed special feature of the mode coupling theory of critical conductivity in a fluid is the singular behavior of the crossover function.

A study has been carried out of the possibility of measuring the critical viscosity in a very thin layer of fluid, so as to avoid the disturbing effect of gravity. We propose the application of electrostriction to portion the layer. This increases the fluid density locally. Upon removal of the electric field the fluid density will relax back to its normal value at a rate determined by the viscosity.

A two-term epsilon expansion calculation has been carried out for the critical viscosity exponent. The computation is facilitated by the use of skeleton graphs.
As a preliminary theoretical study for our critical ultrasonics project, we have calculated the crossover frequency dependence at the critical point. Because of the relatively large non-critical background component of the thermal conductivity, as compared to the critical component, crossover is an unavoidable complication in the ultrasonics.

A. Theoretical

At the present time the theory of fast adiabatic equilibration requires an extension to the case of coexisting phases, at a temperature below the critical temperature. An essential difference for this case compared to that of the single phase above the critical point is that, as the fluid changes its temperature, latent heat is released at the interface. This tends to counteract the temperature change and thereby influence the time dependence of the approach to equilibrium. This extension of the theory is greatly needed as a guide for carrying out AFEQ and is receiving attention.

Although we have at our disposal a successful theory for critical ultrasonics along the critical isochore, the other isochores bring in some new features which require further study. In addition, much more work needs to be done on the crossover problem. Both of these problems will be investigated in collaboration with Prof. J. K. Bhattacharjee during his sabbatical leave from the Indian Institute of Technology at Kanpur, India.

The low temperature flight experiment, CHex (confined Helium), Prof. John Lipa, Principal Investigator requires a quantitatively complete theoretical prediction of the rounding of the lambda specific heat, with which the experimental data can be compared. This will also receive our attention during Professor Bhattacharjee’s visit.

B. Experimental

Our collaboration with the laboratory work at NASA/Lewis Research Center is limited to some modest supplementary assistance in connection with a) software for analyzing the interferograms from TEQB and AFEQ, and b) consultation on the fabrication of a low frequency resonator for our ultrasonics program. We regard the dense pattern of interference fringes, modified by the density changes of the fluid in the cell, as analogous to a radio frequency carrier wave on which there has been impressed an audio frequency modulation.

In the latter case, the carrier wave does not furnish any information and it is only the audio signal that is of interest. Similarly, we want to process the interferograms so as to discard the high wave number component of the intensity, so as to reveal the information contained in the longer wavelength modulation resulting from the density changes. This effort is of prime importance and will continue to receive top priority.

The critical ultrasonics project will proceed in two stages. In order to enter the true critical region, unconfused by the crossover to the non-critical background, it is necessary to work with as low a frequency as possible, in the sonic range of a few hundred Hertz. An additional advantage of this is the possibility of putting to an experimental test a cusp-like dependence on the parameters that has been predicted theoretically by the group of Professor Franz Schwabl of the Technical University of Munich.

Low frequencies require long wavelengths, and a large resonator. The first stage of this project consists in fabricating an annular resonator of the Garland-Williams type, but of somewhat larger diameter than used by them. This will be used for a ground-based study, with the vertical height of the horizontal fluid sample limited to one millimeter, so as to minimize the disturbing effect of earth’s gravity. The resulting enhanced surface to volume ratio has the additional benefit of making possible the exploration of the impact of fast adiabatic equilibration on the attenuation of sound. Having a clear demonstration of this effect will be essential to the second phase of the project, designing the flight hardware. Here we will need to choose the parameters for the spherical resonator (primarily its radius) so as to avoid the confusing surface contribution coming from fast adiabatic equilibration. In this way the in-flight data can be expected to yield true bulk measurements of the attenuation and dispersion.
II. MSAD Program Tasks — Ground-based Research

Discipline: Fluid Physics

ACCOMPLISHMENTS:

1. This work is converting to a UPN 694 activity for one flight experiment, one flight experiment definition activity, and some low temperature fluids theory development;

2. Participated in the Critical Fluid Viscosity delta-CoDR;

3. Reviewed CFTE IML-1 results in the context of the Onuki-Ferrell adiabatic fast equilibration description. He concludes that more data is needed to establish the basis for theoretical investigation;

4. Submitted to Physica A a paper extending the adiabatic fast equilibration theory to systems experiencing temperature and pressure pulses along with the consequences for sound attenuation and dispersion in very compressible critical fluids.

STUDENTS FUNDED UNDER RESEARCH:

Total Students: 2

BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:


II. MSAD Program Tasks — Ground-based Research

Effects of Convection on the Thermocapillary Motion of Deformable Drops

PRINCIPAL INVESTIGATOR: Prof. Hossein Haj-Hariri
University of Virginia

CO-INVESTIGATORS:
Prof. A. Borhan
Pennsylvania State University

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

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.

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.

SIGNIFICANCE:
This task will enable the development of a more realistic model of the motion of drops driven by interfacial tension gradients and is expected to provide an accurate prediction of the migration velocity.

PROGRESS DURING FY 1993:

1. The following computational tools have been developed:
   (a) Color function (2D, axisym.) — A Navier-Stokes solver using a passively convected color having a constant value over each liquid phase and varying abruptly at their interfaces. Uses a solution adapted Cartesian grid. Adaptation is based on quadtree data structure. A Total Variation Diminishing (TVD) treatment of the convective terms is utilized. There is no need for surface reconstruction.

   (b) Boundary integrals (2D, axisym.) — A Stokes equation solver with Gauss-quadrature treatment of the nonsingular kernels and an exact treatment of the singular kernels of the integral equations. This is coupled with a boundary-integral treatment of the conduction equation for temperature so that the zero Reynolds number and Marangoni number thermocapillary motion of a deformable drop can be studied.

   (c) Domain mapping — A second Navier-Stokes solution method using the mapping of the drop or the interface of interest onto a known and simple geometry of similar topology. For some simple problems this method is more economical than using the color function.

2. The following problems are being studied:
   (a) Thermocapillary motion of a drop in a cylindrical tube — Using the boundary-integral method the motion of a drop in a tube at Re = 0 and Ma = 0 was studied.

   (b) Thermocapillary motion of a drop in unbounded liquid — Using the color-function approach the well-known problem of Young, Goldstein, and Block was simulated. Also the qualitative effects of non-zero Re and Ma were observed; they cause substantial deformations, further reiterating the importance of the goals of our proposal.
(c) Surfactant-driven motion in an open cavity — Using domain mapping the time-accurate flow in an open cavity subjected to a nonuniform initial distribution of surfactants as well as a surface temperature gradient is simulated.

STUDENTS FUNDED UNDER RESEARCH:
PhD Students: 1

TASK INITIATION: 12/92  EXPIRATION: 11/95
PROJECT IDENTIFICATION: 674-24-05-45
RESPONSIBLE CENTER: LeRC
Interfacial Transport and Micellar Solubilization Processes

**Principal Investigator:** Prof. T. Alan Hatton  
Massachusetts Institute of Technology (MIT)

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

**Task Objective, Description & Significance:**

**Objective:**  
The objective of this research is to refine 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 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 not been possible because the techniques used for measurement of interfacial transport processes are prone to artifacts associated with ill-defined hydrodynamic conditions.

**Description:**  
A testing program to identify the important parameter ranges over which a space-based experimental study would be beneficial would entail the characterization of the solute/solvent/surfactant systems to be studied. This includes screening of the potential solutes in terms of their partitioning behavior, absorption characteristics, concentration-dependent densities, etc. Surfactants and solvents to be studied will also be determined. Aerosol OT and SDS are the two prime anionic surfactant candidates, while cationic quaternary ammonium and the nonionic C\textsubscript{10}E\textsubscript{4} surfactants will also be investigated. A detailed ground-based experimental program will be undertaken to delineate clearly which solution conditions lead to unstable density gradients.

Research Approach — The research to be carried out during the first year will be to develop the new detection and analysis techniques and to test their effectiveness using a range of model solutes under conditions which can be studied effectively in a normal gravity environment. The two subsequent years will be devoted to developing an important database on interfacial transport characteristics and to adapting the entire system to be compatible with whatever constraints are imposed by NASA on the experimental rig to be sent on a space shuttle mission. Concurrent with these developments, the theoretical modeling will be pursued.

**Significance:**  
The results of this study will be used to define space flight experiment requirements.

**Progress During FY 1993:**

Experiments have been carried out under conditions where the solute, methyl nicotinate, was concentrated in the lower regions of the cell and was increasingly more dilute as it diffused into the upper cell regions. Thus a stable, negative density gradient was established. In many cases, however, we would be interested in transport in the reverse direction, in which case the density gradient would be adversely positive, and the system would be prone to buoyancy-driven convective instabilities. In an important class of experiments associated with the water uptake or loss by reversed micelles in response to changes in salt conditions, stable density profiles can never be attained. This is because the conditions necessary for water uptake would require an initial positive density gradient with the
stronger salt concentration in the water layer above the membrane and the weaker solution in the lower cell. For water loss by the reversed micelles, we would be able to establish the desired negative salt gradient readily, but the density gradient in the organic reversed micellar phase would be unfavorable. This is because the water content of the microemulsion phase would be at its lowest near the interface and higher in the upper regions of the cell. These observations point to the need for a microgravity environment for the successful investigation of many important phenomena associated with interaggregate interactions of colloidal and biological importance.

**STUDENTS FUNDED UNDER RESEARCH:**
PhD Students: 2

**TASK INITIATION:** 4/93  **EXPIRATION:** 4/96

**PROJECT IDENTIFICATION:** 674-24-08-11

**NASA CONTRACT NO.:** NAG8-951

**RESPONSIBLE CENTER:** MSFC
Thermocapillary Instabilities and g-Jitter Convection

PRINCIPAL INVESTIGATOR: Prof. George M. Homsy  Stanford University

CO-INVESTIGATORS:
No Co-I's Assigned to this Task

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

OBJECTIVE: RESEARCH APPROACH:
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.

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.

PROGRESS DURING FY 1993:
The Principal Investigator has recently returned from a sabbatical and has started work on this project.
Turbidity of a Binary Mixture Very Close to the Critical Point

PRINCIPAL INVESTIGATOR: Prof. Donald T. Jacobs

CO-INVESTIGATORS:
No Co-I's Assigned to this Task

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

OBJECTIVE:
This experimental activity is intended to measure the light transmitted through a near critical fluid consolute point of a density matched binary mixture.

DESCRIPTION:
1. Develop a room temperature thermal control system for the sample that enables temperature control inside of ± 3μK;
2. Assemble and use 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));
4. Undergraduate students will assist Prof. Jacobs, making the experience a strong educational experience for the students.

SIGNIFICANCE:
The resulting data will be used to confirm and quantify a small exponent "η" in a denominator "(1+(qΔ)^2)^-η" 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.

PROGRESS DURING FY 1993:
A polarized He-Ne laser beam passing through a laser amplitude stabilizer (L.A.S.) will hold the intensity constant to ±0.1% but stray light and temperature can cause the intensity to vary by ±1%. To avoid this problem, dual beams are used where one beam passes around the fluids while the other beam passes through the fluids. These beams are optically chopped with the signal from photodiode detectors (PD) being measured on lock-in amplifiers. The optics have been assembled on an optical bench and the lock-in amplifiers have been connected to the optical chopper, photodiodes, and computer.

The interfacing software that accesses these instruments has been written and tested. These programs were incorporated into a program that allows the computer to set the lock-in amplifiers and then measure the light intensities and room temperature over long periods of time. Early problems with room temperature drift of the light intensity have been solved and the stability of the intensity ratio of the two beams is less than 0.1% over a long period of time. This stability and resolution are quite adequate for the turbidity measurement being planned.

Cell, Thermostat and Temperature Control — The cell holds the critical fluid mixture in a leak-proof environment that also allows light to pass through the fluids. Our intention initially was to modify a cell design used by ZENO
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Discipline: Fluid Physics

to seal Xenon at its critical pressure and density. Due to the difficulty in producing reliable seals in cells of this
type, we have decided to redesign the cell for our fluid mixture. The cell will be filled with a critical composition
and the fluids will be transferred into the cell while still in the one-phase region (at a temperature above 50°C). A
transfer station has been constructed and tested and should be usable with our cell with few modifications. In
addition to the metal and glass cell being machined, a commercial, cylindrical, glass cell is also being used in
preliminary measurements. The thermostat design was modified as the cell design changed and is also ready for
construction. Since we plan on controlling temperature to $10^{-7}$ the cell and thermostat needed to be carefully
thought out.

The temperature control instrumentation has been acquired and interfacing software has been developed. A prototype
program was written that balances the AC Wheatstone bridge using an ESI ratiotransformer as one arm and a lock-in
amplifier as a detector. The program optimizes the time necessary to reach a balance of the bridge. The temperature
sensors were chosen to be ultra-stable Thermometrics thermistors, which are factory tested for stability and are now
here. The final element of the bridge is the (Vishay) standard resistor which has also been received.

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PROJECT IDENTIFICATION: 674-24-05-74

RESPONSIBLE CENTER: LeRC
**II. MSAD Program Tasks — Ground-based Research**

**Discipline: Fluid Physics**

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**Kinetic and Transport Phenomena in a Microgravity Environment**

**Principal Investigator:** David Jasnow  
University of Pittsburgh

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

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**Task Objective, Description & Significance:**

**Objective:**  
Theoretical research will be done in several areas involving kinetic and transport phenomena in a microgravity environment. Specifically, attention is paid to the kinetics of phase separation, to coupled diffusing fields including the motion of and transport through interfaces separating macroscopic phases, and to kinetics of wetting layers.

**Description:**  
Methods proposed include modeling at the coarse grained or semimicroscopic level, and the use of macroscopic interface equations.

**Significance:**  
The methods should allow further elucidation of the crossover region of weak gravity, of transport properties in complex situations and an examination of phenomenological boundary conditions from a somewhat more microscopic level.

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**Progress During FY 1993:**

The main focus within this period has been to observe thermocapillarity effects such as thermomigration within a coarse grained microscopic description. We have begun our studies systematically, intending first to observe and characterize thermomigration effects in diffusive systems during phase separation and in simple droplet configurations before introducing any hydrodynamic effects. Hence we began with diffusive dynamics described at the coarse grained level by a time-dependent Ginzburg-Landau (TDGL) equation for a conserved density or composition which is coupled to a passive temperature field varying linearly across the system. This corresponds to a system with heat diffusion much more rapid than order-parameter diffusion.

We prepared the codes for forward integration of the appropriate dynamical equation for the diffusive concentration field. Within the framework of this model (initially limited to two spatial dimensions), we have observed and characterized droplet and slab migration, finding that (in this dissipative system) a droplet's velocity is proportional to the temperature gradient, and its mobility is proportional to an inverse power of the droplet radius. These effects are entirely due to the temperature variation of the interfacial free energy for a boundary separating the coexisting phases and should be present in any such system with mutual diffusion, although possibly masked by hydrodynamic effects. Several extensions of this work are being begun.

Analytically we have analyzed the coarse-grained equation describing the change of the diffuse interfacial profile (or "kink") separating the two coexisting phases in the presence of a temperature gradient. We found that the temperature gradient couples to the soft, translation mode which may be clamped weakly in a system of finite size.

To explore thermocapillarity effects from another perspective, we have turned to the microscopic level and have adapted microcanonical Monte Carlo techniques for the simulation of phase separation in a temperature gradient.
We developed the codes for the analysis of phase separation and effective transport coefficients far from equilibrium during coarsening in Ising-like models of binary systems with conserved order-parameter dynamics.

**STUDENTS FUNDED UNDER RESEARCH:**

PhD Students: 1

**TASK INITIATION:** 1/93  **EXPIRATION:** 12/95

**PROJECT IDENTIFICATION:** 674-24-05-42

**RESPONSIBLE CENTER:** LeRC
Surfactant-Based Critical Phenomena in Microgravity

**PRINCIPAL INVESTIGATOR:** Dr. Eric W. Kaler
**University of Delaware**

**CO-INVESTIGATORS:**
M.E. Paulaitis
**University of Delaware**

**TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:**

**OBJECTIVE:**
The specific objective is to characterize experimentally and theoretically both the kinetics of phase separation and the metastable structures produced during phase separation in a microgravity environment.

**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.

**SIGNIFICANCE:**
The purpose of this research is to locate and examine critical points and non-equilibrium structures in mixtures of water, surfactants, and supercritical fluids (SCF) as a function of temperature and pressure.

**PROGRESS DURING FY 1993:**
A high-pressure visualization cell has been built to measure multiphase behavior for three-component mixtures of water, surfactants (C12EJ) and supercritical fluids (SCF). The C12Ej surfactants are ethoxylated alcohols. A stoichiometric experimental technique is used in which equilibrium phase compositions and densities are obtained at a pre-set temperature and pressure from measurements of the individual phase volumes of all co-existing phases. The cell allows a maximum pressure of 500 bar and phase compositions are determined with accuracies of +/-0.007 mole fraction. Calibration of the visualization cell is nearly complete. The resulting experiments will hopefully verify and refine previous pressure, temperature and density measurements for the "near-isopycnic" point (isopycnic phases are equilibrium phases having equal densities). A computer program has been written to analyze the data and compute experimental uncertainties. Substantial effort has also been devoted to purifying the C12Ej sample.

Cells have also been constructed to contain mixtures during light scattering and small angle neutron scattering experiments. The cell designs permits pressure jumps into two-phase regions such that both scattering experiments can be carried out during phase separation. A quartz cell for laser light scattering is under construction. The cell has been designed to accommodate a range of scattering angles from 10°-140°. Provisions have been made to allow video imaging of the spinodal ring near 0° scattering angle. The cell for small-angle neutron scattering has been constructed. A unique design was conceived in which the cell is machined from a single sapphire crystal to allow examination of a wide range of q-vectors (0.005 to 0.5 Å⁻¹).
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Discipline: Fluid Physics

A modified Peng-Robinson equation of state has been chosen to model the multicomponent SCF/C\(_2\)E\(_4\)/H\(_2\)O phase behavior. When a complete set of parameters for the three-component CO\(_2\)/H\(_2\)O/C\(_6\)E\(_3\) system are in hand, multicomponent phase equilibrium calculations will assist in locating the near-isopycnic point mentioned above.

**STUDENTS FUNDED UNDER RESEARCH:**

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**PROJECT IDENTIFICATION:** 674-24-05-75

**RESPONSIBLE CENTER:** LeRC
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, DESCRIPTION & SIGNIFICANCE:

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 see if similar stabilization can be achieved for the thermocapillary (Marangoni) instability by the same means.

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.

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 1993:
An investigation has been completed of the effects of small amplitude forcing, and the results will soon be submitted for publication in a journal. It has been established that nonplanar oscillations can indeed stabilize the Pearson mode of instability, which typically has wavelengths of the order of the layer thickness and exists even when the surface is nondeformable. However, depending upon the operating parameters, another mode of instability, namely the long wavelength mode, can either be stabilized or destabilized.

Formulation of the numerical method for large amplitude forcing is almost complete. It is hoped that preliminary results concerning the maximum achievable stabilization should be in hand by the end of 1993.

TASK INITIATION: 2/93  EXPIRATION: 2/96
PROJECT IDENTIFICATION: 674-24-05-56
RESPONSIBLE CENTER: LeRC
II. MSAD Program Tasks — Ground-based Research  
Discipline: Fluid Physics

Molecular Dynamics of Fluid-Solid Systems

PRINCIPAL INVESTIGATOR: Dr. Joel Koplik  
City College of New York

CO-INVESTIGATORS:  
J.R. Banavar  
Penn State University

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

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 are: (1) static and flowing pure fluids near walls, (2) freezing transition in small pores, (3) the breaking and coalescence of droplet interfaces driven by gravity of flows, and (4) droplet spreading on solid surfaces.

DESCRIPTION:
1. Develop a transportable and robust fluid Molecular Dynamics (MD) simulation code for vector and parallel processing main-frame computer (Cray to connection Machine);
2. Develop realistic molecular model of solid walls, i.e., walls with correct thermal and mechanical behavior, to allow for true physics of wall interactions rather than idealized boundary conditions;
3. Utilize a sub-grant with J. Banavar at Pennsylvania State University to execute some of the work.

SIGNIFICANCE:  
Explore and broaden the science relevance of molecular dynamics to fluid behavior of interest to microgravity science.

PROGRESS DURING FY 1993:

Molecular dynamics (MD) simulations are being used to study problems involving fluid-solid systems that are not straightforwardly addressed either by experiment or continuum fluid mechanics.

Recent experiments have exhibited apparent cusps in fluid free surfaces driven by a converging subsurface flow. We have used MD simulations to examine the fluid behavior in such situations at very small length scales. We find that the curvature of the free surface increases with rotation rate, but instead of a high-curvature steady state we observe the detachment and subsequent entrainment of drops and bubbles of the fluid above the surface. We see no evidence for true cusps or even unusually high stress.

The dynamics of phase separation of binary liquids in confined geometries has been studied using MD and the numerical integration of the exact equation of motion of soft Ising spins undergoing Kawasaki dynamics. We argue that, as long as there is a conservation law for the two species and a planar heterogeneity is present in the initial conditions, composition waves with a wave vector normal to the heterogeneity should be observed. In a narrow channel, systems with one and two fluid layers exhibit distinct behavior. Our results point to the importance of conservation laws and hydrodynamics in determining the growth dynamics.
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Studies are underway of the freezing and flow of a fluid made up of diatomic molecules in a narrow channel. Preliminary results indicate the breakdown of no-slip boundary conditions, non-Newtonian flow characteristics and striking effects of the solid boundary on the rotational correlation functions both in the liquid and the frozen glassy state.

In collaboration with an experimental group at the MARS Center in Naples, we have been studying the suppression of coalescence in flows. In laboratory experiment and MD simulation it is observed that a liquid drop held just above a MOVING bath of the same liquid will not coalesce with it, but when the flow stops the coalescence is quite rapid. Similar effects occur in the presence of a temperature difference between bath and drop. Presumably this behavior is caused by the shear stress transmitted by the background vapor or fluid in the moving case, and by Marangoni effects in the other case, but this explanation has not been systematically checked as yet. Further simulations and complimentary Stokes equations calculations are in progress.

STUDENTS FUNDED UNDER RESEARCH:

PhD Students: 2

TASK INITIATION: 6/90 EXPIRATION: 12/95
PROJECT IDENTIFICATION: 674-24-05-29
RESPONSIBLE CENTER: LeRC

BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:
Fluid Dynamics and Solidification of Metallic Melts (FDSMM)

PRINCIPAL INVESTIGATOR: Prof. Jean N. Koster
University of Colorado

CO-INVESTIGATORS:
No Co-I's Assigned to this Task

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

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.

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 "Hurle" 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).

SIGNIFICANCE:
Fluid physics is the foundation of material solidification. The primary purpose of this research is to develop a unique research capability for flow visualization of metallic and electronic melts. A significant goal of this effort is to design an elevated temperature RAPIV system capable of providing the first flow visualization of the liquid metal convection flow behavior.

PROGRESS DURING FY 1993:
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 particles are currently under development.
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 has been directed toward the design of the appropriate tracer particles; candidate particles are tungsten, coated with SiO₂ or glass coated with gold.

**Students Funded Under Research:**

- MS Students: 2
- Total Degrees: 1

**Task Initiation:** 2/91  
**Expiration:** 2/94  
**Project Identification:** 674-24-05-33  
**Responsible Center:** LeRC
II. MSAD Program Tasks — Ground-based Research

**Thermocapillary Convection in Floating Zones under Simulated Reduced Gravity**

**Principal Investigator:** Prof. Sindo Kou  
**University of Wisconsin**

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

**Task Objective, Description & Significance:**

**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 NaNO₃ rods. Computer simulation of thermocapillary convection will be conducted and the calculated results will be compared with those observed in flow visualization.

**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.

**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.

**Progress During FY 1993:**
The mathematical formulation was derived and was verified experimentally. Flow visualization was conducted on a silicone-oil zone held vertically between two 4 mm diameter Cu rods of different temperatures, using a laser light-cut technique. Computer simulation of heat transfer and fluid flow in the zone was conducted. By using a mathematical formulation, the calculated flow patterns were compared with the distorted ones observed in flow visualization. Very good agreement was obtained.

In addition, the computer model was used to calculate heat transfer, fluid flow and mass transfer in floating-zone crystal growth of NaNO₃ under microgravity.

**Students Funded Under Research:**
Total Students: 2

**Task Initiation:** 12/92  **Expiration:** 12/95

**Project Identification:** 674-24-05-46  **Responsible Center:** LeRC

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II. MSAD Program Tasks — Ground-based Research

BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:
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, Description & Significance:

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.

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.

Progress During FY 1993:
The liquid-solid flow loop has been constructed and testing has begun. Problems with the laser doppler anemometer (LDA) not acquiring enough particles have been rectified. The problem was caused by the particles not being reflective enough; when the particles were made opaque by treating them in a hot-air popcorn popper, the LDA system was able to differentiate between the particles and the liquid. Construction of the immiscible liquids flow loop has been completed, with testing to begin soon. Model development for predicting the radial phase distribution is proceeding. Some initial comparisons of the model with low gravity gas-liquid flow, obtained from Dr. Jean Fabre at the Institute de Mecanique des Fluides de Toulouse in Toulouse France, have shown good agreement.

Students Funded Under Research:
PhD Students: 2

Task Initiation: 12/92  Expiration: 11/95
Project Identification: 674-24-05-72
Responsible Center: LeRC
II. MSAD Program Tasks — Ground-based Research
 Discipline: Fluid Physics

Nonlinear Drop Dynamics and Chaotic Phenomena

Principal Investigator: Dr. L. Gary Leal
University of California, Santa Barbara (UCSB)

Co-Investigators:
E.H. Trinh
Jet Propulsion Laboratory (JPL)

Task Objective, Description & Significance:

Objective: The general objective of the UCSB portion of the research is to apply newly developed knowledge about the dynamical behavior of nonlinear systems, and newly developed methods for the numerical solution of large deformation free-boundary problems in fluid mechanics, to address a number of unexplained or unexplored issues associated with dynamics of levitated drops or bubbles in acoustic and electric fields.

Specific objectives for the analysis of finite amplitude effects for bubble motion include:

1. Establishing conditions for existence of steady deformed shapes for both acoustic and electrostatic levitation. One point of special significance is the difference between the idealized zero-g environment where the drop shape will exhibit fore-aft symmetry along the axis of rotational symmetry, and the shape of a drop in a gravitational field where the fore-aft symmetry is lost.

2. Developing an understanding of modal coupling and instabilities associated with large amplitude deformations. In this part of the research, the interest is with time-dependent oscillations, both from an undeformed and a deformed equilibrium state, and subject to periodic forcing via either the acoustic or electric fields. Again, the effect of asymmetries in the shape associated with levitation in a gravitational field should be considered.

3. Developing an understanding of the conditions for transition between regular and chaotic oscillations, again associated with periodic forcing via either the acoustic or electric fields. The effects of mean deformation are again of special interest, including differences between zero-G and gravitational environment due to the mean asymmetries associated with the latter case.

It should be noted that these general objectives incorporate most of the anomalous observations from previous studies of drop dynamics in the microgravity science program. As indicated in the original proposal, it is the existence of these phenomena that largely motivated and suggested the importance of the present study.

The expected significance of this project is both as a very important contribution to the basic scientific understanding of drop dynamics under nonlinear, large-amplitude deformation conditions (where all previous theories are deficient and few systematic experimental studies have been done), and as a basis for an enhanced ability to manipulate and understand the limits of manipulating levitated materials in containerless process applications.

Description:

From the theoretical side, the proposed project has two basic elements: (a) Derivation and study of law dimensional model systems to obtain an initial framework for prediction of nonlinear dynamical phenomena and (b) numerical study of the full, large deformation fluid mechanics problem using boundary-integral techniques for studying the nondissipative limiting behavior, and boundary-fitted coordinate, finite-difference techniques for the general case. In both cases, similar studies have either recently been carried out (or are being carried out) for deformable (and compressible) gas bubbles in a liquid. Thus, the initial development of methodology for solution of the acoustically driven drop dynamics problem is a relatively straightforward adaptation of existing methods. The
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The electrostatic levitation problem requires some additional modification to include the electric field calculation, but the corresponding governing equations, etc., have already been laid out by previous investigators. Once the problem formulation phase is completed, a detailed investigation of the three major objectives will be carried out, beginning with the problem of steady shapes in a steady levitation mode with and without gravity.

The theoretical work, by itself, can contribute in an important way to explaining previous "anomalous" observation in microgravity-oriented levitation experiments, and to providing a predictive framework that can be used to anticipate additional, as yet undetected phenomena that could be of great significance in material-levitation applications.

However, theory should not be viewed as capable of providing all of the answer. The low-order model systems study is expected to provide a global picture of possible nonlinear phenomena, but the models are often derived from small amplitude expansion theories, and thus, even if the low-order dynamical system is a faithful representation of the full problem in this limiting sense, it cannot be expected to capture all of the possibilities for large-amplitude deformations. Numerical solutions faithfully reproduce the complete physics, but provide only a case-by-case picture, and also suffer from limitations on resolution that make it difficult to capture any high-mode phenomena, or many fully three-dimensional effects.

Thus, experiments still must play a crucial role, and this project is an excellent example of a case where ground-based studies alone cannot be adequate. The ground-based work must always involve an asymmetric deformed base state of deformation, and for large amplitude deformation this will have very significant effects on both the steady state and time-dependent dynamics that cannot be completely assessed via theoretical studies. Thus, flight-based experiments must play a critical role in providing a basis for comparison with the ground-based experimental results. The latter can, of course, be much more comprehensive in scope. A limited set of studies for comparison purposes is an appropriate and realistic role for flight-base experiments. The flight-based experiments can also expand the range of parameters beyond that possible in the ground-based work.

PROGRESS DURING FY 1993:

Progress to Date:
Numerical codes are being applied to self-excited oscillations of highly distorted drops in acoustic fields. Details of the interaction depend on accurate modeling of the time dependent acoustic pressure distribution. Numerical modeling is being coordinated with bubble and drop experiments at JPL performed by Eugene Trinh and Glynn Holt. They are measuring (1) coupling between radial and shape modes in air bubble oscillations in water and (2) finite amplitude effects in water drop oscillations using a hybrid acoustic-electrostatic levitator.

STUDENTS FUNDED UNDER RESEARCH:

Total Students: 1

PROJECT IDENTIFICATION: 694-24-07-01

RESPONSIBLE CENTER: JPL

BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:

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Discipline: Fluid Physics

Absolute and Convective Instability of a Liquid Jet at Microgravity

Principal Investigator: Prof. Sung P. Lin  
Clarkson University

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

Task Objective, Description & Significance:

Objective:  
The objective is to definitively establish the roles 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 reexamining the known theories that can be verified completely only in microgravity. A wide range of Weber and Reynolds numbers will be studied, and any unexpected phenomena in microgravity that may require new interpretation of dynamic capillary force will be examined.

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 1-g, one will be able to unambiguously assess the significance of gravitational and inertial forces relative to the capillary force over a wide range of dynamic flow parameters. The approach will be to design and fabricate a rig to conduct liquid jet experiments during the first year. One-g tests with this rig and numerical modeling will also take place in the first year. Ground-based low-g experiments will take place during the second year in the LeRC 2.2 second drop tower. Data analysis and flight experiment definition is tentatively planned for the third year.

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 1993:  
The progress since the beginning of the grant (January, 1993) includes both theoretical and experimental work.

The theoretical work includes an analysis of the absolute and convective instability of a viscous jet emanating into a viscous gas in a vertical pipe. This analysis was done over a parameter space spanned by the Reynolds, Froude, and Weber numbers, and the viscosity, density, and diameter ratios. The numerical results of the analysis demonstrate that the reduction in gravity tends to enhance the Rayleigh mode of convective instability (i.e., where the liquid jet breaks up into drops of diameter comparable with the jet diameter). On the contrary, the Taylor mode of convective instability (i.e., where jet atomization occurs) is retarded at reduced gravity. The analysis shows that the Rayleigh mode becomes absolutely unstable when the Reynolds number exceeds a critical value, given a set of parameters. The domain of absolute instability is significantly enlarged when the effect of gas viscosity is accounted for.

The experimental work began by scrutinizing the system described in the original proposal. It was decided that the systems would be modified to cover a larger range of parameters. The new system is currently being fabricated.
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The construction of the one-g test rig is in its final stage. It is expected that these ground experiments will start in December. It is anticipated that this one-g can be designed in such a way that its modification for testing in ground-based low-g test facilities (e.g., 2.2 sec Drop Tower) will be minimal.

STUDENTS FUNDED UNDER RESEARCH:
Total Students: 2

TASK INITIATION: 1/93  EXPIRATION: 12/95
PROJECT IDENTIFICATION: 674-24-05-47
RESPONSIBLE CENTER: LeRC

BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:
Cross Effects in Microgravity Flows

PRINCIPAL INVESTIGATOR: Prof. Sudarshan K. Loyalka
University of Missouri, Columbia

CO-INVESTIGATORS:
Prof. R.V. Tompson
University of Missouri, Columbia

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

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 \( L \), 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 the ampoules.

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.

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 the gases and the vapors.

PROGRESS DURING FY 1993:

Theoretical Progress:
A. The Boltzmann equation for a monatomic gas has been solved for rigid sphere molecules and cylindrical geometry, under non-condensing conditions. All phenomenological coefficients have been computed.

B. Initial computations for realistic potentials (monatomic gas), and 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.

Experimental Progress:
A two-bulb apparatus has been designed for the diffusion cell. All necessary components have been purchased. Most of the apparatus has been constructed and/or assembled, and it is currently being tested.
Applications Progress:
Exploration is under way of a computational fluid dynamics program incorporating dummy values for the unknown phenomenological coefficients.

**TASK INITIATION:** 11-92  **EXPIRATION:** 11-95
**PROJECT IDENTIFICATION:** 674-24-05-64
**RESPONSIBLE CENTER:** LeRC
Study of Disturbances in Fluid-Fluid Flows in Open and Closed Systems

**Principal Investigator:** Prof. Mark J. McCready  
**University of Notre Dame**

**Co-Investigators:**  
Prof. H-C Chang  
Prof. D. Leighton  
**University of Notre Dame**

**Task Objective, Description & Significance:**

**Objective:**  
The objective of this effort is to examine the different instabilities that can affect a liquid film during gas-liquid flow.

**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.

**Progress During FY 1993:**

Experiments have been initiated in the rotating, vertical interface, two fluid device that uses a paddle to influence the interfacial disturbances.

Two types of waves have been recently observed:
- Slug-like, which starts with a periodic wave train, and as the rotation rate is increased, a single wave grows to fill the entire distance between the cylinders. This type of mechanism is analogous to slug formation in gas-liquid pipe flows. The controlled nature of the experiment allows for detailed study of the growth of the disturbance that is not possible in a pipe flow. It is hoped that this information will provide insight into the slug formation problem.

- Solitary waves, which start with the formation of first one short-wavelength wave (a solitary wave), then two such waves, then more as the rotation rate is increased. These disappear in sequence as the rotation rate is decreased. The reason for this behavior is that the formation of a wave locally expels some of the less viscous fluid. This makes the average thickness higher elsewhere, which reduces the shear in that phase. To form an additional wave, greater shear is needed; the rotation rate must be increased. Once this is done, more fluid is expelled thus quenching formation of other waves. This process is repeatable and reversible. The importance of this observation is that it may reveal ways of predicting the number/time of non-periodic disturbances in other systems.

**Students Funded Under Research:**

PhD Students: 3

**Task Initiation:** 12/92  **Expiration:** 11/95  
**Project Identification:** 674-24-05-55  
**Responsible Center:** LeRC
Study of Forced Convection Nucleate Boiling in Microgravity

PRINCIPAL INVESTIGATOR: Prof. Herman Merte, Jr. University of Michigan

CO-INVESTIGATORS:
Prof. R.B. Keller
Dr. J.A. Platt
University of Michigan
NASA Lewis Research Center (LeRC)

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

OBJECTIVE:
This task represents a continuation of previous experimental work examining the effects of buoyancy on forced convection nucleate boiling. 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. Understanding when this mechanism will dominate, and the effectiveness of boiling in such circumstances, is important in making accurate predictions of microgravity boiling behavior. This information will also increase the understanding of boiling in cases where gravity is present.

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 the KC-135 microgravity 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. Analyses have been performed in both areas of study to develop physical explanations for the observed results.

SIGNIFICANCE:
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 that results in reduced heat transfer. The program seeks to enhance understanding of the fundamental mechanisms that constitute convection boiling.

PROGRESS DURING FY 1993:
A study of the effects of orientation on forced convection flow boiling has been completed. Forced convection-dominated and buoyancy-dominated conditions were observed and a parameter, the two-phase Richardson number, was identified as the relevant indicator of the dominant regime.
Modifications to the test facility, to extend the range of test conditions that can be achieved, are proceeding:
1. A new pump and flow meter have been ordered that, when installed, will double the liquid flow rate in the loop.
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2. The temperature and pressure control systems are being changed from analog to digital control; the new system will be more precise and easier to operate, and will be better-suited for an aircraft experiment.

3. A new cooling system, to replace the existing tap water system, will be designed and built. Besides being suitable for aircraft operation, the new system will also increase the range of subcoolings that can be achieved.

A new experimental effort has recently been started, examining the effect of heater length-to-width ratio on boiling heat transfer rates and on the departure from nucleate boiling.

<table>
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<tr>
<th>STUDENTS FUNDED UNDER RESEARCH:</th>
<th>TASK INITIATION: 11/91</th>
<th>EXPIRATION: 2/96</th>
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<td>MS Students: 1</td>
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BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:

Proceedings:

Critical Fluid Viscosity Measurement

**PRINCIPAL INVESTIGATOR:** Dr. Michael R. Moldover  
National Institute of Standards and Technology (NIST)

**CO-INVESTIGATORS:**
Dr. R.F. Berg  
National Institute of Standards and Technology (NIST)

**TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:**

**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.

**DESCRIPTION:**
1. Develop a low frequency, low shear rate viscometer with μK 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 300 μK while measuring viscosities to 0.5% 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.

**SIGNIFICANCE:**
The data will provide complementary results with Critical Fluid Light Scattering Experiment (CFLSE) to test the mode-coupling theory of critical phenomena and provide guidance to renormalization group theory development on dynamic critical-point fluid behavior.

**PROGRESS DURING FY 1993:**

Initial preparations for a flight experiment to measure the viscosity of xenon close to its critical point with accuracy sufficient to eliminate uncertainties currently associated with the analysis of 1-g experiments were made. The measurements will provide the first direct observation of the predicted power-law divergence of viscosity in a pure fluid. The measurements will also strengthen Zeno's test of mode coupling theory by greatly increasing the reliability of the extrapolation of viscosity to low reduced temperatures.

The low-gravity flight experiment will be the final stage of a program whose completed ground-based stages are: (1) theoretical studies by one of the principal investigators (MRM) and coworkers, (2) critical viscosity measurements of binary liquid mixtures, (3) critical viscosity measurements of pure fluids in 1-g, and (4) development of a suitable vibration-insensitive viscometer. Major accomplishments of the past year are listed below.

The SRD summarized in detail the apparatus, operation, and flight requirements for the oscillating screen viscometer. For the first time, several novel technical features, such as nonlinearity and spring softening in the electrostatic drive, were described. The crucial issue of sample equilibration by using the constant volume heat diffusion equation was quantitatively assessed.
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The experiment's scientific objectives was described in a report to the director of the Microgravity Science and Applications Division at NASA Headquarters. This report combined for the first time the best available xenon light scattering data with recent NIST viscosity results to assess the current status of tests of mode coupling theory.

The use of electrostriction to locate the critical temperature was experimentally investigated. This included calculations of the effect's expected amplitude and time constant, design, construction, and loading of an appropriate critical point capacitor cell, and optimization of the electronic circuits used to observe the electrostriction. For the first time, electrostriction was observed in a pure fluid.

A calibration procedure for the oscillating screen viscometer which will provide the accuracy needed for the flight measurement was developed. The procedure, which makes use of the viscometer's wide bandwidth and a novel hydrodynamic similarity argument, allows the viscometer to be self-calibrating. To successfully demonstrate the validity of this procedure the oscillator's transfer function with CO₂ at temperatures spanning a range of 35 K and densities varying by a factor of 165 was measured. Achieving agreement with best available viscosity data required understanding a 1% anelastic effect present in the oscillator's torsion fiber.

BIBLIOGRAPHIC CITATIONS FOR FY 1993:

**Journals:**

Control of Oscillatory Thermocapillary Convection in Microgravity

PRINCIPAL INVESTIGATOR: Prof. G. Paul Neitzel
Georgia Institute of Technology

CO-INVESTIGATORS:
No Co-I's Assigned to this Task

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

OBJECTIVE:
The objective is to experimentally investigate active control of oscillatory thermocapillary convection in planar layers. The investigative strategy is: 1) Establish the appropriate basic state for the "thin" rectangular flow geometry described in the subject proposal, 2) Establish and detect the oscillatory thermocapillary convection in the form of hydrothermal waves, and 3) Suppression and control of the hydrothermal waves.

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 CO\textsubscript{2} 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. The duration and time of pulsing will also be compared with the predicted estimates.

PROGRESS DURING FY 1993:
Progress to date has been related to optimizing the design of a final hardware configuration. A preliminary apparatus has been designed and fabricated, based on available data and simple model calculations. Results from experiments performed with this apparatus are presently being used to remedy shortcomings observed in the experiments and improve the hardware design.

The objective of the initial experiments was to investigate the establishment of a suitable basic state subject to oscillatory instability. Silicone oils with viscosities of 1 and 2 centistokes were used as the test fluids. These experiments have confirmed, for some layer depths, the appearance of steady, multicellular states observed previously by other experimenters. For other layer depths, however, a steady, unicellular structure is observed. The appearance of the steady, multicellular state is suspected to be due to the influence of buoyancy and is an undesirable feature from the standpoint of control oscillatory instability.

The Smith and Davis theory which originally motivated these experiments has been modified to include the influence of buoyancy. Preliminary calculations of stability limits using this theory have indicated that there may be a range of dynamic Bond number (and hence, layer depth) for which the earliest appearing instability would be that of the oscillatory, hydrothermal-wave variety rather than steady, multicellular flow. The results appear to be in qualitative agreement with the range of parameters for which multicellular flow was absent in the early experiments. Further experimentation is now required to verify this result.
The two silicone oils to be used in these experiments have been characterized, in the sense that surface tension has been measured over a temperature range which will include temperatures employed in the experiments. These measurements were performed by the Microgravity Advanced Research Support (MARS) Center in Naples, Italy as a courtesy to the Principal Investigator.

The design modifications based on the preliminary tests described above, will permit more precise control of the depth, among other things, which is necessary due to the profound influence of the dynamic Bond number noted in the earlier experiments.

STUDENTS FUNDED UNDER RESEARCH:
PhD Students: 2

TASK INITIATION: 2/93  EXPIRATION: 2/96
PROJECT IDENTIFICATION: 674-24-05-61
RESPONSIBLE CENTER: LeRC
II. MSAD Program Tasks — Ground-based Research

Discipline: Fluid Physics

Industrial Processes

PRINCIPAL INVESTIGATOR: Prof. Simon Ostrach
Case Western Reserve University

CO-INVESTIGATORS:
Prof. Y. Kamotani
Case Western Reserve University

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

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.

DESCRIPTION:
The commercial processes and related basic processes that 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.

SIGNIFICANCE:
The role of gravity in those processes are being studied by using experimental, numerical, and analytical techniques, and the potential benefits of microgravity are being assessed. Based on these studies, microgravity related tasks will be proposed.

Results for FY 1993:
The accomplishments of this effort are:

1. A theoretical model for the process of bubble and drop formation in a flowing liquid, which is applicable for both terrestrial and microgravity environments, has been developed. Two different flow systems were considered, a co-flow system and a cross-flow system. A paper based on the co-flow study has been accepted for publication. An experiment is being designed for parabolic flights to experimentally study the process of bubble generation in microgravity.

2. A test setup to study coating flows has been built. Tests are being conducted to verify our theoretical model developed earlier for coating flows and the data taken so far show good agreement with the theory.

3. Effects of g-jitter on the dynamics of liquid-gas interfaces in open containers in microgravity have been studied numerically. A paper describing the results is being prepared. An experiment is being designed to investigate the effect of g-jitter on oscillatory thermocapillary flow.

4. The growth-in-gel mechanism during zeolite growth is being investigated. We have identified that the behavior of the gel portion within the reaction mixture during the growth and the mass transfer of the nutrients to the growing crystals are very important to obtain large crystals in microgravity. A paper describing the gel behavior in one-g has been published. We are analyzing the gel behavior in microgravity.
5. A rotating electrochemical cell is being designed to study the mass transfer and the effect of rotation on the growth of dendrites on the electrode surface. The apparatus is designed to simulate rotating batteries which have a potential for microgravity applications.

6. Natural convection in circular cylinders filled with a liquid metal is being investigated experimentally. The experiment is designed to study the effect of buoyancy during the Bridgman growth of crystals. Oscillatory convection in cylinders heated from below has been studied and a paper on the experiment has been accepted for publication.

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PROJECT IDENTIFICATION: 674-24-05-30
RESPONSIBLE CENTER: LeRC

BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:


Proceedings:
Two-Phase Interfaces in Weak External Fields.

PRINCIPAL INVESTIGATOR: Prof. Jerome K. Percus

CO-INVESTIGATORS:
No Co-I's Assigned to this Task

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

OBJECTIVE:
This endeavor expects to establish a theoretical framework that describes the interfacial response of fluids that have attractive and repulsive contributions, in interparticle interactions, to external fields — particularly a gravitational field. The most interesting result will be the elucidation of how gravitational forces at the microscopic level play a key role in interface dynamics.

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. Gravitational external fields will be the central emphasis, but the formalism will hopefully be more general;
3. Equilibrium followed by non-equilibrium descriptions are to be developed. It is expected customary as well as new analytical strategies will be utilized;
4. One post-doctoral fellow will support this work.

SIGNIFICANCE:
A description should develop out of this work that provides a mathematical connection with macroscopic hydrodynamics.

PROGRESS DURING FY 1993:
Activity during FY93 focused principally on thermal equilibrium aspects of two-phase interfaces in weak external fields. Before digging into the armory of uncontrolled approximation methods, it is crucial to make sure that the questions being asked are meaningful in at least some model context. For this purpose, we have reexamined analytically and numerically the hydrodynamic limit of a Lennard-Jones-like pure fluid with independent long and short range scaling, attending as well to metastable regimes, with their implications for dynamical processes. Closely related to underlying two-phase droplet substructure is the behavior of microemulsions in low gravity. These are macroscopically controlled mainly by curvature rather than by surface energy; hidden field representations of the surfaces have had increasing success, and are being incorporated into our studies. The theory of complex internally anisotropic fluids, two-phase or at boundaries, is still quite controversial: here too, curvature terms play an important role, and we are examining in detail the construction of physically relevant interfacial Hamiltonians.

We have started to investigate complex dynamics—e.g., formation and growth of droplets—under low gravity. There is also a large literature on stationary current carrying states under strong external fields, and we are trying to bridge the physical and conceptual gap between the strong and the weak, initially by examination of a number of scaling relations that have been suggested.
II. MSAD Program Tasks — Ground-based Research

Discipline: Fluid Physics

TASK INITIATION: 1/93  EXPIRATION: 12/95
PROJECT IDENTIFICATION: 674-24-05-76
RESPONSIBLE CENTER: LeRC

II-325
Containerless Capillary Wave Turbulence

**PRINCIPAL INVESTIGATOR:** Dr. Seth J. Putterman
University of California, Los Angeles

**CO-INVESTIGATORS:**
M.B. Barmatz
Jet Propulsion Laboratory (JPL)

**TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:**

**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. 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.

**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.

**PROGRESS DURING FY 1993:**

**Progress to Date:**
Seth Putterman has built and put into use a laser scattering apparatus to calibrate the wire probe measurements of the turbulent state in his laboratory. A new means of imaging surface deformation has been developed. This method avoids the problems presented by caustics. Programs for analyzing and simulating capillary wave turbulence have been tested out on non-linear lattices.

**STUDENTS FUNDED UNDER RESEARCH:**
Total Students: 1

**BIBLIOGRAPHIC CITATIONS FOR FY 1993:**

**Journals:**

Dynamics of Superfluid Helium in Low Gravity

**Principal Investigator:** Graham Ross
**Lockheed Missiles & Space Co.**

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

**Task Objective, Description & Significance:**

**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.

**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 KC-135. 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 KC-135 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 KC-135 flight to verify the accuracy of the computer model.

**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 1993:**
Contract was awarded Oct. 5, 1993. Preliminary discussions have been held with Bob Chave and Peter Mason of JPL, the technical monitor for this project, regarding the use of an existing SFHe dewar and components from its float package. This dewar was not available at the time of the proposal, but may offer a significant savings in time and schedule if components can be used.

Code has been developed and compared with ground SFHe tests.

**Task Initiation:** 10/93  **Expiration:** 10/96
**Project Identification:** 674-24-07-18
**Responsible Center:** JPL

II-327
**II. MSAD Program Tasks — Ground-based Research**

**Discipline: Fluid Physics**

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*Microscale Modeling of Microgravity Multiphase Flow*

**Principal Investigator:** Dr. Paul H. Rothe, Creare, Inc.

**Co-Investigators:**
- Dr. G.B. Wallis, Dartmouth College

**Task Objective, Description & Significance:**

**Objective:**
The general objective is to investigate the feasibility of modeling adiabatic multiphase flows on earth in facilities of microscopic scale. The specific objectives of this contracted effort are to build, instrument, and test a microscale-sized test loop containing a multiphase flow. This loop—designed and operated for appropriate flow conditions and geometries—will potentially scale space-based multiphase flow systems.

**Description:**
The principal investigator will identify, via the appropriate scaling arguments, the important non-dimensional quantities governing multiphase flow. The principal investigator will demonstrate, by dynamic similitude, how closely on-orbit conditions can be simulated with this microscale system. A microscale system that is appropriately sized for the proper range of flow conditions will then be designed and built. A comparison will be made between microscale data from this loop and the following: representative data already available from ground tests and low-gravity aircraft tests at larger sizes, existing theory, and in-house numerical models.

**Significance:**
A successful effort will demonstrate a method by which on-orbit, low-gravity, multiphase flow experiments can be complimented by low cost, Earth-based microscale experiments.

**Progress During FY 1993:**
The Principal Investigator and his colleagues have completed a feasibility study of the use of tiny "microscale" facilities on the earth to model multiphase flow in microgravity. They have unified existing small tube data as well as adding original data from facilities designed specifically to advance microscale modeling concept. One of these microscale facilities was at Creare and the other was at Dartmouth College. Data from these small facilities compare well with current flow regime prediction methods. Predictions of multi-phase pressure drop were also compared to measured values. The tubes of interest had a diameter of the order of 1 mm and are readily available from commercial sources. A draft final report is anticipated to be submitted in October 1993.

These tests complement the available data from microgravity aircraft experiments, which are typically more costly to obtain and are subject to modeling difficulties due to the transient nature of the gravity environment. The microscale modeling approach offers potentially cheaper testing over a wide range of fluids and fittings. It is the intention of this effort to promote that both aircraft and microscale testing should be fully utilized before full-scale flight experiments are pursued.
**II. MSAD Program Tasks — Ground-based Research**  
**Discipline:** Fluid Physics

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**PROJECT IDENTIFICATION:** 674-24-05-70  
**RESPONSIBLE CENTER:** LeRC
II. MSAD Program Tasks — Ground-based Research

Ground Based Studies of Thermocapillary Flows in Levitated Drop

PRINCIPAL INVESTIGATOR: Dr. Satwindar S. Sadhal
University of Southern California

CO-INVESTIGATORS:
E.H. Trinh
Jet Propulsion Laboratory (JPL)

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

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 be 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.

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
II. MSAD Program Tasks — Ground-based Research  

Discipline: Fluid Physics

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.

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 1993:
1.0 Technical Results
The work, while being in its preliminary stage, has the following main analytical results.

1.1 Pure Conduction in an Oblate Spheroid
In the case of the spheroidal drop, for analytical purposes, it is necessary to identify the interface by a single coordinate. To this end, the oblate spheroidal coordinate system works out quite well. The very first problem of pure conduction in a spheroidal drop with axisymmetric spot heating has been solved analytically. The results are included in a preliminary draft of a paper. The main results are the expressions for the temperature field in the drop and the exterior gaseous medium.

1.2 Outer Acoustic Streaming Around a Spheroid
Some work has been initiated on the study of acoustic streaming around a spheroid. The purpose here is to generalize the work of Lee & Wang (2) who treated the problem of streaming around a sphere and around a cylinder in the region outside the boundary layer. So far the asymptotic analysis of the pressure field has been carried out. The immediate objective is to calculate the tangential component of the acoustic velocity on the surface of the spheroid. This is to be applied to the Stokes equations for the velocity field.

1.3 Thermoacoustic Streaming from a Sphere
This work consists of a study of the thermal effects of the steady streaming motion induced by a solid sphere. The thermal field is generated by an oscillating boundary condition on the surface of the sphere. The interesting feature here is that when the thermal frequency is 'in tune' with the acoustic field, the convective heat transfer term remains nonzero when the energy equation is averaged over time. This leads to a net flow of energy through the equatorial plane of the sphere.
1.4 Experimental Study
Ultrasonic and ultrasonic electrostatic hybrid levitators have been used to develop techniques for controlling the rotation of suspended free drops and for measuring the internal flow within the sample. Tracer particles have allowed the measurement of a slow circulation within isothermal levitated drops.

A singular perturbation analysis has been carried out for small values of the streaming Reynolds number \((U \delta R \Sigma \sim 1)\) along with \(M = R \delta \Sigma \sim 1\). There is equal heating and cooling of the regions above and below this plane, respectively. A result for the net flow across the equatorial plane of the sphere has been obtained.

A paper resulting from this work has been submitted for the 1994 International Heat and Mass Transfer Conference (Brighton, UK).

Conference Effort in Microgravity
The Principal Investigator has taken a role in organizing technical sessions the National Heat Transfer Conference under the auspices of the American Society of Mechanical Engineers. At the last conference (Atlanta, August 8–11, 1993), he chaired two sessions and edited the volume for the proceedings.

**BIBLIOGRAPHIC CITATIONS FOR FY 1993:**

**Presentations:**

Studies in Electrohydrodynamics

**Principal Investigator:** Dr. Dudley A. Saville
Princeton University

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

**Task Objective, Description & Significance:**

**Objective:**
This research investigation is designed to conduct experiments to strengthen the experimental foundation for the theory through further studies of the deformation of drops and the stability of fluid cylinders in strong electric fields. Part of the work consists of developing systems where the conductivity can be controlled using ionic surfactants so as to allow exploration of regions as yet unstudied. Another part of the work will focus on developing a scientific rationale for microgravity experiments. In addition to their scientific value, the results of the study will have a wide range of technological applications since electric fields offer a means to control fluid motions by exerting tractive forces directly on an interface.

**Description:**
Theoretical studies will center on adapting the existing leaky dielectric theory for the stability of a fluid cylinder (Saville, 1971 and 1972) to account for the pinning of the contact lines at the upper and lower boundaries. Some work will need to be done to ascertain the importance of interfacial tension gradients on jet stability. A simple model (Vizika and Saville, 1991) which suggests that such gradients play a minor role in our experiments on drop deformation has already been developed. However, the thrust of our work is experimental.

**Research Approach** — The research to be carried out during the first year will be to develop the new detection and analysis techniques and to test their effectiveness using a range of model solutes under conditions which can be studied effectively in a normal gravity environment. The two subsequent years will be devoted to developing an important data base on interfacial transport characteristics and to adapting the entire system to be compatible with whatever constraints are imposed by NASA on the experimental rig to be sent on a space shuttle mission. Concurrent with these developments, the theoretical modeling will be pursued. The research study consists of experimental and (a limited amount of) theoretical work necessary to prepare a Science Requirements Document (SRD) for microgravity experiments. It appears that the need for a microgravity environment can be justified. Nevertheless, the possibility that some of the work can be accomplished in drop towers will be investigated, which may be feasible using systems with short relaxation times. For this purpose, less viscous fluids will be essential.

The study will focus on:
- The use of ionic surfactants to control the conductivity of low dielectric constant liquids. This will provide a means to explore the role of fluid conductivity in the deformation of drops and the stability of fluid cylinders.
- The stability of fluid cylinders in longitudinal fields with isopycnic systems. This will furnish experience with this geometry and provide some information to test the leaky dielectric model for the stability of fluid columns. To the extent possible, ionic surfactants to control fluid conductivity will be used.
- The deformation of droplets using fluids whose conductivity has been adjusted using a non-ionic surfactant.

**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 consequently there are large gaps in our knowledge. Experimental investigations focused on the effects of fluid conductivity and viscosity are almost non-existent. Moreover, in addition to scientific interest, there is a wide range of applications where electrohydrodynamic phenomena play important roles.

**PROGRESS DURING FY 1993:**

The experimental foundation of electrohydrodynamics—fluid motions driven by strong electric fields—is weak, and the model proposed by G.I. Taylor (the leaky dielectric theory) is relatively untested. Recent work shows some promising areas of agreement, but the range of fluid properties covered is small; the fluids are, for the most part, quite viscous, and the range of electrical conductivities is limited due to the need to use isopycnic systems to avoid sedimentation and deformation due to hydrostatic pressure effects.

**STUDENTS FUNDED UNDER RESEARCH:**

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**BIBLIOGRAPHIC CITATIONS FOR FY 1993:**

**Journals:**


**Presentations:**

Solute Nucleation and Growth in Supercritical Fluid Mixtures

PRINCIPAL INVESTIGATOR: Dr. Gregory T. Smedley
Jet Propulsion Laboratory (JPL)

CO-INVESTIGATORS:
No Co-I's Assigned to this Task

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

OBJECTIVE:
The objective of the proposed research is to develop a ground-based experiment for measuring the nucleation and growth rates of solid particles formed during expansions of supercritical fluid mixtures.

DESCRIPTION:
1. Determine experimental parameters for supercritical expansion processes. These include thermodynamic process paths, equilibrium solubilities, particle size and growth rate estimates.
2. Make optical measurements of solid particle nucleation and growth rates in fluid mixtures of CO₂ and naphthalene that are initially prepared in a supercritical state.
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.

SIGNIFICANCE:
These measurements will contribute to the fundamental understanding of how particle formation and growth occurs in rapidly expanded supercritical solutions. The experiment is intended to lead to the future development of a microgravity experiment.

PROGRESS DURING FY 1993:
The necessary contractual arrangements are still being processed to fund this proposed research which was accepted under the 1991 NRA on Microgravity Fundamental Science.

TASK INITIATION: 4/94  EXPIRATION: 4/97
PROJECT IDENTIFICATION: 674-24-05-69
RESPONSIBLE CENTER: LcRC

II-335
Behavior of Unsteady Thermocapillary Flows

PRINCIPAL INVESTIGATOR: Prof. Marc K. Smith  
Georgia Institute of Technology

CO-INVESTIGATORS:
Prof. J.N. Koster  
University of Colorado

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

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.

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.

SIGNIFICANCE:
This understanding will assist fluid system designers optimize system designs in which thermocapillary flows are important.

PROGRESS DURING FY 1993:

Theoretical Work:
The theoretical portion of this project has concentrated on describing the thermocapillary instability of a thin or liquid layer scales contained in a rectangular enclosure. The assumption that the average thickness of the liquid layer is smaller than the streamwise or spanwise length scales introduces two small parameters called aspect ratios into the description of this flow. A singular perturbation analysis for small values of the aspect ratios allows us to compute the outer solution, which is the flow field away from the sides of the enclosure. The computed velocity field is known in terms of the layer thickness. Then using the kinematic condition on the free surface, we have derived an evolution equation for the shape of the free surface. This is a nonlinear partial differential equation in terms of time and the streamwise and spanwise directions.

To solve the evolution equation, we need boundary conditions along the container walls. These are found by looking at the inner boundary layer problems for the flow near the container walls. Without having to solve for these flows explicitly, we can develop effective boundary conditions for the layer thickness at these walls. The result is a well-posed evolution equation for the shape of the free surface of the liquid layer.

Our analysis of this equation started with finding the steady-state shape for a two-dimensional container. The evolution equation reduces to the one found previously by Sen and Davis. The equation is solved numerically using a spectral method based on a Chebyshev polynomial expansion for the free-surface position. The nonlinearity is dealt with using a Newton-Kantorovich iteration scheme that has been described by Boyd. The numerical solution agrees very well with the exact solution that can be found for this special case.
The next task was to develop the methodology to determine the stability of this steady-state flow. A linear stability analysis was done to form an eigenvalue problem that was solved numerically. The added difficulty here was that the basic state is only known numerically. The result for the two-dimensional container was complete stability as expected.

Presently, we are considering a three-dimensional container. The evolution equation has been extended to the spanwise direction. The effective boundary conditions for the free surface on the sidewalls are being derived. Once these are obtained, the basic state and stability will be found as before. Future work will include considering the geometry of a thin annulus, which is the three-dimensional rectangular container wrapped around once. This will mimic the flow in a float-zone, which is a geometry of some commercial interest. Then we can also consider the flow in a float-zone, which is a geometry of some commercial interest. Then we can also consider the flow in a long narrow cylinder itself. Finally, we plan to include the effects of inertia to leading order in the evolution equation for the free surface. This can be done by using a Karman-Pohlhausen method to approximate the streamwise velocity profile.

Experimental Work:
The goal for this first year is to set-up and begin the experiments on transparent liquids using optical techniques. So far, the optical test cell has been designed and constructed. It is an open, rectangular Plexiglas container about five inches long and half an inch wide. The two ends of the cell are held at different fixed temperatures, while the lower surface, which is Plexiglas, simulates an insulated surface. The container will be filled with a layer of silicone oil to a depth of at most 1 cm. This oil will be driven in a thermocapillary motion by the temperature difference of the ends of the cell.

A significant portion of the resources was devoted to acquisition of the laser doppler velocimeter. This device has been installed in the laboratory and is being configured for use in the transparent liquid experiments.

The visualization of the free-surface deflection is performed by recording the reflection of an incident laser beam on the free surface. As the free surface deflects, the reflected beam moves. The motion of this beam can be directly related to the free surface deflection and orientation. The current technique is able to make a video record of the beam motion during the course of the experiment.

After the transparent liquid experiments are completed, measurements of the velocity field in a liquid metal, probably gallium, will be performed using the x-ray flow visualization technique.

| STUDENTS FUNDED UNDER RESEARCH: |
| PhD Students: 1 |
| TASK INITIATION: 1/93 EXPIRATION: 1/96 |
| PROJECT IDENTIFICATION: 674-24-05-62 |
| RESPONSIBLE CENTER: LeRC |
II. MSAD Program Tasks — Ground-based Research

Discipline: Fluid Physics

Capillary Containment of Liquids in a Microgravity Experiment

PRINCIPAL INVESTIGATOR: Prof. Paul H. Steen
Cornell University

CO-INVESTIGATORS:
No Co-I's Assigned to this Task

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

OBJECTIVE:
The objectives of this research are to explore the range of conditions for which fluid motion can enhance the stability of a free or partially-free body of liquid against capillary instabilities, and to determine the motions generated when a geometrical configuration becomes unstable and results in rupture.

DESCRIPTION:
Linear stability analysis is used to predict stability conditions, and experiments attempt to confirm theory in a plateau apparatus. Auxiliary experiments and theory will focus on aspects of stability and capture in thin-film systems.

PROGRESS DURING FY 1993:

This study has resulted in:
1. The discovery and mapping out, in a variety of contexts, of the windows in parameter space where hydrodynamic shear forces can stabilize, according to linear stability theory, capillary break-up in long cylindrical interfaces; rod flow (isothermal and thermocapillary-driven); tube flow; and core-annular rod flow. In particular, the physical mechanism of stabilization has been identified.

2. The designing, building, and testing of an apparatus capable of shear-stabilization in a parameter range where theory suggests stabilization may occur. Experiments show reasonable agreement with theory. In the course of the experimental investigation, a pressure-drop method of fine-tuning neutral buoyancy has been discovered.

3. The development of a simpler analog experimental system, the soap-film bridge, by means of which many of the fundamental influences of motion on stability may be studied. The soap film bridge has illustrated details of the collapse phenomenon never before documented.

STUDENTS FUNDED UNDER RESEARCH:

Total Students: 2

TASK INITIATION: 1/93  EXPIRATION: 12/95
PROJECT IDENTIFICATION: 674-24-05-27
RESPONSIBLE CENTER: LeRC
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, Description & Significance:
Objective:
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. The objective is to identify these circumstances theoretically by solving linear and nonlinear stability problems, and to try to locate them in experiments conducted with a dynamic Plateau apparatus.

Description:
For the approach, linear stability analysis is used to predict stability conditions, and experiments attempt to confirm theory in a Plateau apparatus. Auxiliary experiments and theory focus on aspects of stability and capture in thin-film systems.

Significance:
Overall, the goal of this work is an understanding of the influence of flow on shape. The breakup of liquid bridges and columns, and the coalescence of droplets, occurs in technologically important processes involving—among others—phase separation near the critical point of liquid mixtures, quenching of miscibility gap molten metal alloys, and float-zone crystal growth. Motion often influences the crucial shape formation process in application.

Progress During FY 1993:
1. The theory of static capillary surfaces has been applied to liquid bridge configurations beyond the Plateau-Rayleigh limit. An analysis of multiply unstable states (i.e. states with multiple modes of instability) gives insight into what must be done in order to stabilize those states. While developing a scheme for classifying shapes, a wavenumber invariance property of configurations has been discovered.

2. Experiment shows excellent agreement with theory for a range of parameters. On the other hand, the predicted window of stabilization is rather narrow and modifications to the apparatus are required to reach parameters most favorable to stabilization. To date, limited stabilization has been achieved experimentally.

3. Capillary breakup involves the change from a connected to a disconnected state. Fundamental difficulties remain in the quest to capture the trajectory in state space that carries systems through topological change. These issues have been studied in the context of the collapse of a soap-film bridge. A self-similar dynamics of the collapse has been documented and a prediction of satellite bubble size has been developed. These results represent a step towards resolving the fundamental difficulties and have technological implications (i.e. they are relevant to a broad class of systems).

Task Initiation: 1/93 Expiration: 1/96
Project Identification: 674-24-05-57
Responsible Center: LeRC
BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:

Proceedings:
Interactions of Bubbles and Drops in a Temperature Gradient

Principal Investigator: Prof. R. Shankar Subramanian
Clarkson University

Co-Investigators:
Dr. R. Balasubramaniam
NASA Lewis Research Center (LeRC)

Task Objective, Description & Significance:

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. Both theoretical and experimental research is planned on the interactions of drops with each other or with a neighboring boundary.

Description:
The emphasis of the initial effort is on the design, fabrication and set up of the experimental hardware, and to check its performance.

Significance:
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.

Progress During FY 1993:
A search was conducted of potential liquid-liquid pairs for this experiment. These pairs must satisfy requirements regarding densities, immiscibility, interfacial tension, and refractive index. Two systems were identified as promising candidates.

The video equipment for recording the behavior of the drops is being upgraded. Also, work is underway on identifying the proper choice of injection system to deliver drops of the desired size range. A new test cell is being designed for the conduct of the experiments.

A new digital thermometer and a data acquisition system were purchased to replace obsolete/broken equipment. Also, some computer programming was done to customize the data acquisition system.

Task Initiation: 3/93  Expiration: 2/96
Project Identification: 674-24-05-58
Responsible Center: LeRC
**Instability in Surface-Tension-Driven Benard Convection**

**PRINCIPAL INVESTIGATOR:** Prof. Harry L. Swinney  
University of Texas, Austin

**CO-INVESTIGATORS:**  
Prof. W.D. McCormick  
University of Texas, Austin  
Prof. J.B. Swift  
University of Texas, Austin

**TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:**

**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.

**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.

**PROGRESS DURING FY 1993:**  
Preliminary qualitative observations of the onset of convection in thin layers (Ar≈30) indicate that the transition occurs at M≈80, in accord with predictions of linear stability analysis but contrary to an earlier report of onset at much smaller M.

Very qualitative measurements were also made on a 0.2 mm thick layer (Ar≈250) of various viscosities to study the long wavelength instability. The measurements will now be made more precise to determine whether the instability arises at the predicted value of M≈1 for the conditions of the experiment, and to examine the post-onset behavior of the convection.

**TASK INITIATION:** 12/92  
**EXPIRATION:** 11/95  
**PROJECT IDENTIFICATION:** 674-24-05-41  
**RESPONSIBLE CENTER:** LeRC
Crystal Growth and Fluid Mechanics Problems in Directional Solidification

PRINCIPAL INVESTIGATOR: Prof. Saleh Tanveer
CO-INVESTIGATORS:
Dr. G. Baker
Dr. M. Foster

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

OBJECTIVE:
Most of the effort in this task will be in trying 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.

DESCRIPTION:
The effort will primarily consist of a numerical study of possible bifurcations of steady-state solutions that may be steady or oscillatory in two or three dimensions.

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 1993:
During the period of support (January 1, 1993-present), very substantial progress has been made in two different aspects of crystal growth problem.

A. Dendritic Crystal Growth
Most of the current work in existing literature is based on finding a steady state solution and working out its linear stability properties. It is generally agreed that with isotropic surface tension there are no steady state solutions that are close to the well-known Ivantsov parabola. Anisotropy in surface tension causes selection. It is surmised by some that the convective linear instability of noise superposed on the steady solution leads to dendrite micro-structures. However, such theories ignore nonlinear interactions that leads to coarsening and can perhaps explain a steadily propagating tip and side branching as of a time dependent phenomenon, not relying on any underlying steady state assumption. Along these lines, we have made specific progress in two limits of small and large Peclet numbers.

In the small Peclet number limit, we (Kunka, Tanveer, Foster, 1993) have derived equations valid in various regions of the temperature field, without resorting to an adhoc quasi-stationary assumption. In an $O(1)$ region around the tip, we have found that the nonlinear time dependent problem allows exact solutions that describe both tip splitting and side-branching when surface tension is neglected. Further, in a limited way, we have also examined how a small nonzero surface tension changes such solutions. In the limit of small surface tension, we find that all complex singularities move towards the physical domain, which is the source of extreme sensitivity to initial conditions.
II. MSAD Program Tasks — Ground-based Research

Discipline: Fluid Physics

In the large Peclet number limit (Foster, 1993), for a specific ordering of the size of disturbance (super-posed on an Ivantsov solution) and inverse Peclet number, we have reduced the evolution problem to a second-order nonlinear partial differential equation in one space variable. Again, there is no resort to a quasi-stationary hypotheses. Further, we find that the partial differential equation changes nature from elliptic (ill-posed) to hyperbolic in the presence of surface tension.

B. Bridgman Crystal Growth

In this problem, we (Tanveer, 1993) have continued our previous efforts and have obtained explicit expressions for radial segregation and interfacial slope in the limit of large thermal Rayleigh-number. We have also suggested a heat transfer criterion that will minimize these quantities.

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BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:

Proceedings:
Foster, M.R. Temporal development of a needle crystal for large peclet number. (in preparation).

Oscillatory/Chaotic Thermocapillary Flow Induced by Radiant Heating

**Principal Investigator:** Dr. Robert L. Thompson  
**NASA Lewis Research Center (LeRC)**

**Co-Investigators:**
- Prof. K. DeWitt  
- Dr. K. Hsieh  
- D. Van Zandt  
- University of Toledo  
- Sverdrup Technology, Inc.  
- ADF

**Task Objective, Description & Significance:**

**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 the 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.

**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 heat 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.

**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.

**Progress During FY 1993:**
A series of experiments has been conducted to study the onset of oscillatory flows. The critical power of the CO₂ laser at the onset point has been measured at various aspect ratios and dynamic Bond numbers. It was found that, for fixed beam to chamber diameter ratio and fixed Bond number, the critical power decreases when the aspect ratio increases. In addition, the critical power increases with the Bond number as other parameters are fixed. Currently, it is intended to identify the domain of unstable flow on the map of aspect ratio and Bond number. In the meantime, the critical Marangoni numbers corresponding to each point in the unstable domain are being measured.

In parallel to the experimental work, a numerical program for predicting the onset conditions of the oscillatory flow is being developed. The methodology is based on the linear stability analysis. Currently, the numerical solutions have compared well with the analytical results in cases of Benard-Marangoni instability of an infinite layer and the stability of plane Poiseuille flow.
II. MSAD Program Tasks — Ground-based Research

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II. MSAD Program Tasks — Ground-based Research

Discipline: Fluid Physics

Transport Processes Research

PRINCIPAL INVESTIGATOR: Dr. Robert L. Thompson

NASA Lewis Research Center (LeRC)

CO-INVESTIGATORS:
No Co-I's Assigned to this Task

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

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.

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.

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 1993:

In the area of thermocapillary bubble/drop migration, asymptotic results for the bubble velocity that were previously obtained for large Marangoni numbers have been extended to higher orders. The temperature distribution around a migrating bubble has been numerically calculated when the flow field is represented as potential flow. Interferometry has been used to obtain the temperature field in a stably stratified liquid layer distorted by a settling solid sphere.

Measurement capabilities of the LeRC Fluid Physics Laboratory were used to support multiple flight projects. Accurate thermophysical property data such as surface tension (for both liquid-gas and liquid-liquid surfaces) and viscosity with temperature were produced for fluids used in several flight experiments. Optical techniques were developed for particle tracking in fluid flows.

Tests were conducted in the drop tower to examine evaporation from a capillary interface. Unfortunately, the drop tower was closed for refurbishing before suitable results could be obtained.

Analysis was continued of the results of tests in the LeRC drop tower examining the effects of expansions and contractions on water-air two-phase flows.

Extensive tests were conducted in the drop tower to examine both capillary flow in wedges and capillary flow driven by surface wettability. Results to date qualitatively agree with the limited existing predictions but also clearly show the need for both additional tests and extended theories.
II. MSAD Program Tasks — Ground-based Research

An asymptotic analysis was performed to predict characteristic times for fluid reorientation in vessels with interior corners. This is an important parameter for many fluid processes in low gravity.

Analysis was made to predict the wetting speed of a liquid film flowing over a heated flat plate.

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<th>STUDENTS FUNDED UNDER RESEARCH:</th>
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**BIBLIOGRAPHIC CITATIONS FOR FY 1993:**

**Journals:**


**Proceedings:**


**NASA Technical Memoradums:**


**Presentations:**
II. MSAD Program Tasks — Ground-based Research

Discipline: Fluid Physics

Marangoni-Bernard Instabilities in Liquid Layers with Small to Medium Aspect Ratios.

PRINCIPAL INVESTIGATOR: Dr. Robert L. Thompson

NASA Lewis Research Center (LeRC)

CO-INVESTIGATORS:

Dr. J.C. Duh

Sverdrup Tech., Inc

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

OBJECTIVE:
The objective is to determine the critical Marangoni number at the onset of Marangoni-Bernard instabilities (MBI) in liquid layers with small to medium aspect ratios, and to study the post-onset evolution of Bernard convection.

DESCRIPTION:
1. A combined numerical and experimental effort is conducted to study the MBI problem;
2. The experimental study is focused on determining the critical Marangoni numbers;
3. The numerical approach is to simulate the post-onset evolution of convection pattern.

PROGRESS DURING FY 1993:
The experiment apparatus has been integrated. Shadowgraph pictures of the hexagonal pattern were obtained. An IR imaging technique was tested to measure the surface temperature profile and the flow pattern; it also produced satisfactory results.

The numerical investigation of the onset of convection in a liquid layer with small to medium aspect ratio has been completed.

TASK INITIATION: 10/92  EXPIRATION: 9/93

PROJECT IDENTIFICATION: 307-51-00

RESPONSIBLE CENTER: LeRC

BIBLIOGRAPHIC CITATIONS FOR FY 1993:

NASA TM:

Proceedings:
II. MSAD Program Tasks — Ground-based Research

Discipline: Fluid Physics

Computational Studies of Drop Collision and Coalescence

PRINCIPAL INVESTIGATOR: Prof. Gretar Tryggvason
University of Michigan

CO-INVESTIGATORS:
Dr. D. Jacqmin
NASA Lewis Research Center (LeRC)

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

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.

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.

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 1993:
We have conducted extensive computations of the head-on collision of drops, establishing the dependency of the evolution on the governing parameters and, in particular, the sensitivity to the time of coalescence. The simulations show that it is important to predict this time accurately, and currently we are working on a "subgrid" model to predict this time in a physically correct way. We have also initiated work on non-head-on collisions of drops where the evolution is fully three-dimensional.

The thermal migration simulations are still two-dimensional. We have focused on the collective behavior of many drops and found rather remarkable pattern formation processes where the drops line up across the channel as they migrate toward the hot wall. This phenomenon, which has not been found before, has far-reaching implications for material processing.

Next year our focus will be mainly on the fully three-dimensional counterparts of these two problems.

STUDENTS FUNDED UNDER RESEARCH:
PhD Students: 2

TASK INITIATION: 1/92  EXPIRATION: 1/95
PROJECT IDENTIFICATION: 694-24-05-07
RESPONSIBLE CENTER: LeRC
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, DESCRIPTION & SIGNIFICANCE:

OBJECTIVE:
This research program focuses on 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.

DESCRIPTION:
The high-frequency components of the residual acceleration field are modeled as a stochastic or random process; that is, as a succession of random values of the intensity and orientation of the acceleration. Our research is divided into two major parts, one analytical and the other numerical in character. In the first part, a hydrodynamic problem is formulated that explicitly includes a random, time-dependent gravitational acceleration that is modeled as a narrow-band noise. In the second part, numerical algorithms are developed to simulate the random fields and incorporate them into Navier-Stokes equation solvers.

PROGRESS DURING FY 1993:
We have completed the study of the linear stability of a free fluid surface under a fluctuating gravitational field. We have found that the stability analysis can be reduced, in the underdamped limit, to studying the equation of the parametric harmonic oscillator for each of the Fourier components of the surface displacement. Analytic results for the stability of the second moments of the stochastic parametric oscillator have been obtained in the limits of low frequency oscillations, and near the region of subharmonic parametric resonance. We have obtained that an external driving force with a broad frequency spectrum leads to parametric resonance in a wide frequency range. The resonant behavior is not given by the superposition of the resonances produced by each of the frequency components because of the nonlinear coupling between the external force and the oscillator coordinate. Furthermore, the resonant behavior of the second moments is in general weaker than the resonance that would result from any of the frequency components acting alone, at equal area of the power spectrum. Our results apply to other cases in which the linear stability analysis reduces to the Mathieu equation (e.g., the stress-free Rayleigh-Benard problem or thermosolutal convection during directional solidification in a time dependent gravitational field).

A numerical scheme has been developed in integrate the equation of the parametric oscillator driven by narrow-band noise. The method developed is explicit and second order in time, and treats the stochastic contribution exactly. The results of the numerical integration agree very well with the analytic predictions for the stability boundaries of the second moments of the harmonic oscillator.

The cases discussed above involve systems in which the density of the fluid is uniform everywhere except in narrow regions at which the fluid density is large (e.g., interfacial regions). We have extended our study to include systems in which there is a smooth density gradient across the fluid, perhaps due to temperature or composition gradients. The configuration that we have analyzed is that of cavity flow in a laterally heated container. A simplified analytical model has been derived to study the onset of macroscopic fluid flow due to g-jitter, as well as its characteristic onset time and velocity scale as a function of the parameters of the noise. A numerical algorithm to solve the Boussinesq equations with a stochastic gravitational field has been developed and is being currently used to study this type of flow.
II. MSAD Program Tasks — Ground-based Research

**Students Funded Under Research:**

- Total Students: 2
- Total Degrees: 2

**Task Initiation:** 6/91  **Expiration:** 5/94

**Project Identification:** 674-24-05-36

**Responsible Center:** LeRC

**Bibliographic Citations for FY 1993:**

**Journals:**


**Proceedings:**


Studies of the Dynamics of Charged Free Drops

Principal Investigator: Dr. Taylor G. Wang
Vanderbilt University

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

Task Objective, Description & Significance:

Objective:
The dynamic behavior of a charged liquid drop has been the subject of investigation. The basic assumption is that the charge essentially reduces the surface tension of a neutral drop; hence the shapes and stabilities of a liquid drop are greatly affected by the amount of charge presented on the drop surface. Various theories have been developed to predict the dynamic responses of a drop under different charge conditions.

However, two major obstacles make bias-free experiments rather difficult to achieve for ground-based experiments. One is the determination of charge quantity on a liquid sample. The proposed research objectives to be attained at Vanderbilt University in ground-based research are to optimize flight experiments and to advance our fundamental understanding of the dynamics of charged drops.

Description:
The study of the oscillation and rotational dynamics of charged liquid drops sustained by surface tension is interesting from both a fundamental standpoint and a practical standpoint vis-a-vis processing materials in space. The proposed ground-based experiments will help us to further the understanding of drop behavior and to optimize the approved flight experiments.

One of the major technical problems facing all charged-drop experiments is the determination of charge quantity on a liquid sample. To avoid the contamination and source loading problems, a noncontact charge-measuring technique is preferred. From basic physics, there is only one way to determine the potential of a charged object by noncontact means, and that is to measure the strength of the electrostatic field generated by the charge object. With precise calibration based on the geometry of the system and measuring distance, the field strength can be used to obtain the potential of the charged object.

Progress During FY 1993:
Ground-based support studies on oscillating and rotating charged drops are underway at Vanderbilt University in an acoustic levitation chamber.

Study 1.
The dynamics of drop oscillation. The present experiments are predominantly concentrating on studying the dynamics of charged free drops with negligible momentum interaction with the surroundings and drops with uniform surface charges. Acoustic forces will be solely used to levitate such drops. In addition, an immiscible levitation system will also be set up for making measurements on internal fluid flows in oscillating drops. Extensive flow visualization will be used for both qualitative and quantitative studies of drop dynamics.

Study 2.
The dynamics of charged drop oscillation. A very-high-resolution charge-measurement system was developed at Vanderbilt University last year. This charge-measurement system has been incorporated into the ground-based
acoustic levitator to study the dynamics of charged drop oscillations. A high-speed video system was used to determine the forced shape oscillation.

This year, variations of the drop oscillation amplitude decay changes in maximum oscillation amplitude viscosity and charge density will be examined. The freely decaying oscillations of charged drops on removal of acoustic excitation will be characterized. The effect of drop viscosity on drop behavior will be examined by using more viscous fluids like glycerine. Special attention will be devoted to the controlled study and characterization of the instabilities leading to mode coupling and to drop fission during large-amplitude oscillations.

Study 3. The dynamics of charged drop rotation. Rotation experiments will be performed on charged free drops. Earlier rotation experiments on drops in an acoustic levitation system have shown good agreement with theoretical predictions. However, the experiments on the rotating drops have shown only axisymmetric and 20 lobed shapes. It is hoped that charged drops can be stably levitated over a reasonable length of time so that rotation experiments can be performed on them which will provide additional insights into this phenomena. Initially, a high viscosity fluid, like glycerin, will be used in the experiments. Drops will be rotated by acoustic torque.

Study 4. Conduction of combined oscillation and rotation experiments on charged drops. The objective of the ground-based studies will be to perform simultaneous oscillation and rotation experiments on charged free drops and to provide a scientific information base for future SpaceLab experiments.

Study 5. Development of internal flow field diagnostics technology. An immiscible drop levitation system was set up for the quantitative study of internal fluid motion in large drops (1 cm diameter). The proposed flow visualization technique will use the afterglow of UV excited phosphorescent tracer particles to retrieve vectorial information on particle motion. Zinc sulfide-coated, quasi-neutrally buoyant polystyrene particles (~50 μm diameter) will be used as tracer. A UV strobe sheet or a pulsed nitrogen laser sheet (~1 mm thickness) will be used to illuminate and excite the tracer particles in the center plane of the drop. Both real-time video imaging and time-lapsed photography will be used to study internal motion.

STUDENTS FUNDED UNDER RESEARCH:

Total Students: 1

PROJECT IDENTIFICATION: 674-24-07-09

RESPONSIBLE CENTER: JPL
Experimental Study of the Vapor Bubble Thermosyphon

**Task Objective, Description & Significance:**

**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 is one such system, consisting 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.

**Description:**
A transparent VBT will be designed and developed. The microscopic intermolecular force field, a function of the liquid thickness profile, will be measured using microcomputer-enhanced video microscopy based on ellipsometry and interferometry. The temperature field will be measured using the interline absorbed film thickness and small temperature sensors.

Models of the transport processes in a VBT that include the effects of liquid-solid and liquid-vapor intermolecular forces have already been developed. As a 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.

**Significance:**
By studying liquid-film thicknesses and temperatures in a VBT, a better understanding of the process can be gained that will lead toward optimization of VBT designs.

**Progress During FY 1993:**
A preliminary experimental study of vapor bubble thermosyphons has been accomplished. In it measurements of wall temperatures (using thermocouples) and liquid film thicknesses (using an image analyzing interferometer) were made to characterize behavior of the VBT; valuable experience was gained in the operation of such systems. Difficulties were found in finding a proper fluid and container material combination so that the system will have desirable characteristics both optically and thermally. Proper procedures for filling the cells have been found to be important in avoiding contamination. VBT cells for the main experiment have been designed and are currently being constructed.

Some preliminary analytical studies have been performed of the processes occurring in VBTs and the physical mechanisms that limit heat transfer.
II. MSAD Program Tasks — Ground-based Research

**Discipline:** Fluid Physics

<table>
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<th>STUDENTS FUNDED UNDER RESEARCH:</th>
<th>TASK INITIATION: 12/92</th>
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**PROJECT IDENTIFICATION:** 674-24-05-44

**RESPONSIBLE CENTER:** LeRC
Interactions Between Solidification and Compositional Convection in Alloys

Principal Investigator: Prof. M. Grae Worster
Northwestern University

Co-Investigators:
Prof. S.H. Davis
Northwestern University

Task Objective, Description & Significance:

Objective:
The project objective is 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 interslices of mushy layers during solidification.

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, as well as asymptotic and numerical methods, in the development and analysis of predictive mathematical models.

Progress During FY 1993:
Progress has been made in the analysis of the stability of flow through mushy layers driven by expansion during solidification. Such convection is an inevitable consequence of solidification and cannot be avoided by manufacturing materials in space. Although the potential for instability has been proved, it has been found that it is unlikely to occur under typical casting conditions. A suite of experiments has been completed in which the degree of macro-segregation caused by compositional (buoyancy-driven) convection has been quantified. A combined experimental and theoretical study has elucidated important interactions between interfacial undercooling and buoyancy-driven convection that help to explain the propensity for chimneys (freckles) to occur during casting of certain alloys.

Students Funded Under Research:

Task Initiation: 1/93  Expiration: 12/96
Total Students: 1

Project Identification: 674-24-05-63
Responsible Center: LeRC

Bibliographic Citations for FY 1993:

Journals:


II. MSAD Program Tasks — Ground-based Research  Discipline: Fluid Physics

Proceedings:

Presentations:
II. MSAD Program Tasks — Ground-based Research

Discipline: Fluid Physics

Nucleation and Chiral Symmetry Breaking under Hydrodynamic Flows

Principal Investigator: Dr. Xiao-Iun Wu

University of Pittsburgh

Co-Investigators:

No Co-I's Assigned to this Task

Task Objective, Description & Significance:

Objective:
The research objective is designed to conduct an experimental investigation of the nucleation and aggregation phenomenon in hydrodynamic flows. Non-linear theories of irreversible states for nucleation will be applied to explain the favored direction or chirality of flow effects. The experimental data and theory will couple and develop a new explanation for fluid flow effects in aggregating model systems.

Description:
The chiral symmetry breaking during crystallization of simple inorganic molecules under different flow conditions will be explored. Earth-based experiments indicate that the symmetry breaking is a result of the rarity of nucleation events. However, in order to produce the extremely high chiral purity (> 95%) observed in the experiments other mechanisms must also be involved, but as yet these effects are not well understood. In particular, the mechanism by which hydrodynamic flow causes the proliferation of one chiral species while suppressing the other species remains a challenge. Earth-based experiments conducted in the laboratory have been helpful in discriminating different effects, but the results were partially obscured due to gravity. Sedimentation dominates convection at an early stage of growth so that crystals settle before they grow to an appreciable size.

Research Approach — The research to be carried out during the first year will be to develop the new detection and analysis techniques and to test their effectiveness using a range of model solutes under conditions which can be studied effectively in a normal gravity environment. The two subsequent years will be devoted to developing an important data base on interfacial transport characteristics and to adapting the entire system to be compatible with whatever constraints are imposed by NASA on the experimental rig to be sent on a space shuttle mission. Concurrent with these developments, the theoretical modeling will be pursued. The research study consists of experimental and (a limited amount of) theoretical work necessary to prepare a Science Requirements Document (SRD) for microgravity experiments. It appears that the need for a microgravity environment can be justified. Nevertheless, the possibility that some of the work can be accomplished in drop towers will be investigated, which may be feasible using systems with short relaxation times. For this purpose, less viscous fluids will be essential. The study will focus on:

• The use of ionic surfactants to control the conductivity of low dielectric constant liquids. This will provide a means to explore the role of fluid conductivity in the deformation of drops and the stability of fluid cylinders.

• The stability of fluid cylinders in longitudinal fields with isopycnic systems. This will furnish experience with this geometry and provide some information to test the leaky dielectric model for the stability of fluid columns. To the extent possible, ionic surfactants to control fluid conductivity will be used.

• The deformation of droplets using fluids whose conductivity has been adjusted using a non-ionic surfactant. Nucleation and chiral symmetry breaking of sodium chlorate under different flows will be studied experimentally. In the first year, the research will concentrate on measurements of chiral symmetry breaking as a function of nucleation rate. The central issue here is whether the symmetry breaking transition a continuous or an abrupt one.
By improving statistics of our measurements, we hope to answer this question quantitatively. In the second year, experiments will be performed in Couette cells, and chiral symmetry breaking will be measured in different flow regimes, laminar, Taylor vortex flow and turbulence, using these cells. The experiment is not perfect at this stage because the nucleation process is still influenced by sedimentation effect. However, one should be able to observe qualitatively different features of nucleation and chiral symmetry breaking associated with various flow regimes. On the third year, the experimental apparatus, consisting of ten identical Couette cells and a temperature-controlled bath, should be ready for the space mission. With gravitational effect greatly reduced, we hope to demonstrate in the cleanest way that chiral symmetry breaking is a result of competition between nucleation and convection.

SIGNIFICANCE:
By conducting the experiment in space, one greatly minimizes the role of gravity. The chiral symmetry breaking in this case will be due only to the competition between the nucleation rate and convection. An interesting and important aspect of the experiment is to determine if the symmetry breaking transition is a continuous or an abrupt transition as the experimental parameters, such as the flow rate and the degree of supersaturation, are varied. This study will enrich the general knowledge of the nucleation and growth process under hydrodynamic flow and may lead to the production of chirally pure materials.

PROGRESS DURING FY 1993:
Hydrodynamic studies of non-steady nucleation are under way. Cylindrical geometries are receiving the most intensive early investigations.

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BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:


II. MSAD Program Tasks — Ground-based Research

Discipline: Fluid Physics

Presentations:


Wu, X. Shear-induced concentration fluctuations in polymer solutions, Department of Physics, Indiana University of Pennsylvania, Indiana, PA, October, 1992.

Wu, X. Slow dynamics of nematic/isotropic phase transition in silica gels, Liquid Crystal Institute, Kent State University, Kent, OH, September, 1992.
Oscillatory Thermocapillary Convection

PRINCIPAL INVESTIGATOR: Prof. Abdelfattah Zebib, Rutgers University

CO-INVESTIGATORS:
No Co-I's Assigned to this Task

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

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.

DESCRIPTION:
In the domain perturbation approach (valid for small capillary numbers \( \text{Ca} \approx 0 \)), 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 \( \text{Ma} = 0 \)) is known to exhibit a Hopf bifurcation at some critical value of the Grashof number, \( \text{Gr}_{\text{cr}} \). Thus, by studying the combined thermocapillary-buoyant convection for values of

\[
(\text{Ma}, \text{Gr}) \text{ near } (0, \text{Gr}_{\text{cr}})
\]

we seek to determine the stability boundary for onset of oscillatory motion and its nature in the limit of vanishing buoyancy, \( \text{Gr} = 0 \). Both positive and negative values of \( \text{Ma} \) are considered. In the second problem we consider pure thermocapillary driven convection (\( \text{Gr} = 0 \)). The solution to the zeroth order system is known to be stationary. Higher order effects are unknown and will be investigated.

SIGNIFICANCE:
These studies can contribute to our fundamental understanding and potential control of thermocapillary instabilities.

PROGRESS DURING FY 1993:
We have completed the first study and confirmed the stationary nature of thermocapillary convection for a range of Prandtl numbers and aspect ratios. Currently, we are developing computer codes to investigate higher order effects of \( \text{Ca} \). In addition, an alternate domain perturbation approach which explicitly retains the influence of \( \text{Ca} \) on transition is also being developed.

STUDENTS FUNDED UNDER RESEARCH:
Total Students: 1

BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:
II. MSAD Program Tasks — Ground-based Research

Discipline: Fluid Physics

Proceedings:

Presentations:
Zebib, A., Oscillatory convection in crystal growth, Ecole Polytechnique Federale de Lausanne, ETHL, Switzerland March 1993.

II. MSAD Program Tasks — Ground-based Research

Discipline: Materials Science

Foam Metallic Glasses

PRINCIPAL INVESTIGATOR: Prof. Robert E. Apfel

CO-INVESTIGATORS:

R. Adams
C. Boa-Teh

Yale University

Yale University

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

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 materials.

DESCRIPTION:
The experimental program is divided into three major sections: proof of principle with organic liquids that are easy glass formers; tests with the water-tin system, even though it is known that such systems will not form a glass; and 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. Here, electron microscopic analysis of our samples will help to determine the degree of crystal, polycrystalline, and glass formation.

Research Approach — It has long been desired to engineer metallic materials that possess the properties of strength and durability without paying the penalty of excessive weight. To overcome the problems of mechanical failures due to dislocations and grain boundaries and to produce desirable magnetic and electrical properties, materials scientists have experimented with producing metallic glasses through the rapid quenching of thin streams of materials. The ribbons produced have many desirable properties (mechanical and magnetic), but they are not bulk materials, i.e., having as-cast configuration. The research approach here is to produce bulk metallic "foams" by the sudden decompression of a melt that is heavily seeded with a volatile liquid. This dispersed "foaming" liquid will vaporize upon decompression, taking its latent heat of vaporization from the melt, thereby adiabatically and homogeneously cooling it. If the cooling rate is sufficiently great, e.g., one million degrees Celsius per second, the possibility of producing a foam metallic glass exists. The experimental program will be complemented by an analytic/computational study of this highly transient "foaming" process and by comprehensive materials analysis of all product specimens.

There are many constraints in the capabilities of the apparatus, choice of materials, and experimental procedures that will have to be dealt with if we are to have a chance at achieving a successful outcome. We will consider our efforts productive even if we are able to produce a metallic foam with a structure characterized by microcrystallites rather than amorphous structure.

SIGNIFICANCE:
Mechanical and electrical tests with bulk samples and for individual fibers (whiskers) will help to answer questions as to whether such materials will go beyond scientific interest to have practical applications.
II. MSAD Program Tasks — Ground-based Research

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II. MSAD Program Tasks — Ground-based Research

DISCIPLINE: Materials Science

PROGRESS DURING FY 1993:

It has been about 30 years since Paul Duwez and colleagues demonstrated that metallic glasses from the melt can be produced using his "gun technique" if the quench rate is sufficiently rapid, e.g., \(1 \times 10^6\) K/s. Since that time, much experimental and theoretical work has disclosed the conditions necessary to produce and maintain the metallic glass (amorphous) state. David Trumbull has been among the leaders in the field. His work in the late 1940s with metal alloy (mercury) drops and that of Vonnegut with oxide coated tin drops demonstrated that the undercooling of metallic materials followed a path similar to non-metallic materials. Deep undercoolings were possible if heterophase nucleants were either absent or neutralized. Even relatively large samples, e.g., a few grams, could be undercooled if nucleants were removed by appropriate fluxing techniques.

<table>
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<td>RESPONSIBLE CENTER: MSFC</td>
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Memory Effects in the Organometallic Chemical Beam Epitaxy of Compound Semiconductors

PRINCIPAL INVESTIGATOR: Dr. Klaus J. Bachmann  
North Carolina State University

CO-INVESTIGATORS:  
No Co-I's Assigned to this Task

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

OBJECTIVE:  
The goal of this research program was to improve the control of the composition of compound semiconductor heterojunctions during growth via chemical beam epitaxy (CBE). The CBE growth of such materials in a containerless process as offered in space growth is expected to eliminate memory effects due to residual species in the reaction region.

DESCRIPTION:  
ZnSiGeP/GaNP/Si heterojunctions were chosen as a model case. This system is of interest because of its birefringence and nonlinear optical properties.

SIGNIFICANCE:  
This work is not only significant for the improvements in nonlinear optical materials, it is also important as a precursor to molecular beam epitaxy experiments with the wake shield.

PROGRESS DURING FY 1993:  
Alloys of ZnSiGeP as well as alloys of ZnGeP and GaNP have been grown on Si and GaP substrates and extensively characterized. As a result of this work, joint funding has been obtained from the NSF and the Deutsche Forschungsgemeinschaft for a bi-national effort on studying the non-linear optical properties of these materials.

STUDENTS FUNDED UNDER RESEARCH:  
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TASK INITIATION: 3/90  
EXPIRATION: 9/92  
PROJECT IDENTIFICATION: 674-21-06-06  
RESPONSIBLE CENTER: LaRC

BIBLIOGRAPHIC CITATIONS FOR FY 1993:  

Journals:  

Microwave Materials Processing in Microgravity

PRINCIPAL INVESTIGATOR: Dr. Martin B. Barmatz
Jet Propulsion Laboratory (JPL)

CO-INVESTIGATORS:
No Co-I's Assigned to this Task

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

OBJECTIVE:
The Microwave Materials Processing in Microgravity Task objective is to apply the unique capabilities of microwave heating and positioning to process materials in microgravity. The task objectives will include 1) determination of the reaction mechanism, microstructure development and physical properties associated with microwave synthesis of ceramics, 2) development and application of microwave techniques for (a) monitoring the energy absorption during processing and (b) measuring thermophysical properties of materials in microgravity, and 3) theoretical modeling of unique microwave heating and positioning capabilities in a microgravity environment.

DESCRIPTION: There is a recognized need to produce advanced refractory ceramics that have higher melting temperatures and improved mechanical properties (such as strength and toughness). In recent years, ground-based experiments using microwave heating have demonstrated enhanced rates of sintering of ceramic materials leading to new microstructures. The synthesis of ceramics in a microgravity environment could provide the opportunity to produce contamination free ceramics with controlled microstructures that lead to advanced structural applications. Microwave processing can heat many glass and ceramic compositions very rapidly to high temperatures, it can heat them more uniformly than other methods, and it is energy efficient. Of particular interest is the application of microwave to the combustion synthesis process. Other important potential applications are crystal growth, and fiber pulling in space. Microwaves can generate a well defined temperature gradient within a material leading to the possibility of melting only the interior of a cylindrical sample, or leading to a radial gradient of the index of refraction upon solidification. By appropriate monitoring of the microwave parameters during processing one can also measure various sample properties as well as obtain energy absorption information which can be used to characterize the sample reaction and densification mechanisms. Microwaves can also produce unique positioning forces. The ability to microwave heat and position a sample may lead to a new containerless technology that is ideally suited for controlled processing of materials in microgravity.

PROGRESS DURING FY 1993:

Progress to Date:
During this last year, we have (a) developed an advanced transient microwave heating model that calculates the microwave absorption and temperature profile within a sphere as it approaches steady state, (b) demonstrated new technique for determining the dielectric constants of a sphere using our microwave absorption theory, and (c) experimentally measured the spatial dependence of microwave forces needed to containerlessly position a sphere at the center of a cylindrical cavity.

STUDENTS FUNDED UNDER RESEARCH:

PhD Students: 1

TASK INITIATION: 10/81 EXPIRATION: 9/94
PROJECT IDENTIFICATION: 674-25-04-07
RESPONSIBLE CENTER: JPL
BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:

II. MSAD Program Tasks — Ground-based Research

Discipline: Materials Science

Studies of Containerless Processing of Selected Alloys

Principal Investigator: Prof. Robert J. Bayuzick

Vanderbilt University

Co-Investigators:

W. Hofmeister
NASA Marshall Space Flight Center (MSFC)

M. Robinson

Vanderbilt University

Task Objective, Description & Significance:

Objective:
The research is presently focusing on determining the speed of solidification as a function of undercooling in the large undercooling regime.

Description:
This work focuses on the undercooling of refractory metals and alloys by containerless processing. It has both a flight component involving electromagnetic positioning and a ground component involving a continuation of already established drop tube work and electromagnetic levitation (on the bench). Specifics to be addressed are a maximum undercooling in refractory metals and alloys, nucleation frequency in refractory metals and alloys, the effect of undercooling on solidification velocity in refractory metals and alloys, and microstructural development in refractory metals and alloys as a function of deep undercooling/solidification velocity.

Research Approach — The research to be carried out during the first year will be to develop the new detection and analysis techniques and to test their effectiveness using a range of model solutes under conditions which can be studied effectively in a normal gravity environment. The two subsequent years will be devoted to developing an important data base on interfacial transport characteristics and to adapting the entire system to be compatible with whatever constraints are imposed by NASA on the experimental rig to be sent on a space shuttle mission. Concurrent with these developments, the theoretical modeling will be pursued. The research study consists of experimental and (a limited amount of) theoretical work necessary to prepare a Science Requirements Document (SRD) for microgravity experiments. It appears that the need for a microgravity environment can be justified. Nevertheless, the possibility that some of the work can be accomplished in drop towers will be investigated, which may be feasible using systems with short relaxation times. For this purpose, less viscous fluids will be essential. The study will focus on:

• The use of ionic surfactants to control the conductivity of low dielectric constant liquids. This will provide a means to explore the role of fluid conductivity in the deformation of drops and the stability of fluid cylinders.

• The stability of fluid cylinders in longitudinal fields with isopycnic systems. This will furnish experience with this geometry and provide some information to test the leaky dielectric model for the stability of fluid columns. To the extent possible, ionic surfactants to control fluid conductivity will be used.

• The deformation of droplets using fluids whose conductivity has been adjusted using a non-ionic surfactant. Nucleation and chiral symmetry breaking of sodium chlorate under different flows will be studied experimentally. In the first year, the research will concentrate on measurements of chiral symmetry breaking as a function of nucleation rate. The central issue here is whether the symmetry breaking transition a continuous or an abrupt one. By improving statistics of our measurements, we hope to answer this question quantitatively. In the second year, experiments will be performed in Couette cells, and chiral symmetry breaking will be measured in different flow regimes, laminar, Taylor vortex flow and turbulence, using these cells. The experiment is not perfect at this stage...
because the nucleation process is still influenced by sedimentation effect. However, one should be able to observe
qualitatively different features of nucleation and chiral symmetry breaking associated with various flow regimes. On
the third year, the experimental apparatus, consisting of ten identical Couette cells and a temperature-controlled
bath, should be ready for the space mission. With gravitational effect greatly reduced, we hope to demonstrate in
the cleanest way that chiral symmetry breaking is a result of competition between nucleation and convection. This
research focuses on the electrically-driven aggregation of particles and coalescence of droplets. The coagulation of
biological or other solid particles migrating in an electric field will be investigated first; a subsequent study of
droplet coalescence during electrophoresis will be conducted.

In each case, this involves the development of a detailed mathematical description of two-body interactions,
especially for near-contact. These descriptions must account for hydrodynamic interactions arising from the relative
motion of the particles or drops in the solvent, electrokinetic interactions that arise from the convection of fluid
near a charged particle surface (or drop interface), and any significant, non-hydrodynamic interactions such as van der
Waals attraction and electrostatic double-layer repulsion. Ultimately, collision rates are sought that provide the link
between micro- and macro-physics of electrically-driven aggregation. The former is concerned primarily with
two-body interactions, while the latter includes particle/drop size distributions and overall, macroscopic
phase-separation behavior.

Ground-based experiments will be conducted to make observations of electrically-driven particle aggregation and
doncept compared with the theory to be developed. It is anticipated that gravitational effects
encountered in terrestrial experiments will obscure a quantitative comparison with theory for droplet coalescence.
Solidification velocity is measured on electromagnetically levitated specimens by monitoring the progression of the
thermal field developed by recalescence. Velocity and temperature measurement are made with a custom-built
device; it consists of a 1 x 38 lincon array of 4 mm by 0.96 mm photodiodes, two amplification stages, and data
acquisition capability for both velocity and temperature measurements. The time when each detector senses the
solidification front is recorded with a resolution of ten nanoseconds. The surface temperature of the levitated drop
is measured by using the center photodiodes as a pyrometer.

SIGNIFICANCE:
A significant reason for concentrating on refractory metals and alloys is that they are prominent for the wave of the
future. They have potential as high-performance materials and, therefore, are potentially high-value-added
materials—an important characteristic if space processing of materials is to be a reality. Examples of potential are in
the National Aerospace Plane and advanced turbine engine design. There are already vigorous programs across the
nation for such applications, but there is considerable need for a science base to point the way for alloy
development.

PROGRESS DURING FY 1993:
The instrument for measuring velocity and melt temperature was built and applied to studies of pure nickel.
Measurements were made over a range of undercooling from 8% of the equilibrium freezing point to 18%. At
undercoolings less than 10% of the equilibrium temperature, the results agree in form with previous results but
markedly disagree at undercoolings greater than 10%. Previously reported results in the literature overestimate the
solidification velocity of nickel at the large undercooling.

The behavior of Nb-Si alloys has been studied under rapid solidification processing conditions. A combination of
bulk undercooling in a levitated coil with subsequent splat quenching was used to produce microstructural
modifications in these alloys.
II. MSAD Program Tasks — Ground-based Research  
Discipline: Materials Science

STUDENTS FUNDED UNDER RESEARCH:
PhD Students: 1  PhD Degrees: 1

TASK INITIATION: 2/89  EXPIRATION: 2/94
PROJECT IDENTIFICATION: 674-25-08-20
NASA CONTRACT NO.: NAG8-765
RESPONSIBLE CENTER: MSFC

BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:

Presentations:
Transport Phenomena During Equiaxed Solidification of Alloys

**PRINCIPAL INVESTIGATOR:** Prof. Christoph Beckermann  
**University of Iowa**

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

**TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:**

**OBJECTIVE:**
The objective is 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.

**DESCRIPTION:**
Experiments will be conducted with a detailed consideration of the theoretical model. A close coupling between the modeling and experimental efforts is critical to the successful development of each. The goal is to achieve agreement between accurate, unadulterated experimental data and numerical predictions employing un tampered physical property data and real experimental conditions as input to the simulations.

**SIGNIFICANCE:**
This investigation complements NASA-sponsored research on the influence of fluid flow on 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 the transport and towards a more complete numerical simulation model of the transport and solidification phenomena, and (3) a base for defining future microgravity flight experiments.

**PROGRESS DURING FY 1993:**
The objective is to study the macrosegregation and structural inhomogeneities resulting from gravity-induced thermosolutal melt convection and solid sedimentation during equiaxed solidification of metal alloys on a bulk level. Through combined experimental and theoretical work, a numerical simulation model is being developed that allows for the calculation of the individual solid and liquid motions and incorporates the detailed phase interactions on a microscopic scale. Progress has been made in all of the originally proposed tasks:

1. Solidification Experiments
   1. Metal Alloys (NASA Lewis)
   The experimental design was finalized according to the model needs. A student from the University of Arizona was hired for three months during the summer to initiate the fabrication of the test apparatus and casting experiments using Pb-Sn alloys. Crucibles and thermocouples were designed and built to perform well instrumented experiments suitable for input and comparison to the solidification model. Modifications to the Bulk Undercooling Furnace were made to accommodate the added instrumentation. A successful feasibility experiment was run using a 99.999% pure Pb-15 wt% Sn alloy. Cooling rates and thermal gradients were measured along the center of the ingot and along the inner edge of the crucible. Later, the crucible temperature measurements will be used as input to the solidification code and the temperatures measured along the ingot center compared to the code results. A report was prepared.
II. MSAD Program Tasks — Ground-based Research

II. Measurements of Interfacial Transport Coefficients

1. Drag Coefficient of Fabricated Dendrite Models (UC)
   Data from previous settling experiments using fabricated dendrite models were analyzed and newly correlated (see publications below). Additional settling experiments with variation of the dropping angle are underway.

2. Drag, Heat Transfer and Mass Transfer Coefficients of Transparent Model Alloy Dendrites (UI)
   The drag coefficient measurements are completed. The data were found to correspond well to the fabricated dendrite data and the newly developed drag coefficient correlation (see above). Work is almost completed to extend the correlation to multiple crystals by utilizing existing permeability measurements for packed beds of equiaxed crystals.

   A new settling column was designed and constructed that allows for precise control of the temperature and, hence, undercooling of the melt contained within it. This column is presently being used to measure the heat/mass transfer coefficients of NH₄Cl dendrites that grow while they are settling. Progress was also made to develop a suitable correlation of the data for use in the numerical simulation model.

III. Model Studies (UI)

The existing two-phase model was extended to dendritic growth by introducing a new multiphase/multiscale modeling approach. Successful validation of the model was achieved for diffusion-dominated equiaxed and columnar dendritic solidification, and previously measured columnar-to-equiaxed transitions were predicted. This model is now being generalized to include convection and solid transport. For this purpose, the present implementation of the two-phase model within the PHOENICS commercial CFD code is being modified. Promising initial results have been obtained, although much work remains.

STUDENTS FUNDED UNDER RESEARCH:

Total Students: 1

BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:

II. MSAD Program Tasks — Ground-based Research


**Presentations:**

Gravitational Effects on the Development of Weld-Pool and Solidification Microstructures in Metal Alloy Single Crystals

Principal Investigator: Dr. Lynn A. Boatner

Co-Investigators:
S. David
G. Workman

Task Objective, Description & Significance:

Objective:
The objectives of this research are to achieve an in-depth quantitative understanding of the role played by convection-driven heat and mass transport in determining the shape of fusion weld pools, and thereby controlling the nature of the solidification processes that determine weld microstructures.

Description:
By combining ground-based results with those obtained in the low-g and high-g environments provided by KC-135 flights, detailed microstructural information can be obtained by using alloy single-crystal weld specimens, and gravitational effects on weld-pool formation can be delineated and quantified. The results of these investigations are expected to yield not only fundamental advances in our level of understanding of convection phenomena as they relate to weld pool shapes, but, since weld mechanical properties depend on the weld microstructures, the results of the proposed research can have practical applications to construction operations in space.

Research Approach — This research investigation 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 a recently-developed single crystal approach to the delineation of solidification microstructural properties and the new quantitative analytical methods that have been developed in conjunction with this new experimental approach. 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 previous ground-based studies, Czochralski-grown single crystals of the pure ternary alloy 70Fe-15Ni-15Cr (a compositional analog of one of the 300-series stainless steels) will be utilized.

Weld specimens are prepared by orienting the stainless steel single crystals and spark erosion cutting slices of the material such that the planar faces of the samples represent principal crystallographic planes. The principal crystallographic directions lying in these principal planes are then identified using Laue back reflection x-ray methods, and these directions are marked on the sample surface. The resulting oriented specimens are then autogenously welded using either e-beam or laser heating so that the heat source moves parallel to a principal crystallographic axis.

Significance:
The long-term goal of this effort is to provide a firm scientific basis for the development of a comprehensive program of more complex welding and solidification experiments that would be carried out on the KC-135 aircraft, on space shuttle flights, and eventually on Space Station Freedom.
II. MSAD Program Tasks — Ground-based Research

Discipline: Materials Science

**PROGRESS DURING FY 1993:**

Recent investigations, in which the polycrystalline alloy specimens normally used in welding and solidification investigations have been replaced by oriented alloy single crystals, have led to a new level of understanding of how the microstructural development of single-crystal welds is determined by the weld pool shape. These new experimental results have led to the development of analytical modeling capabilities to the point where experimentally-observed dendritic microstructures in single-crystal welds can now be predicted from either mathematical simulations of the weld pool shape or an experimentally-determined weld pool shape. Conversely, methods for calculating the shape of the weld pool, beginning with the experimentally-observed microstructures, have been developed.

Large (-4.0 cm O.D.) single crystals of the pure ternary alloy 70Fe-15Ni-15Cr have been grown by means of the Czochralski technique. The single-crystal boules have been oriented and planar specimens have been cut by electric-arc erosion so that the major face of the samples is a (100) crystallographic plane. The principal crystallographic directions lying in this plane have been indexed and the surfaces of the specimens have been lapped flat and Syton polished. The samples are presently ready for the final phase of preparation for investigations of mass transport in both a 1-g environment and the low-gravity environment provided by the KC-135 aircraft.

**BIBLIOGRAPHIC CITATIONS FOR FY 1993:**

**Journals:**


**Presentations:**

II. MSAD Program Tasks — Ground-based Research

Materials Science

Modeling of Convection and Crystal Growth in Directional Solidification of Semiconductor and Oxide Materials

Principal Investigator: Prof. Robert A. Brown
Massachusetts Institute of Technology (MIT)

Co-Investigators:
D. Bornside
Massachusetts Institute of Technology (MIT)

Task Objective, Description & Significance:

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 method. Both semiconducting alloy (GeSi) and oxide (Bismuth gerenate) crystals are being studied.

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 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.

Research Approach — 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 macroscale 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.

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 1993:**

Today, the vertical Bridgman technique is used for the growth of III-V and II-VI alloy semiconductor crystals and for the production of oxide materials such as bismuth germanium oxide (Bi$_4$Ge$_3$O$_{12}$, BGO). The applications to II-VI materials such as mercury cadmium telluride (HgCdTe) are driven by the very high vapor pressure of the melt, which makes impossible crystal growth by conventional meniscus-defined methods. The applications to III-V materials, such as GaAs and InP, were first motivated by the substantial vapor pressures of these melts, but the relative success of vertical and horizontal Bridgman systems for growth of low dislocation material has led to very vigorous applications of the vertical Bridgman technology.

The vertical Bridgman (VB) method is especially important to the future of space processing. First and foremost, only confined systems are feasible in a low-gravity environment, so that all of the industrial technologies involving meniscus-defined growth methods are not applicable. Moreover, meniscus-defined growth introduces the possibility for thermocapillary convection and the intense mixing that space experiments are meant to avoid. Equally as important is the capability of the VB system for very tightly controlled crystal growth: the enormous expense of space experiments and the infrequent experimental capabilities of the eventual growth of very exotic crystals in space makes it imperative that the crystal growth furnaces are well controlled and characterized.

**Students Funded Under Research:**

PhD Students: 2

**Bibliographic Citations for FY 1993:**

**Journals:**


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, DESCRIPTION & SIGNIFICANCE:

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.

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.

Research Approach — 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.
II. MSAD Program Tasks — Ground-based Research

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 1993:
A combination of computational, theoretical and experimental studies are being conducted which are aimed at improving the understanding of microscale dynamics in cellular solidification and of the onset of dendrite growth. Experiments are planned on cellular solidification using transparent organic alloys. Calculations will be based on novel finite-element methods.

Over the last decade, a host of theoretical and experimental studies of directional solidification in thin systems have been performed, including the research conducted under previous NASA support. These studies have shown that cellular microstructures do not develop with a specific wavelength but are better characterized by a spatiotemporally chaotic pattern with a mean wavelength and dispersion about this mean. This picture of microstructural solidification is supported by the calculations of the dynamics of cellular solidification, by analysis of prototype equations that exhibit pattern formation, and by the careful experiments of thin-film solidification by M.I.T. and others.

STUDENTS FUNDED UNDER RESEARCH:

PhD Students: 2

TASK INITIATION: 7/93  EXPIRATION: 7/96
PROJECT IDENTIFICATION: 674-25-08-25
NASA CONTRACT NO.: NAG8-962
RESPONSIBLE CENTER: MSFC

BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:
Optical Properties for High Temperature Materials Research

**Principal Investigator:** Dr. Ared Cezairliyan  
National Institute of Standards and Technology (NIST)

**Co-Investigators:**  
S. Krishnan  
Intersonics, Inc.  
J. McClure  
National Institute of Standards and Technology (NIST)

**Task Objective, Description & Significance:**

**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.

**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 μm 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.

Research Approach — The approach is based on rapid resistive self-heating of the specimen from room temperature up to the temperature of interest in less than one second by passing a large current pulse through it and simultaneously determining emissivity by the following two techniques: from measurements of the surface radiance temperature and the true temperature of the specimen via Planck's law; and from measurements of the stokes parameters of polarized laser light reflected by the specimen. Reference values for emissivity will be determined in two temperature ranges by selecting metals with melting temperatures in the range 1500-2000°C (such as nickel and titanium) and the range 2500-3000°C (such as niobium and molybdenum).

**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 1993:**  
In the last few years, research conducted at Rice University and at Intersonics has resulted in the development and application of laser polarimetry techniques for the measurement of optical properties of electromagnetically-levitated, high-temperature liquids. The properties, which include normal spectral emissivities and optical constants, n and k, were measured for the noble metals and the platinum-group metals in the wavelength range 0.488-1.064 μm. More recent polarimetric investigations were conducted on several other materials at the 0.6328-μm wavelength. The basic technique involves the determination of normal spectral emissivity as a function of temperature by simultaneous measurements of the Stokes parameters of polarized laser light reflected by the
specimen surface and the surface radiance temperature by radiation thermometry. The working principle of the laser polarimetric technique is that, when quasi-monochromatic light of linear polarization is incident on a specularly-reflecting surface at an oblique angle, the reflected light is generally elliptically polarized. By accurately measuring the polarization state of the reflected light, the optical properties of the surface, including its normal spectral emissivity, can be exactly determined. An unambiguous description of the complete state of polarization is given by four Stokes parameters which are measured by an innovative laser polarimeter.

As yet, the emissivities of relatively few metals (Ti, Zr and Nb) have been measured at comparable wavelengths both by high-speed pyrometry at NIST and by laser polarimetry at Intersonics. Results obtained at the two laboratories for Zr and Nb are in reasonable agreement, whereas for Ti the difference between results is significantly larger than the combined measurement uncertainties. Differences among results for a given metal may be due, at least in part, to differences at the two laboratories relating to specimen heating, specimen geometry and surface conditions (e.g., roughness and residual contamination), specimen purity, etc.

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**BIBLIOGRAPHIC CITATIONS FOR FY 1993:**

**Journals:**


Dynamics of Hard Sphere Colloidal Dispersions

**Principal Investigator:** Prof. Paul M. Chaikin  
Princeton University

**Co-Investigators:**
- Prof. P.G. Debenedetti  
Princeton University
- Prof. W.B. Russel  
Princeton University

**Task Objective, Description & Significance:**

**Objective:**
The object of this research is to understand the fundamental nature of liquid-solid transitions.

**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.

**Significance:**
The ideal system for such study is a set of hard spheres which, according to theory, should order as their density is increased past approximately 50%. The problem is that sedimentation prevents setting the density or attaining equilibrium of the sphere system. Research in microgravity may help overcome this problem. These fundamental studies are aimed at a microscopic understanding of the most basic static and dynamic aspects of liquids, solids, and the solidification process.

**Progress During FY 1993:**
We have made great progress in studying a variety of different colloids suspended against gravity by the counterflow of the solvent; that is, with a fluidized bed. We have found the variation in sedimentation velocity resulting from hydrodynamic interactions and its effect on particle dispersion. We have observed crystallization of fluidized particles due to their Coulomb repulsion.

**Students Funded Under Research:**
Total Students: 2

**Task Initiation:** 4/90  
**Expiration:** 7/93

**Project Identification:** 674-25-05-18  
**Responsible Center:** LeRC
Modeling Directional Solidification in Furnaces/Processes in the Microgravity Materials Science Laboratory

**Principal Investigator:** Dr. Amon Chait

**NASA Lewis Research Center (LeRC)**

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

**Task Objective, Description & Significance:**

**Objective:**

The objectives of this task are to develop a general-purpose numerical model for simulating directional solidification furnaces and to provide a consistent approach to process/sample parameter optimization.

**Description:**

The approach has been: (a) to develop required enhancements for simulating solidification processes with a finite element fluid, thermal, and solutal solver; (b) to develop numerical methodology for process optimization; and (c) to develop new time-dependent, multidimensional, free-boundary algorithms.

**Progress During FY 1993:**

Progress has included the addition of inverse design and optimization code which in turn uses our general purpose fluid or thermal analysis codes. A high-level inverse design and optimization code was added and specifically applied to the Programmable Multi-Zone Furnace (PMZF) configuration. The capability adds, for the first time, the explicit specification of the principal investigator science requirements (e.g., thermal gradients, interface shape, etc.) as an experimental design criterion.

The period of performance for this task ended September 1992.

**Task Initiation:** 10/91  **Expiration:** 9/92

**Project Identification:** 674-25-05-15

**Responsible Center:** LeRC
Fluid Mechanics of Directional Solidification at Reduced Gravity

PRINCIPAL INVESTIGATOR: Prof. Chuan F. Chen
University of Arizona

CO-INVESTIGATORS:
No Co-I's Assigned to this Task

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

OBJECTIVE:
The primary objective of this research is to provide ground-based support for the Casting and Solidification Technology (CAST) flight experiment, flown on IML-1 in January 1992. The secondary objective of the proposed research is to examine the stability phenomena associated with the onset of freckles and the mechanisms for their subsequent growth and decline (to the eventual demise of some).

DESCRIPTION:
The focus is to study the convective motion and freckle formation during directional solidification of NH$_4$Cl from aqueous solution at simulated parameter ranges equivalent to reduced gravity. This will involve state-of-the-art imaging techniques and mathematical models for the prediction of the observed phenomena.

PROGRESS DURING FY 1993:

- Computed Tomography Applied to the Mushy Layer
A 30% NH$_4$Cl-H$_2$O solution was directionally solidified in a 12.7 x 12.7 x 12.7 cm plexiglass tank, generating a mushy layer approximately 2.5 cm thick. The tank was then transported to the Arizona Health Sciences Center for computed tomography scanning. Sixty-six scans were made, each 2 mm thick and containing x-ray absorption data for 350 x 350 voxels. The data were analyzed using Data Explorer on an IBM 340 workstation in the Visualization Laboratory at the Center for Computing and Information Technology at The University of Arizona. Using the method presented by Chen and Chen (JFM 227:567-586, 1991), the x-ray absorption data were converted into solid fraction of the mush. Three-dimensional level surfaces were generated and plotted with false coloring. Solid fraction distribution within the entire mushy zone is clearly exhibited. (A set of computer outputs was submitted with the Annual Technical Report dated January 4, 1993.) The results were presented as a poster in the "Gallery of Fluid Motion" during the 45th Annual Meeting of APS-DFD, November 1992.

We have recently concluded an experiment in which the growing mush was continually examined by CT scan during directional solidification. We are still at the data-reduction stage. Final results will include information on the time evolution of (1) horizontally averaged solid fraction, (2) solid fraction distribution within a 2 mm slice of mush, and (3) three-dimensional level interfaces of solid fraction within the mushy layer. The results will reveal the variations of solid fraction within the mush as it goes through the finger convection period, the onset of plume convection, and, finally, the fully developed plume-chimney flow regime. The results will provide a basis for comparison with the predictions of the linear theory of Worster (JFM 237:649-669, 1992).

- Onset of Chimney Convection in a Mushy Layer Generated in a Hele Shaw Cell
This research was carried out at the institut fur Angewandt Thermo- and Fluiddynamic, Kernforschungszentrum, Karlsruhe, Germany, where Dr. C. F. Chen was a Visiting Scientist from May 25 to August 9, 1992. Directional solidification experiments were conducted in a Hele Shaw cell because of its inherent two-dimensionality and ease of flow visualization. A 27% NH$_4$Cl-H$_2$O solution (liquidus temperature = 20°C) was solidified from below in a cell 22 cm wide x 20 cm high x 0.14 cm thick. The bottom copper surface was maintained at a constant low
temperature. Convection in the liquid region was visualized by either the Schlieren or the differential interferometric technique. A detailed study was carried out on the finger convection phase using the interferometer. Results show that, at the start of the solidification process, the finger convection cells are very slender and are distributed evenly all along the bottom. As the cells grow upward, they coalesce to form larger and larger cells. Eventually, the entire tank is disturbed by such convection. With the appearance of plume convection, finger convection gradually weakens. When the plume convection is fully developed, the finger convection becomes very weak, and the background large-scale convection is completely damped out.

Chimney formation experiments were carried out with the bottom temperature varying from -25°C to -5°C. For cases with the bottom temperature higher than the eutectic temperature of -15.4°C, it was found that the time of onset of plume-chimney convection increases and the ratio of the chimney wavelength to the layer thickness decreases as the bottom temperature is increased. However, when the plume convection is fully developed, approximately 2 hr. into the experiment, the chimney wavelength is approximately 1.3 times the layer thickness, regardless of the bottom temperature. These results were presented at the 45th Annual Meeting of the APS-DFD.

• Cross-Diffusion Effect on the Stability of a Double-Diffusive Layer with a Free Surface

During the first grant year, we studied the effect of surface tension on the stability of a double-diffusive layer (Chen and Su, Phys. Fluids A 4:2360-2367, 1992). During the current grant year, we have been investigating the additional effect of cross-diffusion on the stability of such a system. The problem we studied is that of a binary fluid with a finite Soret effect but negligible Dofour effect. This layer of fluid is subjected to a temperature gradient. We studied the stability of this layer under the combined effect of Soret diffusion and surface tension by linear stability techniques. The effects of the dependence of surface tension on temperature and solute were investigated separately. Calculations were made for Rayleigh numbers< 100 suitable for reduced gravity levels. The results show that, in a parameter range in which the density distribution due to the temperature gradient and the solute gradient arising out of the separation ratio are stable, surface tension effects due to either temperature or solute may cause onset of instability in the oscillatory mode. A paper entitled Effect of Surface Tension on the Stability of a Binary Fluid Layer Under Reduced Gravity by C. F. Chen and C. C. Chen has been submitted to the Physics of Fluids A.

STUDENTS FUNDED UNDER RESEARCH: 5

BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:


Chen, C. F. Convection in the mushy zone during directional solidification. Interactive Dynamics of Convection and Solidification, 139-141, 1992.


II. MSAD Program Tasks — Ground-based Research

Discipline: Materials Science

Presentations:


Microgravity Chemical Vapor Deposition

**Principal Investigator:** Dr. Ivan O. Clark  
**NASA Langley Research Center (LaRC)**

**Co-Investigators:**
- W.A. Jesser  
  University of Virginia  
- P.V. Hyer  
  Lockheed Engineering & Sciences Co.  
- E.J. Johnson  
  Lockheed Engineering & Sciences Co.

**Task Objective, Description & Significance:**

**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.

**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.

**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 1993:**
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.

**Task Initiation:** 11/92  
**Expiration:** 11/95  
**Project Identification:** 674-21-06-09  
**Responsible Center:** LaRC
II. MSAD Program Tasks — Ground-based Research

BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Presentations:
Clark, I.O. Gravitational and thermal diffusion effects in horizontal CVD flows, Ninth American Conference on Crystal Growth, Baltimore, MD, August 2-6, 1993.
Task Objective, Description & Significance:

Objective:
There are two specific tasks involved in this research project:

1. Nucleation by internal oxidation or reduction of transition metal-bearing silicate 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. The environmental control required in the pursuit demands a containerless environment; the ability to ultimately produce controlled-shape parts from the uniform, fine-scale crystallization of a melt would take advantage of microgravity.

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. Containerless processing is crucial to secure the necessary amount of undercooling; a quiescent microgravity environment might further allow large bodies of inviscid melt to be so processed.

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 (ALL). 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 ALL 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-SiO₂ metasilicate compositions near the deep cristobalite (SiO₂)-enstatite (MgSiO₃) 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 Al₂O₃-SiO₂ system, the alumina-rich end producing highly inviscid melts that, if sufficiently undercooled, could produce interesting alumina/mullite glass-ceramics.

**Progress During FY 1993:**

Progress on the Two Tasks of this Investigation:

1. Nucleation by internal oxidation or reduction of transition metal-bearing silicate glasses and melts. Oxidation experiments were completed on a suite of CaO-Na₂O-FeO-MgO-Al₂O₃-SiO₂ glasses (prepared from naturally occurring Columbia River Plateau Basalts) and on synthetic compositions minus Na₂O. These experiments were analyzed by Rutherford backscattering spectroscopy (RBS), optical microscopy, transmitted electron microscopy (TEM) and high energy electron diffraction (HEED). The RBS spectra clearly indicate that the oxidation process is accompanied by the flux of Ca²⁺, Mg²⁺ (both specimen types) and Na⁺ (natural basalt specimens) to the free surface with the subsequent formation of a crystalline, calcium-rich orthosilicate (CaₓMgₓ(SiO₄)ₓ) that partially covers the specimen surface. Beneath this surface layer lies a depleted silicate glass, that includes fine precipitates of MgFe₂O₄ spinel. The results indicate that the oxidation occurs by the flux of oxygen into the glass with the oxidation resulting in structural change of the glass, precipitation of the spinel and a release of Ca⁺ and Mg⁺ whose elevated activity at the oxidation reaction front causes their diffusion to the free surface. The kinetics are parabolic (diffusion-limited); the rate-limiting step for the oxidation process is thus either diffusion of molecular oxygen through the depleted glass or diffusion of the divalent cations away from the reaction front towards the free surface (the fastest diffusion path) due to the relative polymerization of the starting and depleted glasses. From a materials processing perspective, the internal oxidation phenomenon does indeed suggest the possibility to fore internal nucleation of an inviscid melt body via environmental change. We are presently preparing two manuscripts describing this phenomena.

These experiments have been extended to melts in two ways. In cooperation with Intersonics Corporation, spheres of both the natural and synthetic compositions have been aero-acoustically levitated and oxidized above the liquidus for the original melt composition. In the sphere so levitated that remained relatively quiescent, the internal oxidation process is plainly evident, with internal oxidation and consequent homogeneous nucleation of magnesioferrite spinel (as well as Ostwald ripening of these nuclei) and heterogeneous (on the spinel precipitates) of calcium-rich feldspar. We are presently working out a kinetic model for this process in the melts, but the thrust of the model is a form of constitutional supercooling wrought by the redox diffusion process. A manuscript for publication is being outlined. In similar experiments employing electrostatic levitation (experiments performed by W. K. Rhim at JPL), the synthetic specimens successfully levitated but experienced instabilities due to the
environment of the chamber, which we believed caused reduction of specimens: the release of oxygen cause bloating and fracture of the droplets. experiments performed very recently look better; we are presently engaged in analyzing these.

2. Internal nucleation of inviscid pseudobinary silicate melts via metastable liquid-phase immiscibility. MgO-SiO₂ binary melts (metasilicate composition) have been drop-tube processed at approximately 1800°C. Yield of these experiments was surprisingly extensive: approximately 80 vol.% of the fine (<50-μm particles experienced melting in the process (a substantial increase in yield over powder processed at ~1600°C). We are presently collecting statistics on this processed powder, specifically discerning the amount of material that received sufficient undercooling so as to remain amorphous. We are further pursuing thermal analysis experiments on the amorphous powder to ascertain, if possible, the role of amorphous phase separation on the nucleation of glass-ceramic (i.e., spatially uniform, fine-grained crystalline) microstructure in these droplets. Time resolved thermal analysis should allow discrimination of surface nucleation processes (i.e., heterogeneous nucleation on amorphous-amorphous interfaces) from possible homogeneous nucleation processes, as well as determine the scale of phase separation important to promote appropriate crystallization. We anticipate collecting such dynamic data in the next few months.

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Directional Solidification in $^3$He-$^4$He Alloys

**Principal Investigator:** Arnold Dahm  
*Case Western Reserve University*

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

**Task Objective, Description & Significance:**

**Objective:**
The goal is to enhance our fundamental understanding of crystal growth kinetics, liquid-solid interface morphologies, and the stability of alloys.

**Description:**
Systematic studies of directional solidification in $^3$He-$^4$He alloys will be conducted to complement work that has been done in other alloys. Studies will also be carried out in normal $^4$He for comparison. Here, the latent heat is non-negligible, and heat transport plays a role in determining the morphology of the interface.

**Significance:**
The study of morphologies is of intrinsic interest in testing theories of non-linear systems. These studies will provide a base for future flight based research. Study results found to differ from those of classical alloys will guide both theorists and experimentalists in their future studies, and significant differences should result in new ideas for space-based alloy growth experiments.

**Progress During FY 1993:**
At this point we have designed and built our sample chamber, installed a helium dewar system and built a gas handling system. We expect to begin taking data this fall.

The reason that we have not taken data to date is the following. I attempted to find a graduate student to work on the project. The only available student who was interested in condensed matter physics and who I considered acceptable for the project expressed an interest in our proposed work. However, he failed the qualifying exam last May. I then made the decision to hire a postdoc for two years rather than a graduate student for three. I have hired Dr. Chen from the University of Illinois. Dr. Chen comes to us with very strong recommendations, and I am convinced that we will accomplish more work in this mode than with a graduate student for three years.

**Task Initiation:** 1/93  
**Expiration:** 12/96  
**Project Identification:** 674-25-05-25  
**Responsible Center:** LeRC


Modeling of Coalescence

**Principal Investigator:** Prof. Robert H. Davis  
University of Colorado

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

**Task Objective, Description & Significance:**

**Objective:**  
The overall objective of this research is to develop models to predict drop-size distribution evolutions due to droplet collisions and coalescence during processing within the miscibility gap of bimetallic liquid-phase-miscibility-gap materials. When bimetallic liquid-phase-miscibility-gap materials are cooled through the miscibility gap, droplets rich in one of the metals form in the liquid matrix rich in the other metal. Droplet coalescence and phase segregation then occur due to buoyancy, thermocapillary, and other nongravitational mechanisms. Drop-size distribution evolutions in time are calculated by population dynamics models.

**Description:**  
In the population dynamics models, continuous drop-size distributions are discretized into a large number of categories. The formation and destruction of drops in each size category are tracked. Drop motion due to gravity sedimentation, Marangoni migration, Brownian motion, and bulk flow are considered individually or collectively.

**Significance:**  
Classic expressions attributed to Smoluchowski for the collision rate between drops (that are required in the collision kernels opposing in the population dynamics models) are improved to include attractive, repulsive, and hydrodynamic interactions between drops. In particular, trajectory calculations are used to predict collision efficiencies.

**Progress During FY 1993:**  
A computer program has been completed for solving the population dynamics model to follow droplet size evolutions with time in homogeneous dispersions due to collisions arising from gravity sedimentation, Marangoni migration, and/or Brownian motion. Some of the key results are that a bimodal initial distribution will exhibit much more rapid coalescence due to gravity sedimentation or Marangoni migration than will a unimodal initial distribution. A unimodal initial distribution will evolve into a bionodal distribution, then into a shifted and broadened unimodal distribution, and the coalescence may be greatly reduced by antiparallel alignment of the gravity vector and the temperature gradient.

Collision efficiencies for Brownian motion and gravity sedimentation for drops having a range of viscosity and radius ratios have been computed both in the presence and absence of attractive forces. A key result is that, in contrast to rigid particles, spherical liquid drops have nonzero collision efficiencies in the absence of attractive forces.

Theoretical work on combined mechanisms for drop coalescence has been focused on buoyancy and Marangoni motion with the gravity vector aligned antiparallel to the temperature gradient. The collision efficiencies for thermocapillary motion are larger than those for gravity motion, because of the more rapid decay of the velocity fields created by drops undergoing thermocapillary motion, provided that the thermal conductivity of the drops is not too large.
Our results show that there is a finite region in parameter space for which no collisions occur. This is because the interaction of two drops due to thermocapillary migration decays more rapidly with the separation distance \( R \) (as \( 1/R^2 \)) than does the interaction of two drops in gravity motion (which decays as \( 1/R \)). As a result, the large drop which moves toward a small drop below it due to gravity may reach a separation distance where the initially weaker thermocapillary effects just balance the buoyancy-driven relative motion, and so the separation distance then no longer decreases.

Time scales have been determined to phase segregation and drop coalescence, together with criteria for predicting whether or not significant coalescence will occur prior to phase segregation. A computer model is being developed to solve population balances for drop size distributions which show that the phase segregation rate initially increases due to coalescence and subsequently decreases as the large drops migrate out of suspension.

Physical data on a variety of bimetallic and transparent immiscibles have been collected, and dimensionless parameters presenting various effects have been tabulated as functions of drop size. Data on composite Hamaker constant (for van der Waals attraction) and temperature coefficients of interfacial tension (for thermocapillary migration) are sparse, and order-of-magnitude estimates are typically used. Calculations of collision efficiencies have been made for bismuth drops in a zinc melt and for ethyl salicylate drops in diethylene glycol.

The period of performance for this task ended September 1992.
Advanced Photonic Materials Produced by Containerless Processing

Principal Investigator: Dr. Delbert E. Day
University of Missouri, Rolla

Co-Investigators:
C.S. Ray
University of Missouri, Rolla

Task Objective, Description & Significance:
Objective:
The objectives of this research are to: (a) use containerless melting to develop nonlinear optical glasses for use as ultrafast, all-optical switches and other photonic devices for communication and advanced computer application; and (b) investigate and compare the kinetics of nucleation and crystallization for these glasses—prepared by containerless melting—with the kinetics for the same glasses melted in a container.

The glasses that have been reported to possess the potential for nonlinear optical applications, such as the heavy metal oxide (HMO) glasses containing PbO, Bi₂O₃, and Ga₂O₃, are made from highly fluid and chemically corrosive melts which readily crystallize during cooling. These typical characteristics not only decrease the glass-forming tendency for these melts, but develop unwanted color centers, primarily due to materials dissolved from the container. The conventional melting procedure for HMO compositions therefore results in colored and chemically inhomogeneous glasses that are presently unusable for nonlinear optical applications. Containerless processing provides the opportunity to eliminate or suppress the heterogeneous nucleation and crystallization in a melt and increase the tendency for glass formation. Since no container is used, color centers caused by impurities dissolved from a container can be completely eliminated, even in a highly corrosive melt. The kinetics of nucleation and crystallization for HMO glasses prepared with and without a container will be investigated to determine, primarily, the temperature range for nucleation, the temperature for maximum nucleation, and the activation energy for crystallization. The nucleation and the crystallization data will be combined with the measured chemical durability to determine the stability of these glasses for practical applications.

At the completion of the ground-based work, the general plan is to conduct follow-on experiments with containerless melts in space, using compositions that appear most suitable to the operational parameters of whatever levitator furnace is available. The purpose is to develop a glass with improved nonlinear optical properties by taking advantage of the reported observation that a glass prepared in a microgravity environment is chemically more homogeneous than an identical glass prepared on Earth. This observation from earlier space experiments can be verified by data from the present experiments, and the mechanism(s) of melt homogenization in the absence of gravity can be determined. The kinetic parameters for nucleation and crystallization determined for the space- and Earth-melted glasses are expected to yield a better scientific understanding for the relative role of heterogeneous and homogeneous nucleation in the processes of glass formation, nucleation, and crystal growth in fluid melts. Since an acoustic field is used to levitate and position melts for containerless processing, the proposed research will also attempt to identify any effect an acoustic field may have on the homogenization, nuclei formation, and crystal growth during a containerless glass-forming melt.

Research Approach: Initially, the investigation of nonlinear optical glasses started with HMO compositions in the system PbO, Bi₂O₃ and Ga₂O₃. The starting composition was chosen after discussions with Dr. William Dumbaugh at Corning, Inc., who has considerable experience with HMO glasses. Depending upon the initial results, other compositions containing TiO₂, Nb₂O₅, or TeO₂, which have the potential of yielding a high nonlinear refractive index and high infrared transmission cut-off (>8μm), will also be studied.
Glasses are prepared in containers of different materials, such as Pt, Au, and Al₂O₃, to determine which container has the highest corrosion resistance to these melts and will yield the best glass. Hot-pressed PbO, Bi₂O₃, and Ga₂O₃ samples of suitable composition will also be containerlessly melted and cooled at Intersonics, Inc., using their newly developed acousto-acoustic levitator/furnace. The critical cooling rate for glass formation, R_c (container) measured by the pendant-drop technique (spherical glass melt hanging from a thermocouple bead), will be compared with the R_c measured for containerless melt R_c (containerless) at Intersonics.

The ratio of R_c (container) to R_c (containerless) will be used to determine the improvement in glass formation for the containerless melt at 1-g. The kinetic parameters for nucleation and crystallization for these glasses (prepared with and without a container) will be measured by differential thermal analysis (DTA), and the crystallized phase(s) will be identified by XRD. The size and distribution of crystallized phase(s) will be determined by SEM and EDAX. Structural features of the glasses, such as cation coordination and oxygen bonding, which can be important to the kinetic parameters for crystallization will be investigated by NMR, XPS, and IR spectra.

Selected properties, such as the glass transition temperature, softening temperature, thermal expansion coefficient, density, refractive index, microhardness, chemical durability, and the cut-off wavelength in the VIS and IR, also will be measured. The nonlinear refractive index will be measured only for the glasses that have the best combination of the above mentioned properties, and a nonlinear optical glass with optimized thermal, mechanical, nucleation, and crystallization properties will be developed at 1-g.

If flight opportunities become available, the optimum composition developed for nonlinear optical application from the ground-based investigation will be used in flight experiments. Hot-pressed spherical samples (6 mm to 8 mm in diameter) will be melted containerlessly in space and cooled at different rates. All of the properties measured for the 1-g glasses will also be measured for the post-flight samples. A comparison of these properties for Earth- and space-melted glasses will reveal the improvement in the optical and other related properties due to better chemical homogeneity for glass melted in space.

Any difference in the number, size, and distribution of nuclei in the Earth- and space-melted glasses can also be determined, the knowledge of which can be useful to understanding the mechanisms of glass formation and crystallization in melts processed in space and on Earth.

**Progress During FY 1993:**

Measurements of selected properties for the heavy metal oxide (HMO) glasses containing PbO, Bi₂O₃, Ga₂O₃ have been completed. The results have been summarized in a technical paper which has been submitted to the Journal of American Ceramic Society for publication.

Investigations on the crystallization behavior of these HMO glasses continuing using differential thermal analysis (DTA) and high temperature x-ray diffraction (XRD) analysis. While the high-PbO containing glasses crystallize a single compound PbO, high Bi₂O₃ containing glasses undergo a series of phase transformations as the temperature is increased. At a relatively low temperature (~400°C) the first crystals to appear in these high-Bi₂O₃ containing glasses are PbO which then reacts with the glass matrix to form 2PbO.3Bi₂O₃ at ~450°C, and finally 2PbO.12Bi₂O₃ at ~525°C. Thermogravimetric analysis (TGA) in air of these glasses shows a weight gain above 500°C, which is possibly due to the formation of PbO₂ from PbO (oxidation). No such weight gain is observed if the TGA is performed in an inert atmosphere such as nitrogen or argon. The activation energy for crystallization for the glasses which yield a single DTA crystallization peak (high-PbO containing glasses) has been determined using Kissinger method and is ~ 420 kJ/mol.
II. MSAD Program Tasks — Ground-based Research

The glass structure of these HMO glasses is not clearly known. It has been planned to investigate the structure of these glasses using ir and Raman measurements in a collaborative program with JPL. The ir measurements are now in progress at UMR, and the Raman measurements are being conducted at JPL. It is also of interest to investigate whether glass formability and the properties of these HMO glasses improve in containerless melting. Samples of different composition have been provided to JPL to perform experiments on melting and cooling these samples in electrostatic levitator/furnace.

STUDENTS FUNDED UNDER RESEARCH:

Total Students: 1

BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:


Presentations:
Modeling Internal Radiative Transport in Crystal Growth Processes

**Principal Investigator:** Dr. Jeffrey J. Derby  
University of Minnesota

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

**Task Objective, Description & Significance:**

**Objective:**  
Radiation energy transfer is an important feature in the high-temperature processes used to produce many optical and semiconductor crystals. The goal of this research program is to understand the effects of internal radiation, i.e., radiation within a participating medium, in several crystal growth systems of technological importance.

**Description:**  
We develop and employ finite element methods to accurately predict radiation transfer in semitransparent media for the complicated, multidimensional geometries of crystal growth systems.

**Significance:**  
These methods are extremely accurate and robust and are applicable to systems of any optical thickness. The prediction of radiation transport is combined with previous heat transfer and hydrodynamic analyses of crystal growth to provide predictive capabilities for these processes.

**Progress During FY 1993:**  
We have employed Galerkin finite element methods for modeling the vertical Bridgman growth of semitransparent crystals. Our prior work has identified the strong influence of internal radiation on the shape and position of the melt/crystal interface and the temperature gradients surrounding it. An important result of our past analyses showed that the coupling of internal radiation through the crystal with conduction through the ampoule walls promoted melt/crystal interface shapes which were highly deflected near the ampoule wall. This "radiative interface effect" had been observed, but not adequately understood, in prior experimental systems. The curvature of the melt/crystal interface in these systems was found to be much more pronounced than that observed during the Bridgman growth of opaque crystals, where the interface deflection at the ampoule wall is attributed to the thermal conductivity mismatch between ampoule and charge. Calculations demonstrated that a flatter overall interface shape could be achieved through optimization of ampoule material properties and furnace temperature profiles.

During the past year, we have extended our model to consider the rigorous analysis of semitransparent crystal and melt domains with different optical properties. This important modification will allow us to address such topics as radiative supercooling --- a morphological instability postulated to be caused solely by internal radiant heat transfer.

**Students Funded Under Research:**

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**Task Initiation:** 5/91  
**Expiration:** 10/93  
**Project Identification:** 674-21-06-07  
**Responsible Center:** LaRC
II. MSAD Program Tasks — Ground-based Research

BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:

Presentations:
Derby, J.J. Computer-aided analyses of internal radiation, conduction, and convection in crystal growth processes, Departmental Seminar Series, Dept. of Chemical Engineering, University of Wisconsin, Madison, WI, April 15, 1993, invited.
Derby, J.J. Finite element analysis of the growth of semitransparent crystals via the Bridgman method, Workshop on Control of Bridgman Growth of CdZnTe Crystals, University of Virginia, Charlottesville, VA, June 24-25, 1993, invited.
Derby, J.J. Computer-aided analyses of high-temperature materials processing systems: Crystal growth and ceramics sintering, Chemical Engineering Program Seminar Series, Univ. of California at San Diego, La Jolla, CA, September 15, 1993, invited.
Derby, J.J. Finite element analysis of the growth of large, single crystals of semitransparent materials, TI, Dallas, TX, September 16, 1993, invited.
Derby, J.J., and Kuppurao, S. Effects of internal radiation during the melt growth of semitransparent crystals: Radiative supercooling, American Institute for Chemical Engineering Annual Meeting, St. Louis, MO, November 7-12, 1993.
The Effect of Gravity on Natural Convection and Crystal Growth

PRINCIPAL INVESTIGATOR: Prof. Graham D. deVahl Davis
University of New South Wales

CO-INVESTIGATORS:
Dr. H. deGroh III
NASA Lewis Research Center (LeRC)

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

OBJECTIVE:
The objective is to measure experimentally-natural thermal convection during Bridgman growth.

DESCRIPTION:
Measurements are made at varying gravity-driven levels to quantitatively determine how convection affects parameters of critical importance to the crystal grower — such as interface shape and radial and longitudinal segregation.

SIGNIFICANCE:
The work will produce an accessible, experimentally verified numerical code that is capable of accurately determining levels of convection in real systems, at varying gravity levels and directions, as well as the effects of this convection on the solidification process.

PROGRESS DURING FY 1993:
To supply and supplement numerical modeling efforts of other flight programs such as the MEPHISTO flight experiment of Dr. Abbaschian.

STUDENTS FUNDED UNDER RESEARCH:
Task Initiation: 11/92 Expiration: 10/95
Task Initiation: 11/92 Expiration: 10/95
Total Students: 1

PROJECT IDENTIFICATION: 674-21-05-09
RESPONSIBLE CENTER: LeRC

BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:


Proceedings:

NASA Tech Briefs:
Reverse Micelle Based Synthesis of Microporous Materials in Microgravity

**PRINCIPAL INVESTIGATOR:** Prof. P.K. Dutta

**Ohio State University**

**CO-INVESTIGATORS:**

- Dr. C.T. Kresge
  - Mobil Research & Development Corporation
- Dr. R. Ansari
  - NASA Lewis Research Center (LeRC)
- W. Meyer
  - NASA Lewis Research Center (LeRC)

**TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:**

**OBJECTIVE:**
The emphasis of this research program is on crystalline microporous materials, characterized by internal channels and cavities of molecular dimensions.

**DESCRIPTION:**
Typically, microporous materials are synthesized by hydrothermal methods in which an amorphous gel converts to a crystalline form via complex solid-solution, polymerization-depolymerization and nucleation reactions. The reactants necessary to form microporous materials in the core of reverse micelles are introduced.

**SIGNIFICANCE:**
These materials are important in various industries—petroleum processing, separation, environmental—and could also be potentially useful as electronics materials. The novel micellar environment has the potential for the nucleation of new frameworks. In addition, the micelles provide the opportunity to examine the evolution of nuclei into crystals. The necessity for microgravity arises since particles formed by intermicellar interactions settle out long before crystal formation. For the most relevant class of microporous materials, namely aluminosilicate zeolites, the process of nucleation and crystal growth can require days and therefore sustained microgravity conditions are required.

**PROGRESS DURING FY 1993:**
The emphasis of this research program is on crystalline microporous materials, which are characterized by internal channels and cavities of molecular dimensions. These materials are important in various industries, including petroleum processing, separation, environmental and could also be potentially useful as electronic materials. Typically, microporous materials are synthesized by hydrothermal methods in which an amorphous gel converts to a crystalline form via complex solid-solution, polymerization-depolymerization and nucleation reactions.

In this project, we are introducing the reactants necessary to form microporous materials in the core of reverse micelles. The novel micellar environment has the potential for the nucleation of new frameworks. In addition, the micelles provide the opportunity to examine the evolution of nuclei into crystals. The necessity for microgravity arises since particles formed by intermicellar interactions settle out long before crystal formation. For the most relevant class of microporous materials, namely aluminosilicate zeolites, the process of nucleation and crystal growth can require days and therefore sustained microgravity conditions are required.

However, in order to establish the feasibility of using reverse micelles as reactants under earth based conditions, we have been focusing on zincophosphate microporous materials. Unlike aluminosilicates, these nucleates very rapidly under mild conditions therefore provide the opportunity to establish the methodology. Surfactants that have been examined for making reverse micelles include AOT (12 bis (2-ethyl hexyloxycarbonyl) -1- ethane sulfonate) and
Igepal (polyoxoethylene nonylphenyl ethers). With both these systems, we have been able to synthesize microporous zincophosphates with sodalite-type frameworks. The experiments involve making up two separate micellar solutions containing aqueous zinc and phosphate solutions, respectively, dispersed in hexane/cyclohexane via the detergent. Upon mixing these solutions, the zinc and phosphate micelles interact with each other and crystalline products are found to settle out within a few minutes to hours, depending on the concentrations of the reactants. Powder X-ray diffraction of the solids confirmed the formation of sodalite framework. The morphologies were examined by scanning electron microscopy and indicated the presence of agglomerated particles made up of crystallites measuring ~1000 Å. Quasielastic light scattering shows that the initial micelles are of dimensions of ~50 Å for the AOT system and 3500 Å for the Igepal system. These particles grow with time to excess of ~1000 Å in case of AOT and ~10000 Å in case of Igepal. The crystals begin to settle as the particles size gets larger. These initial studies establish that reactants introduced as micelles can definitely interact with each other and gradually grow to form crystalline micorporous materials.

Our present focus is to isolate and characterize the early stages of crystal formation in order to obtain information on structure of nuclei. Having established the feasibility of this procedure, we have begun experiments on aluminosilicates in reverse micelles which will be the main focus of our experiments in microgravity.

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II-404
Growth of Nonlinear Optical Thin Films/Vapor Processes

**Principal Investigator:** Dr. Donald O. Frazier
**Co-Investigators:**
B. Penn

**NASA Marshall Space Flight Center (MSFC)**

**Task Objective, Description & Significance:**

**Objective:**
One major goal of this program is to grow high-quality thin films and single crystals of nonlinear optical organic and polymeric materials by vapor transport processes. Organic materials are of interest for use in all optical communication and signal-processing systems.

**Description:**
This program is designed to grow high quality thin films and single crystals of nonlinear optical organic materials in low gravity by vapor transport processes. Since there are many derivations possible among organics, screening is important for tailoring thin films (particularly polymeric films) and crystals for the appropriate properties. Our strategy is to screen compounds (theoretically and experimentally), synthesize, purify, and grow thin films and bulk crystals by vapor transport processes. These processes are considered sensitive to gravity levels. We will then characterize these films and crystals for second- and third-order optical effects, and determine gravitational influences on quality. We will then seek flight opportunities to improve quality when appropriate.

**Research Approach — The approach is to concentrate on exploiting low-gravity processing following a thorough ground-based research program. The ground-based program will provide a basis for the design and analysis of space-based experiments and determine growth condition and property relationships needed to grow high-quality thin films and single crystals, reproducibly and in quantity. For thin-film preparation it is important to determine the best substrate to optimize film homogeneity and orientation in unit- and low-gravity environments.**

**Significance:**
The development of organic materials for use in nonlinear optical (NLO) devices is of interest because their optical nonlinearities are usually considerably larger than those of conventional inorganic materials. Since there are many derivations possible among organics, screening is important for tailoring crystals for the appropriate properties.

**Progress During FY 1993:**
Research to date has resulted in the preparation of MNA-polydiacetylene thin film by solution polymerization having third-order properties which exceed industry standards. Also, new compounds were synthesized and thin films prepared having second- and third-order properties. Cooperative funding continued through Joint Ventures (JOVE, Northwestern State University and New Mexico Highlands University) and Historically Black Colleges and Universities (HBCU, Dophus E. Milligan Science Research Institute and Alabama A&M University). Articles on syntheses of new compounds and determination of nonlinear optical properties have been accepted for publication. A new high-vacuum system has been completed to prepare thin-films on prepared substrates, and a horizontal furnace is also near completion for bulk-growth by physical vapor transport. Some work has begun on determining the feasibility of mechanically strengthening brittle organic compounds by growing composites.
II. MSAD Program Tasks — Ground-based Research

BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:


Presentations:


PATENTS:
Patent Status: Pending U.S. Patent #: Undetermined
Paley, M.S., Donovan, D.N., Frazier, D.O., and McManus, S.P. "Solution-state photo-deposition of thin films of a polydiacetylene derivative of 2-methyl-4-nitroaniline that exhibit outstanding third-order optical nonlinearity"
Growth of Nonlinear Optical Crystals by Melt and Solution Processes

PRINCIPAL INVESTIGATOR: Dr. Donald O. Frazier
NASA Marshall Space Flight Center (MSFC)

CO-INVESTIGATORS:
B. Penn
NASA Marshall Space Flight Center (MSFC)

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

OBJECTIVE:
One major goal of this program is to grow high-quality single crystals and composites of nonlinear optical organic and polymeric materials by the Bridgman-Stockberger process. This may include float-zone and liquid-encapsulated float-zone methods. Organic materials are of interest for use in all optical communication and signal processing systems.

DESCRIPTION:
The strategy is to screen compounds theoretically and experimentally, synthesize, purify, and grow crystals by melt or solution processes. These crystals will then be characterized for second- and third-order optical effects, and gravitational influences on crystal quality will be determined. Flight opportunities will be sought to improve crystal quality when appropriate.

Research Approach — The approach is to study Bridgman-Stockberger and, where desirable, liquid-encapsulated Bridgman-Stockberger organic crystal growth from the melt. Additional focus is on methods for exploiting expected low-gravity-processing advantages of increased stability of low-surface-tension melt zones and reduced convective flows for float-zone crystal growth.

Although organics are desirable for use in optoelectronic devices, their use is hindered by the fragility of organic single crystals. There have been demonstrations that the strength of organics can be improved by aligning the photonic component in a polymer matrix. Bulk composites of aligned nonlinear optical organic crystals in low-molecular-weight transparent polymers having improved mechanical strength and fewer defects might be prepared by growing these composite materials in microgravity by directional solidification. Ground-based research in this area includes identifying mixtures appropriate for developing such composites.

SIGNIFICANCE:
The development of organic materials for use in nonlinear optical (NLO) devices is of interest because their optical nonlinearities are usually considerably larger than those of conventional inorganic materials. Since there are many derivations possible among organics, screening is important for tailoring crystals for the appropriate properties.

PROGRESS DURING FY 1993:
Most of the major characterization equipment, e.g., Nd:YAG laser, is in place for nonlinear optical characterization. Extension of theoretical screening includes bulk-phase modeling. Cooperative funding continued through Joint Ventures (JOVE, Northwestern State University and New Mexico Highlands University) and Historically Black Colleges and Universities (HBCU, Dolphus E. Milligan Science Research Institute and Alabama A&M University). Articles on syntheses of new compounds and determination of nonlinear optical properties have been accepted for publication. A horizontal furnace has also been completed for bulk-growth by physical vapor transport. Work is in progress to mechanically strengthen brittle organic compounds by growing composites in the melt and crystallization in solution.
II. MSAD Program Tasks — Ground-based Research

Discipline: Materials Science

STUDENTS FUNDED UNDER RESEARCH:

PhD Students: 1

TASK INITIATION: 1/91  EXPIRATION: 1/94

PROJECT IDENTIFICATION: 674-21-08-13

NASA CONTRACT NO.: In-house

RESPONSIBLE CENTER: MSFC

BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:


Presentations:


PATENTS:

Patent Status: Pending  U.S. Patent #: Undetermined

Patent Status: Approved
Pending  U.S. Patent #: Undetermined

Patent Status: Pending  U.S. Patent #: Undetermined
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, DESCRIPTION & SIGNIFICANCE:

OBJECTIVE:
The objective is the study of Materials Science phenomena and their interaction with gravity.

DESCRIPTION:
The phenomena being studied include diffusion, coarsening, solution crystal growth, physical vapor transport, pattern formation in solidification or 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).

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 1993:
Mixing phenomena in variable g-fields have been extensively modeled to show the transitions from diffusive to turbulent mixing as the driving force is varied.

TASK INITIATION: 10/92  EXPIRATION: 9/93
PROJECT IDENTIFICATION: 674-21-05-02
RESPONSIBLE CENTER: LeRC
II. MSAD Program Tasks — Ground-based Research

Discipline: Materials Science

Microgravity Materials Science Laboratory

PRINCIPAL INVESTIGATOR: Mr. Thomas K. Glasgow

NASA Lewis Research Center (LeRC)

CO-INVESTIGATORS:
No Co-I's Assigned to this Task

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

OBJECTIVE:
The Microgravity Materials Science Laboratory (MMSL) laboratory was established to provide to researchers from industry, academia, and the government access to specialized equipment and experienced personnel, to aid in the development of space flight concepts, experiments, and hardware. The laboratory is equipped with a number of unusual experimental and computational facilities; it is staffed by engineers from several disciplines and by technicians who are familiar with microgravity practices.

DESCRIPTION:
The MMSL may also be used in preparation for experiments to be conducted in other ground-based, reduced-gravity environments such as those provided by drop-tubes, aircraft, or rockets. The computational facilities may be used to model the expected fluid-flow and heat transfer in microgravity experiments.

SIGNIFICANCE:
In the MMSL, investigators are encouraged to take the first step toward defining space flight experiments for later performance on the Space Shuttle or on Space Stations Freedom. Another use of the laboratory is for postflight investigations.

PROGRESS DURING FY 1993:

MMSL was used primarily by Lewis researchers, although cooperative projects have been performed with Westinghouse, GTE, Marshall Space Flight Center (MSFC), Case Western Reserve University (CWRU), Cleveland State University (CSU), University of Colorado, Texas Instruments, University of Toledo, Ohio State University, and the Space Experiments Division. The MMSL provides facilities for the Programmable Multi-Zone Furnace (PMZF) project, the Advanced Furnace Technology ATD project, the Laser Light Scattering ATD project, and the Surface Light Scattering Instrument ATD project.

STUDENTS FUNDED UNDER RESEARCH:

Total Students: 3

BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:

II. MSAD Program Tasks — Ground-based Research

Discipline: Materials Science


Presentations:

Proceedings:


II. MSAD Program Tasks — Ground-based Research

Discipline: Materials Science

Combustion Synthesis of Materials in Microgravity

PRINCIPAL INVESTIGATOR: Prof. Irvin Glassman
Princeton University

CO-INVESTIGATORS:
No Co-I's Assigned to this Task

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

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 fluids builds on previous theoretical developments at Princeton.

DESCRIPTION:
A comprehensive experimental and theoretical program is planned to synthesize metallic and nonmetallic nitrides (especially titanium nitride) under microgravity conditions, and to understand the underlying combustion mechanisms. The process 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.

SIGNIFICANCE:
Microgravity prevents particle settling, agglomeration, and liquid/metal film formations; permits the use of suspensions and particles of specified characteristics; and facilitates the production of nitride powders of high purity, uniformity and specificity. The novel use of supercritical nitrogen provides a supply of reactant, and—at conditions near the critical temperature and pressure—not only avoids bubble formation that occurs when liquid nitrogen is used, but also provides—because of the very high isothermal compressibility near the critical point—a means for greatly changing reactant density through only a modest change in pressure.

PROGRESS DURING FY 1993:
To date, exploratory experiments characterizing the reaction of titanium powders with gaseous and liquid nitrogen have been conducted. Initial powder packing densities have been varied in order to probe the effect of porosity on the yield of nitride product and on the structure of the combustion wave. Combustion waves that transform titanium to titanium nitride when compacted titanium powders are immersed in liquid nitrogen have been demonstrated confirming Russian reports of this phenomenon. Examination of the combustion of slurries of metal particles in liquid nitrogen has begun. Companion studies on the reactions of single particles of zirconium in gaseous environments have also commenced.

STUDENTS FUNDED UNDER RESEARCH:

Total Students: 1

TASK INITIATION: 11/92 EXPIRATION: 10/95
PROJECT IDENTIFICATION: 674-26-05-09
RESPONSIBLE CENTER: LeRC

II-412
Noncontact Thermal, Physical Property Measurement of Multiphase Systems

PRINCIPAL INVESTIGATOR: Dr. Robert H. Hauge
Rice University

CO-INVESTIGATORS:
J.L. Margrove
Rice University

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

OBJECTIVE:
Measurement methods for high-temperature materials in the liquid state are made difficult or impossible due to reactions with the support or container materials. Levitation methods (electromagnetic, electrostatic, aerodynamic, and acoustic) provide a containerless approach to sample positioning. Electromagnetic levitation also provides in situ heating and stirring of the sample. Our goal is to develop noncontact techniques of measurement for all relevant physical and chemical properties of the liquid state. Properties measurements which we are currently emphasizing are measurements of temperature, sample optical constant, density, surface tension, heat capacity, enthalpy, and thermal conductivity. The equipment allows for all of the above measurements to carry out of the same sample. Measurements of thermal conductivity are compromised by electromagnetic stirring forces and will require the quiescent environment of microgravity for accurate measurement.

DESCRIPTION:
Measurement techniques for the properties are as follows:
1. Temperature: A simultaneous measurement of sample brightness centered at 632 mm and the surface emissivity is made with an integrated multicolor pyrometer ellipsometer. This gives a noncontact measure of temperature with no assumptions. The surface refractive index and extinction coefficient are also obtained.

2. Density: A triggered high-resolution video camera image of the liquid drop is obtained with backlighting of the sample by a 670-mm 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 measured by following the fluctuation of reflected laser light.

4. Enthalpy and heat capacity: Drop calorimetry is currently used to measure the total sample heat content as a number of temperatures.

5. Thermal conductivity: Initial efforts are being made to determine the time-dependent temperature profile of a sample relative to spot heating with a CO₂ laser.

6. 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.

PROGRESS DURING FY 1993:
New current concentrator designs have been shown to provide efficient electromagnetic (EM) coupling to the sample with symmetric positioning forces.
An integrated video camera, ellipsometer, and multicolor pyrometer instrument has been designed and is in a construction and testing phase.

<table>
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Fluid Flow in Partly Solidified Systems

PRINCIPAL INVESTIGATOR: Prof. Angus Hellawell
Michigan Technological University

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

Task Objective, Description & Significance:

Objective:
This research is concerned with the occurrence of local macroscopic convective patterns that develop in bulk liquids during alloy solidification and lead to segregation in the solidified material.

Description:
Studies will be conducted of channel formation in molten salts (NaF-NaCl) and organics (SCN). We intend to correlate the thermal, compositional, and dimensional conditions for channel formation in the salts and organics and compare the results with those already available for lead-based alloys and the aqueous system.

Significance:
Thermosolutal plumes are interesting scientifically because they represent a convective stability problem, and industrially because their occurrence in metal casting causes the defect known as "frackle."

Progress During FY 1993:
Thermosolutal plume flow has been studied in some detail in metallic, aqueous and organic systems. Analysis of directly observed flow velocities in the transparent systems indicates that these are around 0.1 m s⁻¹ in metals. The development of polycrystalline structure in segregation channels has been demonstrated in an aqueous analogue. The present work is directed towards the measurement of the density of dendrite fragments which are distributed by plume/channel flow, their survival and growth to form equiaxed crystals. It is anticipated that these observations will provide a physically realistic model for predicting the grain structure of castings.

Students Funded Under Research:

Total Students: 2

Task Initiation: 12/89  Expiration: 2/93
Project Identification: 674-25-05-19
Responsible Center: LeRC

Bibliographic Citations for FY 1993:

Journals:


Steube, R. S., and Hellawell, A. An alternative approach to modeling the grain structure of castings. ASME, Micro/Macro Scale Phenomena in Solidification, 218, 73-83, 1993.
II. MSAD Program Tasks — Ground-based Research

Discipline: Materials Science

Non-Equilibrium Phase Transformations

PRINCIPAL INVESTIGATOR: Dr. Kenneth A. Jackson
University of Arizona

CO-INVESTIGATORS:
B. Zelinski
University of Arizona

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

OBJECTIVE:
Recent computer simulations have resulted in a breakthrough in the "fundamental understanding of the physics associated with phase changes. This includes solidification, crystal growth, condensation from vapor, etc." The goal of this program is to continue computer simulations and to initiate an experimental study to explore the implications and insights provided by this breakthrough.

DESCRIPTION:
The experiments involve an analysis of microsegregation that requires a microgravity environment to minimize the effects of convection. A program that combines simulations with experimental studies is viewed as essential, not only in providing new insights for the experimental work, but also in keeping the simulations relevant to the real world.

Research Approach — The real world implications of this breakthrough will be explored, since the insight provided can suggest new experimental studies and new approaches to old problems. Published studies of rapid crystallization of silicon and of metals following laser annealing can be compared, in detail, with the new simulations. In ceramic glass-forming systems where phase transformations usually take place far from equilibrium, even though the absolute growth rates are not rapid, the segregation phenomena are not understood. The phases that form and the amount of segregation and second phase transformation depend on the transformation temperature. The study of these effects will involve using the growth of the commercially-important cordierite phase in magnesium-aluminum silicates as the model system. In this system, the sensitivity of nucleation to surface defects could be mitigated by using containerless processing. The Czochralski growth of silicon and the growth of many crystals from solution exhibit an orientation of the incorporation of impurities.

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. Near equilibrium, phase transformations depend primarily on the thermodynamics of the system; the redistribution of components caused by the phase transformation is predictable from the appropriate phase diagram. However, far from equilibrium when the transformation proceeds at a rate such that there is little time for diffusion, there is little redistribution of the components caused by passage of the interface. The role of thermodynamics in the rapid-growth regime and the nature of the transition from one of these regimes to the other have not been understood.

The computer simulations have clearly demonstrated the difference between the diffusionless and the diffusion-dominated regimes of growth. There is still much to be done in this area, and the studies to date are but a beginning: they demonstrate that this method of simulation can indeed capture the essence of transformations that occur in the real world. This simulation method can be used to explore, in detail, the transition between the diffusionless regime and the diffusion-dominated regime. It can be used to study the progress of a series of isothermal transformations that span the regime from above to below the glass transition temperature. It can be
used to predict growth rates, transformation temperatures, and transformation-induced segregation effects. It can be used to study the early stages of interface instability, including not only the growth rate dependence of the distribution coefficient, but also the effects of having a crystal composed of discrete atoms growing into an alloy that has randomly-distributed atoms rather than a crystal bounded by a mathematical surface growing into an alloy of uniform composition. It can be used to explore eutectic growth.

**Progress During FY 1993:**

Recent computer simulations have resulted in a breakthrough in understanding this phenomena. Based on a simple Kossel-Stranski model for an alloy, which is equivalent to the Ising spin-1 model, and using Monte Carlo methods to evaluate the kinetic behavior, the important aspects of the experimental observations on real systems have been reproduced. The computer simulations were first directed to the diffusionless regime, which corresponds to growth far from equilibrium, where the phase transformation rate is fast compared to the rate of atom diffusion. It was discovered that the transformation is governed by the total free-energy differences between the two phases, rather than by individual transformation probabilities. This result provides a new framework for comparison with experimental results in the rapid growth regime. This regime includes splat quenching, laser and electron beam melting, rapid quenching of small droplets to achieve fine dispersions of uniform composition for subsequent sintering, phase transformations in glass-forming systems, and nucleation and the early stages of growth in alloy systems and in multicomponent ceramic or glass systems.

When there is significant diffusion as the interface passes, segregation in the simulations is presented by the liquidus and solidus lines of the phase diagram. As the growth rate increases relative to the diffusion rate, the distribution coefficient approaches unity, in agreement with experiment. The distribution of dopants found following the laser melting of silicon where the effective distribution coefficient was found to be hundreds of times larger that the equilibrium value was reproduced. Interface instabilities, interface breakdown, and the early stages of cellular or dendritic growth in our simulations have been observed. The results of these computer simulations have a direct bearing on any phase transformation that is occurring far from equilibrium in a material with more than one component. This includes not only rapid growth techniques such as splat quenching and laser zapping, but also the evolution of dendritic and cellular structures, most phase transformations in glassy systems, thin film deposition far from equilibrium, and non-equilibrium growth from solution including sectoring and striation effects.

**Students Funded Under Research:**

| PhD Students | 2 |

**Bibliographic Citations for FY 1993:**

**Journals:**


**Proceedings:**


II. MSAD Program Tasks — Ground-based Research

Discipline: Materials Science

Presentations:
Jackson, K.A. A perspective on crystal growth, American Association for Crystal Growth, Baltimore, MD, August, 1993.

PATENTS:
Patent Status: Pending  U.S. Patent #: Undetermined
Jackson, K.A. "Growth of a Superconductor Material in a Fluxed Melt and Article of Manufacture."
Combined Heat Transfer Analysis of Crystal Growth

**Principal Investigator:** Dr. Mohammad Kassemi
**NASA Lewis Research Center (LeRC)**

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

**Task Objective, Description & Significance:**

**Objective:**
Radiation heat transfer affects crystal growth in both space and ground-based processing. The role of radiation heat transfer is even more prominent in the low-gravity environment of space where convection heat transfer is minimized. The technical objective of this task is to provide a quantitative understanding of the effects of radiation heat transfer on crystal growth.

**Description:**
A radiation heat transfer model is developed based on a node-to-node exchange for a generally absorbing, emitting, scattering media, and for multidimensional, complicated geometries encountered in crystal growth. A combined conduction, convection, radiation model will be developed and applied to different class of crystal growth problems in which either the solid, the melt, or both of the phases are semitransparent to thermal radiation. The radiation model will take into account wavelength-dependant semitransparency of crystals, such as BSO. The model will be applied to existing experiments and will be used to demonstrate the role of radiation in the low gravity environment.

**Significance:**
The advantage of the this task's approach is that the radiation model can be easily incorporated into existing finite-difference and/or finite-element formulations.

**Progress During FY 1993:**
A radiation exchange model was developed which calculates radiation heat transfer in an absorbing, emitting scattering medium based on a node-to-node exchange.

The radiation model was successfully combined with an in-house prepared finite difference code to study the interaction of radiation with natural convection under both 1-g and low-g conditions. Results of the simulations were compared to limiting benchmark solutions available in the literature to verify the accuracy of the radiation model.

The verified radiation model is currently being incorporated into a finite element code for crystal growth. The model has been expanded to include the effect of the changing geometry (as the interface deforms and moves) on the radiation exchange.

The model will be used to investigate the effect of radiation exchange and crucible radiative properties on solidification of BSO, a semitransparent oxide crystal.

The model will be expanded to include the effects of wavelength-dependant radiative properties and will be used to investigate the role of radiation in other relevant crystal growth experiments.
BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:

Presentations:
Fundamentals of Thermomigration of Liquid Zones through Solids

**PRINCIPAL INVESTIGATOR:** Prof. Michael J. Kaufman

**CO-INVESTIGATORS:**
- R. Abbaschian
- A. Gokhale

**TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:**

**OBJECTIVE:**
Currently, significant resources in terms of expert manpower as well as direct costs are being expended to develop new high-temperature structural intermetallics, such as MoSi2 and NiAl, and fast semiconductors, such as GaAs. Despite considerable efforts in these areas, the intrinsic properties of many of the intermetallics remain poorly understood to the degree needed to moderate their development. 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.

Conventional crystal growth techniques have not proven successful in this regard because of 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, 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.

**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.

Research Approach — The experimental studies will be performed in three stages. In the first stage, experiments at the University of Florida (UF) will be used to define the proper solute/solvent, temperature/thermal gradient combinations for TGZM and also to develop the requisite instrumentation for monitoring interface position and movement using ultrasonic methods and interface temperature using Seebeck measurements. In addition, during this initial stage the research team at UF will provide input to the designers of the PMZF concerning the requirements for the effective utilization of TGZM in space-based processing. In the second stage, the preliminary experiments at UF will be repeated under the more controlled conditions of the PMZF in order to gain an understanding of the effect of convective-diffusive mass transfer on interfacial kinetics and interface stability under earth gravity. These will be compared with the third stage microgravity experiments which will be carried out on the PMZF in the space shuttle middeck where purely diffusive transfer will dominate so that the effects of convection can be ascertained.
It is anticipated that significant, hitherto unavailable data on liquid diffusivities will also be generated during these experiments. Also, the low-growth temperatures characteristic of the TGZM process circumvent the common problems of crucible contamination and loss of stoichiometry due to vaporization as frequently encountered using other, more conventional crystal-growth techniques. Obviously, such low-growth temperatures require much lower power inputs and result in enhanced safety during both ground operations and space flight. 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.

**SIGNIFICANCE:**
This research is intended to circumvent these problems by using the relatively unique technique of TGZM to grow the single crystals of interest.

**PROGRESS DURING FY 1993:**
TGZM is a relatively novel and significantly underutilized technique of moving a liquid zone through a solid. Since the first paper on TGZM by Pfann more than 35 years ago, various aspects of the method (e.g., length of the liquid zone, composition of the zone, rate of travel of the zone, and shape of the zone) have been modeled by Tiller. The technique differs from other zone melting techniques in that the zones are much smaller, have a high solute content, and are made to move typically by the imposition of a stationary temperature gradient. Significantly, the process is usually carried out at temperatures well below the melting point of the candidate crystalline material.

**STUDENTS FUNDED UNDER RESEARCH:**
**PhD Students:** 2

**BIBLIOGRAPHIC CITATIONS FOR FY 1993:**

**Journals:**


Proceedings:


PATENTS:

Jayashankar, S., and Kaufman, M.J. "Tailored Silicide/ Silicon Carbide Composites by Mechanical Alloying and In-Situ Displacement Reactions."

Kinetics of Phase Transformations in Glass Forming

**Principal Investigator:** Prof. Kenneth F. Kelton

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

**Task Objective, Description & Significance:**

**Objective:**
The objectives of this research are to develop computer models for realistic simulations of first-order phase transformations, in particular crystallization of liquids and glasses, and to design experiments to test those models. This research will lead to improved methods for the analysis of kinetic studies of these transformations, allowing, for example, kinetic parameters for nucleation and growth to be determined from peak profile studies of nonisothermal differential scanning calorimetry (DSC) measurements of crystallization. These new techniques will have wide applicability for phase transformation studies. In particular, they will allow real-time experiments of phase stability and transformation to be carried out in a microgravity environment.

**Description:**
Glasses (primarily silicate based) that devitrify polymorphically by homogeneous nucleation are being studied experimentally and by computer modeling. Lithium disilicate (Li₂O·2SiO₂) is used for most calculations and experimental measurements since the necessary kinetic and thermodynamic parameters are best known in that system. Other glasses studied include soda-lime silicate (Na₂O·2CaO·3SiO₂) and barium disilicate (BaO·2SiO₂). The computer modeling of glasses (primarily silicate-based ones) is done at Washington University. The silicate glasses are prepared by Drs. C. Ray and D. Day of the University of Missouri, Rolla; they also study the devitrification kinetics experimentally with differential thermal analysis (DTA). Transmission electron microscopy (TEM) and DSC measurements are made at Washington University.

Research Approach — This research is a theoretical investigation of the nucleation and crystallization kinetics in glass forming systems. The importance of nonsteady-state nucleation and viscosity in glass formation is being investigated. Methods are being developed for the calculation of nonsteady state crystallization using isothermal and non-isothermal annealing. Calculations utilizing these methods on Li₂O·SiO₂ and Na₂O·2CaO·3SiO₂ will be performed, and several anomalous experimental results that appear to contradict the theoretical calculations will be investigated and explained.

**Significance:**
The computer models describing nucleation and growth under isothermal and non-isothermal conditions by simulating directly the evolution of the non-equilibrium cluster distribution will have wide applicability for phase transformation studies. In particular, they will allow real-time experiments of phase stability and transformation to be carried out in a microgravity environment.

**Progress During FY 1993:**
The computer models describing nucleation and growth in bulk systems have been written and tested. They have already been used for a variety of investigations. Several of the proposed projects have been completed and are listed below.
A recent suggestion that the temperature of the maximum nucleation rate can be estimated from DSC studies of glasses that were preannealed at different temperatures was investigated by simulating those experiments with our computer model. The proposed method was shown to be valid when the nucleation and growth regions are well separated, such as for lithium disilicate glass, but not true when these regions overlap.

Estimates of the macroscopic growth velocity from the kinetic and thermodynamic parameters appropriate for describing the nucleation rate and the transient time were made and checked against experimental data and against cluster growth calculated directly from the rate equations for nucleation. The calculated growth velocity agreed within the probable experimental error of the nucleation data, demonstrating that the same atomic mobility governed both processes. This work, done in collaboration with M. Weinberg, is being prepared for publication.

A general investigation of the effect of different quench rates on glass formation and stability was carried out using parameters that were appropriate for lithium disilicate glass. The stability of the glasses was demonstrated to increase markedly with increasing quench rate due to a smaller number of quenched-in nuclei and a smaller mean nuclei size. Upon reheating, the nucleation rate was predicted to attain a larger value than the steady state value at a higher temperature than the steady state peak. This was shown to arise naturally from the complex relaxation behavior of the cluster distribution. Finally, a method frequently used to account for transient nucleation in analyzing isothermal transformation data was found to be invalid.

Quantitative fits were made to DSC peak profiles as a function of scan rate for lithium disilicate glass, using the kinetic and thermodynamic constants obtained from nucleation data, demonstrating the validity of our approach. Calculations of changes in DSC peak height, width and temperature, when the glasses were first scanned through the nucleation temperature zone, were in good agreement with experimental data. Since the samples used were powdered, finite sample size effects were shown to be important. These results, made in collaboration with C. Ray, are being readied for publication.

We have shown that all existing methods for analyzing nonisothermal DSC or DTA data for nucleation and growth transformations are fundamentally wrong by using them to analyze data produced by our model for glasses with known kinetic and thermodynamic parameters. A manuscript describing this work is in preparation.

STUDENTS FUNDED UNDER RESEARCH:
PhD Students: 2

BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:

Proceedings:
II. MSAD Program Tasks — Ground-based Research

Modeling/Experimental Studies of Droplet Pushing in Miscibility-Gap Alloy Solidification Under Low-g Conditions

PRINCIPAL INVESTIGATOR: Prof. Dr. William B. Krantz
University of Colorado

CO-INVESTIGATORS:
No Co-I's Assigned to this Task

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

OBJECTIVE:
The focus of this study is to understand the mechanism of droplet rejection by an advancing solidification front during directional solidification of a miscibility-gap alloy.

DESCRIPTION:
The first phase of this research is to obtain qualitative and quantitative information on the droplet-pushing mechanism by observation of the solidification front in transparent organic miscibility-gap systems. The second phase is to develop a mathematical model of droplet pushing that predicts the critical drop diameter as a function of processing conditions and fluid properties. The final phase is to determine the proper form of the thin film model for the droplet-pushing problem.

SIGNIFICANCE:
The droplet-pushing phenomenon is thought to be an important factor governing the formation of microstructures in these alloys.

PROGRESS DURING FY1993:
This research investigates a mechanism of great importance to the formation of microstructure in a class of materials known as "miscibility-gap alloys". Such an alloy is formed by cooling an initially homogeneous melt through a region of liquid/liquid immiscibility until a solidified dispersion is formed. These alloys have potential applications as superconductors or as optoelectronic materials. Obtaining such desirable properties in these materials requires that micrograins of one phase be uniformly dispersed in the other phase. Problems associated with density-driven massive phase separation have led to considerable effort to form such alloys in a low-gravity environment. However, several experiments indicate that coarsening of the dispersion and phase separation still occur to an undesirable extent under low-g conditions owing to pushing of microdroplets by the advancing solidification front in the continuous phase as well as several other mechanisms. It has been observed that for a given solidification rate, there is a critical droplet size below which it is pushed and not engulfed by the front. Droplet pushing is a wetting dynamics phenomenon governed by the relative magnitude of the interfacial energies between the three coexisting phases at the solidification front. Previous models of this phenomenon have concentrated on solid particle pushing. The objectives of this research are to formulate a mathematical model appropriate to droplet pushing with special emphasis on the relationship between physical properties of a specific material and the driving force for wetting dynamics and then to validate the model with experimental data on a well-characterized transparent organic miscibility-gap system which solidifies in a manner similar to metals.

Experiments were performed at the NASA Marshall Space Flight Center in cooperation with Dr. Donald Frazier. Succinonitrile (SCN) was chosen as the primary component of the mixtures to be studied because of its crystallographic properties which cause it to solidify in a manner similar to metals. Camphene was chosen as the second component because it has a low monotectic composition (~3 wt%). SCN-ethanol mixtures were also studied.
in order to test the hypothesis of Grugel and coworkers that droplet pushing occurs in this system. The experiments were performed on a gradient-stage microscope in which the solidification front of thin horizontal samples was observed during directional solidification. Direct measurements of critical drop size were made using a micrometer eyepiece. Individual 35mm photographs were taken to capture qualitative features of interest. Time-sequence 35mm photographs were taken to capture the evolution of certain events. Evolution over longer periods of time was captured using a video camera.

Important properties of the SCN-camphene system were measured to permit interpreting the gradient-stage-microscope experiments. The monotectic temperature was measured using a differential scanning calorimeter. The densities of the two liquid phases formed prior to solidification were measured using a vibrating U-tube digital density meter. The viscosities of the liquid phases were measured using a falling needle viscometer. The latter studies resulted in a substantive improvement to the falling needle viscometer technique which was published in the Review of Scientific instruments. The interfacial tensions were measured using a pendant-drop tensiometer. Substantive improvements in the pendant-drop tensiometer technique were also made by the PIs; a paper on these improvements is being submitted to the Review of Scientific Instruments.

The gradient-stage microscope experiments on the SCN-ethanol system did not display droplet-pushing for the same conditions as had been observed by Grugel and coworkers. It was found that the observed morphology was markedly dependent on the width of the test cell. Droplet bridging across the cell occurs in thin cells (50 μm). In thicker cells (150 μm) an irregular fiber structure is formed similar to that reported by Kaukler and Frazier for SCN-water alloys and by Song and Hellawell for SCN-benzene alloys.

SCN-camphene mixtures of monotectic composition were solidified at growth rates in the range of 0.1 to 1 μm/s with a temperature gradient of 36.5° C/cm and a cell thickness of 150 μm. Although drop pushing was observed in those experiments, a unique critical drop size for engulfment by the solidification front was not observed; rather a distribution of sizes for the engulfed drops was observed. We believe that the critical drop size is nonunique for a given solidification rate owing to solute-diffusion effects on the local growth rate and the shape of the solidification front. An interesting observation was that under some conditions, the droplets engulfed by the solidification front would continue to grow to form thin fibers. For solidification under hypermonotectic conditions, the solidification front became unstable; droplet pushing in combination with the nonplanar solidification front then caused bunching together of several droplets. These experiments also suggested a means for mitigating the effect of drop pushing on coarsening in these miscibility-gap alloys. It was observed that mechanical vibration of the apparatus could cause the solidification front to engulf very small droplets. This observation suggested that the application of mechanical vibrations of controlled amplitude and frequency might be used to produce fine-grained alloys from these materials. A paper summarizing these experimental studies has been submitted to the Journal of Crystal Growth.

In order to interpret the results of the experimental studies, a mathematical model was developed to describe the drop pushing. This model built upon prior studies of the pushing of solid particles. As such, it included the effects of a mobile liquid surface and a freely deformable solidification front; in particular, this model allowed for the influence of the different thermal conductivities of the drop and matrix material on the shape of the solidification front. This model incorporated a more rigorous treatment of the disjoining pressure effects in the thin film which separates the pushed drop from the advancing solidification front. This model shows that the mobile drop interface reduces the drag on the drop so as to cause more than a 100% increase in the critical drop size over that predicted by models for solid-particle pushing. Interestingly, gravitational forces were predicted to have a negligible influence on the predicted critical drop size for engulfment. Thermocapillary motion owing to the drop being in a temperature gradient was also shown to have a negligible effect on the critical drop size. The interfacial tension between the solidification front and the melt was predicted to have a significant effect on the critical drop size. Lower values of the interfacial tension permit the solidification front to deform in such a way as to lower the drag on the droplet. A decrease in the viscosity of the drop caused an increase in the critical drop size owing to reduction in the drag force.
The thermal conductivity of the drop relative to that of the surrounding matrix liquid was shown to have a pronounced effect on the critical drop size. When the drop thermal conductivity is higher than the matrix conductivity, the solidification front is depressed beneath the drop and the critical drop radius is decreased significantly. This is because the solid/melt interface is curved around the drop more tightly, thus increasingly the hydrodynamic drag force and lowering the disjoining pressure gradient. When the drop conductivity is lower than the matrix conductivity, a hump is formed in the solid/melt interface and the critical drop size is increased. Preliminary modeling studies of solutal effects indicate that these may have a very significant effect on the critical drop size owing to their influence on the supersaturation in the matrix near the solidification front. A paper summarizing these modeling results will be submitted to the Journal of Crystal Growth in the near future.
Containerless Property Measurement of High-Temperature Liquids

PRINCIPAL INVESTIGATOR: Dr. Shankar Krishnan

CO-INVESTIGATORS:
P.C. Nordine

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

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 (T_m+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.

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 Molelectron 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 Molelectron 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.

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 1993:
An automated rotating analyzer pulsed dye laser spectroscopic ellipsometer was designed and built during the first three months of the project. It consists of a nitrogen laser-pumped pulsed dye laser and a rotating analyzer polarization state detector (PSD). The incident laser beam and PSD axes are placed at an angle of 135° in the horizontal plane and perpendicular to two windows on the electromagnetic levitation chamber. Specimens are levitated and melted at the intersection of these axes, using specially designed levitation coils that do not obscure the equatorial plane of the levitated drop. Incident laser light of a fixed polarization state is reflected into the PSD and analyzed. The PSD includes light collection and focusing optics, a beam splitting Glan-Thompson prism mounted in a computer-operated rotation stage, and a pair of photodiodes.

The new apparatus was successfully used to determine the optical properties of liquid aluminum at wavelengths from 360,990 nm (1.2-3.5 eV) at a temperature of 1,580 K. Data at eight wavelengths were obtained over the temperature range from 1,200 K-1,800 K. Structure in the optical properties of liquid aluminum was demonstrated to occur at photon energies of ca. 1.5 eV (820 nm wavelength). This is the first demonstration that liquid aluminum has structure in the optical properties and displays the nonfree electron behavior characteristic of solid aluminum. Error analyses have been conducted to establish the accuracy of results. Further analysis of the results is in progress and a paper on this work is being prepared for publication. Experiments are in progress to develop electromagnetic levitation coils and conditions for measurements on liquid zirconium and nickel.

Measurements on liquid and solid Zr have been completed. Liquid Zr exhibits 4 peaks in the optical conductivity in the 1.3-3.5 eV photon energy range, including two closely spaced peaks in the near infrared. The spectral emissivity of liquid and solid Zr have been measured in the wavelength range 0.35-0.95 μm. Preliminary measurements have also been conducted on rare-earth liquid metals including Gd, Y, and Lu. New measurements are being planned on liquid Ni-Zr and Ni-Nb alloys and other materials of interest to the TEMPUS PI's.
II. MSAD Program Tasks — Ground-based Research  

Task Initiation: 5/92  Expiration: 5/95  
Project Identification: 694-25-07-05  
Responsible Center: JPL  

II-431
II. MSAD Program Tasks — Ground-based Research

Chemical Vapor Deposition of High-to-Superconducting Films in a Microgravity Environment

PRINCIPAL INVESTIGATOR: Prof. Moises Levy
University of Wisconsin

CO-INVESTIGATORS:
B. Sarma
University of Wisconsin

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

OBJECTIVE:
The basic goal of this research is to grow and process single-crystal, high-temperature superconducting thin films under microgravity conditions.

DESCRIPTION:
Superconducting thin films will be grown by chemical vapor deposition in three different transport reactors in order to investigate the effects of gravitationally-induced convection currents on the superconducting characteristics of the films. Different orientations with respect to gravity will be used.

SIGNIFICANCE:
It is believed that microgravity will allow for unique growth conditions for vapor-phase transport and for the formation of single crystalline materials during postprocessing to the superconducting phase.

PROGRESS DURING FY 1993:

A compact chemical vapor deposition MOCVD system with a single organometallic precursor was designed, built and tested. A model for the vaporization and mass transport of mixed organometallics from a single source for thin film MOCVD was developed. It was shown that a stoichiometric gas phase can be obtained from a mixture of the organometallics in the desired mole ratios, in spite of differences in the volatilities of the individual precursor compounds. Proper composition and growth rate can be obtained by ensuring that the organometallics are completely sublimed after the slotted borosilicate tube containing the compacted mixed organometallics has traversed the short heating zone of the vaporizer. \( Y_{1.18}\)Ba\(_2\)Cu\(_3\)O\(_7\) superconducting films with \( T_c \)'s around 90K were obtained on [100] Y stabilized cubic zirconia and [100] MgO substrates. Under ideal conditions the high \( T_c \) films were oriented with the c-axis perpendicular to the substrate plane. This is the preferred orientation for achieving large current densities in the plane of the film. The pressure of the argon carrier gas was between 5 and 7 torr and that of the oxygen gas was also within this range. The combined flow rate was between 200 and 400 standard cubic centimeter per minute.

The compact MOCVD system may be rotated in order to determine the effect of gravitational fields on the growth behavior of the high \( T_c \) superconducting films. In the first orientation investigated, the compacted precursor vaporizer tube was moved downward through the heating zone and the heated substrate was held upside down in the reactor arm of the U shaped system. Thus the gas flow rate past the reactor was against the gravitational field. It was found that the central part of the circular film was about 250 nm thinner than the outer part for a 1000 nm average thickness film. Thus fluid hydrodynamics affected the diffusion controlled growth rate of the films even at the low pressures of the MOCVD system. A microgravity environment may allow the use of lower gas flow velocities thus making the diffusion controlled growth rate process more uniform along the diameter of the films. Two other configurations of the MOCVD system with respect to the gravitational field are being investigated.
II. MSAD Program Tasks — Ground-based Research

Discipline: Materials Science

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BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:


II. MSAD Program Tasks — Ground-based Research  

Microstructural Development during Directional Solidification of Peritectic Alloys

**Principal Investigator:** Dr. Thomas A. Lograsso  
**Co-Investigators:**  
R. Trivedi  
J. Verhoeven

**Task Objective, Description & Significance:**

**Objective:**  
The mechanisms of and conditions required for the formation of peritectic solid dendrites, cells, or planar growth in between the primary solid dendrites and the transition between these structures will be investigated. In addition, alternating bands of solid phases have been observed in peritectic systems. However, the precise conditions under which these bands form and the mechanism for the formation of these bands are not clearly understood. It had been postulated that the development of these different structures is caused by fluctuations in the composition of the liquid. A model will be developed that will predict the compositional changes that occur under the assumption of no convection in the liquid. This model will be compared to experimental results to evaluate the importance of convective effects on the formation of various structural characteristics.

**Description:**  
This research investigation will focus on:

- 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; and
- Determining the effect of convection-free conditions on the formation of a banded structure during directional solidification of a two-phase peritectic alloy.

**Research Approach** — The research approach is to look at two systems, Sn-Cd and Pb-Bi. Exploratory experiments will also be carried out on Ag-Sn alloys, a system that has not been studied and which may be more experimentally tractable. These systems will allow all four possibilities of stabilizing and destabilizing density gradients to be observed. In addition, the Sn-Cd and Pb-Bi systems have been well characterized in previous ground-based investigations.

**Significance:**  
This research effort will establish the ground-based data required for the identification of the appropriate alloy system, composition, and growth condition for microgravity processing.

**Progress During FY 1993:**

Theoretical models for the formation of bands have been based on pure diffusive regimes, whereas convection effects are believed to be important in the regime where bands form. Recent experimental studies have confirmed the presence of significant convection effects in the liquid ahead of the bands. The formations of bands imply that the interface is not moving at a constant rate that is imposed externally. Rather, it oscillates with time with an average velocity that may be equal to the externally imposed velocity. The precise physics that causes the oscillation in the interface motion is not yet established, but it appears that convection effects in liquid play a key role in causing the
interface to oscillate. During the upcoming year, the growth condition regimes, temperature gradient, and growth rate, under which planar, cellular, and dendritic microstructures develop in peritectic alloys, will be established. The following systems will be investigated, since they represent a range of stabilizing and destabilizing density gradients: Sn-Cd, Pb-Bi, and Ag-Sn. A model will be developed to predict the response of solidification morphology to the absence of convection in the melt. These investigations will establish the ground-based data required for the identification of the appropriate alloy system, composition, and growth conditions for microgravity processing.

TASK INITIATION: 7/93 EXPIRATION: 7/96
PROJECT IDENTIFICATION: 674-25-08-28
NASA CONTRACT NO.: NAG8-963
RESPONSIBLE CENTER: MSFC
II. MSAD Program Tasks — Ground-based Research

Discipline: Materials Science

Numerical Investigation of Thermal Creep and Thermal Stress Effects in Microgravity Physical Vapor Transport

PRINCIPAL INVESTIGATOR: Dr. Daniel W. Mackowski
Auburn University

CO-INVESTIGATORS:
R. Knight
Auburn University

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

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.

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.

Research Approach — 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.
SIGNIFICANCE:
Qualitative theoretical arguments strongly indicate that thermal creep and thermal stress could lead to a significant convective flow of fluids within the ampoule.

PROGRESS DURING FY 1993:
A code to examine binary PVT in cylindrical ampoules has been developed and is being verified against previously published modeling results.

STUDENTS FUNDED UNDER RESEARCH:  
MS Students: 1

TASK INITIATION: 6/93  EXPIRATION: 6/95
PROJECT IDENTIFICATION: 674-21-08-18
NASA CONTRACT NO.: NAG8-977
RESPONSIBLE CENTER: MSFC
II. MSAD Program Tasks — Ground-based Research

Polymerizations in Microgravity: Traveling Fronts, Dispersions, Diffusion and Copolymerizations

**PRINCIPAL INVESTIGATOR:** Prof. Lon J. Mathias
University of Southern Mississippi

**CO-INVESTIGATORS:**
R. Lockhead
University of Southern Mississippi
J. Pojman
University of Southern Mississippi

**TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:**

**OBJECTIVE:**
Polymerization studies of bulk-phase diffusion and polymerization in monomer and comonomer mixtures or in monomer droplets in a non-solvent are being performed to study density effects on polymerization.

**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 copolymer 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.

Research Approach — 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 comonomer 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.

**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 copolymer 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 copolymers available for the first time for comonomer compositions which normally lead to random or long-block copolymers under good mixing.

**PROGRESS DURING FY 1993:**
Workers in the former USSR have studied traveling thermal fronts in vinyl polymerizations under 5,000 atm of pressure. Recently, the feasibility of traveling fronts in solutions of benzoyl peroxide in methacrylic acid at ambient pressure was demonstrated at USM Laboratory. After fronts were initiated with a heated wire, the heat of
polymerization spreads to an unreacted region, causing decomposition of the benzoyl peroxide which in turn initiates further polymerization. Work so far on traveling fronts suggests four areas of investigation which could be best addressed by experiments in a low gravity environment:

- Study onset of double-diffusive convection via light scattering under both normal gravity and low gravity;

- Develop a model relating polymerization rate and convective mixing to front velocities and conditions (this area applies to all three projects);

- Determine and model the mechanism of front pulsations; and

- Attempt copolymerization where composition varies along the direction of front propagation.

**STUDENTS FUNDED UNDER RESEARCH:**  
PhD Students: 3

**TASK INITIATION:** 6/93 **EXPIRATION:** 6/95

**PROJECT IDENTIFICATION:** 674-26-08-10  
**NASA CONTRACT NO.:** NAG8-973  
**RESPONSIBLE CENTER:** MSFC
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:
No Co-I's Assigned to this Task

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

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. Product purity and particle size are two important concerns in producing the desired form of the advanced ceramic. The ceramic products should have the correct particle size for both efficient handling of the ceramic, and should be in the appropriate size range for subsequent processing and/or function. Control of the chemical reactions and particle size is necessary for the achievement of these goals.

DESCRIPTION:
Titanium diboride (TiB₂) is synthesized according to the reaction Ti+2B=TiB₂. The system was chosen for its relative simplicity, allowing the fundamentals of the reaction to be accurately evaluated. TiB₂ will be 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 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 will be completed in order to examine their effect on the reaction.

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.

PROGRESS DURING FY 1993:
Fine powders of TiB₂ in the 1-10 μm size range have been produced via the combustion synthesis reaction. The variables of composition (inert dilution), green density, atmosphere pressure, position, sample mass, and reactant particle size have been examined to find their effect on the combustion synthesis reaction. An intermediate reaction that occurs only in a reactive environment has been isolated and analyzed. Future work includes conducting these reactions in a microgravity environment in order to examine the effects of gravity and convection on the combustion synthesis reaction.

STUDENTS FUNDED UNDER RESEARCH:
Total Students: 3  MS Degrees: 1

TASK INITIATION: 1/93  EXPIRATION: 12/96
PROJECT IDENTIFICATION: 674-26-05-10
RESPONSIBLE CENTER: LeRC
II. MSAD Program Tasks — Ground-based Research

Discipline: Materials Science

Diffusion, Viscosity, and Crystal Growth in Microgravity

PRINCIPAL INVESTIGATOR: Prof. Allan S. Myerson
Polytechnic University, New York

CO-INVESTIGATORS:
A. Izmailov
Polytechnic University, New York

Task Objective, Description & Significance:

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.

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.

Research Approach — 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.
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.

PROGRESS DURING FY 1993:
Crystallization is a phase separation phenomena that involves two distinct steps: nucleation, or the birth of a new crystal, and crystal growth, or the growth of an existing crystal. Both nucleation and crystal growth can only occur when the solution is supersaturated. A supersaturated solution is a metastable state where the concentration of the solute exceeds the solubility at the given temperature. The metastable, or supersaturated, region is bound on one side by the saturation line (binodal) and on the other side by the spinodal line that marks the limits of the metastable zone. At concentrations above the spinodal, the solution is unstable and will rapidly phase separate.

The diffusion coefficients of KDP and ADP have been measured over the entire undersaturated concentration range. Initial measurements in the supersaturated region have begun. Viscosity, metastable limit, and thermodynamic property measurements in the supersaturated region are also underway.

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BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:


II. MSAD Program Tasks — Ground-based Research

Discipline: Materials Science

An Electrochemical Method to Measure Diffusivity in Liquid Metals

PRINCIPAL INVESTIGATOR: Prof. Ranga Narayanan  University of Florida

CO-INVESTIGATORS:

T. Anderson  University of Florida
A. Fripp  NASA Langley Research Center (LaRC)

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

OBJECTIVE:
A research program will be conducted which will use coulometric titration to obtain benchmark values of oxygen diffusivity in liquid metals. An important aspect of the program is the use of low gravity to significantly reduce the adverse effects of gravity-driven convection in the melt during the diffusivity measurement. Another science objective of this study is to establish a clearer picture of the constitutive behavior of "Fickian diffusion" of oxygen in liquid metals.

The accurate measurement of molecular diffusivity in high-melting-point liquid metals and alloys is of interest from a fundamental viewpoint largely because molecular diffusivity is an important physical property. For the same reason, the behavior of molecular diffusivity with temperature can only throw more light on the constitutive behavior of the binary system.

The "strength" of natural convection of liquid metals in enclosures can be measured by its effect on the mass transfer of a tracer such as oxygen. For this, it is necessary to know accurately the molecular diffusivity of the tracer in the liquid metal and compare it to the effective diffusivity.

DESCRIPTION:
A variety of cell designs and operating procedures in an isothermal environment will be tested and the results compared with measurements in low gravity to determine the magnitude of the errors introduced in I-g.

Research Approach — In this study, an existing experimental "cell" will be used that has been slightly modified by the investigators. The principle of the method is quite straightforward. As an example, consider measuring the diffusivity of oxygen in tin. The experimental arrangement consists of two electrochemical cells sharing a common working electrode. A representation of the cell is given by:

\[
Cu,CuO \parallel YSZ \parallel Q \text{ in Sn} \parallel YSZ \parallel Cu, CuO
\]

Here yttria-stabilized zirconia (YSZ) is the solid state electrolyte. In these experiments, one cell will be used to establish a known boundary condition, usually a negligibly small concentration, while the second cell will be operated in an open circuit mode to measure the concentration at the opposite boundary. Rhenium wire welded to copper leads will be used to contact the melt while copper wires will contact the copper/copper oxide reference electrode.

The top electrolyte will have a small overflow port to accommodate the expansion of tin upon heating. Care must be taken to avoid leakage of oxygen from this or other sources. This is accomplished by ensuring that the oxygen partial pressure in the inert gas surrounding the cell is nearly equal to that above the melt by pretreating the gas blanket. Temperature gradients in this so-called isothermal experiment must be eliminated in order to avoid natural convection under Earth's gravity. Under Earth's gravity, it will also be 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 should be from the bottom or that oxygen addition should be from the top. There will be the possibility
of an oxygen leakage into the tin from the overflow port. It might appear at first glance that top depletion is a desired mode of operation. However, this is potentially unstable since a top-heavy arrangement follows. In addition, establishing a high oxygen concentration at the top with the hole in the electrolyte will noticeably give rise to a radial gradient in the vicinity of the hole.

SIGNIFICANCE:
The availability of these benchmark values will be useful for many reasons, including assessing the reliability of different experimental designs and operational procedures for measuring diffusivity on Earth, defining the appropriate constitutive behavior of "Fickian diffusion" in liquid metals, and interpreting the results of flow visualization measurements which use oxygen as a tracer.

PROGRESS DURING FY 1993:
In the last 25 years, coulometric titration procedures have been used to determine values of the diffusivity of elements in liquid metals. The electrochemical cells use highly conductive solid state electrolytes that, under potentiostatic or galvanostatic operation, can establish a known boundary condition at the melt electrolyte interface and, under open circuit operation, can sense the conducting species concentration. Recent results by these investigators indicate that convection driven by thermal or solutal gradients is present in most ground-based experiments. Diffusivity measurements by necessity involve concentration gradients and, if these gradients are not collinear with the gravitational field, will certainly cause fluid convection that adversely affects the diffusivity measurements. Since mass diffusivities are small in value, concentration boundary layers tend to be formed near solute source/sink locations, and this only enhances the solute gradients. Thus even small changes from collinearity are expected to cause considerable convective flow. Indeed, even if the concentration gradients are parallel to the gravity vector, one has conditional stability. Now, diffusivity measurements in most liquid metals are conducted at high temperatures. Therefore, there is also a potential problem of introducing small thermal gradients which are not collinear to the gravity vector. Consequently, there is more than one reason that could cause convection on Earth. This makes low gravity necessary to conduct accurate measurements. Diffusivity measurements using solid state electrolytes have been performed in a variety of geometries by examining either the steady or transient response of an electrochemical cell to constant flux or constant concentration boundary conditions. Data possess considerable scatter, with significant discrepancies between various workers. Previous work by these investigators have shown the presence of solutal and thermal convection during diffusivity measurements in Sn.

STUDENTS FUNDED UNDER RESEARCH:
PhD Students: 3

BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:


II. MSAD Program Tasks — Ground-based Research

Discipline: Materials Science


Proceedings:

Crystal Growth and Segregation Using the Submerged Heater Method

Principal Investigator: Prof. A.G. Ostrogorsky

Co-Investigators:
- G. Müller
- E. Monberg

Task Objective, Description & Significance:

Objective:
The submerged heater furnace will be used to study "close-to-convection-free" solidification in ground-based studies and to test the theory of a zone melting diffusion controlled situation. Use of the submerged heater will reduce radial temperature gradients in the melt and act as a convection baffle according to preliminary experiments. Effective Rayleigh numbers will be reduced, thus reducing buoyancy-driven convection.

Description:
The research plan is to use the Submerged Heater Method in ground-based experiments for fundamental studies of mass transport in the melt at low Ra numbers. The effect of the equilibrium coefficient k on convective interference 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 = 0.5, to k = 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.

Research Approach — At Rensselaer Polytechnic Institute, the growth experiments will be performed using the programmable 18-zone Mellen furnace. The furnace will be modified to incorporate the submerged heater and the necessary additional instrumentation. Experiments at the Institut für Werkstoffwissenschaften VI of the Universität Erlangen-Nürnberg will be carried out using a high pressure Czochralski puller, which has been modified for growth with the SH method (the submerged heater is mounted on the seed lift). The presently-used, single-zone guard heater will be replaced with a multizone tubular furnace. Should a need arise, one of the two available programmable 23-zone Linn furnaces will be adopted for the SH method during the course of the work.

Significance:
The Submerged Heater Method (SHM) reduces the radial temperature gradients in the melt and at the same time acts as a convection baffle. Preliminary experiments carried out using the SHM indicate that the Rayleigh number (i.e., the ratio between buoyancy and viscous forces in the melt) is reduced by a factor of ~ 10^4, compared to the Vertical Bridgman configuration without the submerged heater. The effect of this is drastically to reduce buoyancy-driven convection in a melt.

Progress During FY 1993:
In the past six months, we made modifications to our experimental equipment and conducted preliminary numerical simulations of the heat and mass transfer occurring during growth by the Submerged Heater Method (SHM).
The 22-zone computer controlled Mellen Electro-Dynamic Gradient furnace (EDG), is presently being modified for growth by SHM in vertical Bridgman (VB) configuration. In the past six months, the following modifications were made:

- The first four zones have been removed, leaving 18 independently controlled tubular heating elements. This was done in order to secure sufficient room for the axial motion of the crucible.

- A Techno-isel 100 cm long linear slide with a stepper motor was installed to enable positioning and lowering of the crucible. The linear slide is driven by a IM 483 stepping drive which allows micro-stepping, 400 to 51,200 steps per revolution.

- A 7.5 cm diameter fused silica liner (tube) was used to provide inert atmosphere for the furnace hot zone. Two stainless steel caps were used to seal the ends of the silica tube. The cups contain the necessary feedthroughs for linear motion, the connection to the vacuum pump, inert gas supply, and thermocouples.

The present design of the submerged heater (baffle) does not allow growth from sealed ampoules because of the holder (shaft) and the thermocouples. This may be a key obstacle for the future use of the SHM in space experiments. Therefore, as a part of this research project, we will attempt to develop a submerged baffle (dummy submerged heater) that can be used in sealed silica tubes.

We are in the final stage of a low-temperature transparent furnace that we plan to use to develop the suspended baffle. The preliminary model experiments will be carried out using transparent salt melts. This will reduce the cost of the experiments and will allow us to observe the position and shape of the phase boundary, the position of the baffle, etc. We expect that these model experiments will help us to learn more about the melt convection and segregation.

Spectral element code NEKTON and finite element code FIDAP were used to model melt convection and segregation. Vertical Bridgman (VB) growth configuration was considered with and without the submerged heater.

**STUDENTS FUNDED UNDER RESEARCH:**  
**Task Initiation:** 4/93  **Expiration:** 4/96  
**Project Identification:** 674-21-08-15  
**NASA Contract No.:** NAG8-952  
**Responsible Center:** MSFC

**BIBLIOGRAPHIC CITATIONS FOR FY 1993:**

**Journals:**


Containerless Processing for Controlled Solidification Microstructures

PRINCIPAL INVESTIGATOR: Prof. John H. Perepezko
University of Wisconsin

CO-INVESTIGATORS:
No Co-I's Assigned to this Task

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

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 of selected alloys as an experience base for microgravity experiments.

DESCRIPTION:
The degree of liquid undercooling attainable in a laboratory scale (3-m) 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.

PROGRESS DURING FY 1993:
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.

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 transition (LÆa+b) in which a and b 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-equiaxial 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 (~100mg) 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 To 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 good agreement with experimental results. The model is currently undergoing further refinement to include transient nucleation effects as well as the influence of heat flow on microstructural development.

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 produced from the liquid

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provided high undercooling is achieved. With specially prepared samples it has been possible to access the thermodynamic driving forces involved in metastable t phase solidification. It also has been determined T_{oI} 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 processing 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.

The added capability of the calorimetric system will allow direct measurement of the droplet temperature as a function of falling distance. The calorimeter will provide the capacity for experimental verification of the models for the minimum undercooling required to produce the t phase. The establishment of the undercooling level needed for the formation of t allows for the possibility of controlling t phase nucleation through the use of inoculants. By matching the crystal structure and lattice constant of the inoculants particle to that of the t phase, the undercooling level needed to form t may be substantially reduced, allowing for its formation under a wider variety of conditions.

<table>
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**BIBLIOGRAPHIC CITATIONS FOR FY 1993:**

**Journals:***


II. MSAD Program Tasks — Ground-based Research

Thermosolutal Convection and Macrosegregation in Dendritic Alloys/The Role of Gravity in Macrosegregation in Alloys

PRINCIPAL INVESTIGATOR: Prof. David R. Poirier
University of Arizona

CO-INVESTIGATORS:
A.J. Pearlstein
University of Illinois

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

OBJECTIVE:
The objective was to develop a mathematical model of solidification that simulates the formation of channel segregates or freckles. The model simulates the entire solidification process, from the initial melt to the solidified cast, and the resulting segregation is predicted. Emphasis is given to the initial transient when the dendritic zone begins to develop, and the conditions for the possible nucleation of channels are established.

DESCRIPTION:
In setting up the conservation equations for the mushy zone, it was necessary to derive the momentum equation because, in analyzing dendritic solidification, the mushy zone is modeled as a porous medium with a spatially-varying fraction of liquid — unlike the uniform porosity in most porous media. There were also fundamental issues related to both the energy equation and the solute conservation equation that had to be addressed.

PROGRESS DURING FY 1993:
A finite element model is used for the simulations. It uses a single system of equations to deal with the all-liquid region, the dendritic region, and the all-solid region. The dendritic region is treated as an anistropic porous medium. The algorithm uses the bilinear isoparametric element, with a penalty function approximation and a Petrov-Galerkin formulation. The final report for this grant was submitted.

* Use of Rotation to Suppress Thermosolutal Convection in Dendritically Solidified Binary Alloys
We have shown (Oztekin and Pearlstein 1992) that uniform rotation about a vertical axis leads to significant suppression of the onset of buoyancy-driven convection during the plane-front solidification of Pb-Sn alloys cooled from below. For dendritic solidification of the same alloy, rotation suppresses the onset of convection in the mushy zone and overlying liquid. In both cases, significant suppression is obtained at modest rotation rates.

For Hg_{1-x}Cd_xTe, a pseudobinary alloy of interest due to its electro-optical properties, we have completed a stability analysis (Oztekin and Pearlstein 1993a) of plane-front solidification. Since the density of pure HgTe varies nonmonotonically with temperature, the density of Hg_{1-x}Cd_xTe also varies nonmonotonically with temperature for sufficiently small CdTe mole fractions (Oztekin and Pearlstein 1993b). Thus, adjacent to the liquid-solid interface there can exist a sublayer in which the thermal stratification is destabilizing. Hence, the onset of convection differs significantly from that in previous investigations (e.g., Pb-Sn by Coriel et al.). For Hg_{1-x}Cd_xTe, rotation also stabilizes plane-front solidification (Oztekin and Pearlstein 1993c).

The first graduate student supported under this grant was Alparslan Oztekin, who completed the requirements for the Ph.D. in Mechanical Engineering at Illinois early in March 1992. He is the first author of each of our papers to which reference is made. At the time of his graduation, Alp accepted a position at MIT as a Postdoctoral Research Associate with Bob Brown. Since then, Hanjie Lee, a Ph.D. student, has been supported by the grant. He is working on the case when the solidification proceeds in an ampoule with side walls, in which case the interface is
II. MSAD Program Tasks — Ground-based Research

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no longer flat. He is developing code for computation of the axisymmetric base state and for studying the linear
stability of the axisymmetric base state with respect to fully three-dimensional disturbances. He has now passed the
Ph.D. Qualifying Exam, and is working full-time on his dissertation.

BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:

Oztekin, A., and Pearlstein, A.J. Coriolis effects on the stability of plane-front solidification of dilute Pb-Sn binary

Oztekin, A., and Pearlstein, A.J. Stability of plane-front solidification of a binary liquid in which a density maximum

1993.

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:
J.C. Heinrich
University of Arizona

Task Objective, Description & Significance:

Objective:
Because of the inherent complexity of phenomena associated with macrosegregation, an understanding of macrosegregation must be obtained through theoretical and experimental research wherein these effects can be isolated. In terrestrial experiments, however, the convective phenomena occur simultaneously, which complicates the study of the dendritic growth.

Description:
This is a theoretical and experimental program to study thermosolutal convection during dendritic solidification of alloys. The research contains the following components:

1) Directionally solidified lead-tin alloys are being prepared and examined to study the influence of primary arm spacings, spatial distribution of volume fraction interdendritic liquid, thermal gradients, solidification rate, specimen diameter and magnetic field on the axial and radial macrosegregation;

2) Similar studies will be extended to growth conditions, where the driving force for thermosolutal convection changes during solidification;

3) The terrestrial and low-gravity experiments will be guided by computer simulations, and the experimental observations will be quantitatively compared to the simulations;

4) A goal is to identify the growth conditions for low duration low-gravity experiments for isolating the influence of thermosolutal convection from the convection caused by solidification shrinkage during dendritic solidification.

Significance:
Macrosegregation is a compositional inhomogeneity on a macroscopic length scale, and it is a serious problem in casting processes. In terrestrially processed castings, it is caused by the effects of: a) interdendritic fluid flow driven by solidification shrinkage, b) thermosolutal convection of the melt in both the mushy zone and the all-liquid zone, and c) convection of dendrite fragments and consequential equiaxed grains.

Progress During FY 1993:
Thermosolutal plume flow has also been studied in some detail in metallic, aqueous and organic systems. Analysis of directly observed flow velocities in the transparent systems indicates that these are around 0.1ms⁻¹ in metals. The development of polycrystalline structure in segregation channels has been demonstrated in an aqueous analogue. The present work is directed towards the measurement of the density of dendrite fragments which are distributed by plume/channel flow, and their survival and growth to form equiaxed crystals. It is anticipated that these observations will provide a physically realistic model for predicting the grain structure of castings.
II. MSAD Program Tasks — Ground-based Research

Student Funds Under Research:
Total Students: 1

Task Initiation: 2/93 Expiration: 1/96
Project Identification: 694-25-05-09
Responsible Center: LeRC

Bibliographic Citations for FY 1993:

Journals:


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, Description & Significance:

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.

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.

Research Approach — 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.
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 1993:
An experimental technique that uses nonisothermal DTA or DSC has been developed for determining the temperature range for nucleation and the temperature for maximum nucleation in a glass. The present technique, which requires in situ isothermal nucleation at different temperatures (for a fixed time) prior to crystallization by DTA or DSC, requires fewer samples and is much faster than the technique commonly used for this purpose. The glasses investigated to date by this technique are Li$_2$O.2SiO$_2$ (LS$_2$), Na$_2$O.2CaO.3SiO$_2$ (NC$_2$S$_3$), and BaO.2SiO$_2$ (BS$_2$), and the results obtained for all these glasses are in close agreement with those reported in the literature. This technique provides not only the data for the temperature range for nucleation and the temperature for maximum nucleation of a glass, but predicts whether there is an overlap between the nucleation and growth-rate curves for the glass (as a function of temperature).

A computer model describing nucleation and growth under isothermal and non-isothermal conditions has been developed at Washington University in St. Louis by directly simulating the evolution of nonequilibrium cluster distribution in a melt. This model justifies the experimental approach described above and, when applied to the LS$_2$ glass, yields the temperature range for nucleation and the temperature for maximum nucleation that are in excellent agreement with those determined by the present experimental technique. The model is now being tested for the NC$_2$S$_3$ and BS$_2$ glasses, for which the experimental data have already been obtained.

The effect of DTA/DSC scan rate on crystallization of the LS$_2$ glass was determined by scanning the glass through the nucleation zone (defined as a region of significant nucleation) at different rates followed by crystallization of the glass at a fixed heating rate. No change in temperature and profile of the crystallization peak was observed for this glass when the nucleation scan rate, $a_n$, was higher than 3 °C/min. For $a_n < 3$ °C/min, the crystallization temperature decreased, and the height of the crystallization peak increased with decreasing $a_n$. This indicates that no new nuclei are formed in the LS$_2$ glass when the DTA/DSC scan rate exceeds 3 °C/min. This result is also in close agreement with that determined theoretically using the computer model developed in this research.

The following experimental and theoretical investigations are now in progress:

- Check the validity of the nonisothermal thermoanalytical equations commonly used to determine the kinetic parameters for crystallization;

- Determine the limit (above which no new nuclei are formed) of DTA/DSC scan rate for the NC$_2$S$_3$ and BS$_2$ glasses; and

- Investigate the effects of particle size, nucleating agents, preexisting nuclei (resulting from different quench rates), and non-equilibrium viscosity on crystallization of the LS$_2$, BS$_2$, and NC$_2$S$_3$ glasses.
Nonisothermal crystallization measurements for the Li$_2$O.2SiO$_2$ glass as a function of various parameters such as the sample particle size, nucleation temperature and time, and nucleation and crystallization heating rate have been completed. The results were analyzed using the computer models developed at the Washington University for the non-isothermal phase transformation of the Li$_2$O.2SiO$_2$ glass.

**BIBLIOGRAPHIC CITATIONS FOR FY 1993:**

**Journals:**


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, Description & Significance:

Objective:
The objective of this research is to develop an improved understanding of solid state sintering by investigating vapor phase sintering of ceramics. The microgravity environment offers the unique opportunity to compare behavior of dispersed particles with those in contact in a powder compact. From these comparisons, the relative contributions of particle interaction by volume diffusion in the gas and coalescence controlled by grain boundary mobility can be assessed.

Description:
The proposed research combines a study of the effects of enhanced vapor phase sintering in powder compacts with those of dispersed particles. Ideal systems for space-based experiments will be determined from initial ground-based research. Thermodynamic calculations and screening experiments will be performed to find systems that have a high enough vapor pressure for: 1) short-time microgravity experiments such as in a drop tower; 2) longer experiments in existing space-flight facilities; and 3) that would pose no threat to the mission specialists. Gas solid systems will be chosen for microgravity experiments that clearly show differences between the microstructure changes or coarsening that occurs in a powder compact and that in a dispersed system.

Significance: The significance of the proposed research is that it will lead to an improved understanding of not only solid state sintering of ceramics, but coarsening or grain growth behavior in other solids as well. The research will also establish the fundamental principles necessary for the fabrication of controlled porosity ceramics for many potential applications.

Progress During FY 1993:

This research intends to use microgravity as a tool to study the effects vapor transport on the growth of solid particles suspended in a weightless environment compared to growth of severely constrained particles in dense powder compacts. Mass transport by gases during solid state sintering of ceramics and metals does not lead to densification, the usual goal of the sintering process. Virtually all technically-important ceramics are made from sintered powders. In many cases, the powder or the atmosphere can contain impurities that promote gas transport; for example:

$2 \text{Si}_3\text{N}_4(\text{s}) + 7 \text{O}_2(\text{g}) = 6 \text{SiO}_2(\text{g}) + 8 \text{NO}_2(\text{g})$.

On the other hand, sintering in an atmosphere that promotes vapor transport can lead to potentially important materials of controlled porosity and pore size. High temperature filter materials can be produced by sintering Al$_2$O$_3$ in an HCl atmosphere due to enhanced vapor transport:

$\text{Al}_2\text{O}_3(\text{s}) + 6 \text{HCl}(\text{g}) = 2 \text{AlCl}_3(\text{g}) + 3 \text{H}_2\text{O}(\text{g})$.

Whether intended or not, vapor transport during sintering is an important phenomena in the manufacture of ceramics and metals by powder processing. Models exist that predict microstructure evolution by vapor transport. But these
models do not work for every system. The reason for this is thought to be the constraint of neighboring grains. Microgravity offers a unique environment to compare the effects of gas transport on the sintering of unconstrained particles compared to those in powder compacts.

Most ceramics studied to date have involved high temperatures (1000°C–1800°C) and reactive gases (HCl, H2). To increase the likelihood of simple microgravity experiments in space, model solids are being sought that have high vapor pressures and show the same sintering effects at low temperatures as oxides in high temperature reactive gases. Compounds desired are those that are relatively nontoxic and are relatively stable in humid air. The literature was searched for such compounds. Several organic compounds including benzoic acid, salicylic acid, and cinnamic acid were identified as having potential. The ammonium halides and ice are inorganic compounds that have the requisite vapor pressures at low temperatures. Fine particle preparation of these various compounds is being pursued as well as preliminary studies of sintering and grain growth. Results suggest that several of these compounds show the same phenomena as ceramic materials at high temperatures in reactive atmospheres.

TASK INITIATION: 12/93 EXPIRATION: 12/95
PROJECT IDENTIFICATION: 674-26-05-08
RESPONSIBLE CENTER: LeRC
Electrostatic Containerless Processing

**PRINCIPAL INVESTIGATOR:** Dr. Won-Kyu Rhim
Jet Propulsion Laboratory (JPL)

**CO-INVESTIGATORS:**
A. Rulison NRC, RRA

**TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:**

**OBJECTIVE:**
The Electrostatic Containerless Processing Task objective is to utilize the advanced capabilities provided by the electrostatic levitator for the investigation of thermophysical properties, nucleation kinetics, and selecting metastable phases from undercooled metals, semiconductors, glasses, and ceramics.

**DESCRIPTION:**
Thermophysical property of undercooled melts are very important in order to understand the solidification processes and to select certain metastable states which exhibit desirable physical and chemical properties for engineering applications. However, achieving deeply undercooled melts of refractory materials in the crucibles is normally impossible due to the heterogeneous nucleation caused by the container walls. Therefore, it became important to isolate the sample materials from any container walls in the clean environment and yet to be able to process and measure thermophysical parameters in a noncontact way.

**SIGNIFICANCE:**
In the present task a High Temperature/High Vacuum Electrostatic Levitator (HTHVESL) is used to levitate various materials (such as metals, semiconductors, glasses, and ceramics), to melt, and to create undercooled states. Approximately 3 mm size samples are levitated in vacuum in a quiescent state and various processing and diagnostic methods are applied in non-contact way. The HTHVESL has the following important capabilities: (a) it provides quiescent and clean processing environment without inducing convective flows or contaminating the sample. (b) both conducting and nonconducting materials can be processed. (c) sample temperature can be independently controlled without interfering levitation force, and (d) it provides wide open sample viewing for various diagnostic instruments.

**PROGRESS DURING FY 1993:**
There has been a significant breakthrough in high-temperature electrostatic levitation technology in 1992. The basic capabilities of melting and solidification during levitation have been verified using various high-density materials, such as: In (T_m=157 °C), Sn (232 °C), Bi (271.44 °C), Pb (327 °C), In (0.69%) Sb (492.5 °C), Al (670 °C), Ge (938 °C), Cu (1083 °C), Ni (1,455 °C), and Zr (1,855 °C).

This preliminary work was followed by an extensive undercooling-nucleation study jointly with Vanderbilt University. Using two zirconium samples of different origin and purity, over 300 runs of superheating-undercooling-recalescence processes have been completed. A patent application is in progress for the high-temperature electrostatic levitator of Won-Kyu Rhim and Sang K. Chung.

Six silicate based glass-ceramics samples have been processed while accurate processing temperature was recorded. These samples were sent to Professor Reid Cooper at the University of Wisconsin for analysis. This is the second successful demonstration of processing non-conducting materials using the High Temp./High Vacuum Electrostatic Levitator.
Eutectic alloys of Ni 47 wt % Zr, and Ni 51.9 wt % Nb have been processed primarily to obtain the cooling curves covering superheated as well as undercooled regions of the melts. These alloys have been investigated in collaboration with Professor W. Johnson and Dr. David Lee of Caltech.

A paper on "A Non-Contact Measurement Technique for the Specific Heat and Total Hemispherical Emissivity of Undercooled Refractory Materials" has been completed and submitted to the Rev. of Sci. Instru. for publication.

A paper entitled "A Critical Assessment of Electromagnetic and Electrostatic Levitation for Containerless Processing in Microgravity and under Earthbound Conditions" which will be coauthored with J. Szekely, J. Watkins, E Egry, and B. Feuerbacher is in progress.

Methods for measuring the specific density, surface tension, and viscosity of undercooled melts have been established and the capability demonstrations of these methods have been initiated.

A three color pyrometer which will operate in a wide temperature range (300 - 3000°C) is being constructed assisted by Professor R. E. Spjut of Harvey Mudd College.

**BIBLIOGRAPHIC CITATIONS FOR FY 1993:**

**Journals:**


**Proceedings:**

**PATENTS:**
Rhim, Won-Kyu. "High Temperature Electrostatic Levitator."
II. MSAD Program Tasks — Ground-based Research

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, DESCRIPTION & SIGNIFICANCE:

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.

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.

Research Approach — 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.
- Clarify advantages and limitations of containerless processing for the production of A-15 phases in the bulk in selected Nb-based system where A-15 phase has differing degrees of stability. To attempt to obtain higher supercooling transition temperatures in the bulk than have been previously possible.

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.
II. MSAD Program Tasks — Ground-based Research

Discipline: Materials Science

Progress During FY 1993:
A broad base of experience has been obtained in the containerless, microgravity processing of niobium alloys. In addition, work with pure refractory metals has defined undercooling limits in the 105-meter drop tube and has contributed to the understanding of nucleation in a containerless, microgravity environment. Approximately 4,500 samples have been processed in the 105-meter drop tube to date.

Task Initiation: 1/78 Expiration: 1/95
Project Identification: 674-28-08-02
NASA Contract No.: In-house
Responsible Center: MSFC
Growth Kinetics of Physical Vapor Transport Properties: Crystal Growth of Mercurous Chloride.

**PRINCIPAL INVESTIGATOR:** Dr. N.B. Singh
**Westinghouse Electric Corporation**

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

**TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:**

**OBJECTIVE:**
The objective of this task is to study the relationship between growth parameters during the growth of mercurous chloride by the physical vapor transport (PVT) technique, and to identify the affects of convection on the crystal growth process.

**DESCRIPTION:**
The approach has been to purify and grow mercurous crystals under various conditions.

**SIGNIFICANCE:**
This research deduces relationships between growth parameters and crystal quality.

**PROGRESS DURING FY 1993:**
A method for the purification of Hg$_2$Cl$_2$ was developed. Materials with less than 10ppm metallic impurities could be produced by this method. Growth velocity was observed to be dependent on crystallographic orientation. The condensing flux at the solid-liquid interface was orders of magnitude lower than the flux calculated by the Hertz-Knudsen transport equation. A condensation coefficient of approximately 0.01 is required to explain the growth velocity data of mercurous chloride. Theories based on the Hagen-Poiseuille equation could not explain the fluid dynamic phenomena. Growth rate of mercurous chloride was observed to be dependent on the orientation of the ampoule with respect to g-vector. Crystals grown at low thermal Rayleigh number (decreased convective effects) showed better homogeneity.

The period of performance for this task ended September 30, 1992.
II. MSAD Program Tasks — Ground-based Research

_Placeholder_ Discipline: Materials Science

Crystal Nucleation, Hydrostatic Tension, & Diffusion in Metal and Semiconductor Melts

PRINCIPAL INVESTIGATOR: Prof. Frans Spaepen

Harvard University

CO-INVESTIGATORS:

M.J. Aziz

Harvard University

D. Turnbull

Harvard University

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

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.

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.

PROGRESS DURING FY 1993:

Work on the undercooling of liquid silicon is continuing. A new chamber, with better vacuum, gas control, heating elements and observation port was constructed. The undercooling experiments are carried out by coating a small amount of silicon with a flux, placing it on a refractory substrate, melting it, and measuring the undercooling, right before recalescence, with a thermocouple. Work has been concentrated on finding an appropriate chloride or fluoride flux for removal of heterogeneous nucleants. So far, decomposition of the fluxes has been the main obstacle. Currently high silica chlorosilicate and fluorosilicate liquids and glasses are being investigated. Experiments on new substrates and with heating in high vacuum are also underway.

We entirely rebuilt our 3m Pyrex drop tube. It now has a new spray assembly for producing the droplets (<0.5 mm), easier access to the top for loading, better gas handling facilities, and catch trays at different levels. Experiments have been carried out on zinc to check the temperature evolution during the drop obtained from heat flow calculations. Experiments have been started on the solidification of Ga-Mg-Zn droplets. This system is a known quasicrystal former which was studied extensively in our laboratory and has a conveniently low melting temperature. Study of the microstructure and identification of the large number of phases in the solidification products is underway.

A theory for the temperature dependence of the solid-liquid interface and its link to the structure of the interface has been developed. It was used to reanalyze Turnbull's data on the homogeneous nucleation of Hg crystals from the melt. This analysis showed that the origin of the interfacial tension is a substantial drop in the entropy of liquid due to localization near the crystal; the enthalpy change is small.

A general approach to barrier crossing in multicomponent nucleation has also been developed. The analysis is reformulated to include all paths of nucleation. Starting with simple measurement theory, he defines a global nucleation rate that corresponds to what is observable experimentally. This rate can be calculated at steady state and estimated for arbitrary times, provided the rate matrix and the free energy obey certain constraints. When applied to

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II. MSAD Program Tasks — Ground-based Research  

Discipline: Materials Science

the special case of a quadratic barrier, the global nucleation rate is smaller than the standard result based on the rate of saddle-point nucleation. Cases with more general barriers can also be treated by the same method.

STUDENTS FUNDED UNDER RESEARCH:  

PhD Students: 1

PROJECT IDENTIFICATION: 694-25-07-07

RESPONSIBLE CENTER: JPL

BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:

II. MSAD Program Tasks — Ground-based Research

Levitation Undercooling and Nucleation Studies

PRINCIPAL INVESTIGATOR: Dr. Eugene H. Trinh
Jet Propulsion Laboratory (JPL)

CO-INVESTIGATORS:
Dr. Kenichi Ohsaka
Jet Propulsion Laboratory (JPL)

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

OBJECTIVE:
The principal objectives of this task are to utilize containerless manipulation technology to perform (a) undercooling and heterogeneous nucleation experiments on pure metals and alloys as well as organic and inorganic model materials, (b) measurement of the physical properties of undercooled melts, and (c) investigation of undercooled and solidification of phase-separated model materials and eutectic alloys.

DESCRIPTION:
The research approach is to use ground-based ultrasonic and electromagnetic levitation techniques, together with novel noncontact property measurement methods, to undercool millimeter-size samples in a containerless manner; to characterize the levitation environment to determine possible "dynamic" nucleating effects; and to develop limited theories to investigate specific alloy systems.

PROGRESS DURING FY 1993:
FY1992 research tasks have included (a) the measurement of the specific heat of Ti-Cr alloys in stable and undercooled liquid ranges, (b) the initiation of the thermal diffusivity measurement of levitated melts, and (c) the initiation of a total hemispherical emissivity measurement task using electromagnetically levitated samples.

FY1993 tasks include (a) the continuation of the total hemispherical emissivity measurement, (b) the studies of undercooling using electromagnetically levitated melts of Al-Cu alloys in comparison with electrostatically levitated samples to uncover possible "dynamic" effects on nucleation, and (c) the experimental study of ultrasonically levitated phase-separated model materials (aqueous solutions of succinonitrile).

BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:
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, DESCRIPTION & SIGNIFICANCE:**

**OBJECTIVE:**
Concurrent with our work on designing a spaceflight experiment, ground-based experimental and theoretical studies on Ostwald ripening will be undertaken in an effort to provide comparison experiments to those performed in microgravity and provide a theoretical understanding of the Ostwald ripening process. We are thus investigating the nature of transient Ostwald ripening in solid-liquid systems and developing a first-principles theory for ripening in systems with volume fractions of coarsening phase above 0.1. Such volume fractions are closer to those employed in the ground-based experiments and will permit theory to be compared with the results of the spaceflight experiments over a wider range of volume fractions than it is currently possible. Finally, the transient Ostwald ripening experiments will allow us to determine the time required for the spaceflight experiment.

**DESCRIPTION:**
Previous NASA-sponsored work clearly showed that solid-liquid mixture 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 thermo-physical 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. The objective of this project it is to plan and perform a microgravity experiment on Ostwald ripening in solid-liquid mixtures. 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.

**SIGNIFICANCE:**
The late-stages of a first-order phase transformation process are usually characterized by the growth of second-phase domains with low interfacial curvature, at the expense of domains with high interfacial curvature. This process, also known as Ostwald ripening or coarsening, occurs in a wide variety of two-phase mixtures ranging from a multiphase solids to multiphase liquids, and has a significant impact on the high temperature stability of many technologically important materials. Unfortunately, an understanding of the dynamics of ripening processes is not in hand. Many of the recent theories for the effects of a finite volume fraction of coarsening phase on the kinetics of Ostwald ripening have proposed divergent expressions for the dependence of the coarsening rate of the system on the volume fraction of coarsening phase. As there are virtually no experimental data of sufficient quality to differentiate between these theories, the controversy over the dependence of the coarsening rate of the system on the volume fraction remains unresolved.
PROGRESS DURING FY 1993:

The dynamics of particle coarsening in high volume fraction two-phase mixtures was investigated using numerical simulations. The multi-particle diffusion problem was solved using a multipole expansion method which is valid up to any order multi-pole. The simulations were performed using this multipole expansion truncated at the monopole and dipole level. The dipole terms allow the particles to migrate due to the non-symmetric concentration gradient, but they still remain spherical. The initial particle spatial distributions are chosen as random, both with or without a depletion zone surrounding each particle. Particle spatial correlations were studied in terms of pair correlation functions and the structure function. In the late-stage the particle spatial correlation functions found using different initial conditions agree well, suggesting the existence of a unique spatial correlation function for a coarsening system at a given volume fraction. We also find that the particle correlations in the late-stage can not be explained by a simple depletion zone model. The form of the scaled time independent structure function at small and large wave numbers was also investigated. The slope of the structure function in a log-log plot seems to approach 4 at small wave numbers and is -4 at large wave numbers. There exists oscillations in the structure function at large wave numbers which are related to the spherical shape of the particles and the particle size distribution. The amplitude of the temporal power law for the average particle radius was found to increase with volume fraction faster than the predictions of other statistical mechanical theories.

Experiments are being performed to examine the kinetics of transient Ostwald ripening in solid-liquid systems. We are currently using mixtures consisting of solid silver particles in a silver-copper liquid. The initial particle size distributions are being set by the particle size distribution of the single crystal silver powders. We intend to compare the results of these experiments to our theoretical calculations of transient Ostwald ripening.

BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:

Containerless Liquid Phase Processing of Ceramic Materials

Principal Investigator: Dr. Richard J.K. Weber
Containerless Research, Inc.

Co-Investigators:
P.C. Nordine
Intersonics, Inc.

Task Objective, Description & Significance:
Objective:
This research uses the control of chemistry and nucleation achieved by containerless liquid-phase processing to study nonequilibrium 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 containerless 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.

Description:
High-temperature liquid-phase processing will be achieved by aeroacoustic levitation in combination with laser-beam heating. The two groups of materials chosen for this work are: (a) rare-earth borides and (b) refractory oxides, for example, alumina. The major focus of the work will be on borides.

Metastable phase formation and epitaxial growth from molten rare-earth borides will be investigated. This unique group of materials is particularly hard and refractory; the atomic size differences between boron and rare-earth elements combined with the directional bonding between them result in compounds which exhibit a wide variety of crystal structures. Marginally unstable compounds are predicted to exist which will be accessed by nucleation of undercooled melts onto isostuctural seed crystals. Amorphous phase formation will also be investigated by deep undercooling and possibly quenching of the higher melting borides.

Nonequilibrium phenomena displayed by undercooled oxide melts will also be investigated. It has already been found that the solidification behavior of undercooled liquid aluminum oxide differs greatly in oxygen and argon atmospheres. Environmental effects on molten oxides will be investigated further by conducting containerless experiments in atmospheres of different oxygen potentials.

In addition to the materials research on borides and oxides, the investigators will undertake processing of materials provided by NASA-supported scientists at several other institutions during the first year of this project. These scientists are also interested in the processing and properties of oxide melts.

Progress During FY 1993:
The 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. Containerless high temperature liquid-phase processing is achieved by cw CO₂ laser beam heating of 0.2-0.4 cm diameter specimens using the aero-acoustic levitation technique.
Work to characterize the behavior of liquid aluminum oxide and materials formed by liquid phase processing under conditions of controlled oxygen pressure was continued. Processed material has been provided to scientists at several other institutions and they are working to characterize it. Effect of trace impurities are being investigated using Raman spectroscopy and laser induced fluorescence by Dr. Abi at JPL, and NMR spectroscopy by Dr. Brent Poe at the CNRS Facility, Orleans, France. The results of these investigations show that the material is extremely high purity and work is in progress to establish a reference purity material which can be used for doping aluminum oxide materials. A paper describing the undercooling behavior of liquid aluminum oxide has been accepted for publication in the Journal of the American Ceramic Society.

Work to achieve melting of boride materials and eliminate the formation of borate slag was continued. A "conical nozzle" gas jet levitator has been constructed and it is showing promising early results for levitation melting of these air sensitive materials. Work will be continued to achieve melting of the less refractory borides.

The effects of ambient oxygen pressure on the melting behavior of Y.Ba.Cu.O ceramic superconductor materials was investigated in collaboration with scientists from Argonne National Laboratory. The work followed up on preliminary experiments conducted in the spring this year. The solubility of yttrium oxide was shown to depend on the ambient oxygen pressure such complete melting and dissolution of the Y2O3 formed by peritectic decomposition of YBa2Cu3O7 occurred easily in the material processed under argon (pO2 ca. 1x10^-4 atm.), enabling deep undercooling to be achieved. Processing in air or oxygen resulted in yttrium oxide precipitation and undercooling was not observed unless specimens were heated and held for several minutes. A paper describing this work has been submitted to the Journal of Materials Research.

Collaborative experiments to process materials provided by other NASA investigators were continued. Professors Day and Ray from University of Missouri-Rolla provided dense specimens of heavy metal oxide glass for levitation melting in the aero-acoustic levitator. These materials have proved to be difficult to melt without loss of liquid and complete melting has not yet been achieved. Mr. James Olive and Timothy Dassler graduate students from Vanderbilt University conducted processing experiments on ceramic superconductors, earlier work which was conducted this year has been accepted for publication in the Journal of Materials Research. The Vanderbilt team used an array detection pyrometer to investigate the melting, undercooling and solidification behavior of ceramic superconductor materials. Initial experiments were partially successful but the levitation stability was poor due to electronics problem with the levitator. Additional work is being planned for December.

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II. MSAD Program Tasks — Ground-based Research

Discipline: Materials Science

Glass Forming Ability and Crystallization of Glass

PRINCIPAL INVESTIGATOR: Dr. Michael C. Weinberg
University of Arizona

CO-INVESTIGATORS:
No Co-I's Assigned to this Task

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

OBJECTIVE:
The objectives are: (a) to identify compositions of glasses which could benefit from processing in microgravity and (b) to develop a theoretical framework to applicable to crystallization kinetics in 1-g and μg.

DESCRIPTION:
Both theoretical and experimental work are carried out in parallel; the latter includes glass preparations, in situ crystal growth measurements, and various surface and bulk diagnostics.

PROGRESS DURING FY 1993:
The relative importance of bulk and surface nucleation was studied as a function of cooling rate in the free-surface conditions relevant to containerless processing for both isothermal and non isothermal environments.

TASK INITIATION: 10/83   EXPIRATION: 12/92
PROJECT IDENTIFICATION: 674-26-04-02
RESPONSIBLE CENTER: JPL

II-471
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:**
- M. Wargo  
Massachusetts Institute of Technology (MIT)
- S. Motakef  
Massachusetts Institute of Technology (MIT)
- D. Sadoway  
Massachusetts Institute of Technology (MIT)

**TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:**

**OBJECTIVE:**
The objective of this program involving the system BSO/BTO 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 control adequately their stoichiometry, crystal defect formation and confinement-related contamination. The ultimate success of the proposed research, considered an essential element for the evaluation of the potential selenites in device application, appears fundamentally dependent on the conduct of controlled crystal growth experiments in a reduced gravity environment. Convective interference, otherwise unavoidable, is projected to be substantially suppressed under microgravity conditions, and the defect structures resulting during growth are expected to approach equilibrium values.

**DESCRIPTION:**

Research Approach — The development phase of the research is concerned with:

- Numerical models of heat and mass transfer in support of hot-zone design and search of parameter space for growth optimization for BSO/BTO;

- Design and construction of a hot-zone; identification of processing conditions optimized for growth in reduced gravity environment;

- Development of (quantitative) analytical techniques and procedures capable of resolving gravity-related effects in BSO/BTO grown on Earth and in space;

- Determination of critical thermo-physical properties of BSO and BTO melts, in support of numerical modeling [with the assistance of the Crystal Growth Laboratory (Dr. C.D. Brandle) of AT&T Bell Laboratories in Murray Hill, N.J.];

- Design of a series of experiments directed at optimizing the scientific data output achievable from growth of doped and undoped BSO/BTO in a reduced-gravity environment;

- Conduct, on Earth, of growth experiments, identical with those to be executed in reduced-gravity environment.

The post-flight research phase at MIT is to focus on:

- Comprehensive crystal analysis directed at the identification of microgravity-related changes in growth behavior and its effects on segregation and defect formation;
II. MSAD Program Tasks — Ground-based Research

Discipline: Materials Science

- Analysis of BSO/BTO grown in a microgravity environment for application specific crystal properties as well as for performance in basic device configuration [in collaboration with Hughes Research Laboratory, Malibu, CA (Dr. M. Klein)].

SIGNIFICANCE:
Knowledge gained from the space experiment 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 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.

PROGRESS DURING FY 1993:
In designed space growth experiments involving doped and undoped BSO and BTO, it is considered possible to establish controlled growth perturbations and, through their analysis, to gain insight into cause and effect relationships for defect generation during growth on Earth. Space experiments are similarly to be used to obtain for these opto-electronic materials small quantities as reference standards for a data base of physical properties.

Discussions of the design, conduct and analysis of the proposed space growth experiments with members of the opto-electronic industrial research community have been productive and indicated broad-based interest as well as the desire to contribute actively to the success of the proposed research undertaking.

TASK INITIATION: 1/93  EXPIRATION: 1/96
PROJECT IDENTIFICATION: 674-21-08-19
RESPONSIBLE CENTER: MSFC
II. MSAD Program Tasks — Advanced Technology Development

Noncontact Temperature Measurement

PRINCIPAL INVESTIGATOR: Ali Abtahi
Jet Propulsion Laboratory (JPL)

CO-INVESTIGATORS:
No Co-I's Assigned to this Task

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

OBJECTIVE:
The task objective is to develop new techniques for making accurate non-contact temperature measurements. The techniques developed in this task will use the thermal radiation emanating from samples to determine the radiance temperature. Several different techniques are used to correct the radiance temperature measurement to arrive at the true sample temperature. These techniques include polarimetric determination of the sample emissivity, emissivity correction by measurement at two colors, and a polaradiometric technique that eliminates the need for emissivity measurements and measures true temperature directly.

Non-contact temperature measurements are required for materials processing applications where the temperature must be determined without interfering with the sample surface, eliminating nucleation sites for undercooled substances and inaccuracies due to the presence of thermally conductive probes.

Three devices were procured or constructed during 1993. The first was the Division of Amplitude Polarimetric Pyrometer, which was delivered to JPL and tested. Several modification became necessary to install the system on an electromagnetic levitator. These modification were performed and the device is fully prepared to operate on the electromagnetic levitator. This enables measurements of temperature and emissivity on significantly undercooled liquid metal drops.

The two color pyrometer was delivered to JPL. It proved to be non-functioning and had to be extensively modified. All components had to be replaced and the device was tested showing that the modifications were successful in eliminating the original problems.

A new concept was developed based on polaradiometric true temperature measurements. The hardware for this device was developed and a prototype was constructed which is currently undergoing final evaluation.

PROGRESS DURING FY 1993:
Progress to Date: DAPP — The DAPP receiver has been installed on the levitator chamber. Due to the limited space available around the chamber the head of the DAPP had to be installed on a separate platform from the laser. This creates a problem with the alignment of the system. To alleviate this problem the mount of the laser and polarizing optics had to be specially modified and this is currently in progress. Originally a different modification had been planned that had to be abandoned because of the space available around the levitation chamber.

The problems with the electromagnetic levitation system have been largely resolved. Especially solid samples can be levitated successfully and are relatively stable. Liquid samples require the presence of a gas in the chamber for stability, so that runs involving the liquid metal samples will be done with argon in the chamber. As soon as the laser and polarizer are mounted properly the initial (angular) alignment will be performed and test on levitated samples can commence.
II. MSAD Program Tasks — Advanced Technology Development

ô 2-Color Laser Pyrometer
A completely redesigned system has been built and currently is undergoing testing and calibration. This system is based on the same principle as the original unit from Quantum Logic however none of the original subsystems proved suitable and thus every part was redesigned, procured, and assembled again.

The main problem of the original system (optical anisotropy of the transmitter and receiver) have been eliminated completely. A few minor modifications will be made to make the system easier to use and align. These modifications are: the construction of a light-tight box around the receiving and transmitting systems to eliminate the need for a darkened room as well as use of a bigger aperture lens and smaller pinhole to make the sample alignment less critical.

The development of this system is behind schedule due to the need for complete redesign of every single component. A great deal of time was lost in trying to make the original components work properly. As soon as this system is properly calibrated it will be installed in the same levitator chamber as the DAPP to enable a back to back comparison of the two systems.

ô Polaradiometer
The polaradiometer design developed earlier could suffer from an inability to perform properly on diffuse samples. This inability was a result of the fact that reflection of light from a point source at a diffuse surface is the result of specular reflection from a large number of randomly oriented facets, while the emission process from these facets takes place with a hemispherical intensity distribution. The difference in character of the emission and reflection processes causes the polarimetric characteristics of the light received at the receiver to be biased. To eliminate this problem with the polaradiometer a separate measurement of the polarimetric characteristics of the emitted radiation is needed. This measurement can be combined with the standard nulling technique used in the polaradiometer to arrive at the true temperature of the surface.

The design of the polaradiometer is completed however the selection process of the optical components and the procurement of said components is still in progress.

Since a majority of the remaining time this year can be devoted to this task it is likely to be completed successfully.

Task Initiation: 10/86 Expiration: 9/93
Project Identification: ATD
Responsible Center: JPL
II. MSAD Program Tasks — Advanced Technology Development

Microwave Furnace Development

**PRINCIPAL INVESTIGATOR:** Dr. Martin B. Barmatz  Jet Propulsion Laboratory (JPL)

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

**TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:**

**OBJECTIVE:**
The objective of this Advanced Technology Development (ATD) proposal is to build a highly efficient, cold-wall, direct-heating microwave furnace that will permit fast heating of microwave-absorbing materials and quick cooling of these materials when the microwaves are removed. The task objectives will include 1) development of advanced energy saving methods for (a) continuous tracking of the cavity resonant mode during processing, (b) continuous matching of source and cavity impedance during processing, 2) design, fabrication and evaluation of various cavity geometries and excitation techniques, and 3) modeling of the microwave heating process for various sample shapes and sizes.

**DESCRIPTION:**
In the future, many materials processing studies in space will require specialized furnaces, particularly at high temperatures. Requirements for these furnaces include fast, controlled sample heating, high precision sample temperature control, and fast, controlled sample cooling. Furthermore, there are constraints that a microgravity furnace should meet; i.e., they should be small, light weight, and very energy efficient. Properly designed microwave furnaces can deliver most of the generated energy directly to the material being processed, thus leading to improved energy efficiency. The design requirements for energy efficiency include critical coupling of microwave energy from source to cavity, high electrically conducting cavity walls compared to sample conductivity, and continuous tracking of the microwave resonance during processing. Furthermore, microwaves are able to directly heat the interior of the materials, which differs from conventional heating methods, where only the sample surface is directly heated. In some cases, this microwave volumetric heating ability can lead to more uniform processing, while in other cases it may lead to an internal temperature profile with the highest temperature in the center of the material. These and other microwave heating characteristics will permit new and novel scientific studies to be performed. The furnace designs developed during this ATD task will allow unique microwave heating properties as well as precision sample temperature control capabilities to be applied to microgravity processing. By the proper choice of sample shapes, such as spherical or cylindrical, one can also theoretically predict the unique heating behavior of the sample interior during processing.

**PROGRESS DURING FY 1993:**

Progress to Date:
During this last year, we have (a) completed development of a fast frequency tracker for a traveling wave tube (TWT) system, (b) designed and fabricated a prototype electronically controlled impedance matching system in collaboration with the Oregon Institute of Technology, and (c) developed a microwave absorption model for a cylindrical sample in a cylindrical cavity.

**STUDENTS FUNDED UNDER RESEARCH:**

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**TASK INITIATION:** 10/91  **EXPIRATION:** 9/94

**PROJECT IDENTIFICATION:**  ATD

**RESPONSIBLE CENTER:**  JPL
II. MSAD Program Tasks — Advanced Technology Development

BIBLIOGRAPHIC CITATIONS FOR FY 1993:

**Journals:**

II. MSAD Program Tasks — Advanced Technology Development

Advanced High Brilliance X-Ray Source

**PRINCIPAL INVESTIGATOR:** Dr. Daniel C. Carter
**NASA Marshall Space Flight Center (MSFC)**

**CO-INVESTIGATORS:**
- W. Gibson
  - State University of New York, Albany
- M. Kumakhov
  - I.V. Kurchatov Institute of Atomic Energy
- X. He
  - Universities Space Research Association (USRA)

**TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:**

**OBJECTIVES:**
The primary objective of this research is to produce the first x-ray generator and Kumakhov lens system optimized in design for 8KeV x-rays.

**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.

**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 1993:**
A small number of third-generation optical systems have been fabricated using three different fabrication techniques. Modeling computations have been carried out to determine the optimum design for a lens collecting 0.2 radians (12 degrees) of 8.0 KeV x-rays from a 4 micron divergent source at a source distance of 5mm.

**TASK INITIATION:** 4/93  **EXPIRATION:** 3/96
**PROJECT IDENTIFICATION:** ATD
**NASA CONTRACT NO.:** NAS8-399
**RESPONSIBLE CENTER:** MSFC
Ultrasonic Monitoring of Interfaces in Directional Solidification

PRINCIPAL INVESTIGATOR: Dr. Archibald L. Fripp

CO-INVESTIGATORS:
J.N. Carter
A. Lam

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

OBJECTIVE:
The objective of this research was to develop an ultrasonic interfacial monitoring technique which would not only monitor the interface position in real-time but would also measure the interface shape while the crystal is growing.

DESCRIPTION:
Ultrasons have been used to examine materials during and after growth to identify and locate flaws. Flaw detection, or non-destructive testing (NDE) has been used extensively to examine castings and welds for voids and cracks. This method relies on a difference in the acoustical impedances between the two phases that gives rise to a partial reflection of the incident energy. Previous measurements during growth have only been when the acoustical pulse, from a PZT transducer, was launched directly into the solid phase of the material being melted. The solid was kept in intimate contact with the transducer whereas at some point, largely separated from the transducer, a section of the material was melted. This system, while demonstrating the efficacy of ultrasound interface monitoring, is not easily adaptable to an actual Bridgman growth system because of the high temperatures involved would lead to the destruction of the transducer. This effort was to employ an ultrasound method, using waveguides, to locate and track the interface during the true Bridgman growth of electronic materials.

PROGRESS DURING FY 1993:
Progress to date has been primarily in waveguide development. The use of a solid silica waveguide, in pulse/echo mode, resulted in multimode reflections which made the return signal difficult to detect. The current approach is the use of bundled small silica waveguides to synthesize an aperture similar to that of the solid rod. Since each rod is of small cross-section, the bundle as a whole does not support the manifold of modes accessible to the solid one piece waveguide. This effect enables the detection of a return echo signal with relative ease. This technique has been successfully tested on model systems which include the solidification water and of mercury.

STUDENTS FUNDED UNDER RESEARCH:

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TASK INITIATION: 10/91  EXPIRATION: 9/93
PROJECT IDENTIFICATION: ATD
RESPONSIBLE CENTER: LaRC

BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:
Microgravity Combustion Diagnostics

**Principal Investigator:** Paul S. Greenberg  
**Co-Investigators:**  
- Dr. D.W. Griffin  
- Dr. R.L. Vander Wal  
- Dr. K.J. Weiland  
**NASA Lewis Research Center (LeRC)**  
**Sverdrup Technology, Inc.**

**Task Objective, Description & Significance:**

**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.

**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.

**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 1993:**
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.
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.

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.

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. These modules have been evaluated using rotating targets as a velocity standard. A particle seeder has been designed and fabricated to enable measurements to be performed in actual flows. Concurrently, a digital phase-locked-loop processor has been designed and constructed, and has been demonstrated to perform well on strictly periodic signals. This system is now to be tested on aperiodic signals resulting from actual flow-field conditions. For particle image velocity measurements, a special compact, folded resonator Nd:YAG laser capable of delivering a rapid pulse sequence has been built under contract. This system has been demonstrated to achieve the required pulse energies and temporal resolution, and is presently being integrated into a seeded flow system.

BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:


Proceedings:
II. MSAD Program Tasks — Advanced Technology Development

Presentations:
Vander Wal, R.L. High resolution degenerate four-wave mixing in the $\gamma(0,0)$ band of NO, 24th International Symposium on Combustion, 1993.


Small, Stable, Rugged Microgravity

**Principal Investigator:** Frank T. Hartley  
Jet Propulsion Laboratory (JPL)

**Co-Investigators:**
- P. Zavracky  
Northeastern University
- P. Dolgin  
Jet Propulsion Laboratory (JPL)

**Task Objective, Description & Significance:**

**Objective:**
The objective of this ATD task is to build a novel micromachined accelerometer that is capable of measuring accelerations from $10^{-2}$ g to $10^{-5}$ g with a better than $10^{-5}$ g accuracy ($<10^{-9} g/\sqrt{Hz}$) for the frequencies from $10^{-4}$ to 20 Hz. The device should have low temperature sensitivity and have a build-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.

**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 $10^{-4} Hz \approx 150$ minutes, but the accuracy will 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.

**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 1993:**
Completed or Underway:
The micromachined accelerometer is assembled from four parts, and all four parts have been manufactured and tested in their preassembled states. The breadboard for the data acquisition and control electronics has been built.

**Students Funded Under Research:**
Total Students: 1

**Task Initiation:** 10/92  
Expiration: 09/95

**Project Identification:** ATD

**Responsible Center:** JPL
II. MSAD Program Tasks — Advanced Technology Development

BIBLIOGRAPHIC CITATIONS FOR FY 1993:

Journals:

Patents:
Patent Status: Pending U.S. Patent #: Undetermined
II. MSAD Program Tasks — Advanced Technology Development

Stereo Imaging Velocimetry

PRINCIPAL INVESTIGATOR: Mary Jo B. Meyer
NASA Lewis Research Center (LeRC)

CO-INVESTIGATORS:
M. Bethea
NASA Lewis Research Center (LeRC)

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

OBJECTIVE:
The objectives of this project are to develop a stereo imaging velocimeter that will: (a) Measure fluid velocities between 1.0 mm/sec and 10.0 cm/sec with an accuracy of +1.0% of full field for 150-micron seed particles in a 3.0-cm field of view, (b) Have streamlined data processing which processes 100 time steps of consecutive stereo images to obtain 3-D velocity fields in ten minutes or less, (c) Be able to track at least 100 particle pairs per frame, (d) Require minimal apriori assumptions about the flow, and (e) Initiate tracking and matching without human intervention.

DESCRIPTION:
The approach to successful implementation is to choose the most promising of current techniques as our starting point, and develop or modify algorithms to accomplish particle centroid finding, tracking, and matching, as well as camera calibration. These four capabilities 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 moving in known trajectories. User interface for both the front-end and postprocessing will be created. The velocimeter will be tested on real fluid experiment(s).

SIGNIFICANCE:
Stereo imaging velocimetry will permit the collection of quantitative, three-dimensional flow data from any optically transparent fluid which can be seeded with tracer particles. This includes such diverse experiments as the sturdy of multiphase flow, bubble nucleation and migration, pool combustion, and crystal growth — all of which are part of NASA's microgravity science program.

PROGRESS DURING FY 1993:
A neural net implementation for deconvoluting images of overlapping particles was invented, coded and tested. Results showed a substantial improvement in yield, where yield is defined as the number of correct velocity vectors found per the number of seed particles available for interrogation. This work is significant because the extent to which we are able to identify and decompose overlapping particles determines our particle seeding density (particles per unit volume of fluid), which directly affects the spatial resolution of the SIV system.

An empirical-fit camera calibration routine has been devised, coded and tested for two-dimensional systems. With it we are able to determine 2D positions with an accuracy well under 1% of full field. We plan to extend the routine to 3D systems.

We modified the code for the SIV centroid-finding routine and, as a result, the time required to reduce image data to centroid has been decreased by more than an order of magnitude.
II. MSAD Program Tasks — Advanced Technology Development

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PROJECT IDENTIFICATION: ATD
RESPONSIBLE CENTER: LeRC

BIBLIOGRAPHIC CITATIONS FOR FY 1993:

**Journals:**


II. MSAD Program Tasks — Advanced Technology Development

Laser Light Scattering

Principal Investigator: William V. Meyer
Case Western Reserve University

Co-Investigators:
Dr. R. Ansari
Case Western Reserve University

Task Objective, Description & Significance:

Objective:
One objective of the Advanced Technology Development (ATD) program has been to provide sturdy, miniaturized laser light scattering (LLS) instrumentation for use in microgravity experiments.

Description:
User requirements have been assessed, the capabilities of existing and prospective laser light scattering hardware have been explored, and both have been coordinated to participate in the hardware and software advances needed for a flight hardware instrument.

Progress During FY 1993:

We have successfully breadboarded and evaluated an engineering version of a single-angle glovebox instrument which uses solid state detectors and lasers, along with fiber optics and for beam delivery and detection. Additionally, we have provided the specifications and written verification procedures necessary for procuring a miniature multi-angle LLS instrument which will be used by the flight hardware project which resulted from this work and from this project's interaction with the laser light scattering community.

Students Funded Under Research:

Total Students: 2  Total Degrees: 2

Task Initiation: 10/88  Expiration: 6/93

Project Identification: ATD

Responsible Center: LeRC

Bibliographic Citations for FY 1993:

Journals:

Proceedings:

II. MSAD Program Tasks — Advanced Technology Development

PATENTS:

Patent Status: Approved  U.S. Patent #: Pending
Surface Light Scattering Instruments

**Principal Investigator:** William V. Meyer

**Co-Investigators:**
- Prof. J.A. Mann
- Dr. P. Tin
- R.B. Rogers

**Task Objective, Description & Significance:**

**Objective:**
The objective is the development of an instrument capable of detecting fluid surface phenomena (liquid/liquid and liquid/vapor), such as local temperatures and interface temperature gradients, as well as surface tensions and volume viscosity. This development is maturing with design and fabrication of many portions of the new instruments well underway.

**Description:**
We started with a traditional room-sized surface light scattering instrument. Our instrument has evolved to a state where the vibration sensitive bulk optics’ high power, large space requirements in the traditional surface light scattering instrument have been replaced by a low power, compact laser-diode surface light scattering system. This includes a new “cat's eye” optics design that is immune to the sloshing of the surface being examined. This had been a major problem that kept this useful instrument from seeing commercial applications. A fiber optics version of the instrument is well underway; the ability to detect surface tension gradients (and temperature gradients), using a set of acousto-optic modulator crystals placed in series in the fiber optics line, is being implemented.

**Significance:**
This instrument will allow noninvasive, noncontact measurements of surface tension. From this, one can extrapolate the local surface temperature (compared to bulk surface temperature) of a known clean sample. Viscosity information is also provided. Until we implemented the auto-tracking optics, vibration encountered in a regular laboratory environment made this instrument difficult to use. This means that a commercial version of this instrument will now likely become a reality. Brookhaven Instruments (among others) has already expressed an interest in making this happen, and has designed—and is providing—a custom version of their next-generation correlator with this in mind. Additionally, this instrument will be valuable for reduced-gravity studies of critical phenomena and interface characterization.

**Progress During FY 1993:**
We implemented one of several versions of a "cat's eye" auto-tracking optics design. The transmission version is working and the reflection versions are being tested. We now have a fiber optic array ready for use in one instrument design. We also have the dual acousto-optic modulators working for use in surface tension detection and instrument design. Our laser diodes are working well with the system, providing low noise for data analysis. Algorithms for flat surfaces are complete, and algorithms for curved-surface analysis are being written and tested. We are awaiting the arrival of several fusion-spliced, fiber optic polarization-maintaining couplers. We have a Langmuir trough at Case Western Reserve University ready for testing the instrument on interfaces. We also have a high vacuum system ready at Sandia National Laboratory ready for testing the instrument on liquid metals (lead/tin alloys). We have designed, and have had fabricated, a neutral-density filter that will allow us to extend the surface ripplon amplitude of the instrument to about two microns. This works by changing phase information in the
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scattered light to amplitude information, hence overcoming the problems of distinguishing what multiple of the (wavelength of light)/2 one is observing when working with large amplitude ripples on the surface of fluids.

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BIBLIOGRAPHIC CITATIONS FOR FY 1993:

**Journals:**

II. MSAD Program Tasks — Advanced Technology Development

Multizone Furnace Control Algorithm Development

**Principal Investigator:** Bruce N. Rosenthal
**NASA Lewis Research Center (LeRC)**

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

**Task Objective, Description & Significance:**

**Objective:**
The objective of this AID is to quantify the performance of a multivariable control system for eventual application to multizone crystal growth furnaces.

Multivariable control techniques are important because they can handle explicit coupling among furnace components rather than treating the coupling dynamics as disturbances. The major justification for use of multivariable furnace control is that thermal interactions exist between furnace heating elements. In a multizone furnace these interactions may be strong enough that accounting for them explicitly by the controller can improve performance. It has been our experience working with multizone furnaces that these interactions exist and they can be significant. Not only do interactions occur between heating elements, but also the ampoule, insulation and outside disturbances contribute to interactions as well. Until now we have not quantified the extent of these interactions and the ability of multivariable control methods to handle the complex nature of thermal interactions expected in multizone furnace systems.

**Description:**
Our approach is to quantify multivariable methods developed under prior AID research by studying the performance of multivariable control on a multizone test article. This will help us understand the controller performance in terms of multizone heating. This research will involve two major tasks. The first task will concern the design and construction of the multizone test article. The second task will concern the application and testing of the controller. The controls aspect of this project will involve the expertise of Professor Celal Batur from the University of Akron.

**Significance:**
Quantification will establish a baseline performance index for use of this technology in future applications.

**Progress During FY 1993:**

Significant Progress:
In November 1993, a statement of work was agreed upon between NASA Headquarters and Lewis Research Center. Once the technical direction of this AID was established, detailed design and fabrication of the multizone test article began. The test article frame was constructed from 3/4 inch angle iron. The frame measuring 9x9x12 inches was made by welding the angle to form a box structure. Attachments to the box frame were made to allow for movable side walls at two opposite ends. Half-inch thick Sali type refractory insulating boards are being used for the test article walls. Chromalox brand heating element, delivered in August, are being used for the linear multizone heaters. They have since been installed in the test article frame and have checked out for compatibility with existing power supplies. Initial configuration is setup for testing eight zones. The test article is mounted on a swivel support allowing the unit to be positioned at different angles with respect to gravity. Installation is more than 50% complete.
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Planned Activities:
Remaining construction includes the fabrication of thermocouple supports and fabrication of representative test specimen. Positioning of thermocouples and samples are a variable to be studied. Other variables include the position of the side walls, the emissivity of the side walls, the thermal conductivity of the side walls, the spacing between heating elements, the temperature of the heating elements, the orientation with respect to gravity of the test article. A design of experiments will be done to define an approach for the testing. Testing began in November 1993.

**STUDENTS FUNDED UNDER RESEARCH:**

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**PROJECT IDENTIFICATION:** ATD

**RESPONSIBLE CENTER:** LeRC

**BIBLIOGRAPHIC CITATIONS FOR FY 1993:**

**Proceedings:**

II. MSAD Program Tasks — Advanced Technology Development

Multi-Color Holography

PRINCIPAL INVESTIGATOR: Mr. William K. Witherow
NASA Marshall Space Flight Center (MSFC)

CO-INVESTIGATORS:
No Co-I's Assigned to this Task

TASK OBJECTIVE, DESCRIPTION & SIGNIFICANCE:

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.

DESCRIPTION:
In this system, two fluid parameters will be varied simultaneously, and this technique will measure the variations by using two different frequency lasers.

SIGNIFICANCE:
More complete multivariable research on fluid science experiments will be enabled by this new capability.

PROGRESS DURING FY 1993:
In the past year a test cell was constructed that allowed control of temperature gradients. Concentration gradients are created in the test cell by placing layers of fluids with increasing concentration. Tests were performed using the breadboard system at MSFC. The system at MSFC consists of two wavelengths (HeNe - 633 nm and HeCd - 442 nm). Tests were conducted to determine the accuracy of the temperature control of the test cell. Experiments were conducted in which temperature gradients only, concentration gradients only, and combinations of temperature and concentration gradients were introduced into the test cell. Experiments to determine the overall sensitivity of the system were performed by raising the temperature by a small amount and allowing the temperature of the test cell to become isothermal. The fringes seen in the system would move a certain distance dependent upon the amount of the temperature change. Similar experiments were conducted with concentration changes. Initial experiments contained sugar solutions so that the multicolor holographic technique could be characterized with known gradients. The temperature gradients were of the order of 0.07°C/cm and the concentration gradients were of the order of 0.5 g sugar/(cm liter). These weak gradients were used to demonstrate the capabilities of the system with gradients that are typical of spaceflight experiments. Data was collected from these experiments on video tape. The video data was used to begin development of computer software to perform analysis from the multi-color system.

Phase shifting interferometry has been incorporated into the multi-color holography technique because of the improvement of accuracy and sensitivity that it provides. Preliminary software has been developed that provides a phase map from the phase shifting interferometry data. One problem with producing a phase map from phase shifting interferometry data is phase wrap. The phase wrap is a consequence of the phase map algorithm. A new technique of unwrapping the phase is being developed at MSFC.

Components were purchased by Metrolaser to build a miniaturized breadboard system. Construction of the system was completed in July 1993. The system used fiber optics, a diode laser, and a diode-pumped frequency-doubled Nd-YAG laser (diode laser - 680 nm and Nd-YAG laser - 532 nm). By using current technology lasers the power...
consumption, weight, and size are minimized. Use of fiber optics eliminates the need for mirrors which also reduces the weight and minimizes table space for beam layout and pathlength matching requirements. The size, weight, and power consumption of the system are being kept small in anticipation of designing a spaceflight system. Tests have begun with this system to ensure that it is capable of constructing holograms. A minor setback occurred during the system check out. One of the output optical fibers was broken and had to be sent back to the manufacturer for repair. Work will resume when the system returns to Metrolaser.

The theory for using three wavelengths to separate concentration and temperature gradients has been developed. A third wavelength will allow three separate comparisons of the data to be made. This will allow an increase in the accuracy of the data by applying statistical methods. Also three lasers would provide a redundancy in a spaceflight system. The redundancy would allow data to be collected in the event of one of the lasers failing. However, while developing the theory, it was found that the beam intensity ratios during construction of the hologram will have to be lower to improve the efficiency of the hologram during reconstruction.

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  Biotechnology .................................................... III-497
  Combustion Science ............................................. III-500
  Fluid Physics ..................................................... III-503
  Materials Science ................................................ III-507

- Ground Research Tasks
  Benchmark Science ............................................. III-515
  Biotechnology .................................................... III-516
  Combustion Science ............................................. III-518
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- ATD
  Advanced Technology Development .......................... III-537

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III-495
Flight: Benchmark Science


Flight: Biotechnology


III. MSAD Program Tasks — FY1993

Bibliographic Citations


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Derby, J.J. Computer-aided analyses of internal radiation, conduction, and convection in crystal growth processes, Departmental Seminar Series, Dept. of Chemical Engineering, University of Wisconsin, Madison, WI, April 15, 1993, invited.

Derby, J.J. Finite element analysis of the growth of semitransparent crystals via the Bridgman method, Workshop on Control of Bridgman Growth of CdZnTe Crystals, University of Virginia, Charlottesville, VA, June 24-25, 1993, invited.


Derby, J.J. Computer-aided analyses of high-temperature materials processing systems: Crystal growth and ceramics sintering, Chemical Engineering Program Seminar Series, Univ. of California at San Diego, La Jolla, CA, September 15, 1993, invited.
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Bibliographic Citations


III. MSAD Program Tasks — FY1993

Bibliographic Citations


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Appendix

A  Table of Acronyms .................................................. A-1
B  Guest Investigator Index .......................................... B-1
C  Principal Investigator Index ..................................... C-1
Appendix A: MSAD Program — FY1993

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.

<table>
<thead>
<tr>
<th>Acronym</th>
<th>Definition</th>
</tr>
</thead>
<tbody>
<tr>
<td>AADSF</td>
<td>Advanced Automated Directional Solidification Furnace</td>
</tr>
<tr>
<td>AAL</td>
<td>Aero-acoustic Levitation</td>
</tr>
<tr>
<td>ACRT</td>
<td>Accelerated Crucible Rotation Technique</td>
</tr>
<tr>
<td>AO</td>
<td>Announcement of Opportunity</td>
</tr>
<tr>
<td>AOMS</td>
<td>Advanced Optical Monitoring Systems</td>
</tr>
<tr>
<td>APCF</td>
<td>Advanced Protein Crystallization Facility</td>
</tr>
<tr>
<td>APCG</td>
<td>Advanced Protein Crystal Growth</td>
</tr>
<tr>
<td>ATD</td>
<td>Advanced Technology Development</td>
</tr>
<tr>
<td>BDPU</td>
<td>Bubble, Drop and Particle Unit</td>
</tr>
<tr>
<td>BTF</td>
<td>Biotechnology Facility</td>
</tr>
<tr>
<td>CADAP</td>
<td>Computer-Aided Dendrite Analysis Program</td>
</tr>
<tr>
<td>CAST</td>
<td>Casting and Solidification Technology</td>
</tr>
<tr>
<td>CBE</td>
<td>Chemical Beam Epitaxy</td>
</tr>
<tr>
<td>CCD</td>
<td>Charge-coupled Device</td>
</tr>
<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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<tr>
<td>CGF</td>
<td>Crystal Growth Furnace</td>
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<tr>
<td>CM-1</td>
<td>Combustion Module</td>
</tr>
<tr>
<td>CNES</td>
<td>Centre Nationale d'Études Spatiales</td>
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<tr>
<td>CoDR</td>
<td>Conceptual Design Review</td>
</tr>
<tr>
<td>CPF</td>
<td>Critical Point Facility</td>
</tr>
<tr>
<td>CSA</td>
<td>Canadian Space Agency</td>
</tr>
<tr>
<td>CVD</td>
<td>Chemical Vapor Deposition</td>
</tr>
<tr>
<td>CVTE</td>
<td>Chemical Vapor Transport Experiment</td>
</tr>
<tr>
<td>CW</td>
<td>Continuous Wave</td>
</tr>
<tr>
<td>DARA</td>
<td>Deutsche Agentur für Raumfahrtangelegenheiten</td>
</tr>
<tr>
<td>DARTFire</td>
<td>Diffusive and Radiative Transport in Fires</td>
</tr>
<tr>
<td>DLR</td>
<td>The German Aerospace Research Establishment</td>
</tr>
<tr>
<td>DPM</td>
<td>Drop Physics Module</td>
</tr>
<tr>
<td>DSC</td>
<td>Differential Scanning Calorimetry</td>
</tr>
<tr>
<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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<td>EDS</td>
<td>Energy Dispersive Spectroscopy</td>
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<tr>
<td>EHD</td>
<td>Electrohydrodynamic</td>
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<tr>
<td>Acronym</td>
<td>Description</td>
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<td>---------</td>
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<tr>
<td>ESA</td>
<td>European Space Agency</td>
</tr>
<tr>
<td>FDSM</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>FVCS</td>
<td>Fluid Experiment/Vapor Crystal Growth System</td>
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<tr>
<td>TFEU</td>
<td>Free-Flow Electrophoresis Unit</td>
</tr>
<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>
</tr>
<tr>
<td>GBX</td>
<td>Glovebox</td>
</tr>
<tr>
<td>GFFC</td>
<td>Geophysical Fluid Flow Cell</td>
</tr>
<tr>
<td>HRT</td>
<td>High-Resolution Thermometer</td>
</tr>
<tr>
<td>ICE</td>
<td>Interface Configuration Experiment</td>
</tr>
<tr>
<td>IGDE</td>
<td>Isothermal Dendritic Growth Experiment</td>
</tr>
<tr>
<td>IML</td>
<td>International Microgravity Laboratory</td>
</tr>
<tr>
<td>IWG</td>
<td>Investigator Working Group</td>
</tr>
<tr>
<td>JPL</td>
<td>Jet Propulsion Laboratory</td>
</tr>
<tr>
<td>JSC</td>
<td>Johnson Space Center</td>
</tr>
<tr>
<td>KSC</td>
<td>Kennedy Space Center</td>
</tr>
<tr>
<td>LARC</td>
<td>Langley Research Center</td>
</tr>
<tr>
<td>LDV</td>
<td>Laser Doppler Velocimetry</td>
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<tr>
<td>LRC</td>
<td>Lewis Research Center</td>
</tr>
<tr>
<td>LIF</td>
<td>Large Isothermal Furnace</td>
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<tr>
<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 Phenomènes Intéressants de la Solidification sur Terre et en Orbit [Interesting Phenomena of Solidification on Earth and in Orbit]</td>
</tr>
<tr>
<td>MGM</td>
<td>Mechanics of Granular Materials</td>
</tr>
<tr>
<td>MMSL</td>
<td>Microgravity Materials Science Laboratory</td>
</tr>
<tr>
<td>MP</td>
<td>Microgravity Pressure</td>
</tr>
<tr>
<td>MPA</td>
<td>Microgravity Pressure, Ambient</td>
</tr>
<tr>
<td>MSA</td>
<td>Microgravity Science and Applications (Program)</td>
</tr>
<tr>
<td>MSAD</td>
<td>Microgravity Science and Applications Division</td>
</tr>
<tr>
<td>MSFC</td>
<td>Marshall Space Flight Center</td>
</tr>
<tr>
<td>MSL</td>
<td>Microgravity Science Laboratory</td>
</tr>
<tr>
<td>NASA</td>
<td>National Aeronautics and Space Administration</td>
</tr>
<tr>
<td>NASDA</td>
<td>National Space Development Agency of Japan</td>
</tr>
<tr>
<td>NIST</td>
<td>National Institute for Standards and Technology</td>
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<tr>
<td>NLO</td>
<td>Nonlinear Optical</td>
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<tr>
<td>NRA</td>
<td>NASA Research Announcement</td>
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<td>NRC</td>
<td>National Research Council</td>
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### Table of Acronyms

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<tr>
<th>Acronym</th>
<th>Description</th>
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<tr>
<td>OARE</td>
<td>Orbital Acceleration Research Experiment</td>
</tr>
<tr>
<td>OLSMA</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>PCG</td>
<td>Protein Crystal Growth</td>
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<td>PDA</td>
<td>Phase Doppler Pneumometer</td>
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<td>PJIV</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>
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<tr>
<td>RAPIV</td>
<td>Radiographic Particle Image Velocimetry</td>
</tr>
<tr>
<td>RDR</td>
<td>Requirements Definition Review</td>
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<tr>
<td>RPCVD</td>
<td>Remote Plasma-enhanced Chemical Vapor Epitaxy</td>
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<tr>
<td>RTE</td>
<td>Rainbow Schlieren Deflectometry</td>
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<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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<tr>
<td>SCF</td>
<td>Supercritical Fluid</td>
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<tr>
<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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<tr>
<td>SQUID</td>
<td>Superconducting Quantum Interference Detector</td>
</tr>
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<td>SSCE</td>
<td>Solid Surface Combustion Experiment</td>
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<tr>
<td>SSFF</td>
<td>Space Station Furnace Facility</td>
</tr>
<tr>
<td>STDCE</td>
<td>Surface Tension Driven Convection Experiment</td>
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<tr>
<td>STEP</td>
<td>Satellite Test of the Equivalence Principle</td>
</tr>
<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>
</tr>
<tr>
<td>TGDF</td>
<td>Turbulent Gasjet Diffusion Flames</td>
</tr>
<tr>
<td>TME</td>
<td>Test and Measurement Equipment</td>
</tr>
<tr>
<td>TMS</td>
<td>Thermal Maneuvering System</td>
</tr>
<tr>
<td>USML</td>
<td>United States Microgravity Laboratory</td>
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<td>USMP</td>
<td>United States Microgravity Payload</td>
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<tr>
<td>VCGS</td>
<td>Vapor Crystal Growth System</td>
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<tr>
<td>VDA</td>
<td>Vapor Diffusion Apparatus</td>
</tr>
<tr>
<td>VIBES</td>
<td>Vibration Isolation in a Box Experiment System</td>
</tr>
<tr>
<td>XRD</td>
<td>X-ray Diffraction</td>
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Appendix B: MSAD Program — FY1993

University of Alabama Guest Investigators (cf. p. II-25)

Academic: Domestic

W. F. Anderson — Vanderbilt University
E. Arnold — Rutgers University
J. K. Baird — University of Alabama, Huntsville
C. W. Carter, Jr. — University of North Carolina, Chapel Hill
W. J. Cook — University of Alabama, Birmingham
E. W. Czerwinski — University of Texas, Medical Branch
G. K. Farber — Pennsylvania State University
J. R. Knox — University of Connecticut
K. L. Krause — University of Houston
A. McPherson — University of California, Riverside
E. J. Meehan — University of Alabama, Huntsville
E. F. Meyer — Texas A&M University
T. L. Nagabhushan — Schering-Plough Research Institute
R. J. Naumann — University of Alabama, Huntsville
D. H. Ohlendorf — University of Minnesota
W. M. Rosenblum — University of Alabama, Birmingham
V. K. Senadhi — Temple University
L. Sieker — University of Washington
P. B. Sigler — Yale University
M. Sundaralingam — Ohio State University
D. Voel — University of Pennsylvania
W. W. Wilson — Mississippi State University

Academic: Foreign

S. Abara — Kyoto University (Japan)
C. Betzel — European Molecular Biology (Germany)
L. T. Delbaere — University of Saskatchewan (Canada)
G. G. Dodson — University of York (England)
Appendix B: MSAD Program — FY1993

University of Alabama Guest Investigators (cf. p. II-25)

J. Drenth — University of Groningen (The Netherlands)
W. G. Lever — The Australian National University (Australia)
P. G. Righetti — University di Milano (Italy)
D. Yang — McMaster University (Canada)

Industry: Domestic

Y. S. Babu — BioCryst Pharmaceuticals, Inc.
D. J. Duchamp — The Upjohn Company
D. S. Eggleston — Smith Kline Beecham Pharmaceuticals
H. M. Einspahr — Bristol-Meyers Squibb
B. C. Finzel — The Upjohn Company
N. D. Jones — Eli Lilly Research
M. A. Navia — Vertex Pharmaceuticals, Inc.
B. H. Rubin — Eastman Kodak Company
F. R. Salemme — 3-Dimensional Pharmaceuticals, Inc.
R. M. Swee — Associated Universities, Inc.
P. P. Trotta — Schering-Plough Research Institute
K. D. Watenpaugh — The Upjohn Company

Government: Domestic

D. Carter — NASA Marshall Space Flight Center (MSFC)
R. S. Snyder — NASA Marshall Space Flight Center (MSFC)
K. Ward — Naval Research Laboratory (NRL)
Government: Foreign

G. I. Birnbaum — National Research Council of Canada (Canada)

J. Fontecilla Camps — CENG LCCP/LIP (France)

A. Yonath — DESY/MPG (Germany)
<table>
<thead>
<tr>
<th>Name</th>
<th>Institution</th>
<th>Address</th>
<th>Office Ph</th>
<th>Fax</th>
</tr>
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<tbody>
<tr>
<td>Prof. G.J. (Reza) Abbaschian</td>
<td>Department of Materials Science and Engineering</td>
<td>132 Rhines Hall, Gainesville, FL 32611-2066</td>
<td>904 392-1454</td>
<td>904 392-6359</td>
</tr>
<tr>
<td>Dr. Davood Abdollahian</td>
<td>S. Levy Incorporated</td>
<td>3425 S. Bascom Avenue, Campbell, CA 95008-4870</td>
<td>408 377-4870</td>
<td></td>
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<tr>
<td>Ali Abtahi</td>
<td>Mail Stop 183-401 Jet Propulsion Laboratory</td>
<td>4800 Oak Grove Drive, Pasadena, CA 91109</td>
<td>818 354-5353</td>
<td>818 393-5039</td>
</tr>
<tr>
<td>Prof. Guenter Ahlers</td>
<td>Director, Center for Nonlinear Sciences Quantum Institute</td>
<td>University of California, Santa Barbara, CA 93106</td>
<td>805 893-3795</td>
<td>805 893-4970</td>
</tr>
<tr>
<td>Dr. Iwan D. Alexander</td>
<td>Center for Microgravity and Materials Research</td>
<td>University of Alabama, Huntsville, AL 35899</td>
<td>205 895-6887</td>
<td></td>
</tr>
<tr>
<td>Prof. Robert A. Altenkirch</td>
<td>College of Engineering</td>
<td>P.O. Drawer DE, Mississippi State University, MS 39762-5543</td>
<td>601 325-2270</td>
<td>601 325-8573</td>
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<tr>
<td>Prof. John L. Anderson</td>
<td>Department of Chemical Engineering</td>
<td>Carnegie Mellon University, Pittsburgh, PA 15213</td>
<td>412 268-2230</td>
<td>412 268-7139</td>
</tr>
<tr>
<td>Dr. J. Barry Andrews</td>
<td>Materials Science and Engineering</td>
<td>BEC 360, University of Alabama, Birmingham, AL 35294-4461</td>
<td>205 934-8450</td>
<td>205 934-8485</td>
</tr>
<tr>
<td>Prof. Robert E. Apfel</td>
<td>Robert Higgins Professor of Mechanical Engineering</td>
<td>Yale University, New Haven, CT 06520</td>
<td>203 432-4346</td>
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<tr>
<td>Principal Investigator</td>
<td>Institution</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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<td>2158 GG Brown, Ann Arbor, MI 48109-2125</td>
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<tr>
<td>Dr. Klaus J. Bachmann</td>
<td>Materials Research Center</td>
<td>Department of Chemistry</td>
<td>919 515-2538</td>
<td>919 515-3419</td>
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<td></td>
<td>Campus Box 7919, North Carolina State University</td>
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<td>Raleigh, NC 27695-8204</td>
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<tr>
<td>Dr. M. Yousef Bahadori</td>
<td>Thermal Sciences Division</td>
<td>Science Applications International Corporation</td>
<td>310 781-8723</td>
<td>310 781-8730</td>
</tr>
<tr>
<td></td>
<td></td>
<td>21151 Western Avenue, Torrance, CA</td>
<td></td>
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</tr>
<tr>
<td>Dr. Martin B. Barmatz</td>
<td>Technical Group Leader</td>
<td>Jet Propulsion Laboratory</td>
<td>818 354-3088</td>
<td>818 393-5039</td>
</tr>
<tr>
<td></td>
<td></td>
<td>4800 Oak Grove Drive, Pasadena, CA</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Dr. James C. Baygents</td>
<td>Department of Chemical Engineering</td>
<td>Harshbarger Building, Room 120, University of Arizona</td>
<td>602 621-6044</td>
<td>602 621-6048</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Tucson, AZ 85721</td>
<td></td>
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</tr>
<tr>
<td>Prof. Robert J. Bayuzick</td>
<td>Department of Applied and Engineering Science</td>
<td>Materials Science and Engineering Program</td>
<td>615 322-7047</td>
<td>615 343-8645</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Box 1580, Station B, Vanderbilt University</td>
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<td>Nashville, TN 37235</td>
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<tr>
<td>Jeanne L. Becker, Ph.D.</td>
<td>Dept. of Obstetrics and Gynecology</td>
<td>University of South Florida</td>
<td>813 254-7774</td>
<td>813 254-0940</td>
</tr>
<tr>
<td></td>
<td></td>
<td>4 Columbia Drive, Tampa, FL 33606</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Prof. Christoph Beckermann</td>
<td>Dept. of Mechanical Engineering</td>
<td>University of Iowa</td>
<td>319 335-5681</td>
<td></td>
</tr>
<tr>
<td>Principal Investigator</td>
<td>Address Details</td>
<td>Office Phone</td>
<td>Fax</td>
<td>Other Details</td>
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</tr>
<tr>
<td>Dr. Lynn A. Boatner</td>
<td>Head, Ceramics and Interfaces Section&lt;br&gt;Solid State Division&lt;br&gt;Mall Stop 6056&lt;br&gt;Oak Ridge National Laboratory&lt;br&gt;P.O. Box 2008&lt;br&gt;Oak Ridge, TN 37831</td>
<td>Office Ph: 615 574-5492 Fax: 615 574-4814</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Prof. Melvyn C. Branch</td>
<td>Center for Combustion Research&lt;br&gt;Engineering Center, Campus Box 427&lt;br&gt;University of Colorado&lt;br&gt;Boulder, CO 80309-0427</td>
<td>Office Ph: 303 492-6318 Fax: 303 492-2863</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Prof. R. Malcolm Brown</td>
<td>Department of Botany&lt;br&gt;University of Texas&lt;br&gt;Austin, TX 78712</td>
<td>Office Ph: 512 471-3363</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Prof. Robert A. Brown</td>
<td>Head, Chemical Engineering Department&lt;br&gt;Chemical Engineering &amp; Materials Processing Center&lt;br&gt;Massachusetts Institute of Technology&lt;br&gt;Cambridge, MA 02139</td>
<td>Office Ph: 617 729-3822 Fax: 617 253-5726</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Prof. John C. Buckmaster</td>
<td>101 Transportation Building&lt;br&gt;University of Illinois&lt;br&gt;104 South Mathews Avenue&lt;br&gt;Urbana, IL 61801</td>
<td>Office Ph: 217 333-1803</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Dr. Charles E. Bugg</td>
<td>Center for Macromolecular Crystallography&lt;br&gt;262 BHS, THT 79&lt;br&gt;University of Alabama, Birmingham&lt;br&gt;Birmingham, AL 35294</td>
<td>Office Ph: 205 934-5329 Fax: 205 934-0480</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Dr. Daniel C. Carter</td>
<td>Chief, Biophysics Branch&lt;br&gt;Microgravity Science and Applications Division&lt;br&gt;ES76&lt;br&gt;NASA Marshall Space Flight Center&lt;br&gt;Marshall Space Flight Center, AL 35812</td>
<td>Office Ph: 205 544-5492 Fax: 205 544-1777</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Dr. Ared Cezairliyan</td>
<td>Program Leader, Metallurgy Division&lt;br&gt;124 Hazard Building&lt;br&gt;National Institute of Standards and Technology&lt;br&gt;Gaithersburg, MD 20899</td>
<td>Office Ph: 301 975-5931 Fax: 301 990-2572</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Name</td>
<td>Address</td>
<td>Office Ph</td>
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</tr>
<tr>
<td>Dr. An-Ti Chai</td>
<td>MS 500-327, NASA Lewis Research Center, 21000 Brookpark Road, Cleveland, OH 44135</td>
<td>216 433-2073</td>
<td>216 433-8660</td>
<td>257</td>
</tr>
<tr>
<td>Prof. Paul M. Chaikin</td>
<td>Department of Physics, P.O. Box 708, Princeton University, Princeton, NJ 08544</td>
<td>609 258-4338</td>
<td>609 258-6360</td>
<td>60, 383</td>
</tr>
<tr>
<td>Dr. Arnon Chait</td>
<td>MS 105-1, NASA Lewis Research Center, 21000 Brookpark Road, Cleveland, OH 44135</td>
<td>216 433-3558</td>
<td></td>
<td>384</td>
</tr>
<tr>
<td>Prof. S. H. Chan</td>
<td>Dept. of Mechanical Engineering, University of Wisconsin, Milwaukee, P.O. Box 784, Milwaukee, WI 53201</td>
<td>414 229-5001</td>
<td>414 229-6958</td>
<td>258</td>
</tr>
<tr>
<td>Prof. Chuan F. Chen</td>
<td>Dept. of Aerospace and Mechanical Engineering, College of Engineering &amp; Mines, University of Arizona, Tucson, AZ 85721</td>
<td>602 621-8199</td>
<td>602 621-8191</td>
<td>260, 385</td>
</tr>
<tr>
<td>Dr. Robert K. Cheng</td>
<td>Applied Science Division, B29C-102, Lawrence Berkeley Laboratory, 1 Cyclotron Road, Berkeley, CA 94720</td>
<td>510 486-5438</td>
<td>510 486-7303</td>
<td>222</td>
</tr>
<tr>
<td>Dr. Norman Chigier</td>
<td>Dept. of Mechanical Engineering, Carnegie Mellon University, Schenley Park, Pittsburgh, PA 15213-3890</td>
<td>412 268-2498</td>
<td></td>
<td>261</td>
</tr>
<tr>
<td>Dr. Talso C. P. Chui</td>
<td>Mail Stop 125-214, Jet Propulsion Laboratory, 4800 Oak Grove Drive, Pasadena, CA 91109-8099</td>
<td>818 354-3104</td>
<td>818 393-4878</td>
<td>145</td>
</tr>
<tr>
<td>Prof. Jacob N. Chung</td>
<td>Dept. of Mechanical and Materials Engineering, Washington State University, Pullman, WA 99164-2920</td>
<td>509 335-3222</td>
<td></td>
<td>263</td>
</tr>
</tbody>
</table>
Dr. Ivan O. Clark  
Information Systems Division  
Mail Stop 473  
NASA Langley Research Center  
5 North Dryden Street  
Hampton, VA 23681-001  
Office Ph: 804 864-1500  
Fax: 804 864-7891  
265, 267, 388

Prof. Julian D. Cole  
Dept. of Mathematical Sciences  
Rensselaer Polytechnic Institute  
Troy, NY 12180-3590  
Office Ph: 518 276-6916  
269

Prof. Paul Concus  
Lawrence Berkeley Laboratory  
University of California, Berkeley  
1 Cyclotron Road  
Berkeley, CA 94270  
Office Ph: 510 486-5508  
Fax: 510 486-5401  
270

Dr. Reid F. Cooper  
Materials Science and Engineering  
University of Wisconsin  
1509 University Avenue  
Madison, WI 53706  
Office Ph: 608 262-1133  
Fax: 608 262-8353  
390

Prof. Sam R. Coriell  
Materials Building 233, Room B-166  
National Institute of Standards and Technology  
Gaithersburg, MD 20899  
Office Ph: 301 975-6169  
273

Arnold Dahm  
Physics Dept.  
Case Western Reserve University  
10900 Euclid Avenue  
Cleveland, OH 44106  
Office Ph: 216 368-3586  
393

Prof. Robert H. Davis  
Department of Chemical Engineering  
Campus Box 424  
University of Colorado  
Boulder, CO 80309  
Office Ph: 303 492-7314  
Fax: 303 492-4341  
275, 277, 278, 394

Prof. Stephen H. Davis  
Department of Engineering Sciences  
and Applied Mathematics  
Northwestern University  
633 Clark St.  
Evanston, IL 60208  
Office Ph: 708 491-5397  
281

Dr. Delbert E. Day  
Graduate Center for Materials Research  
Materials Research Center  
University of Missouri, Rolla  
Rolla, MO 65401  
Office Ph: 314 341-4354  
Fax: 314 341-2071  
396
### Principal Investigator Index

<table>
<thead>
<tr>
<th>Name</th>
<th>Institution details</th>
<th>Phone</th>
<th>Fax</th>
<th>Page Numbers</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dr. Lawrence J. Delucas</td>
<td>Center for Macromolecular Crystallography, University of Alabama, Birmingham</td>
<td>205 934-0115</td>
<td>205 934-0121</td>
<td>25, 27</td>
</tr>
<tr>
<td>Dr. Jeffrey J. Derby</td>
<td>Department of Chemical Engineering &amp; Materials Sciences, University of Minnesota</td>
<td>612 625-8881</td>
<td>612 625-8881</td>
<td>399</td>
</tr>
<tr>
<td>Prof. Graham D. deVahl Davis</td>
<td>University of New South Wales, P.O. Box 1, Kensington, AUSTRALIA</td>
<td>612 697-4099</td>
<td>612 663-1222</td>
<td>401</td>
</tr>
<tr>
<td>Dr. Daniel Dietrich</td>
<td>NASA Lewis Research Center Group, Sverdrup Technology, Inc.</td>
<td>216 433-2875</td>
<td></td>
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</tr>
<tr>
<td>Prof. Russell J. Donnelly</td>
<td>Physics Department, University of Oregon</td>
<td>503 346-4226</td>
<td>503 346-4708</td>
<td>147, 149</td>
</tr>
<tr>
<td>Prof. A. E. Dukler</td>
<td>Dept. of Chemical Engineering, University of Houston</td>
<td>713 743-4310</td>
<td>713 743-4323</td>
<td>61, 283</td>
</tr>
<tr>
<td>Dr. Robert V. Duncan</td>
<td>Electrical Standards Division, Sandia National Laboratory</td>
<td>505 844-4843</td>
<td>505 844-4372</td>
<td>7</td>
</tr>
<tr>
<td>Prof. Douglas J. Durian</td>
<td>Department of Physics, University of California, Los Angeles</td>
<td>310 206-2645</td>
<td>310 825-0897</td>
<td>284</td>
</tr>
<tr>
<td>Prof. P.K. Dutta</td>
<td>Department of Chemistry, Ohio State University</td>
<td>614 292-4532</td>
<td>614 292-1685</td>
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<tr>
<td>Principal Investigator</td>
<td>Institution and Address</td>
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<tr>
<td>Prof. Charles Elbaum</td>
<td>Physics Department, Brown University, Providence, RI 02912</td>
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<tr>
<td></td>
<td>Office Ph: 401 863-2186 Fax: 401 863-2024</td>
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<tr>
<td>Prof. C.W. Francis Everitt</td>
<td>Hansen Experimental Physics Laboratory, Stanford University, Stanford, CA 94305</td>
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<tr>
<td></td>
<td>Office Ph: 415 725-4103 Fax: 415 725-8312</td>
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<tr>
<td>Prof. Gerard M. Faeth</td>
<td>Department of Aerospace Engineering, University of Michigan, Ann Arbor, MI 48109-2140</td>
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<tr>
<td></td>
<td>Office Ph: 313 746-7202</td>
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<tr>
<td>Prof. A. Carlos Fernandez-Pello</td>
<td>Department of Mechanical Engineering, University of California, Berkeley, CA 94720</td>
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<tr>
<td></td>
<td>Office Ph: 415 642-6554</td>
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<tr>
<td>Prof. Richard A. Ferrell</td>
<td>Department of Physics and Astronomy, University of Maryland, College Park, MD 20742-4111</td>
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<tr>
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<td>Office Ph: 301 405-6148 Fax: 301 314-9465</td>
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<tr>
<td>Prof. Merton C. Flemings</td>
<td>Head, Department of Materials Science and Engineering, Massachusetts Institute of Technology, Cambridge, MA 02139-4307</td>
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<tr>
<td></td>
<td>Office Ph: 617 253-3233 Fax: 617 258-6886</td>
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<tr>
<td>Dr. Donald O. Frazier</td>
<td>Mail Code ES74, NASA Marshall Space Flight Center, Marshall Space Flight Center, AL 35812</td>
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<tr>
<td></td>
<td>Office Ph: 205 544-7825</td>
<td></td>
<td></td>
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<tr>
<td>Lisa E. Freed, M.D., Ph.D.</td>
<td>Information Systems Division, MIT E25-342, Massachusetts Institute of Technology, Cambridge, MA 02139</td>
<td></td>
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<tr>
<td></td>
<td>Office Ph: 617 253-3443 Fax: 617 258-8827</td>
<td></td>
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<tr>
<td>Dr. Archibald L. Fripp</td>
<td>Information Systems Division, NASA Langley Research Center, Hampton, VA 23681-0001</td>
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<tr>
<td></td>
<td>Office Ph: 804 864-1503 Fax: 804 864-7891</td>
<td></td>
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<td></td>
</tr>
</tbody>
</table>
Appendix C: MSAD Program — FY1993
Principal Investigator Index

Prof. Robert W. Gammon
Institute for Physical Science and Technology
Building 85
University of Maryland
College Park, MD 20742
Office Ph: 301 405 4791 Fax: 301 314-9509

Prof. Stephen Garoff
Dept. of Physics
Carnegie Mellon University
5000 Forbes Avenue
Pittsburgh, PA 15213
Office Ph: 412 268-6877

Prof. Randall M. German
Department of Engineering Science
227 Hammond Building
Pennsylvania State University
University Park, PA 16802-1401
Office Ph: 814 865-4700

Mr. Thomas K. Glasgow
MS 105-1
NASA Lewis Research Center
21000 Brookpark Road
Cleveland, OH 44135
Office Ph: 216 433-5014 Fax: 216 433-5033

Prof. Irvin Glassman
Dept. of Mechanical & Aerospace Eng.
Princeton University
Princeton, NJ 08544
Office Ph: 609 258-3090

Prof. Martin E. Glicksman
Materials Engineering Department
MRC-104
Rensselaer Polytechnic Institute
Troy, NY 12180-3590
Office Ph: 518 276-6449 Fax: 518 276-8554

Prof. Alessandro Gomez
Department of Mechanical Engineering
Yale University
New Haven, CT 06520
Office Ph: 203 432-4384 Fax: 203 432-7654

Dr. John A. Goree
Department of Physics and Astronomy
University of Iowa
Iowa City, IA 52242-1479
Office Ph: 319 335-1843 Fax: 319 335-1753

Paul S. Greenberg
NASA Lewis Research Center
21000 Brookpark Road
Cleveland, OH 44135
Office Ph: 216 433-3621
Dr. Richard N. Grugel  
Center for Microgravity Research & Applications  
Box 6079 Station B  
Vanderbilt University  
Nashville, TN 37235  
Office Ph: 615 343-6965 Fax: 615 343-8730

Prof. Hossein Haj-Hariri  
Dept. of Mech and Aerospace Engineering  
Thorton Hall  
University of Virginia  
Charlottesville, VA 22903  
Office Ph: 804 924-6290 Fax: 804 982-2037

J. Woods Halley  
Physics Department  
University of Minnesota  
116 Church SE  
Minneapolis, MN 55455  
Office Ph: 612 624-0395 Fax: 612 624-4578

Prof. Kevin P. Hallinan  
Mechanical and Aerospace Engineering Dept  
University of Dayton  
300 College Park  
Dayton, OH 45469-0210  
Office Ph: 513 229-2835 Fax: 513 229-3433

Dr. John E. Hart  
Department of Astrophysical, Planetary, and Atmospheric Sciences  
Campus Box 391  
University of Colorado  
Boulder, CO 80309-0391  
Office Ph: 303 492-8568 Fax: 303 492-3822

Frank T. Hartley  
Supervisor, Advanced Test and Measurement Group  
Instrumentation Section  
Mail Stop 125-177  
Jet Propulsion Laboratory  
4800 Oak Grove Drive  
Pasadena, CA 91109  
Office Ph: 818 354-3139 Fax: 818 354-8153

Charles R. Hartzell, Ph.D.  
Director of Research  
Alfred I. DuPont Institute of the Nemours Foundation  
P.O. Box 269  
Wilmington, DE 19899  
Office Ph: 302 651-6800 Fax: 302-651-6888

Prof. T. Alan Hatton  
Chevron Professor of Chemical Engineering  
Department of Chemical Engineering  
Massachusetts Institute of Technology  
77 Massachusetts Avenue  
Cambridge, MA 02139  
Office Ph: 617 253-4588 Fax: 617 253-8723
Appendix C: MSAD Program — FY1993
Principal Investigator Index

Dr. Robert H. Hauge
Chemistry Dept.
Rice University
P.O. Box 1892
Houston, TX 77252

Office Ph: 713 527-8101  Fax: 713 285-5155

Prof. Angus Hellawell
Department of Metallurgical Engineering
Michigan Technological University
Houghton, MI 49931

Office Ph: 906 487-2261

Prof. George M. Homsy
Dept. of Chemical Engineering
Stanford University
Stanford, CA 94305

Office Ph: 415 723-2419

Dr. Wesley C. Hymer
Department of Molecular and Cell Biology
Pennsylvania State University
University Park, PA 16802

Office Ph: 814 865-9182  Fax: 814 865-2413

Marylou Ingram, M.D.
Huntington Medical Research Institute
99 North El Molino Avenue
Pasadena, CA 91101-1830

Office Ph: 818 795-4343

Dr. Ulf E. Israelsson
Mail Stop 125-112
Jet Propulsion Laboratory
4800 Oak Grove Drive
Pasadena, CA 91109-8099

Office Ph: 818 354-9255  Fax: 818 393-6383

Dr. Kenneth A. Jackson
Arizona Materials Laboratory
Department of Materials Science and Engineering
University of Arizona
4715 E. Fort Lowell Road
Tucson, AZ 85712

Office Ph: 602 322-2981

Prof. Donald T. Jacobs
Physics Department
The College of Wooster
Wooster, OH 44691

Office Ph: 216 262-2390

David Jasnow
Dept. of Physics and Astronomy
University of Pittsburgh
Pittsburgh, PA 15260

Office Ph: 412 624-9029  Fax: 412 624-9163

C-10
<table>
<thead>
<tr>
<th>Name</th>
<th>Department/Institute</th>
<th>Address</th>
<th>Contact Information</th>
</tr>
</thead>
<tbody>
<tr>
<td>J. Milburn Jessup, M.D.</td>
<td>Associate Professor&lt;br&gt;Department of Surgery&lt;br&gt;Suite 3A&lt;br&gt;New England Deaconess Hospital&lt;br&gt;110 Francis Street&lt;br&gt;Boston, MA 02215</td>
<td><strong>Office Ph:</strong> 617 632-9817  <strong>Fax:</strong> 617 632-7424 ........................................ 181, 184</td>
<td></td>
</tr>
<tr>
<td>Dr. William L. Johnson</td>
<td>Department of Material Science&lt;br&gt;331 Keck Labs&lt;br&gt;Mail Code 138-78&lt;br&gt;California Institute of Technology&lt;br&gt;Pasadena, CA 91125</td>
<td><strong>Office Ph:</strong> 818 395-4433  <strong>Fax:</strong> 818-795-1547 ........................................ 113</td>
<td></td>
</tr>
<tr>
<td>Dr. K. Kailasanath</td>
<td>Laboratory for Computational Physics&lt;br&gt;and Fluid Dynamics&lt;br&gt;NRL Code 4410&lt;br&gt;Washington, DC 20375</td>
<td><strong>Office Ph:</strong> 202 767-2402 ........................................ 228</td>
<td></td>
</tr>
<tr>
<td>Dr. Eric W. Kaler</td>
<td>Department of Chemical Engineering&lt;br&gt;Colburn Laboratory&lt;br&gt;University of Delaware&lt;br&gt;Newark, DE 19716</td>
<td><strong>Office Ph:</strong> 302 831-3553 ........................................ 298</td>
<td></td>
</tr>
<tr>
<td>Dr. Takashi Kashiwagi</td>
<td>Center for Fire Research, Building and Fire Research Laboratories&lt;br&gt;Bldg. 224, Rm. B258&lt;br&gt;National Institute For Standards and Technology&lt;br&gt;Gaithersburg, MD 20899</td>
<td><strong>Office Ph:</strong> 301 975-6699  <strong>Fax:</strong> 301 975-4052 ........................................ 230</td>
<td></td>
</tr>
<tr>
<td>Dr. Mohammad Kassemi</td>
<td>Ohio Aerospace Institute&lt;br&gt;MS 105-1&lt;br&gt;NASA Lewis Research Center&lt;br&gt;21000 Brookpark Road&lt;br&gt;Cleveland, OH 44135</td>
<td><strong>Office Ph:</strong> 216 433-5031  <strong>Fax:</strong> 216 433-5031 ........................................ 419</td>
<td></td>
</tr>
<tr>
<td>Prof. Michael J. Kaufman</td>
<td>Department of Materials Science and Engineering&lt;br&gt;132 Rhines Hall&lt;br&gt;University of Florida&lt;br&gt;Gainesville, FL 32611-2066</td>
<td><strong>Office Ph:</strong> 904 392-6662  <strong>Fax:</strong> 904 392-6359 ........................................ 421</td>
<td></td>
</tr>
<tr>
<td>Prof. Robert E. Kelly</td>
<td>School of Engineering and Applied Science&lt;br&gt;University of California, Los Angeles&lt;br&gt;408 Hilgard Avenue&lt;br&gt;Los Angeles, CA 90024-1597</td>
<td><strong>Office Ph:</strong> 310 825-5489  <strong>Fax:</strong> 310 206-4830 ........................................ 300</td>
<td></td>
</tr>
</tbody>
</table>
Prof. Kenneth F. Kelton  
Department of Physics  
Campus Box 1105  
Washington University at St. Louis  
One Brookings Drive  
St. Louis, MO 63130  
*Office Ph:* 314-935-6228  
*Fax:* 314-935-6219

Dr. John H. Konnert  
Laboratory for the Structure of Matter  
Code 6030  
Naval Research Laboratory  
4555 Overlook Avenue S.W.  
Washington, DC 20375-6000  
*Office Ph:* 202-767-3267  
*Fax:* 202-767-6874

Dr. Joel Koplik  
Department of Physics  
Levich Institute  
City College of The City University of New York  
Convent Avenue & 138th Street  
New York, NY 10031  
*Office Ph:* 212-650-8162

Prof. Jean N. Koster  
Department of Aerospace Engineering Sciences  
Engineering Center  
Campus Box 429  
University of Colorado  
Boulder, CO 80309-0429  
*Office Ph:* 303-492-6945

Prof. Sindo Kou  
Dept. of Materials Science and Engineering  
University of Wisconsin  
1509 University Avenue  
Madison, WI 53706  
*Office Ph:* 608-262-0576  
*Fax:* 608-262-8353

Prof. Dr. William B. Krantz  
Center of Low-Gravity Fluid Mechanics  
Campus Box 432  
University of Colorado  
Boulder, CO 80309-0432  
*Office Ph:* 303-492-7050

Dr. Shankar Krishnan  
Containerless Research, Inc.  
910 University Place  
Evanston, IL 60261-3149  
*Office Ph:* 708-467-2678  
*Fax:* 708-467-2679

Prof. Jerry C. Ku  
Department of Mechanical Engineering  
Wayne State University  
5050 Anthony Wayne Drive  
Detroit, MI 48202  
*Office Ph:* 313-577-3814
<table>
<thead>
<tr>
<th>Principal Investigator</th>
<th>Affiliation</th>
<th>Location</th>
<th>Office Phone</th>
<th>Fax Phone</th>
</tr>
</thead>
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<tr>
<td>Prof. Richard T. Lahey</td>
<td>Nuclear Engineering and Engineering Physics</td>
<td>Troy, NY 12180-3590</td>
<td>518 276-8579</td>
<td></td>
</tr>
<tr>
<td>Dr. David J. Larson</td>
<td>Research Center A02-026</td>
<td>Bethpage, NY 11714</td>
<td>516 575-4896</td>
<td></td>
</tr>
<tr>
<td>Prof. Chung K. Law</td>
<td>Mechanical &amp; Aerospace Engineering</td>
<td>Princeton, NJ 08544</td>
<td>609 258-5271</td>
<td>258-6233</td>
</tr>
<tr>
<td>Dr. L. Gary Leal</td>
<td>Department of Chemical and Nuclear Engineering</td>
<td>Santa Barbara, CA 93106</td>
<td>805 893-8510</td>
<td>893-4731</td>
</tr>
<tr>
<td>Dr. Sandor L. Leboczky</td>
<td>Code ES75</td>
<td>NASA Marshall Space Flight Center</td>
<td>205 544-7758</td>
<td>544-8762</td>
</tr>
<tr>
<td>Peter I. Leikes, Ph.D.</td>
<td>Laboratory of Cell Biology, Sinai Samaritan Medical School</td>
<td>Milwaukee, WI 53201-0342</td>
<td>414 283-7753</td>
<td>283-7874</td>
</tr>
<tr>
<td>Prof. Moises Levy</td>
<td>Physics Department</td>
<td>University of Wisconsin, Milwaukee</td>
<td>414 229-4168</td>
<td></td>
</tr>
<tr>
<td>Prof. Sung P. Lin</td>
<td>Department of Engineering of Mechanical and Aeronautical Engineering</td>
<td>Clarkson University</td>
<td>315 268-6584</td>
<td>268-6438</td>
</tr>
<tr>
<td>Prof. John A. Lipa</td>
<td>W. W. Hansen Laboratories</td>
<td>Stanford University</td>
<td>415 723-4562</td>
<td>723-8451</td>
</tr>
</tbody>
</table>

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Principal Investigator Index

Dr. Thomas A. Lograsso
Institute for Physical Research and Technology
109 Metals Development Building
Iowa State University
Ames, IA 50011
Office Ph: 515 294-8425  Fax: 515 294-8727  434

Prof. Sudarshan K. Loyalka
Particulate Systems Research Center
0039 Engineering Complex
University of Missouri, Columbia
Columbia, MO 65211
Office Ph: 314 882-8366  Fax: 314 882-2490  313

Dr. Daniel W. Mackowski
Assistant Professor, Department of Mechanical Engineering
College of Engineering
Auburn University
Auburn, AL 36849-5341
Office Ph: 205 844-3334  Fax: 205 844-3307  436

Efstratios Manousakis
Department of Physics
318 Keen Bldg.
Florida State University
Tallahassee, FL 32306-4052
Office Ph: 904 644-3713  Fax: 904 644-8630  160

Prof. Humphrey J. Maris
Physics Department
Brown University
Providence, RI 02912
Office Ph: 401 863-2185  Fax: 401 863-2024  162

Prof. Lon J. Mathias
Departments of Polymer Science and Chemistry
University of Southern Mississippi
Hattiesburg, MS 39406-0076
Office Ph: 601 266-4871  Fax: 601 266-5504  438

Dr. David H. Matthiesen
Materials Science and Engineering Dept.
Case Western Reserve University
10900 Euclid Avenue
Cleveland, OH 44106
Office Ph: 216 368-1366  121, 122, 124

Prof. Mark J. McCready
Department of Chemical Engineering
182 Fitzpatrick Hall
University of Notre Dame
Notre Dame, IN 46556
Office Ph: 219 631-7146  315

Prof. Gareth H. McKinley
Division of Applied Sciences
Pierce Hall, Room 316
Harvard University
Cambridge, MA 02138
Office Ph: 617 496-5167  Fax: 617 495-9837  76

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<table>
<thead>
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<th>Name</th>
<th>Department/Address</th>
<th>Office Phone</th>
<th>Fax Phone</th>
</tr>
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<tr>
<td>Dr. Alexander McPherson, Jr.</td>
<td>Department of Biochemistry, University of California, Riverside, CA 92521-0129</td>
<td>909 787-5391</td>
<td>909 787-3790</td>
</tr>
<tr>
<td>Prof. Herman Merte, Jr.</td>
<td>Mechanical Engineering Department, University of Michigan, Ann Arbor, MI 48109</td>
<td>313 764-5240</td>
<td>313 747-3170</td>
</tr>
<tr>
<td>Prof. Horst Meyer</td>
<td>Department of Physics, Duke University, Durham, NC 27708-0305</td>
<td>919 660-2520</td>
<td>919 660-2521</td>
</tr>
<tr>
<td>Mary Jo B. Meyer</td>
<td>NASA Lewis Research Center, 21000 Brookpark Road, Cleveland, OH 44135</td>
<td>216 433-8165</td>
<td>216 433-5033</td>
</tr>
<tr>
<td>William V. Meyer</td>
<td>Ohio Aerospace Institute, MS 105-1, NASA Lewis Research Center, 21000 Brookpark Road, Cleveland, OH 44135-3191</td>
<td>216 433-5011</td>
<td>216 433-5033</td>
</tr>
<tr>
<td>Dr. Michael R. Moldover</td>
<td>Thermophysics Division, Bldg. 221, Room A105, National Institute of Standards and Technology, Gaithersburg, MD 20899</td>
<td>301 975-2459</td>
<td>301 869-4020</td>
</tr>
<tr>
<td>Prof. John J. Moore</td>
<td>Dept. of Metallurgical and Materials Engineering, Colorado School of Mines, Golden, CO 80401</td>
<td>303 273-3770</td>
<td></td>
</tr>
<tr>
<td>Prof. Allan S. Myerson</td>
<td>Department of Chemical Engineering, Polytechnic University, New York, 333 Jay Street, Brooklyn, NY 11201</td>
<td>718 260-3620</td>
<td>718 260-3136</td>
</tr>
<tr>
<td>Prof. Ranga Narayanan</td>
<td>Department of Chemical Engineering, 431 Chemical Engineering Building, University of Florida, Gainesville, FL 32611-2083</td>
<td>904 392-9103</td>
<td>904 392-9673</td>
</tr>
</tbody>
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Principal Investigator Index

Prof. G. Paul Neitzel
George W. Woodruff School of Mechanical Engineering
Georgia Institute of Technology
Atlanta, GA 30332-0405
Office Ph: 404 894-3240 ........................................... 320

Dr. James R. Norris
Chemistry Division
Building 200/Room E133
Argonne National Laboratory
9700 South Cass Avenue
Argonne, IL 60439
Office Ph: 708 252-3547 Fax: 708 252-9289 .................................... 193

Prof. Simon Ostrach
Department of Mechanical & Aerospace Engineering
418 Glennan Building
Case Western Reserve University
10900 Euclid Avenue
Cleveland, OH 44106
Office Ph: 216 368-2942 Fax: 216 368-6445 .................................... 80, 322

Prof. A.G. Ostrogorsky
Department of Mechanical Engineering
Aeronautical Engineering, and Mechanics
Rensselaer Polytechnic Institute
Troy, NY 12180-3590
Office Ph: 518 276-6975 Fax: 518 276-2623 .................................... 446

Bernhard O. Palsson, Ph.D.
Dept. of Chemical Engineering
Herbert H. Dow Building
University of Michigan College of Engineering
2300 Hayward
Ann Arbor, MI 48109-2136
Office Ph: 313 763-2383 Fax: 313 763-0459 .................................... 195

Prof. Jerome K. Percus
Courant Institute
New York University
251 Mercer St.
New York, NY 10012
Office Ph: 212 998-3130 ........................................... 324

Prof. John H. Perepezko
Department of Materials Science and Engineering
University of Wisconsin
1509 University Avenue
Madison, WI 53706
Office Ph: 608 263-1678 ........................................... 448

Prof. David R. Poirier
Department of Materials Science & Engineering
College of Engineering & Mines
University of Arizona
Tucson, AZ 85721
Office Ph: 602 621-6072 ........................................... 450, 452
Dr. Marc L. Pusey  
Microgravity Science and Application Division  
Mail Code ES76  
NASA Marshall Space Flight Center  
Marshall Space Flight Center, AL 35812  
Office Ph: 205 544-7823 Fax: 205 544-1777

Dr. Seth J. Putterman  
Physics Department  
University of California, Los Angeles  
Los Angeles, CA 90024  
Office Ph: 310 825-2269 Fax: 310 825-5668

Dr. Chandra S. Ray  
Graduate Center for Materials Research  
University of Missouri, Rolla  
Rolla, MO 65401  
Office Ph: 314 341-6432 Fax: 314 341-2071

Prof. Dennis W. Readey  
Coors Professor of Ceramic Engineering  
Dept. of Metallurgical and Materials Eng.  
Colorado School of Mines  
Golden, CO 80401  
Office Ph: 303 273-3437

Dr. Won-Kyu Rhim  
Mail Stop 183-401  
Jet Propulsion Laboratory  
4800 Oak Grove Drive  
Pasadena, CA 91109  
Office Ph: 818 354-2925

Prof. David C. Richardson  
Department of Biochemistry  
Box 3711  
Duke University Medical Center  
211 Nanaline Duke Building  
Durham, NC 27710  
Office Ph: 919 684-6010 Fax: 919 684-8885

Dr. Michael B. Robinson  
ES75  
NASA Marshall Space Flight Center  
Huntsville, AL 35812  
Office Ph: 205 544-7774 Fax: 205 544-2176

Prof. Paul D. Ronney  
Department of Mechanical Engineering  
OHE 430  
University of Southern California  
Los Angeles, CA 90089-1453  
Office Ph: 213 740-0490 Fax: 213 740-8071

Prof. Franz Rosenberger  
Center for Microgravity and Materials Research  
Research Institute  
Room M65  
University of Alabama, Huntsville  
Huntsville, AL 35899  
Office Ph: 205 895-6050 Fax: 205 895-6791
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Principal Investigator Index

Bruce N. Rosenthal
NASA Lewis Research Center
21000 Brookpark Road
Cleveland, OH 44135
Office Ph: 216 433-5027 ................................................... 491

Graham Ross
O/92-05, B/260
Lockheed Missiles & Space Co.
3251 Hanover Street
Palo Alto, CA 94304-1187
Office Ph: 415 424-3488 Fax: 415 424-3315 ........................... 327

Dr. Howard Ross
MS 500-217
NASA Lewis Research Center
21000 Brookpark Road
Cleveland, OH 44135
Office Ph: 216 433-2868 ................................................... 51, 238

Dr. Paul H. Rothe
Creare, Inc.
P.O. Box 71
Hanover, NH 03755
Office Ph: 603 643-3800 Fax: 603 643-4657 ........................... 328

Kurt R. Sacksteder
MS 500-217
NASA Lewis Research Center
21000 Brookpark Road
Cleveland, OH 44135
Office Ph: 216 433-2857 Fax: 216 433-8660 ........................... 244

Dr. Satwindar S. Sadhal
Department of Mechanical Engineering
University of Southern California
Los Angeles, CA 90089-1453
Office Ph: 213 740-0492 Fax: 213 740-8071 ........................... 330

W. Mark Saltzman
Dept. of Chemical Engineering
Room 24, New Engineering Building
Johns Hopkins University
3401 North Charles Street
Baltimore, MD 21218
Office Ph: 410 516-8480 Fax: 410 516-5510 ........................... 203

Dr. David W. Sammons
Center for Separation Science
Bldg. #90, Rm 211
University of Arizona
Tucson, AZ 85721
Office Ph: 602 621-2157 ................................................... 33

Prof. Robert L. Sani
Department of Chemical Engineering
Engineering Center
ECCH 1-43, Campus Box 424
University of Colorado
Boulder, CO 80309-0424
Office Ph: 303 492-5517 Fax: 303 492-4341 ........................... 83

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Principal Investigator Index

Dr. Dudley A. Saville
Department of Chemical Engineering
Princeton University
Princeton, NJ 08544-5263
Office Ph: 609 258-4585 Fax: 609 258-0211

David W. Scharp, M.D.
Department of Surgery
Box 8109
Washington University School of Medicine
4939 Audubon Ave.
St. Louis, MO 63110
Office Ph: 314 362-8320 Fax: 314 361-0426

Prof. Benjamin Shaw
Department of Mechanical, Aeronautical & Materials Engineering
University of California, Davis
Davis, CA 95616-5294
Office Ph: 916 752-4130

Dr. N.B. Singh
Westinghouse Electric Corporation
1310 Beulah Road
Pittsburgh, PA 15235
Office Ph: 412 256-1469 Fax: 412 256-1661

Dr. Gregory T. Smedley
Mail Stop 278-01
Jet Propulsion Laboratory
4800 Oak Grove Drive
Pasadena, CA 91109
Office Ph: 818 354-0583

Prof. Marc K. Smith
George W. Woodruff School of Mechanical Engineering
Georgia Institute of Technology
Atlanta, GA 30332-0405
Office Ph: 404 894-3826

Dr. Robert S. Snyder
Mail Code ES76
NASA Marshall Space Flight Center
Marshall Space Flight Center, AL 35812
Office Ph: 205 544-7755 Fax: 205 544-8762

Prof. Frans Spaepen
Division of Applied Sciences
Harvard University
29 Oxford Street
Cambridge, MA 02193
Office Ph: 617 495-3760 Fax: 617 495-9837

Prof. Paul H. Steen
School of Chemical Engineering
Olin Hall
Cornell University
Ithaca, NY 14853
Office Ph: 607 255-4749 Fax: 607 255-9166

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Principal Investigator Index

Dr. Doru M. Stefanescu
Department of Metallurgical and Materials Engineering
F301 Mineral Industries Building, Box 870202
University of Alabama
Tuscaloosa, AL 35487-0202
Office Ph: 205 348-1740 Fax: 205 348-8574

Dr. Stein Sture
Department of Civil, Environmental and Architectural Engineering
Campus Box 428
University of Colorado
Boulder, CO 80309
Office Ph: 303 492-7651 Fax: 303 492-7317

Dr. Ching-Hua Su
USRA Staff Scientist, Crystal Growth and Solidification Physics Branch
Space Science Laboratory
ES75
NASA Marshall Space Flight Center
Marshall Space Flight Center, AL 35812
Office Ph: 205 544-7776 Fax: 205 544-8762

Prof. R. Shankar Subramanian
Department of Chemical Engineering
Box 5705
Clarkson University
Potsdam, NY 13699-5705
Office Ph: 315 268-6648 Fax: 315 268-6654

Prof. Harry L. Swinney
Dept. of Physics
University of Texas, Austin
Austin, TX 78712-1111
Office Ph: 512 471-4619 Fax: 512 471-1588

Dr. Julian Szekely
Department of Materials Engineering
Room 4-138
Massachusetts Institute of Technology
77 Massachusetts Avenue
Cambridge, MA 02139
Office Ph: 617 253-3236 Fax: 617 253-8124

Dr. Frank R. Szofran
Space Science Laboratory
Microgravity Science and Applications Division
Mail Stop ES75
NASA Marshall Space Flight Center
Huntsville, AL 35812
Office Ph: 205 544-7777 Fax: 205 544-8762

Prof. James S. T'ien
Mechanical & Aerospace Engineering
415 Glennan Building
Case Western Reserve University
10900 Euclid Avenue
Cleveland, OH 44106
Office Ph: 216 358-4581 Fax: 216 358-6445

---

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Appendix C: MSAD Program — FY1993
Principal Investigator Index

Prof. Saleh Tanveer
Department of Mathematics
Ohio State University
231 West 18th Avenue
Columbus, OH 43210-1174

Office Ph: 614 292-5710 Fax: 614 292-1479

Dr. Robert L. Thompson
MS 500-327
NASA Lewis Research Center
21000 Brookpark Road
Cleveland, OH 44135

Office Ph: 216 433-3321 Fax: 216 433-8660

Dr. Eugene H. Trinh
Mail Stop 183-401
Jet Propulsion Lab
4800 Oak Grove Drive
Pasadena, CA 91109

Office Ph: 818 354-7125 Fax: 818 393-5039

Prof. Gretar Tryggvason
Department of Mechanical Engineering
2250 G. G. Brown Building
University of Michigan
Ann Arbor, MI 48109

Office Ph: 313 763-1049

Prof. Jorge Vinals
Super Computer Research Institute
444 Science Center Library
Florida State University
Tallahassee, FL 32306-4052

Office Ph: 904 644-1010

Dr. Frank B. von Swol
Dept. of Chemical Engineering
114 Roger Adams Laboratory
University of Illinois
1209 W. California St.
Urbana, IL 61801

Office Ph: 217 333-8963 Fax: 217 244-8064

Prof. Peter W. Voorhees
Dept. of Materials Science and Engineering
Northwestern University
2145 Sheridan Road
Evanston, IL 60208-3108

Office Ph: 708 491-3537 Fax: 708-491-7820

Dr. Taylor G. Wang
Center for Microgravity Research & Applications
Box 6079, Station B
Vanderbilt University
Nashville, TN 37235

Office Ph: 615 322-7311 Fax: 615 343-8730

Dr. Keith B. Ward
Laboratory for the Structure of Matter
Code 6030
Naval Research Laboratory
Washington, DC 20375-5000

Office Ph: 202 767-2735 Fax: 202 767-6874

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### Appendix C: MSAD Program — FY1993

#### Principal Investigator Index

<table>
<thead>
<tr>
<th>Name</th>
<th>Institution</th>
<th>Office Phone</th>
<th>Fax Phone</th>
<th>Page Numbers</th>
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<tbody>
<tr>
<td>Prof. Peter C. Wayner, Jr.</td>
<td>Isermann Dept. of Chemical Engineering</td>
<td>518 276-6199</td>
<td>518 276-4030</td>
<td>355</td>
</tr>
<tr>
<td>Dr. Richard J.K. Weber</td>
<td>Containerless Research, Inc.</td>
<td>708 467-2678</td>
<td>708 467-2679</td>
<td>469</td>
</tr>
<tr>
<td>Dr. Michael C. Weinberg</td>
<td>Department of Materials Science &amp; Engineering</td>
<td>602 621-6070</td>
<td>602 322-2993</td>
<td>139, 471</td>
</tr>
<tr>
<td>Dr. David A. Weitz</td>
<td>EXXON Research and Engineering Co.</td>
<td>908 730-3536</td>
<td>908 730-3042</td>
<td>93</td>
</tr>
<tr>
<td>Dr. Heribert Wiedemeier</td>
<td>Department of Chemistry</td>
<td>518 276-8444</td>
<td>518 276-8554</td>
<td>140</td>
</tr>
<tr>
<td>Dr. John M. Wiencek</td>
<td>Department of Chemical &amp; Biochemical Engineering</td>
<td>908 235-5323</td>
<td>908 932-5313</td>
<td>212</td>
</tr>
<tr>
<td>Dr. R. Allen Wilkinson</td>
<td>MS 500-327, NASA Lewis Research Center</td>
<td>216 433-2075</td>
<td>216 433-8660</td>
<td>18</td>
</tr>
<tr>
<td>Prof. Forman A. Williams</td>
<td>Dept of Applied Mechanics and Engineering Science, B-010</td>
<td>619 534-5452</td>
<td></td>
<td>54, 250</td>
</tr>
<tr>
<td>Dr. W. William Wilson</td>
<td>Department of Chemistry</td>
<td>601 325-3584</td>
<td>601 325-1618</td>
<td>214</td>
</tr>
</tbody>
</table>
Appendix C: MSAD Program — FY1993

Principal Investigator Index

Mr. William K. Witherow
Microgravity Science and Applications Division
ES74 SSL Bldg. 4481
NASA Marshall Space Flight Center
Marshall Space Flight Center, AL 35812
Office Ph: 205 544-7811 Fax: 205 544-2102

Prof. August F. Witt
TDK Professor of Materials Science & Engineering
Department of Materials Science
RM 13-4134
Massachusetts Institute of Technology
Cambridge, MA 02139-4307
Office Ph: 617 253-5303 Fax: 617 253-5827

Prof. M. Grae Worster
Northwestern University
633 Clark Street
Evanston, IL 60201
Office Ph: 708 491-3345

Dr. Xiao-lun Wu
Department of Physics and Astronomy
University of Pittsburgh
Pittsburgh, PA 15260
Office Ph: 412 624-0873

Prof. Abdelfattah Zebib
Dept. of Mechanical and Aerospace Engineering
Rutgers University
P.O. Box 909
Piscataway, NJ 08855-0909
Office Ph: 908 932-2248 Fax: 908 932-5313

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# Microgravity Science & Applications Program Tasks and Bibliography for FY 1993

The document is an annual report published by the Microgravity Science and Applications Division (MSAD). It represents a compilation of the Division's currently-funded ground, flight, and Advanced Technology Development tasks. The purpose of the document is to provide an overview and progress report for these tasks, including progress reports by principal investigators selected from the academic, industrial, and government communities. The document includes a listing of new bibliographic data provided by the principal investigators to reflect the dissemination of research data during FY 1993. The document also includes division research metrics and an index of the funded investigators.

The document contains three sections and three appendices: Section I is an introduction and contains data, Section II is a compilation of the task reports in an order representative of the ground, flight, and ATD status and the science discipline it represents, and Section III is the bibliography. The three appendices, in the order of presentation, are: A — a microgravity science acronym list, B — a list of guest investigators associated with a biotechnology task, and C — an index of the currently funded principal investigators.

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<td>MSAD-funded principal investigators tasked through NASA research centers</td>
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The document is an annual report published by the Microgravity Science and Applications Division (MSAD). It represents a compilation of the Division's currently-funded ground, flight, and Advanced Technology Development tasks. The purpose of the document is to provide an overview and progress report for these tasks, including progress reports by principal investigators selected from the academic, industrial, and government communities. The document includes a listing of new bibliographic data provided by the principal investigators to reflect the dissemination of research data during FY 1993. The document also includes division research metrics and an index of the funded investigators.

The document contains three sections and three appendices: Section I is an introduction and contains data, Section II is a compilation of the task reports in an order representative of the ground, flight, and ATD status and the science discipline it represents, and Section III is the bibliography. The three appendices, in the order of presentation, are: A — a microgravity science acronym list, B — a list of guest investigators associated with a biotechnology task, and C — an index of the currently funded principal investigators.

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