

NASA Technical Memorandum 87568

**Microgravity Science
and Applications
Program Tasks**

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**Microgravity Science
and Applications
Program Tasks**

Compiled by

Elizabeth Pentecost

National Aeronautics and Space Administration

Washington, D.C.

NASA

National Aeronautics
and Space Administration

**Scientific and Technical
Information Branch**

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NASA TECHNICAL MEMORANDUM

I. INTRODUCTION

The Microgravity Science and Applications Program is directed toward research in the science and technology of processing materials under conditions of low gravity to provide a detailed examination of the constraints imposed by gravitational forces on Earth. The program is expected to lead, ultimately, to the development of new materials and processes in commercial applications adding to this nation's technological base. The research studies emphasize the selected materials and processes that will best elucidate the limitations due to gravity and demonstrate the enhanced sensitivity of control of processes that may be provided by the weightless environment in space. Primary effort will be devoted to a comprehensive study of the specific areas of research which revealed potential value in the initial investigations of the previous decades. Examples of previous process research include growth of crystals and directional solidification of metals in the quiescent conditions in which gravitational fluid flow is eliminated, containerless processing of reactive materials to eliminate reactions with the container and to provide geometrical control of the product, synthesis and separation of biological materials in weightlessness to reduce heat and mass transfer problems associated with sedimentation and buoyancy effects; identification of high vacuum characterization associated with an orbiting wake shield, and minimal knowledge of terrestrial processing methods.

Additional effort will be devoted to identifying the special requirements which drive the design of hardware to reduce the risk in future developments. Examples of current hardware studies are acoustic, electromagnetic, and electrostatic containerless processing modules and electrophoresis separation devices.

In addition to the basic research nature of the program, a lower level of effort is being expended on the business, a logistics and legal implication of rights of data and patents, control of materials, and division of responsibilities when NASA works with commercial ventures aimed at specific products. Examples of current materials research which might lead to commercialization include infrared detector crystals, inertial confinement fusion targets, aligned magnets, and ferromagnetic materials.

The current program emphasis on fundamental processing science and technology in selected areas will continue as the Microgravity Science and Applications Program addresses problems of interest to the public and private commercial sectors which can be resolved by recourse to the space environment.

Emphasis will be placed on the expansion of currently funded activities for ground-based and space-flight investigations to maximize the outputs from these opportunities. Initiatives requiring new hardware will be encouraged at a low level until funds can be made available. The

expansion of current efforts is occurring as a result of focusing support for current space flight investigations on forming facility experiment teams to provide advice and identify future involvement. Emphasis has been placed on experiments involving the Materials Experiment Assembly and Mid-Deck experiments on the Space Shuttle.

II. TASKS

ELECTRONIC MATERIALS

Processing of Complex Compound Semiconductors in Space

North Carolina State University
Dr. Klaus Bachmann
NAG1-354
May 1, 1983 - April 20, 1986

The purpose of this first year ground based research effort was to develop growth and characterization facilities for bulk single crystals of compound semiconductor alloys. As a system of II-VI alloy crystal growth, $\text{Cd}_x\text{Mn}_{1-x}\text{Te}$ was chosen since it has a relatively narrow solidus-liquidus separation serving thus as a suitable first model for zone melting and solution growth. Also, it allows, in principle, perfect lattice matching to $\text{Cd}_x\text{Hg}_{1-x}\text{Te}$ that we want to study as a model for the study of hetero-epitaxial growth from Te-solutions. A vertical zone melting furnace was built and used in the synthesis of $\text{Cd}_x\text{Mn}_{1-x}\text{Te}$ from the elements and crystal growth in carbon coated fused silica crucibles of Bridgman growth geometry. Unseeded single crystals of $\text{Cd}_x\text{Mn}_{1-x}\text{Te}$ have been grown in the composition range $0.80 \leq x \leq 1$. Photoluminescence spectra of the crystals reveal band to band recombination at energies that agree with the reported literature data. In addition, deep luminescence is present at energies corresponding to proportional shifts from the 1.4 eV deep emission observed for bulk single crystals of CdTe. Difficulties still exist with twinning of the crystals. In unseeded growth the crystal axis tends to be in (111) activating twinning only on this plane, but not on the inclined 111 planes. A horizontal zone leveling system for the processing of ingots of 4 cm diameter and 25 cm length has been built and will be used for seeded growth of $\text{Cd}_x\text{Mn}_{1-x}\text{Te}$ and $\text{InAs}_y\text{Sb}_{1-y}$ that will be utilized as substrated insubsequent epitaxy experiments. A liquid phase epitaxy system for the growth of $\text{Cd}_z\text{Hg}_{1-z}\text{Te}/\text{Cd}_x\text{Mn}_{1-x}\text{Te}$ heterostructures is presently being assembled and a high resolution x-ray diffractometer is under construction for structural characterization of bulk single crystals and epitaxial structures and for high precision lattice parameter measurements that will be used to relate crystal properties to their deviation from stoichiometric atom ratios.

Fluid Flow in Crystal Growth: Analysis of the Vertical Bridgman and Floating Zone Process

Massachusetts Institute of Technology
Professor Robert A. Brown
NSG-7645

Research is aimed at the fundamental understanding of the interactions of heat and mass transport and fluid mechanics in crystal growth from the melt, especially in how it pertains to experiments supported by the Microgravity Science and Applications program. Emphasis has been on studies of the vertical Bridgman system designed and constructed at MIT, on small-scale floating zone systems proposed for space flight, and on quantitative prediction of nonlinear transitions in melt/solid interface morphologies leading to the formation of dendrites in directional solidification. Each research project applies new developments in the mathematical modelling of melt crystal growth and convection to problems of central interest for precision solidification of semiconductors and metals.

The analysis of vertical Bridgman growth has led to the complete numerical analysis of transport phenomena in directional solidification and to a detailed comparison of calculations to the GaGe growth experiments of Wang and Witt. Calculations show the central role of the ampoule material in determining the radial temperature gradients in the melt and thus the intensity of convection. Results indicate that diffusion-controlled growth can not be achieved on earth with conventional growth system, but are feasible in microgravity. Analysis is presently underway to understand the effect of externally applied magnetic fields on the structure of convection and on the transitions to time-periodic flow in an earth-bound system. This research is preliminary to full finite element calculations which incorporate the effect of the magnetic field in the model for vertical Bridgman growth.

A thermocapillary model of heat transfer and melt/gas interface shape in small-scale floating zones has been developed to predict zone shape and stability in experimental systems and is a precursor to a complete analysis of the interaction of fluid flow in the melt due to thermocapillary, buoyancy and crystal rotation with one shape and solute segregation. Calculations give the limits of validity of the cylindrical shape approximation often used in analyses. In the next year, research will focus on extending this analysis to include the effects of convection in the melt and on coupling the model more directly with experiments.

The analysis of nonlinear melt/solid interface morphologies in directional solidification has continued in an effort to theoretically predict the transitions between planar, cellular, and dendritic crystal growth. Using a combination of finite element analysis and computer implemented perturbation methods, we have predicted the details of the

formation of large amplitude cells in two dimensional interfaces and are presently analyzing for the onset of side-branching leading to dendritic structures. Research in the coming year will start the extension of these results to three-dimensional morphologies where comparison with experiments is simpler.

Publications

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Solutal Convection and Its Effects on Crystal Growth and Segregation in Binary and Pseudo-Binary System with Large Liquidus-Solidus Separation

Massachusetts Institute of Technology
Dr. Robert A. Brown
Dr. August F. Witt
NSG-7645

This research program is composed of theoretical and experimental studies of solutal convection and its effects on crystal growth and segregation in binary and pseudo-binary systems with large liquidus-solidus separation. Studies are being carried out as an equally funded collaborative effort between the Metallurgy Division of the Centre D'Etude Nucleaire, Grenoble, France, and the Materials Processing Center of MIT under the sponsorship of ESA and NASA, respectively. Both research groups are aimed at advancing the theoretical framework for solidification and at optimizing crystal growth experiments to be conducted in a microgravity environment. Substantial progress toward these goals has been made at both research facilities.

The theoretical program has resulted in the development of a transient simulation of the growth of concentrated alloys in microgravity and has been used to study the dynamics of GeSi, PbSnTe, HgCdTe growth. Finite element analysis has also been developed for studying the interactions of buoyancy-driven convection in the melt caused by temperature and concentration variations with melt-solid interface morphology and alloy segregation in the grown crystal. These calculations are being compared directly to experimental measurements of segregation and interface shape for GeSi growth at Grenoble and GaGe growth at MIT. The results for SiGe indicate that careful design of the thermal system can result in almost diffusion-controlled growth on earth because of the stabilizing influence of the concentration gradient ahead of the solidification front. Single crystal PbSnTe has been successfully grown at MIT and the segregation results support the presence of rampant convection in the melt caused by the unstable density gradient due to the rejection of PbSn at the interface.

Research during the next year will focus on the development of experiment systems for use aboard the Space Shuttle and on the extension of our finite element analysis to calculations of other materials systems under consideration by NASA, e.g. HgCdTe and the low temperature succinonitrile-ethanol system. A fully transient finite element simulation of the dynamics of the coupled melt flow and interface system is also being developed in order to probe the instabilities which limit crystal growth rate for concentrated alloys.

Crystal Growth Research in Space

Langley Research Center

Dr. R. K. Crouch

Dr. A. L. Fripp

In Center

October 1984 - October 1985

The objectives of this work are to develop growth techniques and theory leading to improved bulk growth of semiconductor single crystals. Ground based experiments will be complemented by experiments carried out in the low-g environment provided by the Space Shuttle. Analytical studies and laboratory investigations are being conducted to better define the causes of crystalline defects and inhomogeneities. The compound semiconductor lead-tin-telluride is being used as the modeling material. Theoretical techniques are being developed to predict the thermal and solutal fields which are present during bulk growth from a melt. Techniques for measuring the thermophysical properties of semiconductors at high temperatures have been developed. During the past year electrochemical etching techniques have been developed for delineation of inhomogeneous regions in crystals. Thermal diffusivity measurements have been completed for the solid and liquid phases of PbTe and PbSnTe. Preliminary results have been obtained on the effects on crystal morphology of gravity, interface shape and interaction between the melt and the container. Preflight ground based tests have been completed in the MEA-A2 furnace for a flight scheduled for October 1984. Flight samples have been delivered to MSFC. Investigation into the effects of gravity, interface shape, surface tension, and ampoule interactions will be continued in the coming year. Electrochemical etching and interface demarcation studies will be continued. Results from the MEA-A2 flight will be analyzed and reported. Theoretical calculations will be carried out which include the effects of solutally driven convective flows.

Publications

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Crouch, R. K., et al., "Semiconductor Crystal Growth in the Microgravity Environment," NICE Symposium on Materials Processing in Space, 1984.

Fripp, A. L., et al., "Ground-Based Experiments in Preparation for Directional Solidification Crystal Growth in Space," presented at 6th American Conference on Crystal Growth, Atlantic City, NJ, July 1984.

Crouch, R. K., et al., "Influence of Gravity on the Morphology of Bridgman Grown Lead-Tin-Telluride," presented at 6th American Conference on Crystal Growth, Atlantic City, NJ, July 1984.

Growth of Organic Crystals by Ostwald Ripening

3M Corporation
Dr. William Egbert
Dr. Christopher Podsiadly
Dr. Robert Naumann, MSFC
Joint Endeavor Agreement

The objective of this investigation is to evaluate the growth of various organic crystals by chemical precipitation and Ostwald ripening.

Six precipitation reactors were flown on STS-51A. Five of the reactors contained proprietary materials. The sixth contained urea dissolved in ethanol with toluene as the precipitating agent. The size distribution will be jointly analyzed by NASA and 3M and compared with the model being developed by Baird and Naumann.

The Influence of Low Frequency Mechanical Vibrations on the Growth of Single Crystals

Center for Materials Research - Stanford University
Professor Robert S. Feigelson
Dr. Dennis Elwell
NAS8-34872 (NASA Contact: V. Yost, MSFC)

The optimum conditions for crystal growth are usually achieved either by suppressing convective fluid flows (e.g., by the use of a low-gravity environment) or by over-riding thermal and solutal convection by the use of a strong stirring action. It is particularly difficult to achieve vigorous stirring in melts contained in tall, narrow crucibles which are used for crystal growth by the Bridgman method. We have developed a novel stirring technique which involves subjecting a vertical crucible to a circle in a horizontal plane (without rotation). Use of an amplitude of 3 mm at a frequency of ~6Hz produced complete mixing of a non-uniform aqueous liquid in a few seconds. The mixing time increases rapidly with liquid viscosity, but this appears to be the fastest mixing technique yet devised for low-viscosity liquids in sealed containers.

The mixing action involved the downward flow of liquid in the outer annulus of the liquid, driven by surface waves. When the downward flowing liquid reaches the bottom of the crucible, it is reflected in a central, upward flowing spiral. This flow pattern should be beneficial for crystal growth by the Bridgman method since it will sweep impurities away from the walls and produce a more convex solid-liquid interface. Initial attempts to apply our new stirring technique to CdTe crystal growth did not show significant improvement in the number of crystals nucleated, but the interface shape appeared to be close to that predicted. Flow conditions were not optimized.

The other vibrational stirring studied in this program, axial vibration at 60 Hz, was found to cause relatively little stirring action. The positive influence on crystal quality, which has been observed in some cases, must be due to its effect on processes occurring at the solid-liquid interface.

The Control of Float Zone Interfaces by the Use of Selected Boundary Conditions

Science Applications, Inc.
Dr. Larry M. Foster
NAS8-35108

One of main goals of the Float Zone (FZ) growth project of NASA's Microgravity Science and Applications program is to thoroughly understand the molten zone/freezing crystal system and all the mechanisms that govern this system. To accomplish this, the melt and interface properties, the heat and mass flows, and the dependencies of these on each other and on growth rate and g-levels must be studied.

Since the float zone process involves two solid-melt interfaces, possible gas interfaces, heat and mass transfers, various driving forces and complex heating sources, an analysis of the entire process would be very complex. For an initial investigation, a more feasible approach is to examine each component of the process separately, particularly if mathematical models are to be manageable. The three principal components are: (1) the shapes of the melt and solid-melt interfaces, (2) the heat and mass transfers, and (3) the heating and cooling sources. This study combined facets of all three components.

The purpose of this effort (completed 5 December 1983) was to study and compute the surface boundary conditions required to give flat FZ solid-melt interfaces. The successful completion of this study should provide FZ furnace designers with better methods for controlling solid-melt interface shapes and for computing thermal profiles and gradients.

This study was undertaken in two phases. The first phase was to investigate the solid zones surface boundary conditions required for flat solid-melt interfaces when given the melt zone surface boundary conditions. The second phase complemented the first and was to investigate the melt zone surface boundary conditions required for flat solid-melt interfaces if given the solid zones surface boundary conditions. Dual integral transform methods were used in both phases; in addition, the use of various numerical methods for differential equations and linear systems of equations were required.

Using NASA supplied data, the surface boundary conditions required for flat solid-melt interfaces were studied. In addition, complete documentation and a simple user's guide was provided for all the computer software required during this study.

Crystal Growth of Device Quality GaAs in Space

Massachusetts Institute of Technology
Dr. Harry C. Gatos
NSG-7331
April 1977 - continuing task

The GaAs research evolves about these key thrust areas. The overall program combines: (1) studies of crystal growth on novel approaches to engineering of semiconductor material (i.e., GaAs and related compounds); (2) investigation and correlation of materials properties and electronic characteristics on a macro- and microscale; (3) investigation of electronic properties and phenomena controlling device applications and device performance.

This effort is aimed at the essential ground-based program which would insure successful experimentation with and eventually processing of GaAs in near zero gravity environment. It is believed that this program addresses in a unique way materials engineering aspects which bear directly on the future exploitation of the potential of GaAs and related materials in device and systems applications.

Publications

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Crystal Melts Under Reduced Gravity

Massachusetts Institute of Technology
Professor Harvey P. Greenspan
NAS8-35412 (NASA Contact: I. Yates, MSFC)
December 1982 - December 1984

To date, fluid motion due to thermocapillary convection and centrifugal buoyancy has been studied theoretically and numerically, by adapting specialized programs for rotating viscous fluids. It has been shown that by rotating the crystal melt, thermocapillary flow can be confined to a thin layer at the melt-gas interface even when the interfacial velocity is large enough for non-linear effects to be important.

Theoretical analysis of the basic processes proceeds, but a much larger effort is now being devoted to the numerical simulation of the actual motion in a rotating crystal melt. A numerical program is under development for the study of the fully nonlinear problem. Although the flow is viewed, to start, as one of rapid rotation (in order that certain boundary layer simplifications can be invoked), a more general approach will allow consideration of the situations and conditions of real practical interest.

Aided by appropriate theoretical analysis and insight, we are acquiring a capability to calculate in most circumstances the shape of the melt zone (the free surface and melt-solid interface), the circulation induced by variable surface tension, and the spatial distributions of temperature and chemical concentrations. In addition, it will be possible to explore many special effects of rotation as well as those due to the deposition and desorption of surfactants and variable rheological properties.

Microgravity Silicon Zoning Investigation

Westech Systems, Inc.
Dr. E. L. Kern, Consultant
G. L. Gill
NAS8-34920 (NASA Contact: I. Yates, MSFC)
July 1982 - December 1984

This research program is directed toward the understanding of the float zone crystal growth process, the melt interactions which lead to crystal inhomogeneities, and the influence of microgravity on reducing these inhomogeneities. Silicon was selected as the model crystal because its inhomogeneities lead to known variations in device performance, and because the mechanisms involved in its growth are understood better than for other high temperature crystals. The objective of the program is to understand the growth mechanisms in float zone growth and thereby determine the feasibility and advantages of float zone growth of silicon under microgravity conditions. This will be done by characterizing the growth at $g = 1$, projecting the changes in melt flows due to microgravity, observing these in space growth and determining the effects on defect inhomogeneities.

A Thin Rod Zoner was constructed as a laboratory prototype for flight growth of 5mm diameter silicon crystals, which can be done within the power and cooling capabilities of shuttle flights. A new method of zoning silicon, using resistance heating, has resulted in melting 5mm diameter ingots with less than 100 watts power, under vacuum, and less than 200 watts in an argon zoning atmosphere. The tailored thermal profile resistance heater (Pt./Rh alloy) is being optimized for silicon zoning at 1420°C. Oscillatory melt flows, already characterized at 25mm diameter using r.f. heating, will be characterized at the smaller diameter and related to models. These oscillatory variations lead to striations, which are rapid variations in resistivity and point defect densities.

A different configuration of float zoning silicon is to melt the center of a slice suspended by its edges and recrystallize by slowly cooling and freezing toward the center. This involves a much thinner melt cross-section and much larger surface area than the rod configuration. Using electron beam melting and fast solidification, melt oscillations of 2m sec. period were observed, as compared to 250m sec. for 25mm diameter rods. The instability in this configuration is not predicted by theory. An elliptically heated system has been constructed and the regrowth and melt oscillations will be characterized under better controlled conditions. The effect of solid surface layers stop Marangoni flow will be studied.

Solution Growth of Crystals in Zero-Gravity

Alabama A&M University

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NAS8-32945 (NASA Contact: W.W. Moore, MSFC)

March 5, 1978 - December 31, 1985

In a low-g environment, buoyancy driven convection effects in solution crystal growth are greatly reduced and, thus, one can study diffusion mass transport which in 1-g is masked by convective phenomena. Crystals of triglycine sulfate (TGS) will be grown aboard the Spacelab 3 mission, using a specially developed Fluids Experiments System (FES). The objectives of the experiment are (1) to develop a technique for solution crystal growth in a low-g environment, (2) to characterize the growth environment provided by an orbiting spacecraft and to determine the influence of the environment on the growth behavior, and (3) to determine how gravity in a low-gravity environment influences the properties of a resulting TGS crystal.

Single crystals of TGS have been grown using conventional low-temperature solution crystal growth method and the growth process has been extensively characterized. Various physical properties of TGS solution have been measured. Also, a unique technique of growing solution growth crystals by extracting heat at a programmed rate from the crystal through a semi-insulating sting has been developed and tested in 1-g environment. TGS crystals will be grown by this technique during the Spacelab 3 mission. Data on heat and mass transport in a diffusion-controlled system will be obtained using a laser holography technique. The experiment will be monitored in space as well as on ground by a real time video schlieren system. Analytical techniques are underway to analyze data from holograms. Also, various techniques (optical, electrical, x-ray topography) of characterization of crystals are being tested.

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Growth of Solid Solution Crystals

Marshall Space Flight Center
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Dr. F. R. Szofran
Dr. L. R. Holland, Wyle Labs
In-House NASA Program
October 1977 - continuing task

The major objective of this program is to determine the conditions under which single crystals of solid solutions can be grown from the melt in a Bridgman configuration with a high degree of chemical homogeneity. The central aim is to assess the role of gravity in the growth process and to explore the possible advantages for growth in the absence of gravity. The alloy system being investigated is the solid solution semiconductor $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$ with x -values appropriate for infrared detector applications in the 8 to 14 μm wavelength region. Both melt and Te-solvent growth are being considered. The study consists of an extensive ground-based experimental and theoretical research effort followed by flight experimentation where appropriate.

Experimental facilities have been established for the purification, casting, and crystal growth of the alloy system. Facilities have been also established for the metallurgical, compositional, electrical and optical characterization of the alloys. Crystals are being grown by the Bridgman-Stockbarger method and are analyzed by various experimental techniques to evaluate the effects of growth conditions on the longitudinal and radial compositional variations and defect densities in the crystals.

Theoretical models have been developed for the temperature distribution, and the axial and radial compositional redistribution during the directional solidification of solid solution alloys. The one-dimensional model that treats the variation of the interface temperature, and the interface segregation coefficient and velocity with composition, has been used to establish effective diffusion constants for the alloy system. The temperature and compositional dependence of the liquid and solid phase thermal diffusivities have been measured for the composition range from $x=0$ to $x=0.3$ and are being used in conjunction with the thermal models to predict temperature distributions for various solidification conditions. Phase equilibrium parameters, growth rates and thermal conditions required to obtain various growth interface shapes have been established.

Other tasks currently under study include: (1) theoretical modeling of the effects of growth rate on the shape of the solidification isotherms and radial solute redistribution; (2) design and development of flight cartridges; (3) investigation of correlations between growth parameters and crystal defects; and (4) experimental and theoretical evaluation of the effect of processing conditions on the electrical and optical properties of the alloy crystals.

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HgI₂ Crystal Growth for Nuclear Detectors

EG&G, Inc.

W. F. Schnepple

Dr. L. Van den Berg

H-73830B (NASA Contact: W.W. Moore, MSFC)

April 19, 1984 - October 31, 1985

The objectives of this program are to obtain a benchmark quality sample grown at low-g conditions and to study vapor growth phenomena under space conditions.

Ground-based crystals show a defect structure which impairs their performance as nuclear radiation detectors. These defects may be caused by the gravitational force acting on the crystal in its weakened state at the elevated growth temperature and by irregular convection patterns in the vapor during growth.

The program will be supported by ground-based research aimed at a more detailed understanding and description of the problems associated with the crystal growth process. Mechanical strength measurements have been performed (uniaxial compression tests) which show that the crystals exhibit slip parallel to the c-planes at stresses as low as 1/2 psi. Preliminary calculations using a simple linearized model indicate the oscillating instabilities in the convection part of the vapor transport system are unlikely, even at 1-g, provided that the utmost care is taken in the preparation of the crystal growth source material.

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Float Zone Experiments in Space

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H-34328B
October 1982 - June 1984

The objective of this work has been to evaluate whether or not Marangoni flow could be suppressed in molten metals by the presence of very thin oxide films. Experimental work has been carried out on molten Sn under UHV conditions. A disk floating zone arrangement was developed to allow in situ Auger examination of molten surfaces. An electron energy loss technique was developed which allows detection of continuous tin oxide films of 6 Å or greater. Experiments were planned to detect the effects of oxide formation upon Marangoni flow by measuring (a) temperature profiles, (b) solid liquid interface shapes, (c) macrosegregation, and also to detect (d) the onset of oscillatory Marangoni flow by detecting oscillating temperature variations. The project was terminated before a, b and c were completed. Work on (d) showed that oscillatory temperature variations of frequency $< \sim 10$ Hz were not present in the disk float zone geometry under conditions of $Ma = 4300$ with an oxide free molten surface. The disk float zone geometry was modeled with a finite element analysis and temperature and velocity profiles were determined. Theoretical studies have utilized simplified nonlinear models which indicate that multiple steady state flows exist in some configurations in certain parametric intervals; and system stability is closely related to geometry. In particular, the disk geometry was found to be the most stable.

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Fluid Dynamics and Thermodynamics of Vapor Phase Crystal Growth

Renssalaer Polytechnic Institute

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NAS8-33562 (NASA Contact: D. Schaefer, MSFC)

February 1978 - March 1985

Vapor transport processes are used for the synthesis of bulk and layer-type single crystalline materials for solid state devices. The chemical and structural microhomogeneity of crystals is affected by the mass and heat transfer conditions and by the condensation mechanism. Thus, the inherent properties and performance characteristics of single crystals are essentially established during growth. A detailed understanding of the effects of transport mode and condensation processes on crystal perfection is both of fundamental and practical value.

The ground-based research effort under this program is concerned with systematic studies of the effects of variations (1) of the relative importance of buoyancy-driven convection, and (2) of diffusion and viscosity conditions on crystal properties. These experimental studies are supported by thermodynamic characterizations of the systems, based on which fluid dynamic parameters can be determined. The specific materials under investigation include the GeSe-GeI₄, Ge-GeI₄, HgTe-HgI₂, and Hg_{1-x}Cd_xTe-HgI₂ systems. Mass transport rate studies of the GeSe-GeI system as a function of orientation of the density gradient relative to the gravity vector demonstrated the validity of flux anomalies observed in earlier space experiments. The investigation of the effects of inert gases on mass flux yielded the first experimental evidence for the existence of a boundary layer in closed ampoules. Combined with a thorough thermodynamic analysis, a transport model for diffusive flow including chemical vapor transport, sublimation, and Stefan flow was developed. Similar studies are in progress for the epitaxial growth of Ge using GeI₄ as transport agent. The present task emphasizes the investigation of the effects of ampoule geometry, transport agent pressure, and temperature on the growth rate and morphology of epitaxial layers under horizontal and vertical, stabilizing conditions. In conjunction with simultaneous mass transport rate studies of the bulk growth of the Ge-GeI₄ system, the dominant transport mode and the Stefan flow contribution can be determined and correlated with the morphological characterization of the epilayer growth studies. This system exhibits a significant Stefan flow contribution to the total mass flux.

The materials HgTe and Hg_{1-x}Cd_xTe are characterized by wide single phase stoichiometry ranges which are based on the vacancy formation at Hg sites. Thus, the actual vapor pressure of these compounds is a function of temperature, of composition, and of the sample volume-to-free volume ratio. Systematic vapor pressure measurements of HgTe and Hg_{1-x}Cd_xTe as a function of the above parameters are in progress employing above parameters are in progress employing a dynamic vacuum microbalance technique developed for the studies. The major goals of this task are the derivation of a quantitative relationship between the above parameters, the

determination of precise vapor pressures, and of fluid dynamic parameters under actual transport conditions. The results of these studies are of immediate relevance for mass transport and crystal growth studies of these materials performed under a separate program.

The primary objectives of this program are to provide basic data which, combined with a thorough knowledge of the thermodynamics, will improve the fluid dynamic characterization of vapor transport systems. In addition, this information is required for the conclusive interpretation of on-going experiments in microgravity environment, and for the design of meaningful, future flight experiments.

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Vapor Growth of Alloy-Type Semiconductor Crystals

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NAS8-32936 (NASA Contact: D. Schaefer, MSFC)

March 1978 - September 1986

The present effort is part of a continuing research program directed towards the investigation of basic vapor transport phenomena and of crystal growth properties of electronic materials. The primary purpose of ground-based studies is the development and definition of optimum experimental parameters for flight experiments. The ground-based effort includes the investigation of gravity-driven convection effects on mass transport rates and on crystal morphology for different orientations of the density gradient with respect to the gravity vector, and as a function of pressure and of temperature. In addition to the experimental tasks, theoretical efforts involve the quantitative thermodynamic analysis of the systems under investigation, the computation of fluid dynamic parameters, and the consideration of other possible effects on fluid flow under vertical, stabilizing and microgravity conditions.

The specific experiments to be performed in a microgravity environment include the investigation of vapor transport and crystal growth phenomena of the GeSe-Xenon system and of the mass flux and growth of bulk and layer-type crystals of $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$ using HgI as a transport agent. Both types of experiments are performed in closed, fused silica ampoules.

The primary objectives of the GeSe-Xenon experiments are the determination of absolute mass transport rates under microgravity conditions and their comparison with corresponding ground-based data, with theoretically predicted values, and with flux data of earlier space experiments. With a total of four appropriately designed GeSe-Xenon experiments, a mass flux versus pressure curve in microgravity is to be developed. Another important aspect of these experiments is the investigation of the deposition pattern and crystal morphology of GeSe crystals grown by sublimation under different xenon pressures in microgravity.

The objectives of the $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$ experiments are to determine the positive effects of microgravity on crystal growth of ternary, alloy-type materials in terms of chemical and structural microhomogeneity. Gravity-driven convection effects on mass flux and morphology of bulk crystals have been observed under ground-based conditions. Continued experimental efforts are directed towards the optimization of temperature conditions for the bulk growth of $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$ crystals in microgravity environment.

The major tasks of ground-based studies of the seeded growth of $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$ layers by chemical vapor transport reactions involved systematic investigations of the growth rate, composition, homogeneity, and morphology of HgCdTe layers. These studies include measurements of the effects of substrate orientation relative to the density gradient, of temperature, and of transport agent pressure on the above properties. They are performed under horizontal and vertical, stabilizing conditions with the initial goal to observe the effects of convective interferences on layer morphology and homogeneity, in addition to developing optimum conditions for flight experiments of this system.

The results of the combined experiments are of basic scientific value and of technological significance. It is expected that these experiments will contribute to our understanding of vapor transport and crystal growth processes of binary and ternary materials on earth and in space and to establish conditions for space processing applications.

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Study of Eutectic Formation

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NAS8-34887 (NASA Contact: F. Reeves, MSFC)
May 1982 - August 1984

The objectives of this program are to determine the influence of convection on the microstructure of eutectics and to develop a technique for revealing the longitudinal microstructure of the MnBi-Bi eutectic. Both objectives aim at trying to explain the observed influence of space processing on the microstructure of MnBi-Bi.

A computer program was developed and used to determine the concentration field in front of a growing lamellar eutectic. From this the deviation of the interfacial concentration from the eutectic composition was calculated as a function of eutectic composition, freezing rate, convection, and lamellar spacing. A Hunt-Jackson type treatment yielded the following result:

$$\lambda/\lambda_0 = 1 + 0.00034 \frac{G_u^2}{\Gamma_0^2 D}$$

where λ is the lamellar spacing with convection, Γ_0 is the value without convection, $\Gamma_0 = G_u \lambda_0^2 / D$ is the interfacial velocity gradient and D is the diffusion in the melt. Comparison with realistic experiment values for G_u , λ_0 and D revealed that convection can have a large influence on λ , but probably not under the gentle convection typical of vertical Bridgman-Stockbarger growth in a small ampoule.

Experiments were performed on directional solidification of MnBi-Bi eutectic with spin-up/spin-down (accelerated crucible rotation technique; ACRT). Freezing rates ranged from 1.5 to 60 mm/hr and the rotation rates were 100 or 200 RPM. At low freezing rates spin-up/spin-down changed the MnBi microstructure dramatically, from blades to large irregular dendritic structures. The thickness of the MnBi particles was increased by an order of magnitude. Often no MnBi was present along the center of the ingot. At high freezing rates the chevron fibrous microstructure was maintained, but the thickness of the fibers was increased by about 3X, with the largest effect being along the center of the ingot.

Publications

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Modeling Directional Solidification

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May 1982 - May 1985

The objective of the research is to develop tools of use in explaining the results of directional solidification experiments in space. These tools consist of mathematical and experimental models. Experiments are also being flown at low g to validate the ground-based results.

The technologically important materials selected for solidification in space are high melting and opaque. Consequently one is forced to infer the conditions during growth responsible for the observed microstructure, morphology, inhomogeneities, etc. These inferences can be considerably improved with the aid of appropriate mathematical models and low melting transparent analogs.

Many metals and semiconductors directionally solidified in space had a diameter less than that of the containing ampoule. In fact this occurred whenever the ampoule contained a gas space. This phenomenon has been attributed in a vague way to lack of wetting of the ampoule wall by the melt. The actual liquid behavior by use of low temperature analogs is being observed aboard the KC-135. In the first flight, water was placed in plastic ampoules (not wet) with ceramic end plugs which were wet by the water to simulate a solid-liquid interface. Unfortunately during storage prior to the flight the water began to wet the ampoule wall. Discrete bubbles formed down the axis of the ampoules. New ampoule materials are being used now, with testing for stability prior to the next flight. A theoretical model has also been developed for this phenomenon.

The cause of twinning during crystal growth is one of the last remaining mysteries in the theory of crystal growth. It is especially troublesome in some technologically important materials, such as CdTe, III-V alloys and II-VI alloys. An organic analog, dodecanedioic acid, has been found that exhibits clear twins in crossed polarized light during solidification. Thus far, the freezing rate has no influence on the twin density. Most foreign particles have no effect, but a few reduce twin formation. Preliminary results suggest that mechanical stress may cause the twins to multiply. Additional studies are planned on the influence of mechanical stress, temperature gradient, and soluble impurities.

Naphthalene was selected to study heat transfer and fluid flow in a transparent model of Bridgman-Stockbarger apparatus. In spite of all precautions the natural convection in the melt was asymmetric. It was also gentle and laminar, but somewhat irregular. In spite of the asymmetry in the convection, the interface shape was almost symmetric. The vertical temperature gradients at the interface increased more than linearly with increasing difference in temperature between the heater and the cooler.

Analytical expressions were developed for heat conduction in a Bridgman-Stockbarger apparatus of finite length with different thermal conductivity in the solid and the melt. A finite element program is being developed that takes into account convection for high Prandtl number melts. (Note that all prior work was done in low Prandtl number melts, for which convective heat transfer is not important).

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Heat Flow and Segregation in Directional Solidification

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This research is composed of three major components: (1) development of interface morphology control for automated Bridgman growth of semiconductor systems. In attempts to optimize furnace design for crystal growth in a reduced gravity environment, an analytical approach to heat transfer was developed. It was thus found that charge confining crucibles diminish the ability to control the growth interface morphology through its position within the gradient zone. A heat pipe hot zone system for Bridgman growth, in reduced gravity environment, of crystal with diameters up to 16 mm was developed. Efforts are continuing to maximize the controllability of the growth interface morphology; (2) comparative analysis of segregation during crystal growth in a reduced gravity environment and in the presence of magnetic fields. For growth of Ga-doped germanium in the multipurpose (ASTP) furnace, it was found that the application of transverse magnetic fields (up to 36 kG) does not substantially increase the effective distribution coefficient; i.e., diffusion-controlled segregation observed in reduced gravity environment cannot be reached nor approached by magnetic field induced melt stabilization; and (3) consequences of seeding by melt-back in Bridgman growth under reduced gravity conditions. Experimental and theoretical studies established that steady state conditions cannot be reached during seeding, and that the growth interface temperature (interface location) during the initial stages of growth is a function of melt-back conditions. Numerical calculations were carried out for Ga-doped Ge and for HgCdTe.

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METALS, ALLOYS, AND COMPOSITES

Effect of Undercooling in a Low-Gravity Containerless Environment on the Structure and Properties of Alloys

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The 30 meter and 100 meter drop tubes at the Marshall Space Flight Center offer a unique opportunity to study solidification in a containerless, microgravity environment. Samples are melted in a furnace at the top of the drop tube and solidify during a 4.5 second free fall in the 100 meter drop tube or a 2.6 second free fall in the 30 meter drop tube. Since the sample solidifies during free fall, a container is not necessary. This eliminates the possibility of the nucleation and growth of the solid phase on a container surface and enhances the probability of deep undercooling.

The cooling rate obtained in the drop tubes is lower than cooling rates in atomization and splat-quenching processes. This allows observation of solidification at large undercooling in the absence of a fast quench. Decoupling the rapid solidification process allows the study of the effects of undercooling and recalescence on alloy morphologies. The research therefore has a three-fold purpose. The first is examination of the limits of undercooling of niobium and niobium-base alloys in the drop tube. The mechanisms of homogeneous and surface or impurity catalyzed heterogeneous nucleation are considered. The second purpose is the observation of microstructures resulting from the solidification of samples at large undercoolings and moderate cooling rates. The kinetics of the solidification front and the effects of recalescence are important in this regard. The third purpose is the observation of metastable phase formation in highly undercooled samples. Metastability in a number of niobium-base alloys is of interest but there is specific interest in the possibility of the formation of a bulk high-temperature superconducting metastable phase in the niobium-germanium system.

Approximately sixty specimens of niobium and niobium-germanium alloys have been undercooled. The pure niobium alloys have undercooled $0.19 T_m$ (T_m = absolute equilibrium melting temperature) in agreement with previous work on homogeneous nucleation in pure materials. The majority of niobium-germanium alloys undercooled less than $0.17 T_m$ but, in three cases, undercooling of $0.23 T_m$ was observed.

The microstructures in all cases show the effect of recalescence. Formation of metastable phases, particularly the potential high-temperature superconducting phase, appears to form at the large undercoolings. However, recalescence largely causes transformation to equilibrium although supersaturated phases remain in small quantities.

Some work is continuing on germanium alloys but this binary system has now been well-documented. Attention is presently being given to the niobium-silicon, niobium-tin, and niobium-aluminum systems.

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Measurement of High Temperature Thermophysical Properties

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This task has, as a primary aim, an ongoing evaluation of the experimental procedures needed in the collaborative interaction between Rice University (RICE) and the General Electric Company's Space Systems Division (GE). This effort focuses on technique development and the application of electromagnetic levitation to thermophysical property measurement, including determining heats of fusion, heat capacities, and enthalpy increment functions for extremely refractory materials. Initial emphasis has been on properties of tungsten due to its importance as the highest melting element, and the GE development of an apparatus employing electromagnetic levitation and electron beam heating capable of melting levitating tungsten. RICE has wide experience in high temperature heat content measurements using drop calorimetry. The NBS involvement provides the background expertise in coupling the techniques, calibrating the pyrometric temperature scale, and in ongoing development of automation as prototypes for potential flight experiments.

Experimental studies are needed as high temperature heats of fusion, enthalpy functions and heat capacities (C_p) are essentially all estimated for third row transition metals, and largely unknown for most other refractories with melting points above 200 K. Theory has no predictive capability for these data, particularly at phase transitions, e.g. the change in C_p , solid to liquid, or the value of C_p near the melting point. The Tamman rule, $\Delta H_{\text{fus}}/T = \Delta S_{\text{fus}} \approx 2.3 \text{ cal/mol K}$ provides worse than factor-of-two estimates. There is not even a satisfactory explanation for why $C_p(l)$ is apparently constant for refractory metals and generally much larger than the equipartition limit of $3R$ ($R = 8.314 \text{ J/g.mol}$). Accurate data should provide clues to improve parameters in existing models, or encourage the development of new models. As an example, current models for predicting alloy properties generally require good thermodynamic data of the constituents to obtain even approximate estimates.

Current efforts are concentrating on developing a prototype micro-computer data acquisition system suitable for controlling the ground based system, with consequent improvement in system handling, which can serve as a model for the system needed for flight experiments. Previous and ongoing major efforts include the development of techniques for imaging pyrometry at temperatures above 3500 K, a semiautomatic calorimeter system capable of handling liquid tungsten, and development of a two color imaging pyrometer system. This activity has recently resulted in the first reported direct measurements of the heat of fusion of liquid tungsten, $53.0 \pm 2.3 \text{ kJ/mol}$ at 3695 K. This task and the collaborators' efforts represent the current limits of ground based research, and probe past that limit to experimentation requiring the microgravity of space for success.

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Effect of Undercooling and Modified Microstructure on the Physical Properties of Material Synthesized in the Drop Tube and Drop Tower

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December 16, 1982 - December 15, 1984

The objectives of this research program are (1) to determine the possible existence of metastable phases and novel physical processes suggested by previous experiments on material prepared in a microgravity environment, and (2) to investigate the feasibility of fabricating advanced electronic devices using material with microstructures obtainable in a microgravity environment. The specific alloys and compounds under current study are Au-Ge and immiscible alloys, Nb-Ge compounds, and Ga-Bi immiscible alloys, and Ba-Pb-Bi-O.

In collaboration with Vanderbilt University for microstructure determination, we have been carrying out the investigation by determining the superconducting and normal state transport properties of the materials under different pressures and in different magnetic fields. The simultaneous study on the physical properties and the microstructure of material will provide crucial information about the potential applications of materials processed in a microgravity environment.

For the needs of the study, an ultrasensitive ac magnetic susceptibility apparatus has been developed. It provides a resolution of 10 ppm of a superconducting signal for a sample with a 1 mm^2 cross-section and a capability of separating the 0° phase from the 90° phase of the signal to 5%.

The previous report of superconductivity below 1.5K in the immiscible Au-Ge alloys made during the Skylab Experiment M557 has led to the suggestion of the possible existence of a novel superconducting mechanism. Unfortunately, the present study failed to detect any sign of superconductivity down to 1.15K in the same set of samples. By examining the splat-quenched Au-Ge alloys which possess some of the microstructures in the Skylab samples, we found that there exist at least two metastable superconductive phases in Au-Ge alloys; one with a transition temperature (T_c) of 2.4K and the other 1.6K. The high T_c phase is particularly unstable at 300K. Efforts are being made to identify the superconducting phases by synchrotron radiation, x-ray, SEM, magnetic, and electrical measurements.

In the Nb-Ge samples prepared with the drop tube at MSFC by Bayuzick's group, we have found that an undercooling of $\sim 250^\circ\text{C}$ results in only a T_c improvement of $\sim 1^\circ\text{K}$ for three of the more than 30 samples examined, compared with a T_c of 6°K for the equilibrium Nb-Ge Al5 phase. Comparison is also made of the above samples with the splat-quenched Nb-Ge Al5 phase, which shows a broad superconductivity transition from 6 - 16°k . Microstructure analysis is being made by Bayuzick's group.

The distinct microstructures in an immiscible alloy, prepared in a microgravity environment has raised the prospect of mass-producing advanced electronic devices on an economically competitive basis. As a first step, we have chosen to examine if such a microstructure, i.e., fine dispersion of one constituent in the matrix of another, can indeed be tailored for Josephson devices. We have investigated the heterogeneous superconducting Ba-Pb-Bi-O system by proper and careful heat-treatment to generate the proper microstructure. Indeed, an array of more than 400 Josephson junctions has been obtained naturally, as evidenced by the detection of both the Josephson and Giaever tunnelings. This demonstrates for the first time that immiscible alloys processed in a microgravity environment can provide an economical avenue to the simple fabrication of reliable Josephson-function devices, not just for fast and large computers, nut also for ultrasensitive electromagnetic radiation detectors, not to mention the spin-off for pure scientific pursuit. A binary immiscible alloy system with proper microstrctures obtainable in a microgravity environment is devoid of the complications of many elements and many oxidation states of the Ba-Pb-Bi-O and thus makes possible the easy fabrication of devices. The unusual elevtron scattering in the immiscible Ga-Bi alloys will be examined.

Publications

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The Influence of Containerless Undercooling and Rapid Solid-State Quenching on the Superconductive and Magnetic Properties of Some Clustering Alloy Systems

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The purpose of the research is to investigate the potential of the new 100-meter drop-tube at the George C. Marshall Space Flight Center, for producing materials with new and important properties by virtue of their being rapidly solidified by containerless undercooling. The results which are beginning to emerge from this preliminary low-level-of-effort study are positive in that materials produced so far exhibit evidence of very rapid quenching. The importance of rapid quenching in producing materials of considerable scientific and technological interest has been emphasized in numerous recent publications and international conferences. Many techniques are available for obtaining fast cooling rates -- typically in the vicinity of 10^5 - 10^6 K sec⁻¹; the results of this study are indicating that containerless undercooling, using at first the NASA drop tube and eventually a space-shuttle-serviced orbiting laboratory, will provide the scientific community with another powerful approach for rapid quenching. A special advantage of the drop tube is that the micro-gravity condition is imposed ballistically; it is therefore possible to obtain either spherical samples by allowing solidification to take place in flight (i.e. while the sample is falling), or "splats" by allowing the sample material while still in undercooled liquid form to impact a stationary quench plate. A second important advantage of the drop tube is that it can be used in relatively low cost screening studies prior to in-flight experimentation.

Materials investigated so far in the present program are four representative austenitic (fcc-structured, "nonmagnetic") stainless steels -- AISI 310S, AISI 316, Nitronic 40, and Nitronic 40W. A study of the microstructural and magnetic properties of these materials after processing by various techniques, including containerless undercooling, provides an excellent qualitative indication of the quench-rate potential of the drop tube. Two series of experiments were undertaken:

(1) Control samples were prepared in annealed form and their properties studied. Although the data were collected solely for reference purposes, a study of the magnetic properties of the annealed stainless steels provided some useful and interesting insights into their micro-magnetic character.

(2) Samples were prepared by hammer-and-anvil splat quenching and by drop-tube undercooling and the resulting magnetic and microstructural properties were compared. In annealed form, the alloys selected have the fcc structure; but when rapidly solidified it is possible for them to retain some of the initially formed, characteristically ferromagnetic, bcc phase (frequently referred to as "delta-ferrite"). In a given alloy, the fraction of retained bcc phase can be used as a measure (qualitative at

present) of the rapidity of quench. Calculations based on alloy composition of the expected levels of bcc phase retained in the alloys after rapid cooling, as in weld-deposit cooling or chill casting, indicate that only a few percent of bcc should be present. After very rapid quenching, as in hammer-and-anvil melt quenching, however, the Nitronic 40 alloy for example yielded some 48% bcc phase as determined by X-ray diffractometry. The quench rate in this case was estimated to be some 10^6 K sec⁻¹. On the other hand, after drop-tube processing the same alloy according to magnetic measurements was found to retain some 86% of the bcc phase. It seems, therefore, that drop-tube processing has the potential for much higher quench rates than conventional splat quenching. Furthermore, the product can be obtained in either spherical droplet or splat form. The results of this work was presented (and subsequent publication) in a paper entitled "Magnetic and Structural Studies of Rapidly Quenched Austenitic Stainless Steel Alloys" at the above-mentioned International Cryogenic Materials Conference.

Further research will be aimed at studying other materials and quantifying the quench rate.

Presentations

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Convection During Unidirectional Solidification

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The general aim of this task is the theoretical and experimental study of the fluid flow and solute segregation which occur during directional solidification, including effects of gravity and microgravity. The nature of the fluid flow, its effects on the shape of the crystal-melt interface and the resulting distribution of solutes are examined.

During solidification of a binary alloy at constant velocity vertically upwards, thermosolutal convection can occur if the solute rejection or preferential incorporation at the crystal-melt interface decreases the density of the melt. Numerical calculations of the solute, temperature, and flow fields are being carried out using previously developed algorithms for Schmidt numbers $Sc = 1, 10, 81$. The time periodic flow, which occurs over a narrow range of parameters, is being further investigated.

Convective flow is measured during the unidirectional upward solidification of a transparent materials (succinonitrile) with or without a small concentration of solute (ethanol). Under a narrow range of conditions close to those which produce morphological instability, it is found that there can be a strong interaction between the convective flow and the interface shape in the samples containing solute. Changes in the convective flow pattern, induced by activating auxiliary heaters, are found to change the convection-induced interface shape distortions.

The general problem of the interaction of low fields with crystal-melt interfaces is investigated by linear stability calculations for the case of a flow parallel to a crystal-melt interface. This research, in collaboration with R. F. Sekerka of Carnegie-Mellon University and M. E. Glicksman and colleagues at Rensselaer Polytechnic Institute demonstrates a coupling between hydrodynamic and morphological instabilities. The calculations are in general agreement with experiments carried out by Glicksman and colleagues on succinonitrile.

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Signal Analysis of Gas Tungsten Arc Welds

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Gas tungsten arc welding is a process in which the input parameters such as current, voltage, travel speed, etc. can be easily controlled and/or monitored. However, due to random fluctuations in the welding environment, weld quality is not solely a function of these parameters. In order to improve weld quality assurance, an adaptive method of observing weld quality is desired. For this project, research is being performed to allow the use of dynamic electrical properties of the welding arc as a weld quality monitor.

The electrical properties of the arc are characterized by the current-voltage transfer function. To date, the hardware and software necessary to collect the data at a maximum rate of 45 kHz and to allow the off-line processing of this data have been installed and tested. A study to determine the optimum input current waveform has been completed. Work is now preceeding with bead-on-plate welds to attempt to observe such characteristics of the weld as the fundamental frequency of the puddle. Future work is planned to observe changes of the arc response with changes in joint geometry, base metal chemistry, and shielding gas composition. It is hoped that through this work, enough can be learned about the dynamics of the welding arc to allow the development of a real-time fault-specific weld quality monitor.

Mephisto - Interfacial Destabilization in Metal Alloys

Center for Nuclear Studies of Grenoble

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Dr. Y. Malmejac

The objective of this investigation is to study the destabilizing mechanisms at a solidification interface in order to obtain information on the kinetics and morphologies in the transient and steady state, and to separate the influences of liquid phase instabilities from interfacial instabilities.

Conceptual design of the experiment is complete. Sn-0.01A%Bi to Sn-10A%Bi and Bi-0.01A% Sn to Bi-10%Sn will be directionally solidified at rates varying from 0.0001 to 0.1 cm/sec with thermal gradients varying from 10 to 500 k/cm. A differential seebeck voltage measurements technique has been developed to provide a continuous record of the solid-liquid interface temperature as the solidification rate is varied in order to determine the kinetic coefficients. Signal processing and noise suppression techniques have been demonstrated to allows nanovolt precision which corresponds to mK accuracy for the interfacial temperature.

Undercooling, Remelting, and Coarsening

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October 1, 1984 - September 30, 1985

Solidification of metal alloys with initial high undercoolings generally occurs with rapid recalescence due to rapid heat release of the latent heat of fusion. The recalescence temperature is usually higher than the solidus and/or the thermodynamic " T_0 " temperature of the alloy, depending on the initial undercooling and alloy composition. The rapid heating during recalescence alters the initially solidified structures, in ways that must include partial remelting, ripening, coarsening, and decomposition of the rapidly solidified primary metastable or supersaturated phases.

In this program, the aim is to develop new ways of studying the solidification and remelting phenomena associated with the rapid heating during recalescence of undercooled alloys. The plan is to study these phenomena using (1) emulsions of iron and nickel base alloys in viscous liquid carriers, and (2) rapid heating of metal samples by electric discharge, using a technique similar to the exploding wire process (EWP), except with less power input.

Experimental results in this fundamental program will be combined with theoretical analyses to better understand the solidification of levitated metal droplets with high undercooling, which relates to the solidification process of the planned MIT-EML Space Shuttle experiments, planned for summer 1985.

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Liquid Phase Miscibility Gap Materials

S. H. Gelles Associates

Dr. S. H. Gelles

Dr. A. J. Markworth, Battelle Columbus Laboratories

NAS8-32952 (NASA Contact: D. Schaefer, MSFC)

April 1978 - December 1984

The overall objective of the investigation is to determine the manner in which the microstructural features of liquid-phase miscibility gap alloys develop. The results of such a determination should make it possible to control the microstructures and the resultant properties of these alloys. The long-duration low gravity afforded by the Shuttle will allow experiments supporting this research to be conducted with minimal interference from buoyancy effects and gravitationally driven convection currents.

Ground base studies have been conducted on Al-In, Cu-Pb, and Te-Tl alloys to determine the effect of cooling rate, composition, and interfacial energies on the phase separation and solidification processes that influence the development of microstructure in these alloys. Both isothermal and directional cooling experiments and simulations are being conducted. The ground based activities have been used as a technological base from which flight experiments have been formulated and to which such flight experiments have been compared and judged.

Four flight experiments have been conducted aboard the Shuttle on STS-7 in the Materials Experiments Apparatus (MEA) during June of 1983. Two of these experiments involve aluminum-indium alloys which have been processed isothermally in a configuration that avoids the presence of a free melt surface and thus eliminates a major source of convection, namely that arising from gradients in surface tension along this free surface. A third experiment, also involving an aluminum-indium alloy, is concerned with thermocapillary induced droplet migration. In this experiment, the alloy after homogenization has been cooled to ambient under a controlled temperature gradient. The fourth experiment deals with the melting and solidification of two tellurium-thallium liquid phase miscibility gap alloys. This last experiment is aimed at understanding the influence of interfacial energy and droplet concentration on the phase separation process.

The MEA/Al experiment described above have for the most part been carried out successfully and much valuable information and insight has been obtained. For example, from the results of the isothermal experiments on the aluminum-indium alloys, it can be concluded that the convective flows arising from a free surface make a small contribution to the coalescence process relative to other processes which may be occurring during phase separation. The gradient cool experiment has also produced noteworthy results in that the aluminum-rich droplets produced during the phase separation moved to the cooler end of the sample rather than the hot end as predicted by theory.

Future effort on this program will be devoted to further analysis of the low-g experiments conducted on MEA/A1 and in conducting further ground-based and low-g experiments in liquid phase miscibility gap materials. One such low-g experiment will be conducted on MEA/A2 scheduled for October, 1984.

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Dendritic Growth in Undercooled Melts

Rensselaer Polytechnic Institute
Dr. Martin E. Glicksman

The objective of this investigation is to obtain information relating to the kinetic and morphological behavior of systems solidifying at small undercooling with emphasis on the role of convective and diffusive transport and the influence of gravity.

A large data base has been established for pure succinonitrile which permits a comprehensive check on diffusional dendrite growth theory and the development of "scaling laws" to extend the theory to other material systems. A departure from diffusional-controlled growth has been observed which becomes more significant at smaller undercoolings. A Shuttle experiment is being prepared to test the theory at these low undercoolings where convective effects begin to dominate.

Influence of Melt Convection on Solid-Liquid Interface Under Terrestrial and Reduced Gravity Environments

Rensselaer Polytechnic Institute
Dr. Martin E. Glicksman
NAG3-333 (NASA Contact: Dr. Hugh Gray, LeRC)
October 1983 - October 1985

Solidification and crystal growth processes invariably involve thermal and solutal gradients within a molten phase. In the presence of gravity, such gradients result in convective flows which interact with diffusion fields at the solid-liquid interface. Dendritic growth kinetics has been studied carefully in several transparent model systems which freeze similarly to most metals. Succinonitrile shows a strong influence of convection at supercoolings below about 1K. Conceptual designs are being evolved to ascertain a practical method for carrying out autonomous kinetic and morphological studies in low earth orbit aboard the Space Shuttle. The major challenges now being addressed include: design of a precision thermostatted bath capable of controlling specimen temperatures to $\pm 10^{-3}$ K in orbit; design of a rugged specimen chamber able to produce oriented dendritic growth over a range of thermal supercoolings and permitting optical viewing of the growing crystals for data acquisition; development of an illumination system and photographic method for acquiring high resolution photographs of in situ dendritic growth; design of a microprocessor controlled system capable of executing an autonomous sequence of experiments for a seven day orbital mission. In addition to the dendritic growth tasks, work is also proceeding on basic studies of fluid flows adjacent to solid-liquid interfaces. The behavior of shear flows in vertical annular geometries is under intensive experimental study in a cooperative program with theorists at the National Bureau of Standards. Novel low-frequency eigenstates have been discovered and classified as "coupled modes," inasmuch as they involve interfacial deformation which couple to the fluid flow, and are unknown in systems without deformable interfaces. The dependence of coupled convection modes on interfacial geometry, gravity, fluid properties, and transformation characteristics are now under study for several annular flow arrangements with nominally pure solid-liquid systems.

Publications

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Control of Cast Iron Microstructure

John Deere and Company
Dr. James Graham
Dr. Norman Lillybeck
Dr. Nick Franco, Bethlehem Steel
Dr. Doru Stefanescu, University of Alabama
Joint Endeavor Agreement

The objective of this investigation is to explore the use of microgravity for industrial research in the processing of cast iron.

Following a technical exchange agreement (TEA) with NASA in which a number of solidification experiments were conducted using the KC-135 and F-104 aircraft, an experiment plan has been developed for follow-on experiments using the Shuttle. Three areas of interest have been identified: (1) measurement of thermophysical properties in the melt; (2) understanding of the relative roles of homogeneous nucleation, grain multiplication, and inoculants in forming the microstructure; and (3) exploring the possibility of obtaining an aligned graphite structure in hypereutectic Fe, Ni, and Co. A proposal has been developed and is in the process of being submitted to NASA as a JEA.

The Role of Entrained Surface Oxides in RS-PM Aluminum Alloys on Resultant Structures and Properties

Massachusetts Institute of Technology
Dr. N. J. Grant
NSG-7645

In spite of outstandingly excellent smooth bar test results (tensile testing and fatigue testing), RS-PM aluminum alloys show less than anticipated toughness properties. After eliminating negative variables such as sodium and potassium in lithium containing alloys, hydrogen in all Al alloys, and trapped impurities from the atomization processing the data clearly pointed to fine oxides, usually in stringer form in the hot extruded powder based alloy, as the primary cause of poor toughness properties. The oxide content of aluminum powders increases with: decreasing powder size, deviations from spherical powder shapes, exposure to moist atmospheres either during atomization or in subsequent powder handling (hydrate formation), and alloy compositions which contain significant amounts of lithium, magnesium, cerium, and other reactive elements.

Publications

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The Development and Prevention of Channel Segregation During Alloy Solidification

Michigan Technological University
Dr. Angus Hellawell
NAG3-560 (NASA Contact: Dr. Hugh R. Gray, LeRC)
June 5, 1984 - June 4, 1985

During alloy solidification, interdendritic microsegregation causes density differences between entrapped liquid and bulk liquid which, subject to the direction of heat flow, provides a driving force for convection. One of the consequences of this effect is the formation of long, nearly vertical liquid channels, rich in solute, flowing through the solid-liquid mushy zone somewhat as a river and tributary system. In ingot castings where the heat flow is vertically downwards and the solute less dense than the solvent the convective flow is anti-parallel with the flow caused by contraction on freezing. It is not presently known exactly how and where these segregation channels originate, nor what controls their subsequent spacings and diameters. The object of this project is to identify the mechanisms involved in these two problems. In addition, it is known that certain types of bulk liquid movements can inhibit channel development and the mechanism by which they do so will be examined.

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Containerless Solidification of NiAl

Dupont Corporation
Dr. Michael Kelley
Dr. Edwin Ethridge, MSFC
Technical Exchange Agreement

The objective of this investigation is to achieve solidification of 100% peritectic NiAl₃ phase by undercooling through the L+S region using the MSFC Drop Tube.

The affinity of Al for O₂ results in the formation of oxides which apparently prevented significant undercooling. However, a very unique morphology was found in which the droplets solidified with highly convoluted surfaces and interior porosities. Bulk densities as low as 20% of the normal density were observed for the smallest droplets.

Study of the Reactive-Element Effect in Oxidation of Fe-Cr Alloys Using Transverse Section Analytical Electron Microscopy

Argonne National Laboratory
Dr. Wayne E. King
Dr. Edwin Ethridge, MSFC
Interagency Agreement (DOE)

New insight into the role of trace (<1%) additions of reactive elements like Y, Ce, Th, or Hf to Cr bearing alloys has been gained by applying a new developed technique of transverse section analytical electron microscopy. This so-called reactive-element effect is known to improve the high temperature oxidation resistance of alloys by strongly reducing the high temperature oxidation rate and enhancing the adhesion of the oxide scale: however, the mechanisms for this important effect remain largely unknown. Our results indicate that the presence of yttrium affects the oxidation of Fe-Cr-Y alloys in at least two ways. The reactive element alters the growth mechanism of the oxide scale as evidenced by the marked influence of the reactive element on the oxide scale microstructure. The present results also suggest that reactive-element intermetallic compounds, which internally oxidize in the metal during oxidation, act as sinks for excess vacancies thus inhibiting vacancy condensation at the scale-metal interface and possibly enhancing scale adhesion.

One of the problems is to determine if the reactive-element effect is due to elemental yttrium or the Y_2O_3 particles that form during oxidation of the alloy. In order to investigate this, it is essential to prepare alloys with uniform reactive-element oxide dispersions. Melting and solidification of such alloys has not been successful due to the density difference between oxide particles and liquid metal. Alloys with reactive-element oxide dispersions have been prepared by mechanical alloying of powders. Unfortunately the grain structure of these materials is much different than that of melted and solidified metals thus making direct comparison of dispersion-free alloys quite different. Further it appears to be quite difficult to control the impurity content in alloys prepared by mechanical alloying due to contamination of the powders from the attriting media, impellers, and process control agents that are required for successful mechanical alloying. Therefore we have proposed to prepare melts of Fe-24Cr alloys with various Y_2O_3 dispersions by electromagnetic levitation melting in a microgravity environment.

These experiments are technologically significant because reactive element and reactive element oxide additions have been, and promise to be, very important in commercial high temperature alloys. It is expected that a fundamental understanding of the mechanisms of the reactive element effect will provide valuable input into optimization of alloys for high temperature applications.

Containerless Solidification of Fe and Ni Aluminides

North Carolina State University
Dr. Carl Koch

The objective of this investigation is to obtain a basic understanding of the complex solidification structures found in materials by combining rapid solidification studies in foils and ribbons with undercooling studies in the MSFC drop tube.

Chill block melt spinning apparatus and arc-hammer apparatus are being assembled at North Carolina State. Samples have been prepared for the MSFC drop tube to begin the undercooling portion of the effort.

Theoretical Problems in Materials Science

Institute for Theoretical Physics
Dr. J. S. Langer
Dr. M. E. Glicksman
NSFG PHY 77-27084

The objective of this investigation is to provide interactions between theoretical physicists and material scientists in order to identify problems of common interest in which some of the powerful theoretical approaches developed for other branches of physics may be applied to problems in materials science.

A unique structure has been identified in rapidly quenched Al-14% Mn by Shechtman, Blech, Gratias, and Cahn. The material has long-range directed bonds with icosahedral symmetry which does not form a regular structure but instead forms an amorphous-like quasi-periodic structure. Interactions between NBS and visiting scientists at the Institute were instrumental in determining this structure.

Interactions between Kawasaki, Glicksman, and Voorhees have advanced the theory of Ostwald ripening to account for finite volume fractions of second phase material. Progress is being made in coupling this with nucleation theory to describe the formation and structure of precipitating phases in alloys.

Work is progressing between Langer, Glicksman, and McFadden in applying the theory of pattern formation to the problem of dendrite formation.

Orbital Processing of Aligned Magnetic Composites

Grumman Aerospace Corporation

Dr. David J. Larson

NAS8-35483 (NASA Contact: F. Reeves, MSFC)

September 1983 - February 1986

A series of experiments will be conducted on the Space Shuttle in which aligned, two-phase magnetic composites will be grown by plane front directional solidification. The objectives of this program are to:

(1) identify and quantitatively evaluate the influences of gravitationally driven thermo-solutal convection on contained plane front solidification of binary, eutectic, off-eutectic, and peritectic magnetic composites; (2) to evaluate the effectiveness of micro-g processing as a means of damping convection; and (3) to evaluate the uniqueness of micro-g processing relative to the best means of terrestrial convection damping.

Three flight experiments are presently planned, each processing four independent samples. The first and third flights will consist of Bi-Mn eutectic and off-eutectic experiments, respectively. These experiments will be conducted in the Low Temperature Automated Directional Solidification Furnace (ADSF-I) which will be located in the mid-deck of the Space Shuttle. Co-Sm peritectic and eutectic experiments will be conducted on the second flight. These experiments will be conducted in the High Temperature Automated Directional Solidification Furnace (ADSF-II) on the MSL-2 Carrier in the payload bay of the Space Shuttle. The ADSF systems use the Bridgman-Stockbarger plane front directional solidification technique. This consists of translating a thermal gradient at a programmed velocity down the length of a stationary sample (directional) so that the solidification interface is flat (plane front solidification) and at a constant solidification velocity.

The relationships between the gravity vector, heat transfer, level of thermo-solutal convection and solidification processing parameters are being studied terrestrially by varying the orientation of the gravity vector during solidification processing and by employing in-situ thermal measurement and interface demarcation techniques. These experimental results are compared with existent models of: heat flow, eutectic solidification, and off-eutectic solidification. In addition, a model for the Bridgman-Stockbarger solidification technique including sample, ampoule, and translation, has been developed. These studies and comparative analyses and magnetically sampled and micro-g processed samples will identify the role of gravitationally driven convection in plane front solidification using the Bridgman-Stockbarger technique.

Relationships between solidification processing parameters (including gravity vector), microstructure, macrostructure, chemistry as a function of fraction solidification, crystal structures, and magnetic properties are also being developed. Microstructure and macrostructure are being quantitatively analyzed using quantitative metallographic analysis techniques. Chemistries are being determined using chemical spectrophotometric absorbance, x-ray fluorescence, magnetic and microprobe analyses. Crystallography is being studied using x-ray diffraction. The magnetic

measurements, which are sensitive to all of the above parameters, are used as a structure and processing-sensitive means to determine the impact of gravitationally driven convection and convective heat transfer on an important physical property.

The one-g experimental studies, in conjunction with theoretical analyses, will serve as a comparative base from which to evaluate the effectiveness of micro-g processing as a means of achieving diffusion controlled growth of eutectic, off-eutectic, and peritectic composites. Comparative analyses between the micro-g processed and the one-g magnetically damped results will determine the uniqueness of the orbital processing. Micro-g results to date have shown that existing theory is an inadequate base from which to anticipate results.

Solidification Fundamentals

Case Western Reserve University
Dr. V. Laxmanan
Professor J. F. Wallace
NAG3-417 (NASA Contact: Dr. Hugh R. Gray, LeRC)
March 10, 1983 - continuing task

During the first phase of this on-going task, a broad-based program has been defined aimed at understanding the fundamental aspects of a variety of solidification phenomena, all taking advantage of the unique opportunities offered by the microgravity environment of space. Containerless processing and an understanding of the role of fluids induced by gravitational effects are believed to be the two major objectives justifying an experimental program in reduced gravity. The solidification phenomena which are likely to benefit from such an investigation are: macro- and micro-segregation, columnar-equiaxed transition, pore formation, and undercooling.

The investigations currently underway include work on macrosegregation and directional solidification of Pb-Sn alloys in an apparatus similar to the General Purpose Rocket Furnace (GPRF) currently being flown in the space shuttle. Tentative approval for an experiment entitled "Isothermal Solidification in a Binary Alloy Melt" has been obtained. This will be flown on the D-1 mission in September 1985, following final approval of the flight experiment. Initial design of an experimental capsule aimed at studying bulk undercooling has also been completed.

Publications

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Measurement of the Properties of Tungsten at High Temperatures

Rice University
Professor John L. Margrave
NAS8-33199 (NASA Contact: L. Gardner, MSFC)
November 1978 - March 1985

This research is directed toward the measurement of the thermophysical properties of tungsten and other materials using containerless techniques. Successful levitation studies of liquid silver, gallium and tungsten have been completed. For liquid silver, $C_p = 32.6 \pm 2.1 \text{ J}\cdot\text{g}\cdot\text{atm}^{-1}\cdot\text{K}^{-1}$ over the range of 1281-1549 K and the heat of fusion is $10916 \pm 415 \text{ J}\cdot\text{g}\cdot\text{atm}^{-1}$. For liquid gallium, the heat capacity is $26.5 \pm 0.7 \text{ J}\cdot\text{g}\cdot\text{atm}^{-1}\cdot\text{K}^{-1}$ over the range 587-1630 K. For tungsten, the heat of fusion is $52 \pm 5 \text{ KJ}\cdot\text{g}\cdot\text{atm}^{-1}$. The studies of liquid aluminum are almost complete and are expected to derive new, reliable properties for liquid aluminum.

In preparation for a series of experiments using the cooling curve techniques to establish the high temperature properties of C, ZrC, TaC, Al_2O_3 , ZrO_2 and other materials, we need to obtain values for both the monochromatic spectral emissivity, ϵ_λ , at various temperatures as well as total hemispherical emissivities at various temperatures. Data for refractory materials are very limited and we will use a multi-color pyrometer technique for these studies.

In addition, prototype devices/approaches for the space flight studies will be developed at the GE Space Sciences Laboratory. A multi-year proposal will be developed, making use of the experience in high temperature science at Rice and the expertise in electronics and equipment design at GE.

Containerless High Temperature Property Measurements by Atomic Fluorescence

Midwest Research Institute
Dr. Paul C. Nordine
NAG8-465
March 1984 - March 1987

Laser induced fluorescence (LIF) provides a non-contact method for measuring high temperature properties. It is used in this program, with aerodynamic and/or electromagnetic levitation, and electromagnetic and/or CW CO₂ laser heating to develop and test new methods for containerless high temperature property measurements and process control. The capabilities and limitations of earth-based experiments are demonstrated and the information necessary to design improved experiments in space is obtained.

LIF studies of Al₂O₃, W, Mo, and LaB₆ evaporation and the structure of supersonic levitation jets were carried out and several methods for specimen temperature measurements by LIF were evaluated. Accurate absolute vapor pressure measurements were obtained from spatially resolved boron-atom LIF intensity versus temperature measurements over levitated spheres of LaB₆, a substance that reacts with all of the container materials used in previously reported work. Space-based experiments would allow application of this method to a broader range of materials, including liquids and solids that evaporate incongruently or for which the evaporation coefficient is small.

A new program was initiated to develop LIF methods for measuring temperature and component activities, which will be applied for containerless property measurements on liquids and for process control in synthesis experiments. The temperatures will be calculated by use of Boltzmann's law with measured vapor species velocity and/or electronic state distributions. Component activities will be obtained from relative LIF intensities over a compound and its components at the same temperature. The experiments will use two dye lasers to determine velocity distributions from time of flight measurements on laser pumped metastable atoms and to obtain precise simultaneous concentration measurements for different vapor species or electronic states.

Publications

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Containerless Processing of Undercooled Melts

University of Wisconsin-Madison
Professor John H. Perepezko
NAG3-436 (NASA Contact: Dr. Hugh Gray, LeRC)
September 1, 1983 - August 31, 1985

A main objective of the research is to extend the understanding of the physical mechanisms controlling liquid undercooling and to apply this knowledge to solidification processing methods. An assessment of the undercooling potential of containerless processing will be conducted on droplet samples of high melting temperature metals at NASA drop tube and drop tower facilities as well as in a laboratory-scale apparatus. New insight into nucleation and crystal growth will be obtained in undercooled liquids of high melting temperature iron and nickel-base systems. The processing parameters to be examined include melt superheat, droplet size and particle statistics and droplet surface coating. The solidification behavior is determined by thermal analysis and by structural and metallographic characterization.

During the initial phase of the program research has been developed along complimentary efforts. In one direction a number of samples have been sent to the Marshall Space Flight Center for processing in the drop tube facility. When these samples are returned, a detailed examination will be conducted on the solidification structures. A second direction has been focused upon the development of a laboratory scale drop tube for the processing of fine (10-100 μ m) droplet samples which is essentially completed. With the present design both previously prepared and in-situ atomized droplets may be released in a controlled manner for free-fall within the drop tube environment. Processing temperatures in excess of 1300 C can be attained for the evaluation of melt superheat. The tube chamber environment may be controlled also to introduce different gas atmospheres. In initial trials several runs with Cu and Sb-alloy droplets have been completed successfully and are undergoing microstructural study. To facilitate the evaluation of undercooling a separate thermal analysis examination is in progress on eutectic Sb-Mn droplets.

As part of a multi-year effort the continuing program will emphasize the development of laboratory scale drop tube studies during the next year. Following completion of the studies on Mn-Sb and Cu-Ag alloys, the experience will be extended to higher temperature systems. Further attention will be focused on monitoring the thermal history of drop samples by direct measurement and microstructure study. The influence of particle size on solidification kinetic will also be incorporated into evaluation of the undercooling capability of containerless processing.

Thermal and Solutal Convection Damping Using an Applied Magnetic Field

Grumman Aerospace Corporation

Dr. R. G. Pirich

J. L. DeCarlo

NAS8-34922 (NASA Contact: F. Reeves, MSFC)

August 1, 1982 - August 1, 1985

Samples of eutectic Bi/Mn alloy were directionally solidified in the presence of a transverse magnetic field to determine if gravity-induced convection effects could be reduced or eliminated. Furnace velocity, V , was varied over the range of $0.2 \text{ cm/h} \leq V \leq 50 \text{ cm/h}$ while thermal gradients at the liquid-solid interface were 100 C and 150°C/cm . The microstructure of Bi/MnBi is characterized by a regular, aligned-rod eutectic morphology that is sensitive to growth conditions. This, combined with ferromagnetism of one of the components, MnBi, can be used to determine whether convection effects are significant enough to be effected by the presence of a static, homogeneous, magnetic field.

Morphological, thermal, and magnetic analyses were carried out on samples grown with and without an applied magnetic field. For samples grown at velocities $> 3 \text{ cm/h}$ in a transverse magnetic field, MnBi mean rod diameter and interrod spacings were reduced compared to samples grown without the magnetic field present. Additionally, there was significant undercooling occurring for the applied field case, as evidenced by in-situ thermocouple measurements. Enhanced magnetic coercivities were observed for samples grown above $V = 3 \text{ cm/h}$, in a transverse magnetic field compared to no field growth. Sample properties for those samples grown in a magnetic field at 3.0 kG were found to be similar to previous low-gravity results. Samples grown below 3 cm/h in the magnetic field show little or no deviation from those grown without an applied field. In this low velocity regime, other factors such as rejection of a lower density solute at the interface, may overshadow thermal convection effects and higher fields may be needed to suppress solutal instabilities.

The results obtained during the first year effort suggest future experiments to further quantify and understand the mechanisms involved. As-grown samples will be analyzed to determine whether growing Bi-Mn eutectic in a magnetic field alters the % of HC (high coercivity) phase that would ordinarily be present in one- g_e growth with no magnetic field applied. Growth of the eutectic Bi/MnBi system in an external longitudinal magnetic field will also be performed. This experiment will be compared to present results, obtained by growth in a transverse magnetic field, since the magnetic drag induced by the field on the conductive melt should be sensitive to convective flow patterns. In addition, study of the effects of high external fields (up to 50 kG) on the solidification of the eutectic Bi/MnBi systems will be initiated to determine whether the deviations from diffusion-controlled growth at low growth velocities ($V < 3 \text{ cm/h}$) can be damped. These results will be compared with future low- g shuttle experiments in this velocity regime. These results will be further analyzed more completely using a theoretical model being developed at by Wilcox at Clarkson College. In addition, solidification modeling by Brown at MIT will be considered in the analysis. Another experiment

involves quantifying the effects of external transverse and longitudinal fields on the solidification of off-eutectic and peritectic Bi-Mn and Pb-Bi compositions. Previous one- g_e results at Grumman have shown significant macrosegregation for these compositions due to gravity-induced thermal and solutal convection. We are focusing on whether solutal convection can be reduced by applying various external magnetic field geometries.

Publications

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DeCarlo, J. and Pirich, R., "Effect of Applied Magnetic Fields on Directional Solidification of Eutectic Bi/MnBi," submitted to Met. Trans., 1984.

Presentations

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Pirich, R. G. and DeCarlo, J. L., "Effects of Damped Convection on the Properties of Directionally Solidified Bi-Mn Alloys," presented to 86th American Ceramic Society Annual Meeting, Pittsburgh, PA, April 1984.

Metallic Foams

Johns Hopkins University
Professor Robert B. Pond
NAS8-33021 (NASA Contact: D. Schaefer, MSFC)

The objective of this program is to investigate the formation of metallic foams by use of a blowing agent in the melt.

An attempt was made to produce a metallic foam by melting a mixture of Cu powder and graphite in a SPAR rocket. The graphite combines with the oxide on the copper to form Co which acts as a blowing agent. The result was inconclusive. No foam was formed, but it could not be determined whether the foam failed to form, formed but was lost to surface tension instabilities, or was destroyed by incomplete solidification before re-entry.

Diffusion in Immiscible Melts

Johns Hopkins University
Professor Robert B. Pond
NAS8-35620 (NASA Contact: D. Schaefer, MSFC)
February 1978 - March 1985

The objective of this program is to measure the diffusion coefficients for molten Pb in Zn in the immiscible liquid-phase region.

Diffusion couples of pure Pb and Zn have been prepared using a shear cell. These have been placed in graphite crucibles and encapsulated in stainless steel cartridges and are awaiting the next MEA flight opportunity. In flight, one couple will be soaked for 40 minutes at 440°C (just above the monotectic temperature) and the second couple will be soaked for 40 minutes 820°C (just above the consolute temperature). After the soak both samples will be rapidly quenched by flowing He to minimize redistribution of the immiscible phases. Post flight compositional analysis will be accomplished using x-ray fluorescence in the scanning electron microscope.

Directional Solidification of Monotectic and Hypermonotectic Aluminum-Indium Alloys Under μ -g

Centre d'Etudes Nucleaires de Grenoble
Dr. Claude Potard

The objective of this program is to analyze the mechanisms involved in the composite solid structure formation obtained from a miscibility gap alloy under microgravity. The metallic system aluminum-indium has been chosen for its low critical temperature, broad miscibility gap, and rather well-known thermodynamic properties.

Two experiments were performed aboard the Salyut-7 spacecraft. The purpose of the experiment, "Alliage Immiscible," was the study of the behavior of non-miscible alloys in microgravity. A parallel experiment, "Calibration," concerned the study of the heat exchange between cartridges and furnace in microgravity. The Soviet furnace, MAGMA, was used jointly with data recorder and accelerometer built by CNES. The 2 cm diameter cartridges were equipped with nine thermocouples. Five other thermocouples were installed near the furnace resistor itself. The MAGMA furnace is of the pulling type and works in open air. Two cartridges of the "Alliage Immiscible" containing two samples each were processed. Predispersed Al-In alloys obtained on the ground by quenching were molten and re-solidified in space under controlled conditions. One of the cartridges was processed without pulling and natural directional cooling and the second was pulled at a rate of 0.37 mm/min. The flight conditions were closed to the nominal conditions for the first processed cartridge which was not pulled. The second cartridge suffered from annealing time and gravity levels far from the acceptable limits. The recorded thermal analysis showed the melting and solidification events. They have helped to determine the temperature field inside the molten sample by numerical methods. The radiographic observations revealed non-wetting configurations of the samples and particular distributions of the heavy indium phase. It was shown that accumulations could be interpreted by considering the residual gravity vector, low rate of migration due to Marangoni forces and capture of the globules limited by pressure developed on the solid-liquid front.

The theories of capture of particulates by an advancing front were not able to explain the observations made on SPAR and Salyut-7 experiments. A new model was developed based on the filtration theory considering the globules population: the solid/liquid interface is like a filter of negligible thickness for the globules and its efficiency is limited by a pressure barrier measured for Al-In alloys by ground based experiments. The MEA-3 experiment was consequently adjusted to take into account the new model. The behavior of the globules in an interacting population on the solid front is, of course, an essential factor in the theory. The theory is at present being completed by results obtained by the Laboratoire d'Aerothermique of Meudon. The hydrodynamic and thermal interactions between pairs of globules placed in a temperature gradient have been calculated showing a large reduction of the Marangoni effect compared to the isolated globule. Physico-chemical properties of metallic liquid interface are currently being investigated within a cooperative program between CNES and the University of Grenoble.

Publications

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Presentations

Morgand, P. and Potard, C., "Solidification of Al-In Emulsion in Microgravity," presented at XXV COSPAR Plenary Meeting, Graz, Austria, July 1984.

Metals Electroprocessing in Molten Salts

Massachusetts Institute of Technology
Dr. D. R. Sadoway
NSG-7645

The present study seeks to explain the poor quality of solid electrodeposits in molten salts through a consideration of the effects of fluid flow of the electrolyte. Transparent cells allow observation of electrolyte circulation by a laser schlieren optical technique during the electrodeposition of solid zinc from the molten salt electrolyte, ZnCl_2 - LiCl - KCl . Experimental variables are current, density, electrolyte composition, and cell geometry. Based on the results of earlier electrodeposition studies as well as reports in the literature, these parameters are identified as having the primary influence on cell performance and deposit quality. Current density is varied over the range of 1 mA cm^{-2} to 1 A cm^{-2} ; the composition of the electrolyte is varied from 5 mole percent ZnCl_2 to 40 mole percent ZnCl_2 ; anode-cathode separation is varied from $1/4$ " to 1"; and the electrode orientation varies from vertical to horizontal.

Experiments are conducted to measure the fluid flow patterns and the electrochemical cell characteristics, and to correlate this information with the morphology of the solid electrodeposit produced. Specifically, cell voltage, cell current, characteristic time for dendrite evolution, and dendrite growth directions are noted. Their relationship to electrolyte flow patterns and the morphology of the resulting electrodeposit are derived.

Results to date indicate that laser schlieren imaging is capable of revealing fluid flow patterns in a molten salt electrolyte. On this basis the work on visualization of flow patterns in molten salts is being extended from the field of electrodeposition to the field of solidification. Molten salts have Prandtl numbers much closer to those of molten metals and molten semiconductors than aqueous solutions. Consequently, molten salts may be used as transparent analogs to metals in the study of the solidification process. On the basis of preliminary discussions with NASA/Lewis, plans are being made to design an experimental apparatus for flow visualization studies of the solidification of a molten salt. This apparatus would meet NASA requirements for low gravity experimentation. Plans are also being made to study low gravity electrodeposition to help understand the roles of turbulence and convective forces in this process.

Environmental Degradation of Materials: Surface Chemistry Related to Stress Corrosion Cracking

Syracuse University
Professor James A. Schwarz
NAGW-466

Parallel experiments have been performed in order to develop a comprehensive model for stress cracking (SCC) in structural materials. The central objective of the research is to determine the relationship between the activity and selectivity of the microstructure of structural materials to their dissolution kinetics and experimentally measured SCC kinetics.

Zinc was chosen as a prototype metal system. The SCC behavior of two oriented single-crystal disks of zinc in a chromic oxide/sodium sulfate solution (Palmerton solution) were determined. It was found that:

(a) The dissolution rate is strongly (hkil)-dependent and proportional to the exposure time in the aggressive environment.

(b) A specific slip system is selectively active to dissolution under applied stress and this slip line controls crack initiation and propagation.

As a precursor to potential microgravity experiments, electrophoretic mobility measurements of zinc particles were obtained in solutions of sodium sulfate (0.0033 M) with concentrations of dissolved oxygen from 2-8 ppm. The equilibrium distribution of exposed oriented planes as well as their correlation will determine the particle mobility. It is found that further development of ground-based experiment techniques to the limits possible in the terrestrial gravitational environment.

Graphite Formation in Cast Iron

University of Alabama
Dr. D. M. Stefanescu
NAG8-469 (NASA Contact: R. Mixon, MSFC)
March 1984 - March 1985

In the first phase of the project it was proven that by changing the ratio between the thermal gradient and the growth rate for commercial cast iron samples solidifying in a Bridgman type furnace, it is possible to produce all types of graphite structures, from flake to spheroidal, and all types of matrices, from ferritic to white at a certain given level of cerium. KC-135 flight experiments have shown that in a low-gravity environment, no flotation occurs even in spheroidal graphite cast irons with carbon equivalent as high as 5%, while extensive graphite flotation occurred in both flake and spheroidal graphite cast irons, in high carbon samples solidified in a high gravity environment. This opens the way for production of iron-carbon composite materials, with high carbon content (e.g. 10%) in a low gravity environment.

By using KC-135 flights the influence of some basic elements on the solidification of cast iron will be studied. Ground experiments have shown that no clear planar interface occurs in aluminum rich Fe-C samples. This is probably due to convection of aluminum which has a low density as compared with the base metal. This hypothesis will be checked by low gravity experiments.

The mechanism of flake to spheroidal graphite transition will be studied, by using quenching experiments at both low and one gravity for different G/R ratios. By this procedure flake graphite eutectic-liquid, compacted graphite eutectic-liquid and spheroidal graphite eutectic-liquid interfaces will be frozen, and cerium segregation at the graphite-frozen liquid interface will be determined by means of Auger microscopy. In this way, a monolayer absorption of cerium would be possible to document. It is expected to produce a theory on the eutectic solidification of cast iron, documented by direct experiments, which will explain the formation of various types of graphite, from flake to spheroidal.

Publications

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Theoretical Studies of the Surface Tension of Liquid Metal Systems

The Ohio State University
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Dr. Wei-Heng Shih
NAG-483 (NASA Contact: I. Yates, MSFC)
July 1984 - July 1987

A major goal of this project is to understand the surface tension and other thermophysical properties of liquid metals and alloys from a fundamental viewpoint. The approach is to calculate these quantities by a first principles technique which combines the statistical-mechanical theory of the liquid state with an electronic pseudopotential theory of electrons in metals. The inhomogeneity of the surface is treated using an ionic-density-functional formalism developed with the support of NASA. Of particular interest are the variation of surface tension with temperature and impurity concentration: such variations strongly influence the types of convection which make take place in a low-gravity environment. Some progress has already been achieved in computing the reduction of surface tension due to the presence of low-surface-tension impurities, and the corresponding surface segregation of such impurities. In the coming year, it is planned to concentrate on the surface properties of materials of particular interest to the MSA program: Si, Ga and GaSn alloys.

An additional goal of the program is to gain some theoretical understanding of the high temperature thermophysical properties of liquid metals, particularly high melting point materials which have not been studied extensively from a theoretical viewpoint. We are particularly interested in the properties of liquid Si and Ge (metals in their liquid state) and may extend the program to include elements of the transition metal groups.

Finally, it is planned to begin an investigation of other types of interfaces in liquids. Of particular relevance is the liquid-solid interface between, e.g. the liquid and the solid form of the same element. The surface tension associated with the solid liquid interface plays a major role in theories of crystal nucleation from the melt. We will attempt to generalize the theory of the liquid-vapor interface to the liquid-solid interface.

Publications

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Containerless Studies of Nucleation and Undercooling

Jet Propulsion Laboratory
Dr. Eugene H. Trinh
NAS7-918

The long term research goals are to perform experiments to determine the achievable limits of undercooling, the characteristics of heterogeneous nucleation, and the physical properties of significantly undercooled melts. The techniques used are based on the newly developed containerless manipulation methods afforded by acoustic levitation. Ground based investigations involve 0.1 to 2 mm specimens of pure metals and alloys (In, Ga, Sn, Ga-In, ...) as well as glass-forming organic compounds (O-Terphenyl). Both inert gases and high temperature immiscible liquids (organic oils, molten salts, glasses) are used as host media for sample suspension. Non-invasive measurements techniques for the surface tension, viscosity, density, sound-velocity, and specific heat are being developed and refined in order to probe the physical state of undercooled levitated melts.

A currently operating ultrasonic high temperature apparatus has allowed the ground-based levitation of 1 to 2 mm samples of solid aluminum at 550°C in an argon atmosphere. Levitation and melting of In and Sn samples have also been carried out in the same apparatus at lower temperatures. Present work is concentrating on the undercooling of pure metal samples (In, Sn), and on the measurements of surface tension and viscosity of the undercooled melts via shape oscillation techniques monitored through optical detection methods. The sound velocity of undercooled O-Terphenyl is being measured in an immiscible liquid levitation cells.

Publications

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FLUIDS AND TRANSPORT PHENOMENA

Cloud Microphysics by Thermal Wave Methods

Marshall Space Flight Center
Dr. B. Jeffrey Anderson
David A. Bowdle, USRA
Michael Reischel, USRA
In-House

This experiments series is the first application of a low-gravity experimental technique to the study of cloud microphysics. The low-gravity environment is provided by the parabolic maneuver of NASA's KC-135 aircraft. The primary objective is to compare experimental observations of cloud droplet growth and evaporation in a convection free environment with a numerical model of the process. Beyond that, the work also involves the development and testing of low-gravity research techniques. In particular, passive methods of thermal control have been devised and used effectively in this study.

The experiment hardware has gone through a two stage development process with several successful KC-135 missions completed at each stage. The experimental portion of the program is expected to be completed by the final flight series, to be conducted during July 1984. The numerical model has been debugged and improved and data analysis from recent flights is well underway. The study to date has shown that the method is particularly suitable for looking at interactions between adjoining portions of the cloud drop field and interactions of the drop field with a solid interface. After final analysis of the data, it is expected the results will shed light on the development of cloud droplet size spectra in natural clouds as well as the performance of certain types of cloud physics instrumentation, particularly continuous flow diffusion chambers and cloud condensation nuclei counters.

Publications

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Reischel, M. T., " β -AgI Bipyramids: Growth by Solvent Dilution of Aqueous NH_4I Solution," submitted to Journal of Crystal Growth, 1984.

Presentations

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Forcing," presented at First International Aerosol Conference, Minneapolis,
MN, September 17-21, 1984.

Extension of Ostwald Ripening Theory

University of Alabama, Huntsville
Dr. James Baird
Dr. Robert Naumann, MSFC

The objective of this investigation is to develop models based on the mean field approximation of Ostwald ripening to describe the growth of second phase droplets or crystallites. The models will include time variations in nucleation rate, control of saturation through addition of solute, precipitating agents, changes in temperature, and various surface kinetic effects.

Numerical integration schemes have been developed and tested against the asymptotic solution of Lifshitz, Slyozov and Wagner (LSW). A second attractor (in addition to the LSW distribution) has been found and, contrary to the LSW theory, the final distribution is dependent on the initial distribution. A series of microgravity experiments is being planned to test this and other results from this work.

Modelling of Laser-Enhanced Chemical Vapor Deposition

Massachusetts Institute of Technology
Dr. Robert A. Brown
NSG-7645

Research is directed at development of a detailed model of mass and heat transfer and chemical reaction in the pyrolysis of silane for the growth of thin amorphous silicon substrates incorporating laser heating of the gas phase above the film. Research is in collaboration with the experimental program lead by Dr. John Haggerty of the Materials Processing Center for the development of laser-enhanced chemical vapor deposition. The model will be the basis for evaluation of the relative importances of the decomposition of SiH_4 in the vapor phase, mass transfer of the intermediate species, e.g. SiH_2 , and the evolution of hydrogen gas. Plans are also underway for developing a model for homogeneous nucleation of Si in the vapor phase to model the rate limitations observed at high gas-phase temperatures and high partial pressures of silane.

We have concentrated on an almost one-dimensional model for the coupling of the CO_2 laser beam for heat transfer of the vapor phase with simple kinetic models for SiH_4 decomposition and subsequent absorption of Si vapor on the substrate. Mass transfer in the vapor phase is assumed to be solely by diffusion. Results of this model should qualitatively mimic the operating regions observed experimentally and will be the basis for extending the analysis to two space dimensions to examine the effects of beam shape on the spatial uniformity of the deposited film. The role of convection in the vapor phase caused by the large changes in density in and around the center of the laser beam will be analyzed to evaluate the potential of microgravity experiments for increasing the uniformity of the film and the deposition rate.

Dynamic Thermophysical Measurements in Space

National Bureau of Standards
Dr. Ared Cezairliyan
Dr. A. P. Miller
Dr. M. S. Morse
Dr. L. A. Schmid
H-27954B
April 1981 - continuing task

The objective of this research is to develop techniques for the dynamic measurement of selected thermophysical properties (e.g. heat of fusion, heat capacity, electrical resistivity) of solids and liquids at temperatures above 2000 K in a near zero-gravity environment. The first phase of this project involves establishing the stability (geometrical) limits of specimens when rapidly heated to temperatures beyond their melting point. A test equipment package has been designed and constructed for this purpose, consisting of removable specimen cartridge cells, a battery-bank power supply, a high-speed framing camera, a single-wavelength pyrometer, and electronic switching and control equipment. The operation of the equipment package has been tested in the laboratory in preparation for near-zero-gravity experiments to be performed during a KC-135 flight in September, 1984.

Transport Processes in Solution Crystal Growth

Lewis Research Center
Dr. A.-T. Chai
In-House
Continuing Task

The objective of this effort is to conduct fundamental research in reduced gravity on transport processes occurring during solution crystal growth. Experimental techniques will be developed to monitor and control key parameters at the interface between a growing crystal and the solution from which it grows. Techniques developed in this investigation will lead to an in-space experiment on the Shuttle. The focus of this research effort will be on non-incurative ground-based laboratory measurements of model systems and the definition of requirements for space experimentation.

Initially aqueous solutions will be used for easier control and instrumentation. Model systems with imposed steady flows and simplified boundary conditions will be studied for characterization. Various nonincurative measurement techniques are being investigated for proper applications in a newly built laboratory capable of flow visualization, laser doppler velocimetry, Schlieren photography, specklegram, holographic interferometry, and Raman spectroscopy.

Free Surface Phenomena Under Low- and Zero-Gravity Conditions

University of California-Berkeley

Dr. Paul Concus

Dr. D. Coles, Caltech

Dr. R. Finn, Stanford University

Dr. L. Hesselink, Stanford University

NAG3-147 (NASA Contact: Dr. Tom Labus, LeRC)

February 28, 1985 - January 31, 1986

In a low- or zero-gravity environment the free surface of a liquid can behave in striking, unexpected ways. For example, in a partially filled container, a free surface that is well behaved under terrestrial conditions can rise to an arbitrarily large height or even fail to exist when gravity is absent. This study attempts to answer the central mathematical questions: Under what conditions can free surfaces exist and what are their properties? -- and experimental questions of what means can be devised to observe and to measure the surfaces quantitatively.

Current activity focuses on material selection and the design of optical diagnostic methods for in-space experiments. These experiments will test mathematical predictions of discontinuous transition from existence to nonexistence of capillary free surfaces in certain geometric configurations under zero gravity conditions. Liquids and container materials suitable for achieving the required contact-angle range and optical properties are being investigated and, as well, the effects of contaminants and fluid motion on contact angle. The optical diagnostic technique investigation concerns laser-induced fluorescence, including emphasis on data acquisition, sources and detectors, reliability, data management, and computer control.

Publications

Concus, P. and Finn, R., "Continuous and Discontinuous Disappearance of Capillary Surfaces," Report LBL-15356, 1983.

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Thermocapillary Flows and Their Stability: Effects of Surface Layers and Contamination

Northwestern University
Dr. S. H. Davis
Dr. G. M. Homsy, Stanford University
NAS8-33881 (NASA Contact: I. Yates, MSFC)
June 1980 - June 1983

The objective of the research is to study quantitatively thermocapillary flows in two-dimensional and axisymmetric geometries in order to learn the impact of such flows on float-zone refining configurations.

The work begins with the analytical study of steady flows in slots and zones to obtain predictions of surface curvature, flow and heat transport. It then examines the instability of such flows. Three new instability mechanisms have been identified and the results show that thermocapillary flows of high Prandtl number fluid become convectively unstable while those of low Prandtl number fluid become hydrodynamically unstable. The work includes numerical simulations of steady, high Marangoni number flows in unit aspect ratio boxes and asymptotic solutions in large boxes. Furthermore, the effect of surface contamination on the steady thermocapillary flows have been examined.

This fundamental study has focused on fluid dynamical systems used to understand events in the flow of melts in float-zone geometries both on Earth and in microgravity environments.

Publications

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Mass Transport Phenomena Between Bubbles and Dissolved Gases in Liquids
Under Reduced Gravity Conditions

University of Toledo
Professor Kenneth J. De Witt
J. L. Brockwell, Union Carbide Technical Center
NAG3-34 (NASA Contact: Dr. Tom Labus, LeRC)
November 15, 1983 - January 14, 1986

The long term objective of the experiment is to observe the dissolution of isolated, immobile gas bubbles of specified size and composition in a solvent liquid of known concentration in the reduced gravity environment of earth orbit. Preliminary bubble dissolution experiment conducted both in the NASA Lewis 2.2 sec drop tower and in normal gravity using SO_2 - Toluene system were not completely successful in their objective. The method of gas injection and the lack of bubble interface stability experienced due to the extreme solubility of SO_2 in Toluene has the effects of changing the problem from that of bubble dissolution to one of bubble formation stability and subsequent dissolution in a liquid of unknown initial solute concentration.

Current work involves further experimentation in order to refine the bubble injection system and to investigate the concept of having a bubble with a critical radius in a state of unstable equilibrium. The method of gas injection used in the drop tower tests was by means of a spring-actuated hypodermic syringe. In many of the experiments, the resulting bubble formation rate was either too slow so that the bubble remained attached to the syringe needle tip due to surface tension effects, or the formation rate was too high, such that, after detaching from the needle tip, the bubble quickly crossed and disappeared from the photographic field of view due to inertia effects. Bubble oscillation and coalescence were other consequences of the method of injection, although the system solubility also contributed to the observed phenomena. It is necessary that the injection technique be refined such that the bubble is isolated and immobile. The critical bubble radius concept must be examined since it is needed for initialization for all experiments involving highly soluble gas-liquid systems. In these systems, the high gas solubility generally prevents the formation of a stable gas-liquid interface, so that a bubble can be formed, until a suitable background concentration of the dissolved gas in the liquid has been attained. This technique involves the injection of a single bubble of gas of approximately a prescribed size and composition into a quantity of thermostatted liquid under controlled pressure conditions. The pressure on the liquid is then adjusted to maintain the bubble in a state of unstable equilibrium with the surrounding liquid. As a result of a step increase in the pressure, bubble dissolution is initiated. The rate of mass transfer can be determined from an observation of the change in bubble size with time.

The NASA facilities available for further experimental work are both drop towers (2.2 sec and 5 sec of free-fall time) and the NASA Lear Jet (23 sec of free-fall time). NASA Lewis engineers are designing and

constructing the improved prototype experiments package. The principal investigators will (1) consult in the construction and participate in the running of the experiment; (2) begin a further analysis of gas-liquid mass transfer theory; and (3) participate in the evaluation of the experimental data.

Publications

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Suppression of Marangoni Convection in Float Zones

George Washington University
Dr. Robert F. Dressler

The basic purpose of this program is to demonstrate by means of an earth-based 1-g experiment that the undesirable Marangoni (surface tension) convection can be suppressed or significantly reduced by means of gas jets directed tangentially to the free surface of the liquid in a float zone. These jets will establish the tangential shear stress field over the surface which must be adjusted to equal the counter-stress resultant of the Marangoni shear stress which causes the convection. For proposed materials processing in space (0-g), particularly of important, highly reactive semiconductor materials, e.g. silicon, microgravity will virtually eliminate the unwanted thermal-buoyancy convection in the liquid silicon, but will have no effect in reducing the Marangoni convection. Unless this can be sufficiently suppressed by other means, there may be no significant advantages to the proposed space processing of reactive semiconductors. Although some inert gas such as argon must be used for the corrosive liquid silicon, the earth-based experiment uses air jets and various transparent oils, since the basic principle involved is the same. The first float zone is enclosed in a very small rectangular box with a quasi-planar free surface. Stable Marangoni convection has been achieved and velocities measured photographically. The air jet system with variable velocity and temperature is under construction. Three independent parameters must be optimized to attain maximum suppression: the gas velocity, angle of attack, and gas temperature.

The half zone being used is heated at the top to establish a more thermally stable configuration. Computations show that in the ranges being used, the 1-g effect of buoyant convection is virtually eliminated (only 4% of total velocity) due to the tiny size (1.5 mm x 1.5 mm x 2 mm) of the float zone.

After the quasi-2D zone has been fully investigated, a rotationally symmetric zone will be studied on earth. If the results exhibit significant reduction in Marangoni convection in 1-g, then specific plans and design for a large float zone experiment in the Space Shuttle will be presented to NASA. A space experiment should be easier than the earth-bound experiment because: (1) a large float zone can be maintained, and (2) the shape of the free surface can be made precisely cylindrical (both due to the absence of gravity). Then the adjustments for the gas jets will be easier to effect, and the jets will conform better to the straight (ruled) surfaces, thus minimizing variations in the counter-shears, and unwanted momentum transfer.

Many computer analyzes have been made for the Marangoni flows in the 1-g experiments, and other computations have been made which show that buoyancy convection has successfully been removed. A mathematical research task has been completed, and is being written up for publication, which

analyzes quantitatively the Marangoni flows by means of the modification of boundary-layer theory, and an estimate is given for the change in critical Marangoni numbers when materials, e.g. silicon, with very low Prandtl numbers will be used, which constitute commercially very important applications.

Studies of Model Immiscible Systems

Marshall Space Flight Center
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B. R. Facemire
W. K. Witherow
U. Fanning
Dr. William F. Kaukler, USRA
In-House
October 1, 1982 - October 1, 1986

The objectives of this program task are to use model transparent monotectics to obtain fundamental information applicable to two-phase systems in general, to apply this understanding to materials of interest in the Microgravity Science and Applications program, and to interpret results of flight experimental involving monotectic alloys.

A number of model immiscible systems are in use to study various aspects of two-phase behavior within the miscibility gap and during solidification. Particle growth, coalescence and particle motions are under investigation using a holographic microscopy system. The system is capable of working with particle densities up to 10^7 particles/cm³ through a 100 μ m depth and can resolve particles of the order of 2 to 3 μ m in diameter throughout the entire cell volume. Particle size, distribution changes with respect to time and temperature are observable from sequential holograms. Initial experiments using diethylene glycol/ethyl salicylate (DEG/ES) have demonstrated the usefulness of the technique. The thermal system controls temperature to at least ± 0.001 K over the course of an experiment. A time-lapse film, made from holograms, of a succinonitrile/water solution shows particle size and number distribution changes with time under isothermal conditions. The observations are consistent with Ostwald ripening theory.

Data with respect to solidification of succinonitrile/water solutions are thus far consistent with critical point wetting behavior and Marangoni effects. There is experimental evidence that wetting phenomena are observable by holographic photography. Solid-liquid interfacial free energy differences are, in principle, accessible by film pressure (via ellipsometry) measurements currently in progress. Spinodal decomposition and nucleation studies via differential thermal techniques are underway. These studies, in light of concentration profiles of model solidified ingots, should yield valuable verification of operational limits. Wherever feasible, data in all areas of study will include components for analyzing surface effects (modification of pyrex or quartz surfaces to reverse wetting properties). Experiments to study critical point wetting by direct measurements of contact angles are also in progress. These experiments require a microgravity environment to relieve the hydrostatic effects which distort contact angles governed by weak interfacial forces, and, therefore, rely on KC-135 flights and the drop-tower facility at MSFC.

A temperature gradient stage with high thermal stability is in use to study solidification phenomena in situ using model transparent systems. An unusual monotectic solidification interface morphology is currently under

observation by this technique. Publications to describe this phenomena are near completion. Laser interferometry will proceed in systems having large features to measure concentration gradients in the liquid ahead of an advancing solid/liquid interface. A Bridgeman-Stockbarger type furnace to observe directional solidification of transparent systems is in operation. Solutions observed on the temperature gradient stage will also be observed in the Bridgman furnace to relate microscopic to macroscopic solidification processes. The model systems for study include monotectic and eutectic phase reactions. Many of the experiments will be performed in the lab and the KC-135 to observe the effects, if any, on the fine scale concentration and density gradients in the liquid by the gravity vector.

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Light Scattering Tests of Fundamental Theories of Transport Properties in the Critical Region

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Dr. M. R. Moldover, NBS
NAG3-470 (NASA Contact, Dr. A-T. Chai, LeRC)

The objective of this program is to measure the decay rates of critical density fluctuations in a simple fluid (xenon) very near its liquid-vapor critical point using laser light scattering and photon correlation spectroscopy. Such experiments have been severely limited on earth by the presence of gravity which causes large density gradients in the sample when the compressibility diverges approaching the critical point. The goal is to measure decay rates deep in the critical region where the scaled wavevector is the order of 1000. This will require loading the sample to 0.01% of the critical density and taking data as close as 3 microKelvin to the critical temperature ($T_c = 289.72$ K). Other technical problems have to be addressed such as multiple scattering and the effect of wetting layers.

We have demonstrated the ability to avoid multiple scattering by using a thin sample (100 microns), and a temperature history which can avoid wetting layers, satisfactory temperature control and measurement, and accurate sample loading. Thus the questions of experimental art are solved leaving the important engineering tasks of mounting the experiment to maintain alignment during flight and automating the state-of-the-art temperature bridges for microcomputer control of the experiment.

The experiment consists of measurement of the fluctuation decay rate at two angles for each temperature while simultaneously recording the sample turbidity from the log ratio of transmitted and incident beam intensities. The turbidity data is used to measure the correlation range at each temperature and to locate T_c . Temperature will cover from 100 milliKelvin to the resolution of the bridges, 3 microKelvin, from the critical point.

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Transient Heat Transfer in Zero Gravity Environment

National Bureau of Standards - Boulder Laboratories

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H-27954B

The objective of the project is to provide computer codes and/or correlations for application in the design of transient heat transfer systems. The mathematical models in the computer codes are being tested by experimental data obtained in the laboratory using optical techniques.

The elimination of natural convection (buoyancy) in the experimental data obtained in zero-g (KC-135) facilitates the study of thermal expansion effects which is of interest in the development and testing of the mathematical models of the transient heat transfer. Initial flight in the KC 135 (June 1983) suggested some modifications to the experimental apparatus and the data acquisition technique (high speed video will replace high speed movies for recording optical records generated during the experiments).

A series of KC-135 flights with this experiment on board are scheduled for summer/fall 1984. We anticipate the acquisition of the aforementioned data using interferometry and "beam deflection" optical techniques. The measurements provide time dependent temperature fields above a horizontal heater surface submerged in a static fluid (Freon 13). The heater surface is electrically heated for a time interval ≤ 0.100 sec. during which the data are optically recorded.

Surface Tensions and Their Variations with Temperature and Impurities

National Bureau of Standards
Dr. S. C. Hardy
Dr. J. Fine
H-27954B
April 1981 - September 1984

Surface tension gradients at free surfaces of liquid due to temperature or concentration variations can generate fluid flows. In low gravity these can be significant and can influence the temperature and solute fields in the bulk liquid. Consequently, in materials processing insapce, knowledge of these surface tension gradients can be essential to the understanding of the observed phenomena.

The surface tension of silicon and its variation with temperature are not known accurately. These data are of interest because it is now thought that surface tension driving forces contribute significantly to fluid flow in the Czochralski and float zone crystal growth of silicon. Fluid flows arising from surface tensions are referred to as Marangoni or thermo-capillary flows. They are important in normal gravity and are the dominant type of fluid flow in low gravity space experiments with silicon for which buoyancy convection is negligibly small. Experiments involving free liquid silicon surfaces are presently being developed for future space shuttle flights.

The surface tensions in this work were determined using the sessile drop technique. This method is based on a comparison of the profile of a liquid drop with the profile calculated by solving the Young-Laplace equation. The comparison can be made in several ways; the traditional Bashforth-Adams procedure has been used here in conjunction with recently calculated drop shape tables wich virtually eliminate interpolation errors.

Although previous study has found little difference in measurements with pure and oxygen doped silicon, there is other evidence suggesting that oxygen in dilute concentrations severely depresses the surface tension of silicon. Our measurements in surface tension also show a strong sensitivity to what we believe to be a variation in oxygen pressure. Although the atmosphere in all the experiments was nominally ultra pure argon, somewhat different conditions in each group of measurements produced different concentrations of impurities.

The surface tension of liquid silicon in purified argon atmospheres was measured. A temperature coefficient near $-0.28 \text{ mJ/m}^2\text{K}$ was found. The experiments show a high sensitivity of the surface tension to what is believed are low concentrations of oxygen. Thus one cannot rule out some effect of low levels of oxygen in the results. However, the highest surface tension values obtained in conditions which minimized the residual oxygen pressure are in good agreement with a previous measurement in pure hydrogen. Therefore, depression of the surface tension by oxygen is insignificant in these measurements.

Direct Observation of Critical Point Wetting in Microgravity

University of Alabama, Huntsville
Dr. William F. Kaukler

The objective of this program is to observe the interface shape in single and multicomponent systems at the onset of critical wetting in microgravity using the MSFC drop tower and KC-135 aircraft.

Test cells for the drop facility have been built and tested up to critical point of CCl₄. Low temperature drops have been conducted for two-component systems near the critical consolute point. Contact angle seems to approach 90° near the critical consolute temperature contrary to expectations. It is suspected that since the interfacial energy becomes vanishingly small at the critical consolute temperature, the interface shape has not reached equilibrium in the available low-gravity time.

Surface Tension Induced Instabilities in Reduced Gravity: The Benard Problem

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NAG3-393 (NASA Contact: Dr. A-T. Chai, LeRC)
December 31, 1984 - December 30, 1985

A Benard convection experiment has been set up, and the onset of convection in shallow layers of silicone oil two millimeters or less deep has been studied. The onset has been observed visually or has been determined by the break in the heat transfer curve which accompanies the onset of convection. The outcome of these experiments has been very surprising, from the point of view of theoretical expectations. Surface tension driven convection in the absence of gravity is described by Pearson's study, or the adaption of Pearson's work to the condition in a lab on Earth (considering g) by Nield. Both papers predict the existence of a critical temperature gradient, below which convection will not occur. Our experiments clearly contradict this concept. While our experiments are in complete agreement with theory for fluid layers $> 2\text{mm}$ deep, we observe the onset of convection at temperature differences far below the critical value for fluid depths smaller than 2mm . The discrepancy between experiments and theory increases with decreasing fluid depth. According to theoretical considerations, the effects of surface tension become more important as the fluid depth is decreased. Actually, one observes that the onset of convection takes place in two stages. There is first an apparently surface tension driven instability, occurring at subcritical temperature differences according to conventional theory. This instability forms a weak cellular pattern. If then the temperature difference is increased, a second instability occurs which transform the first pattern into conventional strong hexagonal Benard cells. The second instability is in agreement with the critical temperature gradients predicted by Nield. The first instability is, at least qualitatively, in agreement with a theoretical study by Scriven, who does not only consider the effects of the variation of surface tension with temperature, but also the consequences of the equilibrium value of surface tension, i.e., the consequences of σ not only $d\sigma/dT$.

A drop tower rig has been built to investigate the edge effects of test containers. Results show clearly that sharp edges do contribute to a stable meniscus. A flat free surface is therefore feasible insofar as conducting the Benard convection study in space is concerned. The drop tower rig is also designed to perform tests investigating the effects of low accelerations both parallel and perpendicular to the meniscus. However, problems caused by the malfunction of an optical encoder for positioning delayed that part of the feasibility study. A new and sturdier encoder has been on order. Further tests should resume as soon as the encoder problem gets resolved.

A lab designated for fluid measurements including flow visualization has been built and is near completion. It is expected to be fully functional in early 1985.

Lambda Point Experiment

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NAG2-62 and JPL 955057

Following a major breakthrough in the understanding of cooperative phase transitions pioneered by Kenneth Wilson in the 70's, it became increasingly clear that hard-core, definitive experiment tests of the theory were lacking. A study of the nature of the lambda-transition under reduced gravity conditions provided the greatest potential to advance the experimental knowledge in this area.

Central to the study of cooperative transitions is the idea of asymptotic behavior of various thermodynamic properties in the limit as the temperature interval from a transition is reduced to zero. Most current theoretical predictions are made in this limit. The present experiment is designed to explore the submicrodegree region of this transition using new thermometry technology which pushes the resolution of temperature close to the fundamental limits set by statistical fluctuations. This will open up a whole new region, never before explored, for testing of the theories of cooperative transitions. The tests that will be performed will be more than an order of magnitude more stringent than any performed so far and will shed light on a major area of condensed matter physics. The program will ultimately extend the explored region of cooperative phenomena by an amount greater than the total region so far investigated since the discovery of such transitions in 1863.

In the initial flight experiment, the plan calls for the observation of the temperature dependence of the heat capacity of helium very near the lambda point with a resolution of a few times 10^{-10} deg. This experiment will be conducted in conjunction with JPL, who will provide the helium dewar, flight-tested on Spacelab 2. During the last two years of this program a second dewar will be constructed to allow follow-up experiments with a turn-around time of the order of 1 year, and begin developing additional experiments to probe other properties of helium, among them the superfluid Josephson effect, tricritical point behavior, and transport properties. At a later phase, the use of a low cost, recoverable spacecraft, such as the SPARTAN, will make it more desirable to perform experiments in a quieter environment than the Shuttle.

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Critical Transport Phenomena in Fluid Helium Under Low Gravity

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NAG4-379

The purpose of the research is to study the feasibility of carrying out measurements of certain critical transport properties of pure fluid under conditions of low gravity. These properties are the thermal conductivity, κ , the shear viscosity ζ and the diffusive relaxation time τ , which are predicted to diverge (tend to infinity) as the liquid-vapor critical point is approached. However, in this critical region, the earth's gravity effect becomes very important. As the critical point is approached, the gravity effects increasingly distort the results. The reason for this is that the compressibility of the fluid also diverges and under the influence of gravity causes a vertical density gradient in the fluid, which is significant even when very thin fluid layers (typically 1 mm high) are being used. The result is that the temperature dependence of κ , ζ , and τ tends to flatten off as T_c is approached instead of continuing to increase, and therefore the predictions from the renormalization group and mode coupling theories cannot be subjected to a satisfactory test.

The research consists in developing adequate techniques for the critical shear viscosity measurement of ^3He along the critical isochore. These experiments use a torsional oscillator operating at 150 Hz. This oscillator has a flat cylindrical shape and the space for the fluid has a height of 1.5 mm and a diameter of 5 cm. There are four highly polished aluminum plates in this space, rigidly attached to the frame to increase the surface of contact with the fluid. Introduction of the fluid into the cell dampens the oscillations. From the decay with time, the viscosity is calculated. The first results were achieved in December 1983, and showed the expected beginning of the divergence of ζ and the flattening-off due to gravity. At present more tests are made, a new cell is designed to check the reproducibility and also experiments will be designed for a frequency of 5 kilohertz. According to predictions, a frequency dependence of the viscosity should be observed. Once measurements on the critical viscosity of ^3He , of ^4He and possibly on $^3\text{He} - ^4\text{He}$ mixtures are completed and the performance of the very delicate equipment is considered adequate, a decision will be made whether the viscosity of the thermal conductivity measurements will be technically suitable for a space shuttle flight. (The diffusive relaxation time is obtained as a by-product during thermal conductivity experiments.

Precise Viscosity Measurements Very Close to Critical Points

National Bureau of Standards

Dr. M. R. Moldover

Dr. B. Berg

C-86129D (NASA Contact: Dr. A-T. Chai, LeRC)

A torsion oscillator is being developed to measure the viscosity of fluids at moderate temperatures (0-100°C) and pressures (0-10 MPa) at very low frequencies (0.5 Hz) and very low shear rates (0.05 sec⁻¹). These conditions are required to measure the shear viscosity of fluids extremely close to the critical point. The oscillator, thermostat, vacuum system, and instrumentation have been assembled and are undergoing debugging. Preliminary measurements indicate that the system has a noise corresponding to 0.2% of the viscosity of methanol-cyclohexane mixtures near the consolute point.

Experimental and Theoretical Studies of Wetting and Multilayer Adsorption

National Bureau of Standards
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Dr. J. W. Schmidt
Dr. J. W. Cahn
Dr. R. F. Kayser
H-27954B
April 1977 - continuing task

The recent work with partially miscible binary liquid mixtures has established that the structure of the liquid-vapor interface can undergo a first-order phase transition from incomplete to complete wetting of the vapor as the temperature is raised. A discontinuity in the change of interfacial tension as a function of temperature at the phase transition has been predicted to occur in many systems and to play an important role in the growth of uniform composites from alloy melts at monotectic points. Our measurements are the first to establish the order of the transition. Studies of capillary rise in SF_6 in a unique interferometer have led to the first measurements of the thickness of wetting layers (or equivalently, multilayer adsorbed films) on a solid surface near a liquid-vapor critical point. We have observed instabilities in wetting layers. A theory for the instabilities is being developed and will be checked by both static and dynamic optical experiments. The effect of gravity on the apparent thickness of interfaces (as measured by ellipsometry) is under study.

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Impurity Extraction by Droplets

National Bureau of Standards
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Dr. J. M. Kincaid

The goals of this project are to model and to measure the phase equilibrium properties of a finely divided fluid containing a large number of chemically similar species. Our objective is to develop an accurate, usable model for such phenomena as pollutant extraction of rain clouds, industrial separation in spray towers, and separation in emulsions. The project has been designed as a hierarchy of complementary theoretical and experimental steps.

A theory has been developed to describe the segregation of complex impurities at the interface of a solvent. This phenomenon is important in phase behavior when a large fraction of molecules in a material are near an interface, the situation in a finely divided material. The theory will be modified to account for the effect of surface curvature on the surface tension. The study of mixtures differs from pure fluids not only because of the surface effects but also because composition differences between the droplet and the surrounding vapor can stabilize a droplet with respect to a bulk phase.

The experimental part of the project has been the design and construction of a variable volume, vapor-liquid equilibrium cell to study bulk-phase equilibrium. This apparatus is fully automated and will be interfaced with a laboratory computer. Measurements being made on the properties of a "pentane-like" many-component hydrocarbon mixture will provide a benchmark against which the measurements of mists of the same material will be compared. These measurements will also provide an experimental test of thermodynamic models that describe complex mixtures which have been developed by the principal investigators. The next step in the experimental part of the project is the development of techniques for mist production and design of a low angle light scattering experiment to monitor the development of the mists.

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Energy Stability of Thermocapillary Convection in Models of the Float Zone Process

Arizona State University
Professor G. Paul Neitzel
Professor Daniel F. Jankowski
NAG3-568 (NASA Contact: Dr. Tom Labus, LeRC)
August 5, 1984 - August 4, 1987

This research program is intended to study the energy-stability of thermocapillary convection in models of the float-zone, crystal-growing process. The program is intended to determine stability limits, as functions of pertinent parameters, that will identify conditions which will not allow the existence of an undesirable oscillatory flow instability. Such instabilities may occur in the space processing of semiconductor materials.

The determination of the stability limits will involve two sets of numerical computations: (1) solution of the nonlinear governing equations together with the appropriate boundary conditions to determine the basic state (in general, velocity, pressure and temperature fields and the displacement of free surfaces and interfaces); and (2) solution of a nonlinear Euler-Lagrange systems for the energy-stability limit. Both computations, while difficult, should be within the scope of available computer capability and available concepts in numerical analysis. Finite-element methods are attractive candidates for the numerical work.

Surface-Tension Driven Convection

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Dr. Y. Kamotani
NAG3-570 (NASA Contact: Dr. Tom Labus, LeRC)
February 1985 - May 1985

The objective of this investigation is a detailed study of the transient and steady-state nature of fluid flows in the stable, long-duration environment afforded by the Space Shuttle. The present schedule will result in the flight of this experiment in early 1986.

There are a variety of nongravity forces, as well as gravity itself, that can induce fluid flows in space. Such nongravity driving forces include surface or interfacial tension, g-jitter, thermal volume expansions, and magnetic and electric fields.

The present experiments are directed to examine one of the aforementioned driving forces, i.e., surface-tension gradients. The objective of the research is to study the surface-tension induced convection under reduced gravity and to compare it with normal gravity convection with all other identical conditions. The intensities of such flows and their penetration depth below the free surface are also determined. In this way, the dominant role of a free surface in a reduced-gravity environment is indicated.

The interface between two fluid phases can influence the motion of fluids when either the interface has finite curvature or when the interfacial tension varies from point to point. In both cases, forces appear in the interfacial region that can affect or generate fluid motions.

The relative importance of surface-tension and gravitational forces is usually estimated from the Bond number, $B_0 = \rho g L^2 / \sigma$, where ρ is the fluid density, g the acceleration of gravity, L a characteristic dimension, and σ is the surface tension. From the Bond number it is clear that, on Earth, surface tension is important (Bond number less than unity) only in small-scale configurations, i.e., where L is small. Therefore, most existing work on the effect of surface tension deals with flows in capillaries and thin films or the motion of droplets or bubbles or short wavelength water waves. In space, on the other hand, surface tension becomes a significant force whose influence on fluid motion must be assessed and understood.

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Thermocapillary Motion Research

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In-House

July 27, 1984 - November 26, 1985

The objective of this program is to conduct fundamental microgravity research on interfacial surface tension driven motion by a linear temperature gradient. A vertical linear temperature gradient will be established within a test container filled with a host fluid. The thermal gradient will be positive in the upward vertical direction to avoid any free convective currents. With minimal disturbance, a single immiscible fluid droplet will be introduced into the host fluid. As predicted by existing theory, the droplet will move in the direction of higher temperature due to tangential shear stresses created by surface tension variations along the periphery of the droplet.

The research to be conducted involves experimentally determining the effects of various parameters (such as droplet size, magnitude of the temperature gradient, and fluid properties of both the droplet and host fluids) on the behavior of the droplet migration. Efforts are currently being centered around LeRC's 2.2-second Drop Tower Facility. Fluid combinations, droplet size, and temperature gradients are being analyzed and chosen on the basis of being capable of producing sizeable droplet motion to provide reasonable data in the 2.2-second time period. Available theory provides the models enabling these chosen fluid combinations. If reasonable data is not produced, efforts will be shifted to LeRC's Zero Gravity Facility, where a full 5.0-second microgravity drop period can be attained. Once the behavior of a single fluid droplet is reasonably well known and defined, multiple droplets in a single host fluid will be studied. Two host fluid systems with single and multiple droplets may also be considered for further research.

Morphological Stability and Fluid Dynamics of Vapor Crystal Growth

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Professor Franz Rosenberger
NSG-1534

This research is directed towards a fundamental understanding of the conditions under which crystals can retain morphological stability, i.e. shape stability of the advancing interface, during growth from vapors. Morphological stability (MS) is a necessary condition for the growth of homogeneous single crystals required for numerous device applications. For crystallization from melts, the MS concepts are well developed and are essentially based on heat and mass transfer conditions about the advancing interface. For crystallization from vapors, the MS requirements are more complex and not well understood. The added complexity arises from the fact that anisotropies in interfacial kinetics are typically stronger in crystallization from vapors than from melts. These pronounced anisotropies root in the distinctly lower atomic roughness of most vapor-solid interfaces.

The key insights obtained from this work are: (1) with the incompressibility assumption (uncoupling of Navier-Stokes and energy equation), traditionally made in materials processing fluid dynamics, much of the essential physics is lost in simulations of vapor crystal growth processes; (2) even under zero-g conditions, the mere viscous interaction of diffusion fluxes with container walls leads to nonuniform concentration distributions (which, in turn, can act morphologically destabilizing); consequently (c) on earth, buoyancy-driven convection is always present in closed ampoule systems, irrespective of heating geometry and orientation of the transport flux with respect to "g". Utilizing these insights, the mass and heat transport prevailing about crystals during their growth from vapors are being investigated numerically and experimentally. To facilitate the simultaneous determination of temperature and concentration gradients a novel refractometric technique, utilizing anomalous dispersion, is being developed. The macroscopic morphological evolution of the crystals will be recorded holographically, concurrent with the characterization of the transport conditions.

The interfacial kinetics aspects of MS in vapor growth are being addressed through microscopic studies of grown features and rates. Materials have been chosen, such as CBr_4 , that, depending on the growth temperature, exhibit atomically rough or atomically smooth interfaces. Thus we will be able to correlate the existing, isotropic MS models with the anisotropic model as it emerges from our results. In this part of the

work we have observed, for the first time, surface roughening to occur as a precursor to a solid-solid phase transition. In addition we have expanded the traditional statistical treatment of atomic surface roughness to include surface relaxation. The resulting model predictions agree well with experimental observations, in contrast to the rigid lattice models which fail to yield realistic predictions for vapor-solid systems.

This program is expected to provide sufficient insight into the diffusive-convective limitations of morphological stability in vapor crystal growth to ultimately warrant a purely diffusion-controlled benchmark experiment in space.

Publications

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Horizontal Cylinder?" Int. J. Heat Mass Transfer (submitted).

Automatic Processing of Reactive Polymers

Massachusetts Institute of Technology
Dr. David Roylance
NSG-7645

This project is aimed at exploiting a series of process modeling computer codes which were developed under a previous NASA-MPC project. The codes use finite element techniques to determine the time-dependent process parameters operative during nonisothermal reactive flows such as can occur in reaction injection molding or composites fabrication. In this present project, we are extending the use of these analytical codes to perform experimental control functions; since the models can determine the state of all variables everywhere in the system, they can be used in a manner similar to currently available experimental probes. To date, we have implemented a small but well instrumented reaction vessel in which fiber-reinforced plaques are cured using computer control and data acquisition. The finite element codes have also been extended to treat this particular process. In future phases of the work, we will extend our computer strategy to take more complete advantage of the finite element analysis codes, and we will develop the experimental facility to treat convective flows in which gravitational effects can be important.

Publications

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Thermocapillary and Diffusocapillary Migration of a Fluid Drop

University of Colorado

Dr. Robert L. Sani

NAG3-493 (NASA Contact: A.-T. Chai, LeRC)

November 1, 1984 - October 30, 1985

The migration of bubbles, or drops, plays an important role in many engineering science and space manufacturing problems. In material science processes as in the manufacturing of glasses, etc., gas bubbles can be formed from the by-products of chemical reactions or gas trapped in the interstices of the raw material. In the low-g environment of space, forces other than gravitational must be utilized as a bubble separation technique. It is well-known that gradients in interfacial tension on the bubbles' surface can promote droplet motion in the direction of decreasing interfacial tension and hence provide such a separation mechanism. Thus, the role of thermocapillary and diffusocapillary migration of a bubble, or drop, can be of paramount interest in materials processing in space.

Our ongoing research is focused on the quantitative investigation of such processes by analytical and numerical models. The initial phase of the study dealt with a thorough literature search for pertinent studies on thermocapillary and buoyancy driven bubble migration and an assessment of this literature. Computer algorithms for the available analytical solutions of interest (primarily the thermocapillary migration of a spherical bubble [or bubbles] in Stokes flow) have been developed so that effects of various physical parameters can be easily studied. Such existing analytical solutions are being extended to model concentration effects and in particular the effect of surface active material and to account for some convective transport effects. Concurrently, the development of a Galerkin-finite element algorithm for the discretization and numerical solution of axisymmetric bubble migration problems has been initiated so that significant nonlinear convective transport and surface deformation effects can be investigated.

Publications

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Transport Processes Research

Princeton University

Professor D. A. Saville

Professor W. B. Russel

NAG3-447 (NASA Contact: A. T. Chai and M. L. Millard, LeRC)

July 14, 1983 - July 14, 1985

The objective of this effort is to conduct fundamental research in reduced gravity on transport processes occurring during crystallization and/or during solidification processes. The details of the transport processes in various materials processing modes will be systematically studied. A Shuttle experiment will be planned to provide a better understanding of the role of fluid motion on the formation of morphological patterns and roles of these patterns in solid composition. The approach to the research will involve theoretical modeling of transport processes, non-incurative ground-based laboratory measurements of model systems, and the definition of requirements for space experimentation.

Electrohydrodynamics

Princeton University
Dr. Dudley A. Saville
NAG3-259 (NASA Contact: Dr. A-T. Chai, LeRC)

The purpose of this investigation is to develop and test (in a limited sense) models of electrohydrodynamic processes involving liquids with poorly ionized solutes at high (applied) field strengths. Extant theories which account for the details of physico-chemical processes associated with charged interfaces deal mainly with low field strengths and fully ionized solutes. The model used to describe processes at high field strengths--the leaky dielectric--omits consideration of electric double-layers, adsorption at interfaces, and chemical processes involved in the dissociation and recombination of solute species. Thus, even though the model depicts some features associated with bulk fluid motion, it fails to give a comprehensive picture. One example of the shortcomings of both the classical model of electrokinetics at low field strengths and the leaky-dielectric model for high field strengths is found in the field strength dependence of the mobility of small particles in apolar liquids. The research will provide a more general model of electrohydrodynamic phenomena capable of application in diverse circumstances.

Work on the $O(\beta)$ -problem is essentially complete, and a manuscript has been prepared for publication. As originally envisioned, this effort would furnish a theoretical basis for modeling electrokinetics with poorly ionized solutes. Although the task of constructing a comprehensive model to deal with the partial ionization characteristic of apolar liquids has succeeded, the extension to higher fields has been stalled due to unresolved mathematical difficulties. Specifically, the "inner problem," however formulated, is singular. Thus, although there is a formal solution for the region near the particle, it is as yet indeterminate because the solution to the so-called "outer problem" has eluded us. To resolve the difficulty turns out to be a substantial mathematical problem. One way of proceeding, is to restrict attention to situations with thin double-layers. Here, exponential decay of the solutions will enable us to avoid the singular region at the expense of a somewhat less general result. Accordingly, we have begun the formulation of the problem for fluid interfaces and set aside the intermediate steps for the present.

Binary Miscibility Gap Systems

National Bureau of Standards

Dr. L. A. Schmid

H27954B

April 1981 - continuing task

Immiscible droplets embedded in a host fluid in which a temperature gradient exists migrate toward the hot end of the host fluid because of the temperature dependence of the interfacial energy of the droplet. This thermocapillary migration effect has been exploited in the design of a controllable heat valve which is the thermal analog of the electronic vacuum triode. Studies have also been made of test cells that could use thermocapillary migration to facilitate the study of condensation and dissolution kinetics in miscibility-gap solutions.

Experimental and Theoretical Analysis of Chemical Vapor Deposition with Prediction of Gravity Effects

Aerodyne Research, Inc.

Dr. Charter D. Stinespring

Dr. Karl E. Spear, Penn State University

NAS3-23934 (NASA Contact: Dr. L. M. Anderson, LeRC)

September 29, 1983 - September 28, 1986

The overall objective of this program is to determine feasibility and general design of shuttle-borne experiments for assessing the effects of microgravity/gravity on CVD processes. To meet this objective, it is necessary to develop a quantitative understanding of gravitationally-induced transport processes in CVD systems and to determine the relationship between these transport processes and compositional and structural defects in CVD processes. Based on this knowledge, it will then be possible to recommend and interpret space-based microgravity experiments.

The approach in this program involves a combined experimental and theoretical study to characterize the effects of gravitationally-induced transport on atmospheric pressure silicon epitaxy by SiH_4 pyrolysis. Experimentally, flow regimes in which free convective transport contributes to the CVD process will be identified, and, for these conditions, the flow and deposition process will be characterized. Specifically, this will include measurements of three dimensional temperature variations using in situ Rayleigh scattering, gas phase composition profiles using laser absorption and fluorescence techniques, and deposition rates and defect densities. Subsequently, the free convective transport contribution to the CVD process will be minimized and/or altered while leaving deposition chemistry unaltered, and the characterization will be repeated. Based on these analyses, the effects of gravitationally-induced transport on atmospheric pressure CVD will be assessed.

The theoretical component of the program seeks to develop and validate a combined fluid dynamics/chemistry model of CVD. To accomplish this, the modeling effort has been divided into two components. First, a two-dimensional fluid dynamics code for developing flow with variable fluid properties (parabolic problem) and a 2-D fluid dynamics code for fully-developed flow with variable fluid properties (elliptic problem) will be set up. These will then provide input for the second component which is a mass transport model with equilibrium and rate controlled chemistry coupled with the fluid dynamics. The predictions of this model will be tested against experimental observations.

Publications

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Role of Convection in Grain Refining

Massachusetts Institute of Technology

Professor Julian Szekely

Professor Merton C. Flemings

Dr. N. El-Kaddah

Dr. Y. Shiohara

NSG-7645 (NASA Contact: Dr. Hugh Gray, LeRC)

October 1, 1984 - September 30, 1985

The programmed research has two major components. First, the research effort is aimed at developing a quantitative representation of the heat and fluid flow fields in electromagnetically levitated (positioned) metallic specimens. The work has the following components: (1) a mathematical model has been developed to represent the fluid flow field and the temperature field in a levitation melted specimen under both microgravity and earthbound conditions; (2) experimental measurements have been carried out to study turbulent electromagnetically-driven flow, as produced by the passage of DC current between two electrodes immersed into molten wood metal; (3) these experimental measurements have been compared with theoretical predictions, using both existing turbulence models and a model developed in the course of the present study, thus verifying the model; (4) the findings concerning turbulent electromagnetically driven flows have been broadly disseminated, regarding earthbound application, such as induction furnace design; and (5) the quantitative understanding of turbulent electromagnetically driven flows, which is being developed in this study, is being fully utilized in the design of a space shuttle experiment, which is now planned for early 1985.

Second, the research is comprised of continuation of experimental studies as well as modelling of those studies on undercooling and solidification of levitated metal droplets. Thermal measurements will be made of the undercooling and solidification of levitated metal droplets. Thermal measurements will be made of the undercooling and of the details of the ultra-rapid recalescence. This levitation technique combined with quenching will be used to aid in the development of coherent model for the solidification process related to structural studies, including thermodynamics and kinetics for nucleation and metastable solute partition, kinetics for solid/liquid interface velocity with high undercooling, and heat flow consideration.

These levitation experiments also relate directly to planned MIT-EML (Electro-Magnetic Levitation) Space Shuttle experiments planned for December 1984. This program proposed includes characterization and metallography work for the samples of the Space Shuttle experiments. The modelling work also includes the effects of microgravity on solidification with high undercooling, and the proposed ground-based levitation experimental results will be compared with the results of the Space Shuttle experiments.

Publications

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BIOTECHNOLOGY

Biochemical Assays of Cultured Cells

Michael Reese Research Foundation
Dr. Grant H. Barlow
NAS9-16389
October 1, 1982 - April 30, 1984

The purpose of this program is to perform biochemical assays on conditioned media from cultures originated from cell subpopulations obtained by ground and flight experiments using electrophoretic methodologies.

Subpopulations of human embryonic kidney cells isolated from continuous flow electrophoresis experiments performed at McDonnell Douglas and on STS-8 have been analyzed. These analyses have included plasminogen activator assays involving indirect methodology on fibrin plated and direct methodology using chromogenic substrates. Immunological studies have been performed and the conditioned media for erythropoietin activity and human granulocyte colony stimulating (HGCSF) activity has been analyzed.

The ground based experiments showed resolution of the human embryonic kidney cell into subpopulations as judged by the distribution of plasminogen activator activity. No separation of erythropoietin or HGCSF was evident. Preliminary results from STS-8 samples show an increased resolution over ground base experiments and evidence for an exceptionally high plasminogen activator peak not observed prior to this flight. These are indications that erythropoietin activity is concentrated in one area of the fractionation with the slower mobility cells. No HGCSF activity has been observed which, at this time, is unexplained.

Publications

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Hormone Purification by Isoelectric Focusing

University of Arizona

Dr. Milan Bier

NAS8-32950 (NASA Contact: V. Yost, MSFC)

This research involves four areas: (1) experimental characterization of Earth gravity electroosmosis effects on the Isoelectric Focusing (IEF) process; (2) computer simulation of low-gravity performance of various IEF apparatus configurations; (3) Space Shuttle Middeck flight experiments; and (4) computer optimization of pH gradients obtainable with chemically defined buffers in isoelectric focusing.

Various ground-based research approaches are being applied to a more definitive evaluation of the natures and degrees of electroosmosis effects on the separation capabilities of the Isoelectric Focusing (IEF) process. A primary instrumental system for this work involves rotationally stabilized, horizontal electrophoretic columns specially adapted for the IEF process. Representative adaptations include segmentation, baffles/screens, and surface coatings. Comparative performance and development testing are pursued against the type of column or cell established as an engineering model. Previously developed computer simulation capabilities are used to predict low-gravity behavior patterns and performance for IEF apparatus geometries of direct project interest. Three existing mathematical models plus potential new routines for particular aspects of simulating instrument fluid patterns with varied wall electroosmosis influences are being exercised.

In collaboration with Marshall Space Flight Center, the first middeck flight experiment has been carried out aboard STS-11. The apparatus performed as intended, without any malfunctions. The results were rather surprising, indicating a far more complex role of electroosmosis in IEF than anticipated. At present, a second flight experiment is being prepared for the middeck of STS-16, to be launched in August 1984. In addition, we have initiated the development of a continuous flow recycling IEF apparatus to be used in space. Specifically, this involves the complete definition, design, fabrication, assembly, testing, and delivery project activity sequence for a breadboard model of the space apparatus. This is again being done in cooperation with the Marshall Space Flight Center.

The optimization of the existing computer model of IEF processes continues to automatically adjust concentrations of all components so as to produce linear pH gradients. The pH gradient in a given buffer system is a function of the electrochemical parameters of all components (e.g., their disassociation constants and mobilities), their concentrations, and the applied currents. Various strategies for the optimization of the process are being pursued.

Publications

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Cell Partition in Two Polymer Aqueous Phases

Oregon Health Sciences University
Dr. Donald E. Brooks
NAS8-35333 (NASA Contact: V. Fogle, MSFC)
May 5, 1983 - May 4, 1986

The objectives of this research program are to develop and understand cell partition in a reduced gravity environment as a sensitive, analytical and high resolution preparative procedure for biomedical research.

In a reduced gravity environment the two polymer phases will not separate via density driven settling in an acceptably short length of time. It is to be expected that a certain amount of phase separation will take place, however, driven by the reduction in free energy gained when the interfacial area is reduced. This stage of separation process will therefore depend directly on the magnitude of the interfacial tension between the phases. In order to induce complete phase separation in a short time, we are investigating electric field-induced separation which occurs because the droplets of one phase in the other have high electrophoretic mobilities which increase with droplet size. These mobilities are significant only in the presence of certain salts, particularly phosphates. The presence of such salts, in turn, has a strong effect on the cell partition behavior in dextran-poly (ethylene glycol) (PEG) systems. The addition of the salts necessary to produce phase drop mobilities has a large effect on the interfacial tensions in the systems.

Studies have been carried out to determine the extent to which cell partition is thermodynamically controlled. Experiments on particle partition as a function of the interfacial tension of the phase boundary have shown that the partition coefficient depends exponentially on the tension, as predicted thermodynamically, but that a strong statistical component two to three orders of magnitude greater than thermal energies is superimposed on the process. The origin of this stochastic process presumably lies in the fluid mechanical disturbances generated when the phases coalesce and settle. Control over the rate of phase separation, such as that provided by field-drawn separation in the absence of gravity-driven settling, would allow this hypothesis to be tested. If the stochastic component could be reduced, the resolution of cell separations obtainable via successive partition steps (countercurrent distribution) would be dramatically enhanced.

Finally, a collaborative project with Dr. J. M. Harris has been carried out on the synthesis, characterization and application of PEG ligands useful in affinity partitioning. In particular a series of PEG alkyl esters and ethers were studied as these are probes of hydrophobic properties of cell surfaces useful in providing non-charge-dependent separations.

Publications

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Presentations

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Sharp, K. A., Evans, E. A., and Brooks, D. E., "Cell Partition and Surface Free Energies Related Through Contact Angles in Two-phase Polymer Systems," presented at Third International Conference on Partitioning in Two Polymer Systems, Vancouver, B.C., July 3-8, 1983.

A Study of Blood Flow and of Aggregation of Blood Cells Under Conditions of Zero Gravity - Its Relevance to the Occlusive Diseases and Cancer

University of Sydney, Department of Medicine
Dr. Leopold Dintenfass
MPS77F113

The objectives of this program are: (1) to determine whether the size of red cell aggregates, kinetics and morphology of these aggregates are influenced by near-zero gravity; (2) whether viscosity, especially at low shear rate, is afflicted by near-zero gravity (the latter preventing sedimentation of red cells); (3) whether the actual shape of red cells changes; and (4) whether blood samples obtained from different donors (normal and patients suffering from different disorders) react in the same manner to near-zero gravity. These objectives are to be explored in the experiments to be carried out on STS-19, planned for September 1984. Ground-based experiments will take place using identical blood samples. Eight Australian donors will be used, and twenty-four experiments will be carried out on each set of blood samples.

It is possible that such data, obtained under near zero-gravity, when compared with equivalent data and subsequent procedures could form the basis for diagnostic tests. These subsequent procedures would encompass the response of blood samples or aggregates of red cells to the addition of drugs or agents which have various, even opposite, effects on the aggregation of red cells. Such agents or drugs will include fibrinogen, glucose, triglycerides, snake venom derivatives (i.e., Ancrod), beta blockers, etc. The kinetics of aggregation or disaggregation will be studied in parallel with the viscosity of blood. The results of these tests with compounds at different concentrations may well prove to be distinctive for blood samples from patients suffering from different diseases. It is possible that patients suffering from the same disease might exhibit different responses (in blood rheology) when subgrouped according to their ABO blood groups.

Publications

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New Instrumentation for Phase Partitioning

University of Alabama in Huntsville
Dr. J. Milton Harris
NAS8-35334 (NASA Contact: V. Fogle, MSFC)
July 1, 1983 - June 31, 1986

Cells and molecules can be purified by partitioning between the two immiscible liquid phases formed by aqueous solutions of poly/ethylene glycol and dextran. Such purification can be more selective, higher yielding, and less destructive to sensitive biological materials than other available techniques. Earth's gravitational field is a hindering factor as it causes sedimentation of particles to be purified and shear-induced particle randomization. The present proposal is directed toward developing new instrumentation for performing phase partitioning both on Earth and in microgravity.

One aspect of the program has been a thorough testing of the Ito countercurrent chromatograph obtained from the National Institutes of Health. Examination of the Treffry countercurrent distribution device for automated earth-bound separations will begin. Work has also continued on developing apparatus for performing microgravity experiments. A prime focus of this work is control of the rate of phase separation by manipulating interactions between the polymers and coated container walls; coating methods are also under development. This apparatus will be used to take advantage of the new affinity polymers developed in our other contract.

Publications

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Presentations

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Cell Partition in Two Polymer Aqueous Phases

University of Alabama in Huntsville
Dr. J. Milton Harris
NAS8-35362 (NASA Contact: V. Fogle, MSFC)
August 1, 1983 - July 31, 1986

Partition of biological cells in two phase aqueous polymer systems is recognized as a powerful separation technique which is limited by gravity. A major need of the program is to greatly refine the sensitivity of the technique by developing new polymers that will exhibit a high, selective affinity for specific cell types and which will take advantage of the unique environment of space. Our interest is to synthesize new, selective polymer-ligand conjugates to be used in affinity partition separations.

The two most commonly used polymers in two phase partitioning are dextran and polyethylene glycol. We have begun a thorough review of the chemistry of these polymers, particularly in the area of protein attachment. Preliminary studies indicate the importance in affinity partitioning of minimizing gravity-induced randomizing forces in the phase separation process. The PEG-protein conjugates we are preparing appear to be ideally suited for achieving high quality purifications in a microgravity environment. An interesting spin-off of this synthetic work has been the observation of catalytic activity for certain of our polymer derivatives.

Publications

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Purification and Cultivation of Human Pituitary Growth Hormones Secreting Cells

Pennsylvania State University
Dr. Wesley C. Hymer
Dr. Paul Todd
Dr. Richard Grindeland
Wayne Lanham
Dr. Dennis Morrison, JSC
NAS9-15566

The rat and human pituitary gland contains a mixture of hormone producing cell types. We are attempting to separate cells which make growth hormone (GH) for the purpose of understanding how the hormone molecule is made within the pituitary cell; what form(s) it takes within the cell; and what form(s) GH assumes as it leaves the cell. Since GH has a number of biological targets (e.g., muscle, liver, bone), we are also assessing the activities of the intracellular/extracellular GH by new and sensitive bioassays.

To do this project, we initially try to separate GH cells contained in the mixture by free flow electrophoresis. Our most successful experiments are those which use the McDonnell Douglas continuous flow electrophoresis (CFES) device. These experiments show that subpopulations of GH cells reside within the pituitary; i.e., GH cells have different electrophoretic mobilities. Hormone within these subpopulations apparently exists in different physical forms (on the basis of retention times off HPLC columns) and the molecule must be in a certain form before it can be released from the cell. As yet we have no estimates of the biological activities of these different forms. We tentatively attribute the different cell mobilities to hormone on the cell surface; a phenomenon which is convenient for us experimentally, but one for which we as yet have no satisfactory explanation.

This project assumes relevance to NASA goals since a lack of GH could be a prime causative factor in muscle atrophy. Further, GH has recently been implicated in the etiology of motion sickness in space. Our CFES experiment on STS-8 showed that we could partially separate GH cells in microgravity. However, definitive cell culture studies could not be done due to insufficient cell recoveries. Nevertheless, data from the microgravity experiment strongly suggested that rat GH cells could not secrete hormone after they have been exposed to microgravity. This "lesion" appeared specific to the GH cells; it did not occur in the case of the PRL cell.

Upcoming tasks on this research project involve preparation (via ground-based studies) for another flight experiment to (1) definitively characterize the GH contained/released by subpopulations of cells after exposure to microgravity so that we can (2) establish the purported microgravity-induced GH secretory lesion with certainty.

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Cell Separations in Microgravity and Development of a Space Bioreactor

Johnson Space Center
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In-Center
January 1983 - continuing task

NASA has demonstrated the potential of microgravity for the improved processing of useful products. Among such products are biologicals with pharmaceutical value. The Bioprocessing program at JSC has utilized the McDonnell-Douglas Continuous Flow Electrophoresis System (CFES) to separate mixtures of human kidney cells into about 30 fractions. Several of these fractions produce much larger quantities of plasminogen activators than the other fractions. Plasminogen activators are useful in treating blood clot-produced diseases. The goal of the program is to demonstrate that useful cells from a variety of heterogeneous mixtures can be better separated and cultured in space under conditions which allow their biological products to be collected and purified. To achieve this goal, methods and equipment for culturing and handling cell cultures in microgravity are being developed.

For larger scale culture of cells and collection of biological products, microgravity offers significant promise. At 1-g, commercial culture of mammalian cells to produce pharmaceutical products is dramatically compromised by sedimentation and inadequate transfer of gaseous oxygen. The shear forces accompanying the stirring required to keep microcarriers (with cells attached) suspended and oxygenated can damage cells severely. U.S. and European flight experiments indicate possible increases in growth rate and cell size of eucaryotic cells grown under weightless conditions. Therefore, microgravity offers a potential for improved mass culture of mammalian cells. On the other hand, in microgravity, one cannot rely on head space (air above the liquid phase) to assist in the oxygen supply. A bioreactor optimized for operations in space is now being developed. The current research is focused on determining the optimum cell-to-bead ratios, medium content and proper maintenance conditions required to keep living cell specimens alive and healthy for the entire flight.

The bioreactor development project has recently added a microprocessor/computer system to the JSC prototype for control and data analysis. The effort has been extended to include research at two major universities which are developing state-of-the-art technology and equipment for commercial culture of cells to obtain pharmaceuticals. Appropriate new technology is being combined with the current bioreactor designs and tested to determine what specific features must be included in the fabrication of a bioreactor designed to operate for STS demonstration tests. Considerations include (1) circulation and resupply of culture media; (2) sensors required to monitor temperature, cell growth, mass transport, and oxygen consumption; and (3) inflight control of shear stress on cells, gas transfer in microgravity, diffusion, and intracellular transport. These data and results from the JSC prototype bioreactor test will be used for the design and construction of a small space bioreactor for the Orbiter middeck. The middeck bioreactor will consist of a small version of the

culture vessel and related control system. It will be certified for a flight verification test in 1986 to (1) demonstrate systems operation and improvements or limitations in microgravity, and (2) obtain baseline flight data for future full-scale flight experiments.

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Cell Maintenance Systems

Johnson Space Center
Dr. Dennis R. Morrison
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April 1984 - continuing task

Living human cells require attachment to a suitable surface and special culture conditions in order to grow. These requirements are modified and amplified when cells are taken into a weightless environment. Special handling and maintenance systems are required for routine laboratory procedures conducted in the Orbiter and in the Spacelab.

Methods have been developed to maintain cells in special incubators designed for the Orbiter middeck, however, electrophoresis and other experiments require cells to be harvested off of the culture substrate before they can be processed or used. The cell transport assembly (CTA) was flown on STS-8, and results show that improvements are required to maintain adequate numbers of cells in this device longer than 48 hours. In addition, harvested cells must be concentrated from wash fluids before they can be used or recultured. The life sciences middeck centrifuge probably can be used, but modifications will be required to transfer cells from the CTA and keep the cells sterile. The optimum inflight procedures and timelines must be determined, then incorporated into the design and construction of an advanced CTA which can be used to supply living cells for future CFES and other cell flight experiments.

In addition, after electrophoresis separation or other bioprocessing, the cell must reattach to a suitable surface before they can grow again. The cell attachment test (DSO-0432) on STS-8 indicated that microcarrier beads can be used if the cells and beads are incubated at 37°C. Microcarriers and the incubator were not used on STS-8 to maintain the cells for the 6-day mission. As a result, both kidney and pituitary cells returned from CFES experiments on STS-8 were only 20-30 percent viable. Better methods are clearly needed. The optimum culture experiments must be determined and then used to develop adequate inflight procedures. Automated systems such as the Skylab SO-15 flight hardware and crew operated systems are being evaluated for use on the Space Shuttle, Spacelab, and Space Station research modules.

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Mathematical Models of Continuous Flow Electrophoresis

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NAS8-32614 (NASA Contact: V. Fogle, MSFC)
August 1977 - June 1985

Development of high-resolution continuous flow electrophoresis devices ultimately requires comprehensive understanding of the ways various phenomena and processes facilitate or hinder separation. A comprehensive model of the actual three-dimensional flow, temperature and electric fields shall be developed to provide guidance in the design of electrophoresis chambers for specific tasks and means of interpreting test data on a given chamber.

Part of the process of model development includes experimental and theoretical studies of hydrodynamic stability. This is necessary to understand the origin of mixing flows observed with wide-gap gravitational effects; the suppression of gravity may allow other processes to become important.

To insure that the model accurately reflects the flow field and particle motion requires extensive experimental work. Much of the experimental work can be done under terrestrial conditions if the roles of gravity are appreciated and taken into account properly. Even though the resolution of a terrestrial-based machine may be unsatisfactory, verification of the model will provide the support necessary for the interpretation of microgravity operations. Recommendations will be made for the design and operations of the ground experiments.

Another part of the investigation is concerned with the behavior of concentrated sample suspensions with regard to sample stream stability, particle-particle interactions which might affect separation in an electric field, especially at high field strengths ($>100\text{v/cm}$). Mathematical models will be developed and tested to establish the roles of the various interactions.

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Electrophoresis Technology

Marshall Space Flight Center
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In-House

The objectives of this program are to: (1) analyze the fluid flow and particle motions during continuous flow electrophoresis by experimentation and computation; (2) characterize and optimize electrophoretic separators and their operational parameters; (3) develop innovative methods to accomplish electrophoretic separations in space; (4) investigate advantages of other electrokinetic separation processes such as isoelectric focusing and isotachopheresis; and (5) separate biological cells using apparatus that has been characterized or modified to perform in a predictable manner and according to procedures that have been developed to yield improved separation.

During the past year, a new high-resolution apparatus designed for space has been built as a laboratory prototype. Using a moving wall with a low zeta potential coating, the major sources of flow distortion for an electrophoretic sample stream are removed. Highly resolved fractions, however, will only be produced in space because of the sensitivity of this chamber to buoyancy-induced convection in the laboratory. However, preliminary laboratory experiments and analysis confirm important features of its operation.

The second and third flights of the McDonnell Douglas Astronautics Corporation continuous flow electrophoresis system carried samples developed at MSFC intended to evaluate the broad capabilities of free-flow electrophoresis in a reduced gravity environment. Biological model materials, hemoglobin and polystyrene latex microspheres, were selected because of their past use as electrophoresis standards and as visible markers for fluid flow due to electroosmosis, spacecraft acceleration or other factors. The specific objective of the experiments was to assess the dependence of the separation resolution on the properties of the sample and its suspension solution. Photographs of the space electrophoresis experiments, analysis of the collected fractions and recent laboratory experiments show the sensitivity of resolution to the electrical properties of the sample solution and electrophoresis curtain buffer.

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Kidney Cell Electrophoresis

The Pennsylvania State University
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NAS9-15584
June 1980 - continuing task

The objective of this project is to evaluate materials and procedures for microgravity electrophoresis of living human embryonic kidney cells, to provide ground support in the form of analytical cell electrophoresis and flow cytometry and to analyze cells returned from space flight.

In collaboration with D. R. Morrison, principal investigator, and M. L. Lewis of Johnson Space Center, and G. H. Barlow, co-investigator, of Michael Reese Research Foundation, pre-flight culture media, electrophoresis buffer, fraction collection media, temperature profiles, and urokinase assay procedures were tested prior to flight. Electrophoretic mobility distributions of aliquots of the cell population to be fractionated in flight were obtained. The following protocol was established and utilized:

Cells were prepared in suspension prior to flight in electrophoresis buffer and 10% calf serum. Electrophoretic separation proceeded in electrophoresis buffer without serum in the McDonnell Douglas Continuous Flow Electrophoresis Separator, and fractions were collected into sample bags containing culture medium and concentrated serum. Two separations were performed, and subsequent culturing and biochemical measurements were conducted at Johnson Space Center. Fractions that yielded enough progeny cells were analyzed at The Pennsylvania State University for morphology and electrophoretic mobility distributions. The mobility distributions of progeny cells cultured from four electrophoretic fractions showed that the lowest-mobility fraction studied produced higher-mobility progeny while the other fractions produced progeny cells with mobilities related to the fractions from which they were collected.

Production of Large-Particle-Size Monodisperse Latexes in Microgravity

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NAS8-32951 (NASA Contact: V. Yost, MSFC)
February 1978 - March 1986

The purpose of this project is to produce large-particle-size monodisperse polystyrene latexes in microgravity in sizes larger and more uniform than can be manufactured on Earth.

A latex is a suspension of very tiny (micrometer-size) plastic spheres in water, stabilized by emulsifiers. The objective of this experiment is to grow billions of these tiny plastic spheres, or balls, to sizes larger than can be grown on Earth, while keeping all of them exactly the same size and perfectly spherical. Thus far on several of the Monodisperse Latex Reactor (MLR) flights, the latex spheres have been returned to Earth with standard deviations of better than 1.4%. The little balls making up these latexes can only be grown in quantity on Earth up to about 5 micrometers in diameter while staying monodisperse because of buoyancy and sedimentation effects. They cannot be stirred sufficiently to keep them in suspension during polymerization because stirring causes shear-induced coagulation which destroys the latex. But in microgravity the absence of buoyancy effects has allowed growth of the balls up to 30 micrometers in diameter thus far (during the last flight on STS-11). The MLR has now flown 5 times on the Shuttle, during Missions STS-3, 4, 6, 7, and 11, and three more flights are presently scheduled, to be completed by mid-1985.

This experiment, the MLR, has now produced the first commercial space product; that is the first commercial material ever manufactured in space and marketed on Earth. The 10 micrometer latex manufactured on STS-6 was officially accepted by the US National Bureau of Standard in July 1984, and they plan to market it to reseachers as the United States National 10 micrometer (10 μ m) Standard Reference Material. NBS has also officially requested that NASA produce for them 30 grams of 30 micrometer latex and 80 grams of 100 micrometer latex which they plan to put on the market. It is hoped than this can be accomplished by the end of the next three flights, that is by mid 1985.

Once it is demonstrated that these large-size-monodisperse latexes can be routinely produced in quantity and quality, they can be marketed for many types of scientific applications. They can be used in biomedical research for such things as drug carriers and tracers in the body, human and animal blood flow studies, membrane and pore-sizing in the body, and medical diagnostic tests. Other applications include their use as calibration standards for optical and electron microscopes, Coulter Counters, light-scattering equipment, and many other types of laboratory equipment.

Publications

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GLASS AND CERAMICS

Colloidal Oxide Properties and Kinetic Studies

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NSG-7645

Silica based oxide particles prepared by various methods are compared in terms of their gelation kinetics, densification kinetics, and microstructures. Recent work has shown by scattering techniques that discernable structural evolutions begin at very low temperature. Additional work reported in literature further suggested that the colloidal behaviors and the dried gel structures of the same oxide from different preparation techniques differ fundamentally. These differences significantly affect the densification kinetics and the product microstructure. Extension of this work to mixtures of oxides from different preparation techniques is pursued, with the objective of optimizing microstructures and processing kinetics.

Gravitational segregation of particles of different sizes in a gravitational field is modelled as a diffusion/segregation problem. A structural free-volume model was used to describe the "activation" process of jump statistics. The sizes of the particles, the interstices, and their packing topology are accounted for in the estimation of the free volume. The gravitational field provides a drift force for the segregation. The limit of high concentration is treated as a multilayer segregation problem, for which an "isotherm" of the BET type is derived. Lastly, an attempt frequency is ascribed to particles to account for the vibrational rearrangement during powder packing.

Containerless Processing of Glass Forming Melts in Space

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NAS8-34758 (NASA Contact: V. Fogle, MSFC)
April 1983 - April 1986

The major objectives of this work are to (1) obtain quantitative evidence for the suppression of heterogeneous nucleation/crystallization in containerless melts in micro-g, (2) study melt homogenization in the absence of gravity driven convection, (3) develop the procedures for preparing precursor samples suitable for flight experiments, (4) perform comparative property analysis of glasses melted on earth and in micro-g, (5) determine the feasibility of preparing glass shells in micro-g for use as laser fusion targets, and (6) assess the operational performance of the single axis acoustic levitator/furnace apparatus for processing multi-component, glass-forming melts in micro-g.

If the heterogeneous nucleation/crystallization of a melt is suppressed by containerless melting, then its critical cooling rate (R_c) for glass formation in micro-g will be less than its R_c on earth. The practical consequence of a smaller R_c for glass formation in micro-g is an extension of the compositional limits for glass formation and the possibility of obtaining new glasses by melting in micro-g. For samples returning as glass after containerless melting in micro-g, the ratio of R_c on earth to R_c for micro-g will serve as a quantitative measure of the degree to which glass formation is enhanced, or heterogeneous nucleation is suppressed. Binary calcia-gallia and lead-silicate and ternary calcia-gallia-silica compositions possessing different critical cooling rates will be heated, melted, and quenched in an acoustic levitator/furnace for the MEA/A2 experiment. A wide range of physical, optical, thermal, and mechanical properties will be measured for glasses made in micro-g for comparison with the same properties of glasses made on earth. A borosilicate glass sphere containing an irregularly shaped air bubble will be remelted in micro-g in order to examine the feasibility of producing glass shells of thin uniform wall thickness. Melt homogenization in the convection-free environment of micro-g will be investigated by observing the level of chemical homogeneity achieved in melts made from deliberately inhomogeneous precursor samples.

An important practical task is to determine the suitability of using hot pressed precursor samples for containerless melting experiments in micro-g. Hot pressing has the advantage of being a relatively simple way of preparing precursor samples without chemical contamination from a container. The degree of chemical inhomogeneity that can be tolerated in a hot pressed precursor while still yielding a chemically homogeneous multi-component melt within a reasonable time in micro-g is being determined.

Publications

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Crystallization, Optical, and Chemical Properties of Fluoride Glasses

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JPL-955870

The objectives of this program are to: (1) cooperate with JPL in constructing an ACES apparatus and to prepare samples and experiments for space flight; (2) measure properties needed for the flight; and (3) examine the nucleation and crystallization kinetics and other properties of fluoride glasses on the ground, so these properties can be understood and compared with containerless behavior.

Fluoride glasses have great promise as infrared optical components, especially fibers, because they are transparent to $8\mu\text{m}$ and higher. In order to optimize properties, different glass compositions are needed. Some are hard to form in a container, and may possibly be formable in a containerless furnace. Understanding of crystallization with and without a container could lead to glasses with optimum properties. Chemical durability (attack by water) can limit or extend the applicability of fluoride glasses.

Progress to date includes; (1) melting and casting of fluoride glasses for ground studies and for flight STS-11; (2) measurement of crystallization kinetics with the DSC; (3) examination of microstructure of crystallized sample; (4) cooperation with JPL in building the ACES apparatus; and (5) post flight analysis of video tape, temperatures, and sample microstructure from ACES experiment on STS-11. A report has been prepared.

Publications

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Gel Precursors as Glass and Ceramic Starting Materials for Space Processing Applications Research

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NAS7-100
December 1982 - December 1984

Our overall program objective is to prepare starting materials by the metal organic-derived gel route, for investigations of containerless processing in space. Containerless processing allows no stirring of a melt, thus the starting materials must be compositionally homogeneous. The metal-organic gel route has been used for compositionally homogeneous preparations of glass and ceramics. We are determining its utility in two systems, a sodium tungstate system and a calcium tantalate system. The sodium tungstate system is used to model a low melting oxide and the calcium tantalate system is used to model a high melting oxide.

The technical issues to be solved are the preparation of the gel and the processing of the gel. Gel preparation involves not only determining how or whether a gel can be made but also the compositional range that can be handled through the gel route. Gel processing is the conversion of the gel to a glass or oxide with the complete removal of residual organics and gas bubbles while ensuring compositional homogeneity.

Our approach is to first prepare gels over a wide compositional range. The gels are dried and characterized for chemical and physical properties. The gels are then treated to remove residual organics, primarily by gas phase hydrolysis. Pyrolysis is then used for conversion to glass or ceramic products. These products are also characterized by chemical and physical property measurements and compared to inorganic derived ceramics of identical composition.

In the past year we have investigated the sodium tungstate system. We were not able to make gels but instead produced metal-organic-derived powders for further experiments. Hydrolysis and pyrolysis treatments were used to convert the powders to sodium tungstate ceramics. The metal-organic route was not found to be superior to an inorganic route for this system. However an interesting result was the accidental production of hollow glass shells by containerless pyrolysis of the gels in a high temperature drop tower furnace.

Currently we are examining the calcium tantalate system. We have prepared metal-organic derived gels in the compositional range of from 0 to 0.5. The gel-to-glass or gel-to-ceramic conversion will be the object of our research for the rest of the year.

Glass Formation in Reluctant Glass Formers

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In-House

The purpose of this work is to investigate the crystallization kinetics and glass forming ability of reluctant glass formers. This could ultimately aid the formation of bulk samples of unique glass compositions outside of normal glass forming regions allowing the optimization of certain properties of the glass.

One important aspect of processing in space is the containerless undercooling of molten substances. Theoretically, the extent of undercooling can be greatly enhanced by solidifying in the absence of heterogeneous nucleation resulting from contact with crucibles or molds. The containerless solidification of reluctant glass formers may permit much slower cooling rates to form glasses than is otherwise required.

This work has concentrated on establishing techniques for the measurement of crystallization kinetics and critical cooling rates. The glass formation ability and crystallization kinetics of $\text{Ga}_2\text{O}-43\text{CaO}$ and several $\text{Al}_2\text{O}_3-\text{CaO}$ compositions have been measured. An apparatus has been set up to measure the temperature of spherical samples on a thermocouple at large cooling rates. The time and temperature of nucleation is recorded and the probability of nucleation at various cooling rates can be measured. From this curve, it has been defined that the critical cooling rate to form a glass is that cooling rate at which there is a 95% probability of forming a glass.

Other techniques being developed include superimposing processing variable (temperature) from a fast data acquisition system onto the video from the microscopic examination of sample on a strip heater furnace. From this apparatus, crystal growth rates at various undercooled temperatures will be measured.

Another apparatus under development is an airjet levitator with heating from a 700 watt CO_2 laser. This has been used to levitate molten Al_2O_3 to temperatures as high as 2800° and resolidify it containerlessly. The plan is to utilize this apparatus to investigate the limit of undercooling in the absence of heterogeneous nucleation.

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Rapid Solidification of Magnetic Oxides

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NSG-7645

This research attempts to exploit the enhanced control over micro-structural evolution inherent in rapid solidification processing techniques to create novel ceramic magnetic materials. In addition, the great sensitivity of magnetic properties to local structure provides a powerful probe both for the study of structure and of microscopic solidification mechanisms.

The first system to be studied in this program is the $\text{SrO-Fe}_2\text{O}_3$ binary, which contains the commercially important hard magnetic compound strontium hexaferrite. Eutectic melts have been quenched in a single wheel melt spinner. The products have been analyzed by transmission electron microscopy, Mössbauer spectroscopy, magnetic measurements, and differential thermal analysis.

As-quenched ribbons contain high concentrations of super-paramagnetic particles, 80-250 Angstroms in diameter, in a glassy matrix. This suggests the possibility of crystallizing monodomain (less than 0.8 micron diameter) strontium hexaferrite during subsequent heat treatment, with a resulting increase in coercivity over conventionally processed ferrite magnets. A correspondence has also been observed between magnetic properties, bulk morphology, and thermal history of the quenched material.

This work has demonstrated that magnetic properties can be controlled in solidification processing by varying the quench rate.

Sol-Gel Glasses

Jet Propulsion Laboratory
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June 1982 - continuing task

Multicomponent homogeneous, ultrapure noncrystalline gels/gel-derived glasses are promising batch materials for the containerless glass melting experiments in microgravity. Hence, ultrapure, homogeneous gel precursors could be used to (a) investigate the effect of the container induced nucleation on the glass forming ability of marginally glass forming compositions, (b) investigate the influence of gravity on the phase separation and coarsening behavior of gel-derived glasses in the liquid-liquid immiscibility zone of the non-silicate systems having a high density phase. Because of the molecular scale homogeneity, the gel-derived glass produces extremely fine scale uniform droplets, the coarsening study of the heavy droplets in low viscosity glass matrix on earth and in microgravity will show the applicability or deviation of present theories of coarsening, (c) prepare ultrapure glasses by the containerless melting of noncrystalline ultrapure gels/gel-monoliths.

In the context of the advantages of the containerless melting of gels for the preparation of glasses, the objectives of the present programs are to study the gel formations and homogeneity of gels in the following systems that are important for near infrared and infrared optical communication technology: (i) $\text{SiO}_2\text{-GeO}_2$, (ii) $\text{GeO}_2\text{-PbO/Bi}_2\text{O}_3$. The $\text{SiO}_2\text{-GeO}_3$ system is selected as a model system for the scientific understanding of the physiochemical principles of gel processing controlling the molecular scale distribution of a second component in multicomponent network forming oxide gels. The crystallization tendency and container induced contamination due to high reactivities are obstacles for studying the intrinsic glass formation ability of high $\text{Bi}_2\text{O}_3/\text{PbO}$ containing glasses. Hence, the intrinsic glass formation ability and the role of container induced nucleation will be investigated by the container melting of homogeneous gels in these systems.

The structure and crystallization behavior of gels in the $\text{SiO}_2\text{-GeO}_2$ as a function of gel chemistry and thermal treatment have been investigated. The chemical principles involved in the distribution of a second network former in silica gel matrix being investigated. The procedures for synthesizing noncrystalline gels/gel-monoliths in the $\text{SiO}_2\text{-GeO}_2$, $\text{GeO}_2\text{-PbO}$ systems have been developed. Preliminary investigations on the levitation and thermal treatment of germania silicate gel-monoliths in the Pressure Facility Acoustic Levitator have been done.

Publications

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Bulk Formation of Metallic Glasses and Amorphous Silicon from the Melt

Harvard University
Dr. Frans Spaepen
NAS8-35416 (NASA Contact: L. Gardner, MSFC)
May 16, 1983 - May 15, 1986

The objectives of the research are: (1) to find new procedures and compositions for producing metallic glasses in bulk at slow cooling rates; (2) to attempt to form the amorphous phase of the tetrahedrally coordinated elements (Si or Ge) by undercooling the melt; and (3) to examine the crystal nucleation behavior of pure liquids and glass formers experimentally and theoretically.

By exposing a metallic glass forming melt for an extended period of time to a surrounding molten oxide flux, heterogeneous nucleants (primarily at the surface) can be eliminated. By using metallic glass compositions with a high relative glass transition temperature ($T_g/T_l > 0.6$), such as $Pd_{40}Ni_{40}P_{20}$, homogeneous nucleation also becomes negligible. We have succeeded in obtaining large (5g) masses of this alloy, using a molten B_2O_3 flux. This procedure is more effective and more reproducible than the vacuum treatments that were developed earlier. Presently, bulk glass formation in iron-based glasses is being investigated. It is expected that if an undercooling of about 250K can be achieved in a Ge or Si melt, formation of the amorphous semiconductor phase (rather than the crystal) may be kinetically favored. We are exploring this in experiments in a 10 foot drop tube, and using fluxes. We are investigating the volumetric behavior of undercooled liquid Ga droplet dispersion by dilatometry. There are indications that the oxide coating on the droplets leads to hydrostatic tension in the liquid, which in turn effects the crystallization thermodynamics.

We have developed a theoretical model (both analytical and numerical) for transient nucleation in glass forming melts. Recently, the model, originally designed for isothermal conditions, has been extended to continuous quenching. It is being applied to glass formation in various metallic and oxide systems. A further refinement will be the inclusion of diffusion-controlled interfacial rearrangements governing the growth of the crystal embryos.

Publications

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Physical Phenomena in Containerless Glass Processing

Clarkson College

Dr. R. S. Subramanian

Dr. Robert Cole

NAS8-32944 (NASA Contact: V. Fogle, MSFC)

December 1977 - December 1985

The objective of this work is to study the behavior of gas bubbles inside drops of model fluids and molten glasses in free fall, focusing on their migration and interaction. Such migration will be induced by thermocapillarity, rotation and/or oscillation of the drop, and other means. The results of the experiment are expected to be of use in the development of techniques for mixing and fining glasses in space and in providing a better understanding of how microballoons are formed.

A broad ground-based investigation into the various physical phenomena of importance in the space experiments is under way. Theoretical models of thermocapillary flow in drops, thermal migration of bubbles and droplets, the motion of bubbles inside drops, and the migration of bubbles in rotating liquid bodies are being developed. Experiments have been conducted on the migration of bubbles and droplets to the axis of a rotating liquid body, and the rise of bubbles in molten glass. Also, experiments on thermocapillary motion in silicone oils as well as glass melts have been performed. Experiments are currently being conducted on the migration of bubbles in a thermal gradient, and on their motion inside unconstrained liquid drops in a rotating liquid.

Experiments on bubble and droplet behavior in liquids under low gravity conditions aboard NASA KC-135 aircraft are under way. Space Shuttle experiments on bubble behavior inside a molten glass drop, as well as on the behavior of bubbles inside drops at ambient temperatures are scheduled for flight in 1984.

Publications

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Fusion Target Technology

Jet Propulsion Laboratory
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In-Center
October 1979 - continuing task

The long range objectives of this task are to (1) study the dynamics of liquid bubbles and of the gravitational effects relevant to the production of spherical shells both in the laboratory and in a weightless environment; (2) develop the technology that is pertinent to the production of metallic and metallic glass shells of various dimensions and aspect ratios; (3) develop and construct high temperature and high cooling rate facilities that are needed to produce refractory metallic and metallic glass spheres, and (4) develop technology applicable to the production of a novel high-strength low-weight material by bonding of the spheres.

In order to produce the high-quality spherical shells that are required, three parameters must be controlled accurately: the shell dimensions, shell sphericity and concentricity, and the surface topology of the shell. The present shells fabrication techniques are not set up to study the fundamental physical processes which control those parameters separately. Attempts to conduct experiments on the dynamics of liquid bubbles (molten shells) in laboratories are limited by a strong coupling among the three parameters, time, gravity, and temperature. The work described here will circumvent these limitations and enable detailed study of each of the important processes through use of low gravity environments collectively available in drop towers, in KC-135 flights, in a neutrally buoyant immiscible system, and in an acoustic levitation system.

Publications

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Presentations

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Glass Research

Jet Propulsion Laboratory
Dr. Michael C. Weinberg

Research efforts span three general areas of glass science: glass refining, gel-derived glasses, and nucleation and crystallization of glasses. Gas bubbles which are present in a glass product are defects which may render the glass totally useless for the end application. For example, optical glasses, laser host glasses, and a variety of other specialty glasses must be prepared virtually defect free to be employable. Since a major mechanism of bubble removal, buoyant rise, is virtually inoperative in microgravity, glass fining will be especially difficult in space. On the other hand, the suppression of buoyant rise and the ability to perform containerless melting experiments in space allows the opportunity to carry out several unique bubble experiments in space. Gas bubble dissolution studies may be performed at elevated temperatures for large bubbles with negligible bubble motion. Also, bubble nucleation studies may be performed without the disturbing feature of heterogeneous bubble nucleation at the platinum walls. Currently, ground based research efforts are being performed in support of these potential flight experiments.

Gel precursors can be used to prepare unique metastable glasses and glass ceramics. They offer several special advantages for glass preparation in a containerless experiment in space in that they can be prepared as amorphous, homogeneous materials in an ultrapure state. However, there is evidence that the structure and phase transformation behavior of these materials may be different from the corresponding glasses prepared by conventional glasses. Since these differences can adversely affect optical, mechanical, and other properties, there is need to explore the extent and origins of these differences. Our ground-based research efforts have aimed to elucidate in which systems such differences may be expected to occur and their causes.

The ability to prepare inorganic oxide glasses may be limited by the tendency of the melt to heterogeneously nucleate and crystallize from the container wall. Also, the ability to study free surface crystallization may be impaired by crystallization events initiating at the melt-container interface. In space such events may be avoided through the use of containerless processing. We have aimed to seek simple systems which exhibit a tendency to crystallize at the melt-container interface and to study their ground based crystallization behavior.

Publications

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Oxidation Behavior of Glassy Alloys

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NSG-7645

The objective of this program of research is to investigate the oxidation behavior of high-temperature glassy alloys produced by rapid solidification processing and to study the effects of processing and composition on oxidation behavior. Glassy Ta-44.5at%Ir, Ta-40at%Ir-10at%B and Nb-45at%Ir oxidized rapidly at 700-800°C at an oxygen partial pressure of 10^{-3} atm. The alloys were embrittled during the oxidation process. No apparent oxidation or embrittlement of the Ta-Ir alloy occurred after oxidation for 4h at 500 C at an oxygen partial pressure of 10^{-3} atm. Embrittlement occurred, however, after 100h of exposure under the latter conditions. Alloy embrittlement is associated with the partial or full conversion of the metallic glass to a mixture of crystalline β -Ta₂O₅ and metallic iridium. Hot compaction of glassy alloys of this type must be limited to relatively low temperatures (~500 C) and short times at the low temperatures unless extremely low oxygen partial pressures can be achieved during the compaction process.

COMBUSTION SCIENCE

Solid Surface Combustion at Reduced Gravity

University of Kentucky
Dr. Robert A. Altenkirch
(NASA Contact: Dr. Kurt Sacksteder, LeRC)
December 19, 1984 - December 30, 1985

The spread of a flame in the gas over the surface of a solid combustible involves in an essential way the transfer of heat from the flame to the solid fuel immediately ahead of it. This heat transfer is affected by the character of the gas-phase flame, and so the phenomenon of flame spreading under reduced gravity, in which the flow is generated by gasification of the solid combustible, is apt to be different from what occurs under the Earth's normal gravitational acceleration where the flow is largely buoyancy driven.

An experiment is being designed for the Middeck of the Space Shuttle to aid us in understanding the process of flame spreading in the absence of a buoyancy-driven flow. A chamber approximately 0.035m^3 in volume is to contain either a thin sample of a cellulosic material or a thick sample of polymethyl-methacrylate and an oxidizing environment of O_2 and N_2 . Samples will be ignited at one end, and the ensuing flame spread will be filmed. The spread rate can be determined from the films, and surface and gas-phase temperatures just above the surface will also be recorded. A matrix of eight experiments to be carried out on the Middeck has been identified. These data will help to clarify the mechanism of forward heat transfer in the low gravity flames.

The experimental apparatus is being constructed at NASA's Lewis Research Center and will be tested there, possibly in the Lewis Drop Tower Facilities, and at the University of Kentucky. In addition, methods of data reduction will be developed, and theoretical analysis of the reduced gravity, flame spread problem will be carried out.

Publications

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Particle Cloud Combustion Experiment

Energy Center, University of California, San Diego

Dr. A. L. Berlad

NAG3-381 (NASA Contact: Dr. Kurt Sacksteder, LeRC)

April 1983 - April 1984

Preparation of flight experiment designs is supported by experimental studies of acoustically induced mixing process, optical transmissivities of particle cloud distributions, wall saturation effects and their control through the use of electrically neutral flame tube materials and surfaces, and the pyrolysis-vaporization kinetics of selected organic particulates. Drop tower tests of stabilized particle cloud flames have allowed valuable comparison of $g = 0$ and $g = 1$ (upwards and downwards) stabilized flame propagation. These stabilized flame data will be valuable assists in dealing with the freely propagating particle cloud flame data anticipated through Space Shuttle experimentation.

Supporting theoretical studies emphasize comprehensive flame propagation and extinction relations among premixed single phase (gaseous) flames and premixed particle cloud flames, for both stabilized and freely propagating flames.

Publications

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Buoyancy Effects Upon Vapor Flame and Explosion Processes

Science Applications, Inc.

Dr. Raymond B. Edelman

Dr. Philip T. Harsha

NAS3-22822 (NASA Contact: Dr. Kurt Sacksteder, LeRC)

November 1984 - November 1985

The overall objective of this microgravity project is to develop an experimental and theoretical analyses critical to the understanding of the coupling of buoyancy and turbulence generation and its effect on fuel-air mixing, flame intensity and flame propagation in jet diffusion flames. Collateral objectives include developing information relevant to the control of unconfined fires in outer space and on earth as well as providing information needed to support the development of advanced low momentum burners and combustion chambers for industrial applications.

Current research is on the Phase II feasibility studies. An experiment for ground based investigations has been developed and is being instrumented to provide data to both validate theoretical analyses and to help in the preliminary design of the actual Space Laboratory experiment. The experiment is designed to examine certain effects of buoyancy acting on a diffusion flame in which the flame is directed either upward or downward. This change from negative to positive "g" is observed to significantly alter the flame shape although all other operating conditions are the same for both configurations. However, to perform this experiment a significant coaxial secondary air flow is needed in order to prevent flow reversal when the flame is inverted. The theoretical analysis that has been developed handles the secondary air flow and the extreme change in gravity vector direction. Thus the data will provide a measure of credibility of the analysis which will then be used to assist in the design of the actual zero-g experiment. In addition, the current experiment is being used to establish instrumentation compatible with Space Laboratory capabilities and constraints. As a consequence of the research conducted to date, an analysis of data obtained from a spray diffusion flame was carried out. It was found that certain observations could only be explained if buoyancy effects are included in the analysis.

Publications

Harsha, P. T. and Edelman, R. B., "Analytical Modeling of a Spray Diffusion Flame," AIAA Journal, 1984 (submitted).

A Fundamental Study of Smoldering with Emphasis on Experimental Design for Zero-G

University of California, Berkeley
Professor Patrick J. Pagni
Professor A. Carlos Fernandez-Pello
NAG3-443 (NASA Contact: Dr. Tom Labus, LeRC)
October 1, 1984 - September 30, 1985

The objective of this project is to identify key sets of low-gravity experiments which would critically compliment a larger set of more easily performed normal-gravity experiments to elucidate the phenomena found in smoldering combustion.

It is planned to follow through on the conceptual design of these experiments by participating in the future in the fabrication of the refined apparatus and in the actual data collection and interpretation. Low-gravity experiments are appropriate for smoldering combustion because of the complexity of smoldering which requires every means possible to discriminate among the many chemical and physical mechanisms operative in most smoldering combustion scenarios. Efforts will be primarily analytical, attempting to identify appropriate approximations and dominant dimensionless groups based on existing data and state-of-the-art combustion modelling. Transient stability questions such as ignition, extinction and the choices among charring, tarring, or flaming modes will be included.

Ignition and Flame Spread Above Liquid Fuel Pools

Carnegie-Mellon University

Professor William A. Sirignano

NAG3-404 (NASA Contact: Dr. Kurt Sacksteder, LeRC)

March 1, 1985 - December 1, 1985

The main objectives of this program are to study phenomena of ignition and flame spread above liquid fuel pools, and to determine factors that can improve fire safety. Dominant mechanisms for convective heat transfer and impact on ignition and flame spread above liquid fuel pools will be determined from these efforts.

The approach to be used will be: (1) analytical and computational studies to evaluate scaling factors; (2) experimental design, development, and operation in laboratory at Earth's gravity; (3) experimental design for drop towers at NASA Lewis Research Center; (4) development and operation of drop tower experiments together with NASA Lewis Research personnel and; (5) develop recommendations for Learjet and/or Space Shuttle experiments. Through this research, a mathematical model of two-phase systems has been formulated and is being coded for the computer.

Flammability Limits of Gases Under Low Gravity Conditions

University of Illinois-Urbana

Dr. Roger A. Strehlow

NAS3-23770 (NASA Contact: Dr. Kurt Sacksteder, LeRC)

December 24, 1984 - December 23, 1985

The purpose of this combustion science investigation is to determine the effect of zero gravity on the flammability limits of a premixed methane air flame in a standard 51 mm diameter flammability tube and to determine, if possible, the fluid flow associated with flame passage under zero-g conditions and the density (and hence, temperature) profiles associated with the flame under conditions of incipient extinction.

The purpose of the current phase of the study is to determine the lean flammability limit and the behavior of the flame near the limit for a methane-air mixture using a standard flammability tube in the vertical position under conditions of zero, fractional, and super gravity. This will be accomplished by constructing an appropriate apparatus for placement in NASA's Lewis Research Center Lear Jet facility and flying the prescribed g-trajectories while the experiment is being performed. Data will be recorded photographically using the visible light of the flame. The data that is to be acquired is (1) the shape and propagation velocity of the flame under various g-conditions for methane compositions that are inside the flammable limits and (2) the effect of gravity on the limits. Additionally, if time permits, the effect of varying gravity during flame propagation will be investigated. Real time accelerometer readings for the three orthogonal directions will be displayed in full view of the cameras and the framing rate of the cameras will be used to measure velocities. It is anticipated that fifteen flights will be required and that each of these will have at least six usable constant g-trajectories for experimentation.

At the present time a rig that contains eight parallel flammability tubes on a rotating mount that allow each of the tubes to be placed in the firing position sequentially has been constructed, tested, and approved by the safety committee at NASA Lewis Lab for installation and operation in the Lear Jet facility. This rig has been thoroughly ground tested and the technique of simultaneously photographing the flames and the output of three orthogonal "g" meters has been shown to be viable. The Lear Jet facility has been used to fly zero-g trajectories without the rig on it. It is anticipated that very soon experimental data taking will begin using the rig in the Lear Jet facility.

Publications

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Fundamental Studies of Droplet Combustion at Reduced Gravity

Princeton University

Professor Forman A. Williams

NAG3-380 (NASA Contact: Dr. Kurt Sacksteder, LeRC)

December 23, 1983 - December 22, 1984

The primary objective of this program is to ascertain how best to make use of reduced gravity to carry out scientific investigations of droplet combustion. In earlier work a preliminary conceptual design had been developed for droplet burning experiments for Spacelab. The present project concerns refinement of the earlier work with special consideration given to possible experiments for Mid-Deck Modules of the Space Shuttle.

The program involves a re-evaluation of suitable experiments on droplet combustion to ascertain whether influences of reduced buoyancy on time-dependent processes of heat and mass transfer in the gas or in the liquid on extinction processes or on disruptive burning phenomena are best suited for further investigation. Components of the experimental apparatus, which include a droplet dispensing system, a droplet positioning system, a droplet ignition systems and provision for recording, primarily photographically, the combustion of the individual droplet in a chamber having a controlled gas atmosphere, are being studied in an effort to determine optimal approaches to the experimental design. Methods for data reduction and interpretation are being made more specific, particularly in the context of an objective to calculate overall gas-phase chemical-kinetic parameters for the combustion from observations of extinction conditions in different atmospheres. The work involves ongoing cooperative studies being performed at the NASA Lewis Research Center.

EXPERIMENTAL TECHNOLOGY

Development of Materials Processing Systems for Use in Space on on Low-g Simulation Devices

Marshall Space Flight Center
B. R. Aldrich
W. D. Whitt
In-House
Continuing Task

The objective of this program is to continue to advance the state-of-the-art of materials processing systems for use in space. Materials processing systems will be designed for specific experiment requirements, prototype hardware built and tested to assure that all of the principal investigators requirements are met. Upon successful ground testing using the prototype system, flight hardware will be built.

Advanced furnace systems are continuing to be developed for use in space. Exciting systems are being tested for current experiment applications and modified for future experiment requirements.

Future projects that will be started are: (1) fabrication and testing of the Advanced Automated Directional Solidification Furnace (AADSF) flight hardware; (2) development of a Heat Pipe Furnace (HPF) for use in space. Heat pipes will be tested for space flight qualification in conjunction with the furnace development. The HPF design will be based on the AADSF development and will be of modular design including capabilities of operating with or without heat pipes; and (3) the AADSF furnace will be modified and tested to operate at temperatures up to 1700°C in the heated cavity. This will be accomplished by developing a new hot end heating module and insulation package for the existing AADSF.

Tests in the ASDSF engineering model are continuing. Calibration curves for this furnace are being developed. Refurbishment of the Drop Tower Furnace (DTF) is being accomplished. The DTF will be capable of operating at temperatures up to 1700°C. The sample size will be approximately 3/8 in. dia. x 5/8 in. long. Design improvements for the General Purpose Rocket Furnace (GPRF) for use in the Materials Experiment Assembly (MEA) will be accomplished.

Multimode Acoustic Research

Jet Propulsion Laboratory
Dr. Martin Barmatz
NAS7-918
October 1, 1981 - September 30, 1985

There is a recognized need for high temperature containerless processing facilities that can efficiently position and manipulate molten samples in the reduced gravity environment of space. The primary objectives of this task are to develop theoretical models of new classes of acoustic levitation and provide experimental validation of these models using research levitation devices.

The ultimate goal of this research is to develop sophisticated high temperature manipulation capabilities such as selection of arbitrary axes of rotation and rapid sample cooling. This program will investigate new classes of levitation in rectangular, cylindrical and spherical geometries. The program tasks include calculating theoretical expressions of the acoustic forces in these geometries for the excitation of up to three acoustic modes (multimodes). These calculations are used to (1) determine those acoustic modes that produce stable levitation, (2) isolate the levitation and rotation capabilities to produce more than one axis of rotation, and (3) develop methods to translate samples down long tube cylindrical chambers. Experimental levitators will then be constructed to verify the stable levitation and rotation predictions of the models.

Theoretical analyses carried out under this task have predicted stable acoustic levitation in rectangular, cylindrical and spherical resonators using only one mode of excitation. Levitation using single modes in each of these geometries was experimentally verified in room temperature chambers. The translational and rotational levitation stability of these single mode levitators will now be evaluated at elevated temperatures.

Publications

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Continuous Fiberization of Silicon-Carbide-Nitride Precursor Resin

Bjorksten Laboratories
Dr. Stanley A. Dunn
NAS8-34648

The initial objective of this project was to design and build a laboratory scale extruder and with it to fiberize 5000 continuous feet of silicon-carbide-nitride precursor resin. Subsequently the objective was extended to include the cross-linking and pyrolysis of approximately 100 feet of the resin fiber.

The extruder was designed to accommodate approximately 1 cubic centimeter samples at accurately controllable rates corresponding to take-up from a single orifice of a 25 micron fiber at speeds in the 1000 feet/minute region. Positive feed delivery was achieved with a cylinder and piston driven by a geared down electric motor. Gear reduction was achieved primarily with off-the-shelf parts. Its most noteworthy feature, however, was the compound screw drive on the piston. A sleeve surrounding the piston rod was threaded inside and out to engage both the piston rod and the interior of the cylinder. The two sets of threads were in opposite directions and of slightly different pitches. Thus turning the sleeve caused the piston to progress axially by the difference in the pitches, the piston being keyed to the cylinder to prevent its rotating.

Over 5000 feet of fiber, 20 to 30 micron diameter, were spun from precursor resin samples supplied by MSFC. An electrical motor driven drum mounted on wheels and a lateral track was used to take up the fiber; spinning temperatures were around 100°C. The fiberized resin was then cross linked by exposure to 100% humidity at temperatures from 50 to 100°C. Over 100 eight inch lengths of hydrolyzed fiber were then pyrolyzed to produce the desired silicon carbide/silicon nitride fiber.

The formation of continuous silicon carbide/silicon nitride fiber appears to be feasible.

Levitating Furnace for Containerless Processing in Space

Bjorksten Laboratories

Dr. Stanley A. Dunn

NAS8-33513 (NASA Contact: V. Fogle, MSFC)

August 1978 - December 1984 (no cost extension)

The immediate purpose of this project has been to design, construct and test a levitator which would function reliably in a radiant heated cold wall furnace in a reduced gravity environment.

A prototype levitating/positioning device termed the Sonic Pump Levitator was designed, built and successfully tested in full gravity and in the reduced gravity of the parabolic flight regime of the KC-135. Positioning is achieved by timely and appropriate application of gas momentum from one or more of six sonic pumps. The sonic pumps, which are arranged orthogonally in opposed pairs about the levitation region, are activated by an electro-optical, computer controlled, feedback system.

The sonic pump is a low inertia electro-mechanical transducer which rapidly converts alternating electrical into sonic thence into kinetic energy of a directed gas flow. It was developed to provide the very short response time necessary to keep pace with computerized position keeping demands. It consists of a loudspeaker whose face is sealed by a closure perforated by one or more orifices. The diaphragm of the loudspeaker is the only moving part of the sonic pump, no valves being needed.

Valving is obviated by the fact that the flows of gas into and out of the orifice(s) of the pump naturally follow different paths. The flow during the intake portion of the sonic cycle stems essentially isotropically from the external vicinity of the orifice(s). The exhaust flow on the other hand, proceeds more or less axially from the orifice(s) due to cancellation of the lateral components of momentum of the intake flows in the orifice(s).

The Sonic Pump Levitator is a promising candidate for containerless processing in microgravity. Being a null point device, it imposes minimal perturbation on the levitated, or positioned, target. Functioning as it does by optical feedback controlled jets of air, it would be expected to be free from disturbance caused by high temperature. This expectation is now being examined experimentally.

Electric Field Containerless Processing Technology

Jet Propulsion Laboratory
Dr. D. D. Elleman
Dr. W. K. Rhim
In-House

The primary objective of this task is to develop the science and technology base required to design and construct a high temperature electric field positioning module that could be used by materials scientists to conduct containerless science experiments in the low gravity environment provided by the space shuttle or in future years by space station.

Containerless science modules that employ electric fields to position and manipulate samples offer several advantages over acoustic or electromagnetic systems. The electric field system will operate not only at atmospheric pressures but also in a vacuum, in contrast to the acoustic modules which can only operate in an atmosphere where the acoustic forces are sufficient. The electric field technique puts minimum energy into the sample, whereas the electromagnetic system can deposit energy into the sample through eddy current heat as well as physical mixing in the sample.

Two types of electric field modules have been constructed and tested to date at JPL. One employs a charged sample and uses electrostatic forces to position and control the sample. The second type of module induces electrical polarization of the sample and electric field gradients to position and control the sample. The second technique has some advantages at elevated temperature, in that electric charge loss from the sample due to thermionic emission does not affect its operation.

The electrostatic system has been tested in the reduced gravity, approximately 10^{-2} g, provided by a KC-135 aircraft flying a parabolic trajectory. Liquid samples and solid high density samples at room temperature have been positioned with stability of better than 1 mm during random acceleration perturbations of 10^{-2} g on the aircraft.

Ground-base tests with low density samples have been conducted in vacuum as well as in high temperatures up to 500 C. Future KC-135 flights with high density samples at elevated temperatures are planned.

Publications

Rhim, W. K., Collander, M., Hyson, M., Simms, W., and Elleman, D. D., "Development of an Electromagnetic Positioner for Space Materials Processing," Rev. Sci. Instrum., in press.

Mackin, B., Rhim, W. K., and Elleman, D. D., "Performance Characteristics of a Pulse Charging System," IEEE/IAS, 1984.

Cryogenic Equivalence Principle Experiment

W. W. Hansen Laboratories of Physics
Dr. C. W. F. Everitt
Dr. Paul W. Worden

The purpose of this project is to test the equivalence of inertial and passive gravitational mass in an earth-orbiting satellite. Preliminary work and technology development is being done in a ground based experiment which is expected to test the equivalence principle to a few parts in 10^{13} ; a satellite version might have a sensitivity of one part in 10^{17} .

The ground-based experiment is now well developed. It consists of comparing the motions of two cylindrical test masses suspended in precision superconducting magnetic bearings and free to move along the horizontal (axial) direction. The masses are made of niobium and lead-plated aluminum. A position detector based on a SQUID magnetometer measures the differential motion between the masses. The periods of the masses are matched by adjustment of the position detector until the system is insensitive to common mode signals, and so that the experiment is less sensitive to seismic vibration. The apparatus is contained in a twelve inch helium dewar suspended in a vibration isolation stand. The stand achieves 30 db isolation from horizontal motions between 0.1 and 60 Hz, by simulating the motion of a 200 meter long pendulum with an air bearing. With this attenuation of seismic noise and a common mode rejection ratio of 10^5 in the differential mode, the ground based apparatus should have a sensitivity to equivalence principle violations of one part in 10^{13} . The primary limitation is due to seismic noise.

A version of the experiment to be performed on the Space Shuttle is under study. The earth-based apparatus will be appropriately scaled and modified for operation in zero gravity. The test masses will be about 10 centimeters in diameter. A crucial difference in the orbital experiment is the effect of the gravity gradient of the earth on the masses. This can be eliminated by putting the centers of mass of the test bodies at the same location. If the centers of mass are not coincident, the resulting acceleration can be detected and used as an error signal for a servo loop to drive them into coincidence. The Shuttle version of the experiment should have a sensitivity of about 10^{-15} limited by the vibration environment and gravity gradient field of the Shuttle orbiter. An independent drag-free satellite is necessary for the ultimate version of the experiment which might exceed a sensitivity of 10^{-17} limited by gas pressure effects.

Publications

Worden, P. W., "Measurement of Small Forces with Superconducting Magnetic Bearings," Precision Engr. 4, 139-144 (1982).

Everitt, C.V.F., "Low Temperature Techniques in Gravitational Experiments," in Proceedings of 1983 International School and Symposium on Precision Measurement and Gravity Experiment, Taipei, Republic of China, 1983, (W-T. Ni, ed.), 1983.

Worden, P. W., "Cryogenic Equivalence Principle Experiment: Discussion and Present Status," in Proceedings of Third Marcel Grossman Meeting on General Relativity (H. Ning, ed.), North Holland, 1983, pp. 771-779.

Worden, P. W., "Almost Exactly Zero: The Equivalence Principle," in press.

Modification of the Electromagnetic Levitator (EML) Hardware

General Electric Company
Dr. R. T. Frost
NAS8-34321 (NASA Contact: L. Gardner, MSFC)

The goals of this project are: (1) to study the upgrade requirements and approaches needed for incorporation of an Electromagnetic Levitator (EML) into the shuttle orbiter, (2) to work with members of the Electromagnetic Containerless Processing science working group (SWG) to define future experiments for the EML, and (3) to assist these investigators in further development of ground-based experiment techniques to the limits possible in the terrestrial gravitational environment.

Present work is directed toward (1) upgrading the EML flight apparatus to meet requirements of safety and integration interfaces with the MSL orbiter carrier, (2) development of new experiment components required to carry out approved experiments in undercooled solidification and associated fluid flow studies directed by MIT, and (3) construction, test, qualification and integration assistance for the EML MSL flight package. New components which have been developed and tested include a specimen exchange mechanism, a high current power conditioner/EML filter to operate from the 28 volt MSL bus, and an experiment controller to permit repetitive processing of single specimen, specimen exchange, and manual or automatic initiation of successive experiment cycles.

Publications

Frost, R. T., Flemings, M. C., Szekely, J., El-Kaddah, N., and Shiohara, Y., "Electromagnetic Containerless Undercooling Facility and Experiments for the Shuttle Orbiter," in Proceedings of XXV COSPAR Plenary Meeting, Graz, Austria, July 1984.

Microgravity Materials Science Laboratory

Lewis Research Center
Dr. S. J. Grisaffe
In-House
February 1984 - continuing task

A Microgravity Materials Science Laboratory (MMSL) has been planned, designed, and is being developed. This laboratory will support related efforts to define the requirements for the Microgravity and Materials Processing Laboratory (MMPF) and the MMPF Test Bed for the Space Station. The MMSL will serve as a "check out" and training facility for science mission specialists for STS, Spacelab and Space Station prior to the full operation of the MMPF Test Bed. The focus of the MMSL will be on experiments related to the understanding of metal/ceramic/glass solidification, high perfection crystal growth and fluid physics. This ground-based laboratory will be used by university/industry/government researchers to examine and become familiar with the potential of new microgravity materials science concepts and to conduct longer term studies aimed at fully developing a "1-g" understanding of materials and processing phenomena. Such research will help create new high quality concepts for space experiments and will provide the basis for modeling, theories, and hypotheses upon which key space experiments can be defined and developed. The MMSL will be fully equipped with appropriate materials research facilities and will be supported by the extensive Lewis Research Center materials characterization and computational capabilities.

The laboratory equipment is currently being designed and ordered. Rehabilitation of the laboratory area has been planned and has already begun. Activity is underway to acquaint industrial and university researchers with these plans so as to incorporate appropriate requirements and to make the facilities readily usable.

Advanced Optical Measurement Techniques

Marshall Space Flight Center
Dr. R. B. Owen
Dr. M. H. Johnston
W. K. Witherow
In-House
Continuing Task

This research analyzes four areas: (1) Improved Techniques for Optical Measurement; (2) KC-135 Low Gravity Simulation Flight Preparation; (3) In-Line Holographic Microscopy; and (4) Holographic Optical Schlieren System (HOSS).

The first task provides for laboratory breadboarding of advanced optical measurement techniques which appear promising for use in MPS flight experiments. A laboratory schlieren system has been constructed which simulates a similar optical system on the Fluids Experiment System (FES) which is to be flown on Spacelab 3. This system has been used to study growing triglycine sulfate (TGS) crystals in preparation for this mission and to help train the Shuttle crew members who will grow this crystal in space. In addition, a fiber optics interferometric system useful in detecting minute deformations in crystals has been designed and components have been ordered. It is anticipated that use of fiber optics will eliminate some of the vibration and temperature sensitivity of previous interferometer.

The second task allows improved optical systems and breadboards to be built for experiments and hardware tests on the KC-135 aircraft. A new, more stable Mach-Zehnder interferometer has been developed and components have been ordered. It is anticipated that the new design will eliminate beamsplitter instabilities associated with the present configuration as well as a minor misalignment problem. In the mean time, use of the existing optical system has been quite fruitful.

The third task is concerned with improvements on the optical system being used to study immiscible fluids. Experiments studying the dynamics of nucleation and the separation of immiscible fluids have been conducted. The phenomena of Oswald ripening is still being investigated. Work is also progressing on an optical system that may be able to study the solution concentration gradients in a test volume of separating immiscible fluids.

The four task involves building breadboard reconstruction systems for holograms taken in the HOSS. The HOSS is an early prototype of the Fluids Experiments System (FES) which is scheduled to fly on Spacelab 3. Holograms have been taken of triglycine sulfate (TGS) crystals growing in solution in the FES. These holograms have been reconstructed in various breadboard reconstruction systems to demonstrate the different analysis techniques possible through the use of holography. In addition, the quality of the FES holograms was assessed. The flight hardware was found to be capable of generating good quality holograms while mounted on a ground-based isolation platform.

Design, Fabrication and Testing of an Experimental Apparatus for
Determining Particle Behavior Under Very Low Intergranular Stresses

University of Colorado
Dr. Stein Sture
Dr. Nicholas Costes, MSFC
NAS8-35668 (NASA Contact: V. Yost, MSFC)

Because the constitutive laws for soils are governed mainly by interparticle friction, all aspects of their mechanical behavior depend strongly on gravitational body forces. This fact poses serious limitations on the formulation of a materially objective soil constitutive theory, based on experimentation performed on earth. In particular, the presence of the earth's gravity prohibits the design of controlled experiments to properly simulate a variety of critical phenomena associated with the dynamic response of soils to seismic excitation in a very low effective confining stress field. For these reasons, laboratory-controlled experiments in the Space Shuttle, under essentially zero-gravity conditions, could offer invaluable opportunities for developing a quantitative understanding of fundamental aspects of soil behavior during or after an earthquake, which, in turn, could result in significant technological advances in geotechnical earthquake engineering.

Stress induced stiffness anisotropy becomes significant when the principal stress directions rotate with respect to the soil fabric. This feature may have important practical implications, yet due to the limitations of current experimental and analytical tools, it has not been thoroughly investigated.

A fabric freezing technique for granular materials has been developed and it was used in conjunction with a sub-sectioning procedure to test sand in a set of multiaxial cubical devices. This technique permits the application of arbitrary discrete rotations of the principal stress directions for many stress paths. A multiaxial test program was conducted on dense and loose Leighton Buzzard sand at low confining pressures. It was shown that all stress paths considered in the test program induce significant anisotropy in both the deviator and volumetric stress-strain responses for both dense and loose sand.

Publications

Ketcham, S. A., Sture, S., and Costes, N., "Testing of a Cohesionless Soil at Very Low Confining Pressures," Report under NAS8-33449, University of Colorado, 1980.

Sture, S., "Influence of Gravity Independent Parameters on the Constitutive Behavior of Soils - Soil Behavior at Very Low Effective Stress Levels," Report under NAG2-58, 1980.

Sture, S., "Friction, Dilatancy, and Energy Relations for a Granular Soil," Report to USRA-Boulder, 1981.

Costes, N. C. and Sture, S., "The Potential of In-Space Research in Liquefaction Phenomena and Related Soil Behavior," in Proceedings of International Conference on Recent Advances in Geotechnical Earthquake Engineering and Soil Dynamics, St. Louis, MO, Volume 3, 1981, pp. 929-959.

Botts, M. E. and Sture, S., "Application of X-Rays in Geotechnical Engineering," Report under NAG2-58, University of Colorado, 1982.

Mould, J. C., "Stress-Induced Anisotropy in Sand and the Evaluation of a Multi-surface Elasto-plastic Materials Model," Ph.D. Thesis, University of Colorado, 1983.

Sture, S., "Development of an Experimental Concept of Investigating the Constitutive Behavior of Particulate Materials in the Spacelab/STS Zero-g Environment," Final Report, NAG2-58, 1983.

Mould, J. C., Sture, S., and Ko, H-Y., "Experimental and Analytical Modeling of Stress Induced Anisotropy in Sand," in Proceedings of 5th American Society of Civil Engineers-Engineering Mechanics Division Specialty Conference, University of Wyoming, Laramie, Wyoming, 1984.

Ontuna, A., Budiman, J. S., Sture, S., and Ko, H-Y., "Directional Shear Experiments and Stress Induced Anisotropy," in Proceedings of 5th American Society of Civil Engineers-Engineering Mechanics Division Specialty Conference, University of Wyoming, Laramie, Wyoming, 1984.

Advanced Containerless Processing Technology

Jet Propulsion Laboratory
Dr. T. G. Wang
In-Center
October 1978 - continuing task

The long-range objectives of this task are to: (1) study and advance the science of contactless positioning and manipulation of a high-temperature acoustic chamber; (2) provide technical information to the Acoustics Containerless Experimental System (ACES) engineering team; and (3) develop a set of high temperature ground-based facilities for precursor material processing experiments.

Breadboards for high temperature containerless processing systems will be developed, the principles of operation will be studied, the performance will be characterized, the limitations identified, and the influence of the acoustic field on the samples established.

The subjects to be addressed are experimental and theoretical studies of: (1) acoustic positioning and manipulation capabilities in a high temperature gradient environment (from 25°C to 900°C); (2) acoustic waveforms, harmonic contents, power transfer, sample transport and stability associated with high temperature gradient system; (3) high temperature ground based levitation systems which will allow us to melt, process, and solidify samples without crucibles in the laboratory, (4) KC-135 and laboratory tests of various acoustic geometries which may have spherical applications in the Materials Processing in Space Program; and (5) provide technical information to ACES engineering team and establish the operation conditions for ACES.

Publications

Wang, T. G., "High Temperature Acoustic Levitation," in Proceedings of IEEE Sonics and Ultrasonics 1124 (1983).

Robey, J. and Trinh, E., "Sample Transport of Levitated Objects in Dual Temperature Resonance Chamber," JASA Suppl. 1., 74, S27-28 (1983).

Acoustic Levitation Containerless Processing

Intersonics, Inc.
R. R. Whymark
C. A. Rey
NAS8-33742 (NASA Contact: V. Fogle, MSFC)
April 1983 - April 1986

This research program consists of the development of acoustic containerless processing systems with applications in the areas of research in material sciences, as well as the production of new materials, solid forms with novel and unusual micro structures, fusion target spheres, and improved optical fibers.

The efforts at Intersonics have been largely focused on the containerless processing at high temperatures for producing new kinds of glasses. Also, some development has occurred in the areas of containerlessly supporting liquids at room temperature, with applications in studies of fluid dynamics, potential undercooling of liquids, etc.

The high temperature area holds the greatest promise for producing new kinds of glasses and ceramics, new alloys, and possibly unusual structural shapes, such as very uniform hollow glass shells for fusion target applications. High temperature acoustic levitation required for containerless processing has been demonstrated in low-g environments as well as in ground-based experiments.

Future activities include continued development of the single axis acoustic levitator, similar to the one being utilized by NASA on the Materials Experiment Assembly (MEA) and the development of more advanced high temperature containerless processing systems for use on the Materials Science Laboratory (MSL).

Progress is being made toward even higher temperatures, i.e., above 1500°C, and toward achieving rapid cooling rates in the containerless state. In addition engineering developments are underway to provide more efficient high temperature furnaces, more stable acoustic positioning, and improved control of specimen contamination.

Additional areas of study include extending ground-based levitation to temperatures above 1000°C, and to specimen densities greater than 3. Also, a project is under way to attempt the drawing of fiber optics from a containerless melt.

High-Temperature Controlled Redox Crystallization Studies

Johnson Space Center
Dr. Richard J. Williams
In-House

The crystallization of silicates containing redox sensitive ions (e.g., Fe, Ti, Ce) must be performed under controlled and known redox conditions in order to obtain the maximum scientific benefit from experimental study. Furthermore, many compositions crystallize dense phases which settle during ground-based experiments. This settling influences the texture and chemical evolution of the crystallizing system. The purpose of this investigation is to develop a test system in which controlled redox experiments can be performed in the microgravity environment.

The system will use solid ceramic oxygen electrolyte cells for control, measurements, and production of the required redox conditions. This year we have developed a preliminary design for a prototype, tested the electrolyte and furnace components, and developed a tentative protocol for experiment. By fall, we hope to have established the control parameter and be building a laboratory prototype.

The prototype will be used to study the crystallization of silicates with the composition of meteorite chondrules. We have studied those systems using conventional techniques and note differences between laboratory and natural materials, which can be ascribed to gravity-induced settling in our experiments. The results from the previous studies and our prototype testing will be used as the basis for comparison with flight data. The flight experiment program will be proposed next year for evaluation and funding.

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16. Abstract <p>This report is a compilation of the active research tasks as of the end of the fiscal year 1984 of the Microgravity Science and Applications Program, NASA-Office of Space Science and Applications, involving several NASA centers and other organizations. The purpose of the document is to provide an overview of the program scope for managers and scientists in industry, university, and government communities. The report is structured to include an introductory description of the program, strategy and overall goal; identification of the organizational structures and people involved; and a description of each research task, together with a list of recent publications.</p> <p>The tasks are grouped into six categories: Electronic Materials; Solidification of Metals, Alloys, and Composites; Fluid Dynamics and Transports; Biotechnology; Glasses and Ceramics; and Combustion.</p>					
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