

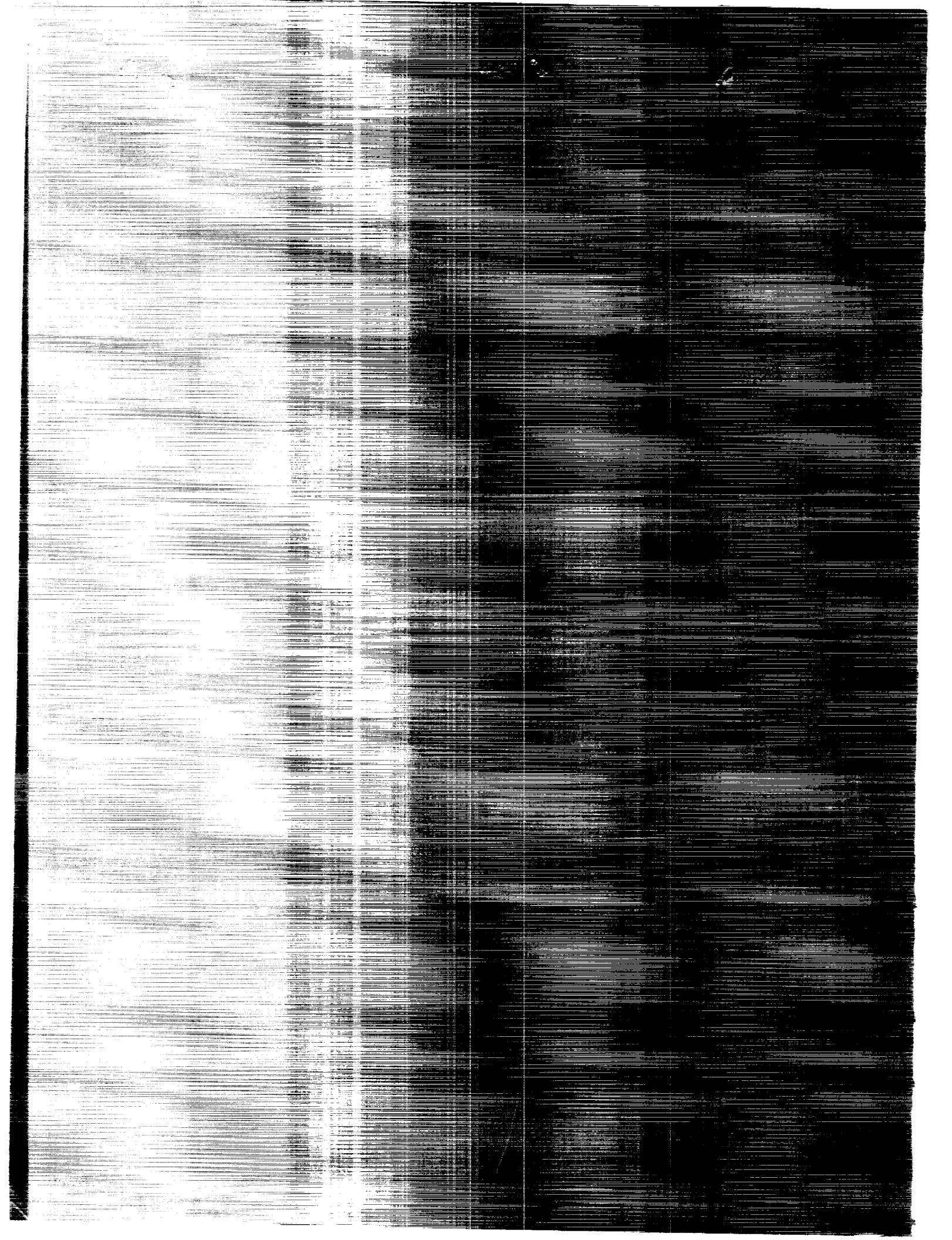
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I. INTRODUCTION

I. INTRODUCTION

The Microgravity Science and Applications (MSA) 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 of 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 electro-static containerless processing modules and electrophoresis separation devices.

The current 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.

The Microgravity Science and Applications Program Task Document covers the period of January 1987 - January 1988. The document includes research projects already completed as well as those now being funded by the Office of Space Sciences and Applications, Microgravity Science and Applications Division, NASA Headquarters.

The Microgravity Science and Applications Division wishes to thank the Universities Space Research Association (USRA) and in particular Ms. Elizabeth Pentecost for her efforts in the compilation and publication of this report.

II. TASKS

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A. GROUND BASED EXPERIMENTS

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1. ELECTRONIC MATERIALS

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

Massachusetts Institute of Technology
Professor Robert A. Brown
NSG-7645 (NASA Contact: R.J. Naumann, HQ)
October 1, 1987 - September 30, 1988

Fundamental understanding of the interactions of heat and mass transport, melt flow and the morphology of solidification interfaces are crucial to the design and interpretation of experiments aimed at microscopically controlled solidification of crystals on earth and in space. This research program focusses on analyses of the transport mechanisms important in macroscopic and microscopic understanding of solidification processes, especially ones of interest to the Microgravity Science and Applications Program at NASA. Research has focused on detailed analysis of the floating zone and directional solidification processes for growth of semiconductor crystals and on the dynamics of microscopic cell formation in two-dimensional solidification of binary alloys. Some of the most significant results follow.

1. We have completed a finite-element-based simulation of the thermalcapillary model for small-scale floating zones tat includes axisymmetric fluid flow in the melt driven by either buoyancy-driven and surface-tension convection and rotation of the feed and crystal rods. The simulation has been used to study systems for growth of silicon, germanium and NaNO_3 . Calculations of solute segregation in these systems is underway.
2. We are completing the development of a numerical simulation for the time-dependent flow and interface morphology in directional solidification for non-dilute alloys. The simulations will be used to predict the composition fields and interface morphology in crystals grown by gradient freeze and the vertical Bridgman Stockbarger method.
3. A study of the existence of a fundamental mechanism for wavelength selection in solidification of two-dimensional cellular interfaces from a binary melt based on large-scale numerical simulations has shown that steadily solidifying structures are possible for a continuous range of wavelengths. This conclusion opposes results for more idealized solidification systems where mechanisms for selecting a specific wavelength of the microstructure exist.
4. We have completed the design and construction of a two-dimensional solidification experiment capable of tracking the development of microscopic interface morphologies in transparent melts and are testing this device for the solidification of an acetone-succinonitrile alloy. We are completing an exhaustive set of theoretical predictions for this system.

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Fundamentals of Electronic Crystal Growth

NASA Lewis Research Center
Dr. Hugh R. Gray
In-House,
October 1, 1987 - Continuing

Post-Flight Analysis of 3M's DMOS Experiment (Roberts, et al)

Ground-base experiments designed to simulate the fluid flow which had occurred during the 3M Company's DMOS-2 (Diffusive Mixing of Organic Solutions) space experiments have been completed. The major conclusion of this collaborative research among researchers from 3M, MSFC, and LeRC are: the solution mixing which occurred during the space experiment can be simulated in ground-based experiments by making the $(\Delta p)g$ products equivalent; a simple model of channel flow driven by hydrostatic pressure differences can be used to estimate mixing rates; the results of these ground-based experiments suggest that some mixing natural convection did occur during the DMOS space experiment.

For future space experiments, convection can be reduced by

- reducing Δp (using deuterated solvents)
- maintaining g parallel to the initial density gradient
- reducing the width of the mixing chambers

The effects of both mean gravitational field (for ground experiments) and g -jitter (for shuttle applications) on fluid mixing have been investigated both numerically and analytically. One of the results of the analytical work has been to show that small scale Kelvin-Helmholtz and Rayleigh-Taylor instabilities can be generated by g -jitter at the interface of the two fluids. These instabilities can cause chaotic mixing of the fluids and greatly affect the nucleation rate of crystals and cause growth defects.

Characterization of Directionally Solidified Lead Chloride (Duval, et al)

In collaboration with the Westinghouse R&D Center, a complete analysis on directionally solidified lead chloride material is being conducted. Efforts are focused on photographic observation of the solid-liquid interface at several G/V ratios (denoting the temperature gradient and the translation velocity, respectively) to study the morphology of the interface and optimize the growth conditions. Future studies will investigate effects of segregation by doping lead chloride with silver.

Effects of Thermal Radiation and Convection in PVT (Duval and Kassemi)

The objective of the present study is to investigate the extent by which thermal radiation can influence and alter fluid motion during physical vapor transport inside an enclosure. This is being accomplished by a systematic parametric study to delineate the intricate interaction between radiation and natural convection. Experimental studies are focused on measurements of crystal growth rates and imposed temperature profiles. Future experiments are being planned to measure the flow and temperature combined theoretical and experimental effort will help design critical microgravity experiments where it is suspected that radiation plays a significant role but up to now has not been addressed.

Mathematical Modeling of Directional Solidification (Chait, et al)

A 3-D fluid dynamics and heat transfer code is now available. This code is a finite-element based solver which can be used in a arbitrary geometry, boundary conditions, initial conditions, etc. The code can handle both steady and transient flows. We are currently utilizing this code for simulating both directional solidification and float zone processes. Solidification is simulated using two approaches. The first method is an enthalpy formulation on a fixed grid, and the second uses an adaptive grid in which the mesh deforms during the solution process. Both approaches can handle different materials properties for the solid and the melt phases, as well as resolving the heat of fusion at the interface. Output includes both numerical and graphical representations of the entire flow and temperature fields, including auxiliary information such as interface shape and location.

The present code is limited to pure materials, with dopant concentrations to be determined during post-processing in a limited way. The full problem of the thermal and solutal fields (with up to two extra solutes), consistent with the phase diagrams and appropriate boundary conditions is expected (with some qualifications) to be attainable within the next year.

An axisymmetric fluid dynamics, heat transfer and one solute solver is also available. This code is finite-element based, and it is specifically designed for simulating directional solidification in cylindrical ampules. The code can predict the flow, temperature and solutal fields for a steady-state solidification of dilute and non-dilute alloys. Full time transient capabilities are currently being added to the code.

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Growth Kinetics of Physical Vapor Transport: Crystal Growth of an Opto-Electronic Material, Mercurous Chloride

Westinghouse R&D Center
Dr. N. B. Singh
NAS3-25274

For the optical and acousto-optic devices refractive index of the material should be very uniform and the optical scattering should be low. This can be achieved by growing homogeneous, extremely pure and stress free crystals. For this reason, the crystal growth and transport behavior is being studied in transparent cylindrical ampoules under 1 g conditions. The present experiment should yield detailed insights into the relationship among convective phenomena, growth kinetics and, subsequently, the high quality of the crystal. The data from the ground-base experiment will be used to develop a flight experiment so that advantage of the microgravity environment to space can be used to enhance the optical homogeneity.

The experiment is being carried out to define the effects of convective phenomena on the growth mechanisms and properties of the opto-electronic crystals grown by physical vapor transport. Mercurous chloride, which exhibits an anomalously slow sound velocity, a wide range of transparency, large birefringence, and very high acousto-optic diffraction efficiency is the material under study. Since the material is transparent and transports congruently we are investigating the relationship between growth parameters, convective behavior and morphology of the solid-vapor interface.

Crystal Growth of Organic and Polymeric Material

Marshall Space Flight Center
Dr. Marcus Vlasse
In-house
January 1987 - Continuing Task

The objective of this work is the crystal growth of bulk single crystal of high quality and perfection for eventual use in non-linear optical applications. The growth of such crystals requires accurate control of the environment at the growth interface, particularly in the liquid phase. Thermal or solutal fluctuations in the fluid phase can give rise to inhomogenities and physical defects in the growing crystal. Convection due to the above fluctuations is thought to be detrimental to the control of the growth process and is generally a cause for many of the ingrown imperfections. The understanding and control of convection in crystal growth processes in ground-based experiments and the use of reduced gravity environment will facilitate the production of single crystals and polymeric films.

The program consists of two tasks: (1) ground-based experiments on the melt growth by directional solidification of several mode substituted diacetylene, and (2) solution crystal growth of the same diacetylenes from organic solvents and growth of L-arginine phosphate from aqueous solutions. This latter material is considered very promising for NLO applications. The aspects to be studied will be the influence of thermal gradients and concentration gradients (solution growth), growth rates, and the influence of convective flows on the growth process and perfection of the grown crystal. Correlations between growth conditions and size and quality of crystal will be established.

Heat and Mass Transfer Control in Directional Solidification

Massachusetts Institute of Technology
Professor August Witt
NSG-7645 (NASA Contact: R.J. Naumann, HQ)
January 1987 - January 1988

The heat pipe based, 3-zone Bridgman growth system, developed under this contract was modified to a) permit monitoring of all functions and growth control through an IBM PC/XT, b) allow for growth of CdTe with vapor pressure control through the installation of an additional hot zone and c) accomodate a superconducting magnet providing for growth with axial fields of up to 30 kgauss.

Ga-doped Ge was grown at a displacement rate of 10 micro m /s in vertical Bridgman/Stockbarger configuration with an applied axial magnetic field of 3 T. A composition analysis of the single crystal grown showed that diffusion controlled segregation (absence of convective interference with mass transport at the growth interface was achieved in quantitative compliance with Tiller et. al.) for the first 2 cm of regrowth. The results suggest that the value of the solute diffusion coefficient in liquid Ge is $1.7 \text{ E } -4$ rather than $2.1 \text{ E } -4 \text{ cm}^2/\text{s}$ as generally assumed in the open literature.

Most recently, we have been successful in developing an optical approach to the rapid determination of the micro-distribution of free charge carriers in doped elemental and compound semiconductors. This technique permits, for the first time, on a microscale the establishment of quantitative cause and effect relationships between crystal growth parameters, growth conditions and crystal properties; it is expected to enhance significantly our ability to assess the potential of a reduced gravity environment for research in electronic materials processing.

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Capillary Convection with Crystal Growth

University of Michigan
Professor Wen-Jei Yang
NAG3-903 (NASA Contact: A.T. Chai)
May 1988 - May 1990

Marangoni effects play a significant role in natural convection within a drop evaporating on a plate. It is proposed to study interfacial "turbulence" (namely Marangoni Instability), internal flow structures and evaporation rate when crystal growth occurs in the sessile drop. Both shadowgraph-schlieren method and holographic interferometry will be employed in the experimental study, while a numerical technique will be used in the theoretical investigation. The effects of surface tension-controlled natural convection and evaporation rate on the growth of crystals will be determined. The crystal growth in both pure-liquid and binary-liquid drops will be investigated. The crystal quality will be evaluated by x-ray diffraction and compared with the quality of crystals grown under buoyancy-controlled natural convection. The study offers the possibility of understanding the origin, formation and eventual suppression of defects in crystal growth from melt or in fabricating new alloys under surface tension-controlled natural convection. It, therefore, has applications in melt (a higher temperature process) or solution (a lower temperature process) growth in order to grow better crystals on earth or in space.

2. SOLIDIFICATION OF METALS, ALLOYS AND COMPOSITES

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Studies of Containerless Processing of Selected Nb-Based Alloys

Vanderbilt University
Dr. Robert J. Bayuzick
Dr. M. B. Robinson, MSFC
NAG8-536 (NASA Contact: M.B. Robinson, MSFC)
July 17, 1985 - July 31, 1988

Research is being conducted on the effect of containerless processing of alloys in a low-gravity environment. The primary goal is the better understanding of deep undercooling and its effect on microstructure and properties. The 100 meter drop tube at the Marshall Space Flight Center is being used to continue and extend work which has already been done to give a firm foundation of earth-based research. Previously, Nb-Ge alloys of compositions ranging from 13 to 35 atomic percent Ge were deeply undercooled. Undercoolings observed were as high as 25 percent of the liquidus temperature (Approximately 530 K). The microstructure and superconducting properties were extensively characterized.

More recently work has focused on Pb-Pt and Nb-Si alloys ranging in composition from 10 to 32 atomic percent alloy additions. Undercoolings ranged from 15 to 27 percent of the liquidus temperature (absolute undercooling as high as 671 K). Investigations included scanning electron microscopy, x-ray powder diffraction, and measurement of the superconducting transition temperature. Higher composition Nb-Pt samples had undercooling limited by nucleation of the Nb₃Pt phase. Solute trapping is indicated at the lower compositions at the higher undercoolings. In Nb-Si, although formation of metastable phases has been reported in the literature, only the equilibrium phases have been noted. However, unique microstructures are observed.

Experiments on bulk samples of pure metals have also been conducted. Droplets were as large as 7 mm in diameter. Undercoolings up to approximately 23 percent of the melting temperature were obtained. These represent the largest absolute undercooling ever obtained in bulk samples. For example, the absolute undercooling in tantalum was about 740 K. The results were consistent and repeatable showing that these large levels of undercooling in large samples are readily obtained in containerless, microgravity experiments. It is clear that nucleation commences near the free surface. However, such surface nucleation cannot be explained by surface energy arguments nor is it thought to proceed from surface oxides, particularly in the case of molybdenum, niobium, and tantalum. Thermal gradients in the drop samples are thought to be responsible for the surface nucleation.

Publications

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Solidification Processing of Dispersed Phase Reinforced Mg Alloy Composites under 1-G and Microgravity Conditions

Massachusetts Institute of Technology
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Professor Julian Szekely
Dr. O. J. Ilegbusi
Dr. Z. Raczynski
NAG3-308

The objective of this research is to develop improved understanding and mathematical modelling of the various processes involved in semi-solid slurry processing of metal matrix composites for space applications and in space processing. Namely: (1) the addition of non-wetting ceramic particulates reinforcement to magnesium alloy melts; (2) the dispersion of those particles in the melt; (3) semi-solid slurry warming of the resulting dispersion; and (4) the control of microstructure during solidification.

The current research is pursuing two complementary and related directions: (1) process development and slurry rheology studies and (2) mathematical modelling of the various processes.

The process development phase of the work was concentrated on the development of experimental facilities for the study of slurry formation and processing. With this facility, we have initiated the evaluation of the factors controlling the rate of particulate inclusion and the formation of porosity in composites. We have developed a facility for injection molding of slurries.

The mathematical modelling component involves the following two key items:

We have developed the equations governing the electromagnetically driven flow of melt-slurry suspension through the combined solution of Maxwell's equations and the non-Newtonian equations of motion.

We have also developed a model representing the behavior of non-Newtonian melt-suspension which is being agitated mechanically using a paddle stirrer.

Important milestones of the project include:

Development for the inclusion and dispersion of ceramic particulates into a molten metal without the usual incorporation of porosity. Patents are being filed on this technology.

Mathematical models have been developed for the non-Newtonian semi-solid slurries for both electromagnetic and mechanically driven flow.

Future directions include: (1) developing uniformity in dispersion distribution; (2) evaluation of interfacial reactions between the reinforcements and molten magnesium alloy matrix; (3) slurry forming and solidification studies, comparing theoretical; (4) mathematical models with experimental measurements, and (5) conceptual design of microgravity experiments.

Publications

Ilegbusi, O. J. and Szekely, J., "On the Flow Criteria for Suspending Solids in Electromagnetically-Stirred Melts," Met. Trans. B, 1987 (in press).

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Braze Metal Flow in Planar Capillaries

Massachusetts Institute of Technology
Professor T. W. Eagar
NSG-7645 (NASA Contact: R.K. Crouch, NASA HQ)
October 1, 1986 - September 30, 1988

There is considerable evidence suggesting that braze joints could be as strong as weldments if the defects at the joint could be eliminated. The goal of the proposed research is to measure the interface morphology of a braze alloy advancing in a planar capillary, to quantify the forces that drive the flow and that produce the instabilities, and to attempt to stabilize the interface by the addition of an appropriate gradient.

Two methods were proposed at the beginning of this study by which this flow may be measured: (1) solder flow in glass capillaries and (2) braze alloy flow in copper capillaries using infrared thermography. A braze alloy flows faster into a thin capillary, presumably due to the relatively greater ratio of interfacial tension to inertial forces. By imposing a geometric gradient in the capillary spacing, it may be possible to stabilize the interface. Another known variable in the speed of braze metal flow is surface roughness, where greater roughness behaves the same as a relatively larger interfacial tension. This suggests that transverse grooves, or gradients in surface roughness may stabilize the braze front. Yet another important variable is the composition of the solid being wetted. Gradients in the thickness of active metal coatings might provide a stabilizing factor in this system. Since buoyancy forces are known to be important in brazing, the orientation of gravity may have an effect on the stability of the interface. Studies of metal flow in different orientations may provide insight into the physics of braze metal flow. Finally, brazing in microgravity may well stabilize the interface merely by reducing the Rayleigh number of the system.

These tests with glass slides, and then the construction of the vacuum IR thermography chamber have consumed the initial period of work. Current research is assessing whether we can measure solder or braze alloy flow in planar capillaries. This second experimental method is now being used to study interface stability under thermal, geometric and compositional and roughness gradients.

Model Immiscible Systems

NASA Marshall Space Flight Center
Dr. Donald O. Frazier
In-House

Since the study of transparent immiscible systems has been an important method of investigating metallic monotectic alloys, it is important to suitably generalize from observations in the model systems to metallic monotectics. Recent work on model transparent systems has focused on homogeneous solution component and surges interactions to assess subsequent effects on macrosegregation during fast quenches through the monotectic temperature. To the extent that the existence of the miscibility gap itself can serve as a "signature" for certain thermodynamic characteristics of the solution, for example, deviations from ideality, it is appropriate to determine as completely as possible the key thermodynamic parameters for at least one such model system. From the model, study directions may arise for specific metal systems, which could result in better control of ingot microstructure and macrostructure.

For the succinonitrile-water system, densitometry shows a significant effect of homogeneous solution equilibration temperature on surface induced composition shifts. These shifts influence monotectic reaction onset in fast-quenched small-volume samples. Relative effects between high and low equilibration temperatures appear to vary with respect to the isopycnic temperature. Equilibration of succinonitrile-rich solutions above the isopycnic temperature in hydrophilic containers generally result in "less undercooling" and larger heat release than equilibrations below the isopycnic temperature. No such ordering exists in similar solutions fast-quenched in hydrophobic containers. We postulate from partial molar volume calculations, that at the isopycnic temperature, all homogeneous succinonitrile-water systems behave ideally. Above and below this temperature, solute-solvent aggregates differ significantly and if strong solute-container affinities are present, these aggregates will influence radial composition profiles with significant specificity.

Fourier Transform Infrared (FTIR) spectroscopy of a succinonitrile-benzene solution has been successful in giving 1-cm^{-1} resolution spectra at 1- to $1.5\text{ }\mu\text{m}$ penetration depth into the bulk phase through a zinc selenide attenuated total reflectance crystal. Determination of preferential wetting properties on zinc selenide with respect to succinonitrile-rich and benzene-rich phases, by use of contact angle goniometer shows that at room temperature, benzene-rich phases preferentially wet zinc selenide. There are differences in homogeneous succinonitrile-benzene surface spectra apparent upon heating and equilibrating at temperatures ranging from near monotectic to critical. These differences are currently under analyses to help define equilibration temperature effects on surface aggregation in these miscibility-gap type systems.

Further on-going investigations use Raman and resonance Raman spectroscopic methods to determine preferred bulk-phase cluster profiles in succinonitrile-based systems at different temperatures. Dr. John Hall at the Dolfus E. Millian Science Research Institute is performing ab initio self-consistent field calculations to determine the optimized geometries of trans and gauche conformers of succinonitrile and the degree of hydration for each conformer. From the models established by this approach, the group at MSFC will perform normal coordinate analyses on complexes using reasonable force fields to duplicate vibrational frequencies observed in the surface FTIR and bulk-phase Raman spectroscopic analyses.

Publications

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Gravitational Effects on Liquid Phase Sintering

Rensselaer Polytechnic Institute

Professor Randall M. German

C. Kipphut

A. Bose

T. Kishi

NAG3-744 (NASA Contact: Dr. G. Santoro, LeRC)

October 1, 1986 - September 30, 1988

The focus of the research is on identification of the gravitational effects on liquid phase sintering. The primary concerns are with macroscale distortion of compacts (slumping, nonuniform shrinkage, and liquid migration) as well as the microscale effects as seen in solid content, contiguity, connectivity, dihedral angle, and grain size gradients due to sintering in a gravitational force. In addition to experimental measurements, theoretical work is in progress to predict the degree of solid-liquid separation possible in attaining the energy minimum associated with grain shape accommodation and gravitational settling. The determination of correction factors necessary in liquid phase sintering grain growth laws are being determined based on contiguity and coalescence measurements. A change in coarsening mechanism is sought associated with the termination of grain coalescence.

To determine the role of gravity in liquid phase sintering, the tungsten heavy alloys have been selected as the study basis. These alloys consist of high contents of tungsten, with a matrix (W-Ni-Fe) that is liquid at the sintering temperature. The large density difference between the liquid and solid phases induces segregation during sintering. The segregation that occurs during sintering can be seen by microstructural gradients and compact distortion. Several test geometries and experimental conditions have been studied to date, including intentional changes in compact height, tungsten content, sintering time, and sintering temperature. It is clear that gravity causes substantial changes during sintering when the liquid content is high. There are gradients in the liquid content, grain size, contiguity, connectivity, and dihedral angle that depend on the alloy content, sintering time, and sintering temperature. Likewise, mechanical property tests have been performed to correlate the microstructure with the expected properties. These studies are being coupled with theoretical calculations of microstructural coarsening and grain shape accommodation to establish the role of grain rigidity (connectivity) and liquid viscosity in determining the slumping conditions. We believe a model for the slumping kinetics is now possible. Furthermore, new concepts in microstructural coarsening are emerging with respect to the role of coalescence and solution-reprecipitation during liquid phase sintering. This work is laying the foundation for critical microgravity experiments to experimentally establish the importance of coalescence to coarsening and compact slumping.

Publications

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Solidification Fundamentals

NASA Lewis Research Center

Dr. Hugh Gray

In-House

1983 - Continuing

The objective of this research program is to obtain a fundamental understanding of gravitational effects during solidification of metals and alloys. Experimental work underway can be divided into three major categories. First, experiments in support of a space Shuttle experiment on macrosegregation behavior in Pb-Sn alloys. Second, experiments aimed at obtaining a somewhat more fundamental understanding of dendritic and cellular growth, using a directional solidification apparatus. Third, experiments aimed at understanding the influence of undercooling on macro-and micro segregation behavior in bulk samples (> 20 grams) of binary Pb-Sn alloys. This experimental work is also being complimented by theoretical work aimed at understanding these fundamental solidification phenomena.

The Role of Natural Convection on Crystallization from Vapor and Solution: A KC-135 and Laboratory Study

Desert Research Institute
Dr. John Hallett
NAS8-34605 (NASA Contact: V. Fogle, MSFC)
January 1987 - January 1988

The objective of this research is to investigate the role of buoyancy induced convection in crystal growth and the differences which occur during growth in the absence of such convection under low-g.

Laboratory studies are being conducted on the role of convection in growth of: (a) ice vapor growth in presence of air (snow crystals); (b) ice growth from solution in presence of NaC ; and (c) sodium sulfate decahydrate from solution. Visualization of flow is achieved by Schlieren/ Mach Zender optics. Enhanced flow can be achieved by a wind tunnel or moving the crystal during growth. The system is uniformly supercooled so that the crystal grows into a well defined environment. Of particular interest is the facet - dendrite transition which occurs at a critical supersaturation/ supercooling, which is dependent on the ventilation velocity. This transition changes to lower supercooling/supersaturation with the absence of convection in low-g in KC-135 flights.

Water drops suspended at the interfaces of mineral oil/carbon tetrachloride (3 mutually immiscible liquids) are uniformly supercooled ($.5$ to $10\text{ C} \pm 0.1\text{ C}$) and nucleated by a single ice crystal with "c" axis oriented vertically or horizontally. Crystals grow through the liquid as thin dendrites parallel to the basal plane, and cease growth on reaching the opposite side.

Slow lateral growth perpendicular to the basal plane subsequently occurs at the water periphery, which ultimately gives rise to dendrites growing parallel to the first, back into the liquid. Final solidification takes place by a solidification front passing through the dendrite mush toward the drop center. No fast dendrite growth or new crystal orientation occurs at the liquid-liquid interface as occurs in capillary tubes or on a metal/glass interface. Preliminary studies of ice crystal growth in thin supercooled films shows a uniform velocity around the periphery, with an apparent change in orientation caused by stress in the boundary film.

These results show that crystallization of suspended spherical drops in low-g should, in addition to reduction of surface diffused impurities, also give a more characterized crystal texture and defect structure. There appears to be a transition in the nature of the crystal texture for thin films, which may occur at a critical thickness of the film.

Publications

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Knight, A. C., Hallett, J., and Devries, A. L., "Solute Effects on Ice Recrystallization: An Assessment Technique," J. Cryobiology 25, 55-60 (1988).

Presentations

Hallett, J. and Harrison, K., "Influence of High and Low Gravity on Convection Around Growing Crystals," AIAA 26th Aerospace Science Meeting, Reno, Nevada, January 1987.

Hallett, J., "Propagation of Vortex Rings and Starting Plumes in High and Low g," AIAA 26th Aerospace Sciences Meeting, Reno, Nevada, January 1987.

The Development and Prevention of Channel Segregation During Alloy Solidification

Michigan Technological University
Dr. Angus Hellawell
NAG3-560 (NASA Contact: Dr. R. L. Dreshfield, LeRC)
July 15, 1986 - July 14, 1989

The object of the research is to identify quantitatively, the conditions under which channel segregation occurs during alloy solidification and to seek methods of preventing such development.

With vertical growth upwards, lead base alloys in the systems Pb-Sn, Pb-Sb and Pb-Sn-Sb have been examined over a range of alloy compositions. The solidification conditions have been carefully recorded and the type of channel formation fully characterized: the results for the metallic systems had been compared with earlier studies of the aqueous NH-Cl system. Channels are considered to originate at, or close to the dendritic growth front. Analysis, in terms of thermal and solutal Rayleigh numbers, indicates a characteristic dimension which is close to that of the interdendritic spacing in both metallic and aqueous systems.

Publications

Hellawell, A., "Local Convective Flow in Partly Solidification Systems," NATO ASI Series E 125, 3-22 (1987).

Sarazin, J. R. and Hellawell, A., "Channel Flow in Partly Solidified Alloys System," in *Proceedings of Symposium on Advances in Phase Transitions*, Pergamon Press, 1987 (in press).

Sarazin, J. R. and Hellawell, A., "Channel Formation in Pb-Sn, Pb-Sn and Pb-Sn-Sb and Comparison with the System $\text{NH}_4\text{Cl-H}_2\text{O}$," Met. Trans. A, 1988 (in press).

Hellawell, A., "Channel Formation During Alloy Solidification," in *Indo-US Scientific Workshop on Solidification Processing*, (R. Trivedi, ed.), ONR, 1988 (in press).

Presentations

Sarazin, J. R. and Hellawell, A., "The Influence of Thermal Gradient and Growth Velocity on the Formation of Segregation Channels," presented at AIME Spring Meeting, Phoenix, 1988.

Whisker Growth Studies Under Conditions Which Resemble Those Available on an Orbiting Space Laboratory

George Washington University
Dr. Herman H. Hobbs
NAG3-642 (NASA Contact: L. Westfall, LeRC)
January 1987 - January 1988

The objectives of this research task are: (1) the determination of advantages, disadvantages, and special circumstances attendant upon annulment of earth's surface gravity during nucleation and growth of metal crystals (especially whiskers), and (2) determination of possible analytical (or practical) uses for electric currents which are a concomitant of the procedure used to levitate the growing whiskers.

Whiskers are grown by the chemical reduction of metal halides in the presence of applied electric fields. The applied fields have a number of effects including levitating forces on the whiskers. The fields can partially (or fully) support the growing whiskers and aids in growth by preventing the young whisker nuclei from falling over into the molten metal halide growth substance. This process is accompanied by an electric current which could be highly useful if current studies yield an understanding of its origin. To further mimic orbital conditions steps are being taken to suppress the convection currents which are usually attendant upon this growth process, and to perform all growth experiments in a vacuum chamber which will permit use of low and quickly variable partial pressures.

Structure of Nickel and Iron Aluminides Prepared by Rapid Solidification and Undercooling

North Carolina State University
Dr. Carl C. Koch
NAG8-475 (NASA Contact: E.C. Ethridge, MSFC)
August 1, 1984 - July 18, 1988

The objective of this investigation is to obtain a basic understanding of the complex solidification structures found in the nickel-base aluminides during rapid solidification and undercooling.

The $\text{Ni}_3\text{-Al}$ and Fe-Ni-Al-C systems have been selected for study. Particular interest lies in fcc-like metastable structures in the Fe-Ni-Al-C system which can be revealed by rapid solidification. Rapid solidification studies are carried out in an arc hammer apparatus and by melt spinning at controlled and variable cooling rates. Undercooling experiments are conducted in the 100 m drop tube at Marshall Space Flight Center. Structural studies use x-ray diffraction and transmission electron microscopy techniques.

Publications

Chen, H. T., Myers, S. A., and Koch, C. C., "Rapidly Solidified Fe-Ni-Al-C Alloys: Metastable Phase Formation," in *Proceedings of the 6th International Conference on Rapidly Quenched Metals*, Montreal, Canada, August 3-7, 1987; to appear in Mat. Sci. Engr., March 1988.

Crystal Growth by Two Modified Floating-Zone Processes

University of Wisconsin

Professor Sindo Kou

NAG8-705 (NASA Contact: S.L. Lehoczky, MSFC)

March 1, 1988 - February 28, 1991

Floating-zone crystal growth under microgravity, though essentially free from natural convection, can still suffer from undesirable Marangoni convection. To effectively reduce this convection while at the same time help produce single crystals of uniform diameter and smooth surface, it is proposed that two modified floating-zone processes be studied. The first of the two processes uses a ring heater in contact with the melt surface and the second a sheet heater immersed in the melt, both (heaters) with careful temperature control during crystal growth. The objective of the research is twofold: to help approach the convectionless condition for zone-melting crystal growth under microgravity, and to insure good diameter control and surface quality of the crystals.

The first part of the proposed work is the direct observation of Marangoni convection in the two processes, using a transparent material of high Marangoni number. The second part is the characterization of the two processes, with emphasis on effects of process variables and search for optimum growth conditions. The third part is the computer modelling of the two processes and the experimental verification of the computer models.

At present the experimental work on the direct observation of Marangoni convection in the two processes, using NaNO_3 and silicone oil is being initiated. Computer models to describe Marangoni convection in the two processes are being developed. Preliminary calculated results for the process involving a surface ring heater have shown that Marangoni convection appears to be reduced significantly.

Metallic Glass Research in Space

Jet Propulsion Laboratory
Dr. Mark C. Lee
Dr. Taylor G. Wang
James L. Allen
NAS7-918
October 1, 1985 - continuing task

The objective of this research is to develop a space experiment to acquire thermodynamic properties of bulk metallic glasses over the entire undercooling region, with emphasis on the temperature region inaccessible by terrestrial techniques.

Ground-based precursory experiments will be designed and performed in such a manner that all the critical parameters for the space experiment will be defined and validated. A logical approach to achieve this goal should include the following ground-based tasks: (1) development of a novel contactless calorimetry technique for specific heat measurements over the entire undercooling region of a bulk metallic glass sample; (2) measurements of specific heats and crystallization kinetics to precisely define the optimal candidate systems for the space experiment; (3) conceptualization of the data analysis technique for the space experiment; and (4) feasibility study of the metallic glass space experiment module.

A noncontact true temperature measurement technique using a laser pyrometer has been developed that allows the accurate ($\pm 2\%$) determination of the absolute temperature of the surface of any diffuse and opaque sample in the temperature range 750 C to 1200 C. This range is currently being extended to over 2000 C, and the accuracy is being improved. An electromagnetic levitation coil has been fabricated that will be used in conjunction with the pyrometer to develop the contactless calorimetry technique. Several candidate systems in several temperature ranges have been identified as easy glass formers. Ground-based measurements of the specific heat have been made for Au-Pb-Sb systems. Preparation for a flight experiment proposal is in progress.

Publications

Lee, M. C. and Allen, J. L., "Noncontact True Temperature Measurement," Mat. Res. Soc. Symp. Proc. 87, 285-293 (1987).

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Lee, M. C., Fecht, H. J., Allen, J. L., Perepezko, J. H., Ohsaka, K., and Johnson, W. L., "The Glass Transition, Crystallization, and Melting in the Au-Pb-Sb Alloys," in *Proceedings of Sixth International Conference on Rapidly Quenched Metals*, 1987 (in press).

Elleman, D. D., Allen, J. L., and Lee, M. C., "Laser Pyrometer for Spot Temperature Measurements," NASA Tech Briefs, in press.

Levitation Studies of High Temperature Materials

Rice University
Professor John L. Margrave
Shankar Krishnan
George P. Hansen
Robert H. Hauge
NAG8-612

This research is a proposed three-year program which is designed to expand capabilities for doing levitation research by moving into the microgravity of space. It will allow the establishment of highly reliable thermodynamic and other properties of elements like silicon and boron in both solid and liquid states, without the risk of container contamination. Also, the phenomenon of super-cooling, nucleation and kinetics of crystal growth which are so important in semiconductor development can be studied without the interference of gravity, vibrations, container impurities and dust. Studies will be conducted which yield monochromatic spectral and hemispherical emissivities of liquid boron and liquid silicon at various wavelengths and temperatures. Also, the densities of the liquids will be determined by a photographic technique at various temperatures.

Research efforts over the past year have been focussed on: (1) design of optimum coils of levitation of good conductors (Hf, HfC, etc.) and of poor conductors (B, Si, SiC, etc); (2) design and construction of an optical system for photographic determination of liquid metals; (3) design and construction of a system for high-speed photography of levitated objects - solid or liquid; (4) development of the background mathematical equations from which surface tensions and viscosities can be related to experimental oscillations of liquid droplets; and (5) development of techniques for determining emissivities of liquid metals over wide ranges of temperature and wave length.

During the second year of the project, tasks to be accomplished are: (1) complete studies of emissivities (Pd, Pt and Ir studies are in progress); (2) measure high-temperature thermodynamic properties of Hf, HfC and ZrC (solids and liquids-in progress); (3) measure high-temperature thermodynamic properties of B, C, Si and SiC (in progress); and (4) determine surface tensions and viscosities of liquid transition metals--Cu, Ni, Ti, Fe, etc.-in progress.

Publications

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Containerless Processing of Undercooled Melts

University of Wisconsin, Madison
Professor John H. Perepezko
NAG3-436 (NASA Contact: H. de Groh, LeRC)
January 1, 1987 - January 1, 1988

A main objective of the research is to evaluate the undercooling and resultant solidification morphologies in the containerless technique of drop tube processing. The degree of liquid undercooling attainable in a laboratory scale (3m) drop tube can be altered through the variation of processing parameters such as melt superheat, droplet size, and gas environment. In a given undercooled molten sample, nucleation and growth kinetics between equilibrium and metastable phases compete in the microstructural development. This solidification behavior is evaluated through metallography, thermal analysis, and x-ray examination in conjunction with a heat flow model of the processing conditions.

Process parameter effects on undercooling and structural evolution are critical in the evaluation of drop tube processing. Under controlled conditions variations of particle size and processing environment have resulted in a multitude of microstructural developments in Ni-53 at% Nb. For example, a reduction in droplet size increased the fraction of droplets which undercooled to the glass transition temperature. This behavior is attributed to the isolation of internal nucleants and the increase in cooling rate which accompanies a reduction in droplet size. In addition, at a constant size distribution, an increase in the thermal conductivity of the gas environment leads to an increased fraction of amorphous powder due to increased cooling rates. Moreover, a change in gas can alter the catalytic potency of the surface through chemical reaction to allow nucleation and subsequent growth of a different metastable phase. Specific nucleation catalysts effects on structure formation have also been identified in Co-Cr alloys. A more thorough identification of these effects including surface chemistry analysis is currently being continued to understand the structure competition in undercooled liquids during containerless processing.

In drop tube processing, a quantitative analysis of the thermal history is required. Direct thermal measurement in drop tube powder processing is difficult, but thermal histories can be evaluated through alternate techniques. Millimeter size droplets of various Fe-Ni alloys can be dropped from a specified superheat, and the falling distance before solidification can be measured by adjusting a copper quench plate height. Undercooling levels at these various heights can then be calculated through a heat flow analysis. The solidified microstructures together with the determined undercooling can then be compared to a calculated metastable phase diagram to check the validity of the result. A compilation of size ranges and alloy compositions has led to the development of a processing-microstructure map to delineate regimes of structural/morphological evolution in drop tube processed Fe-Ni. Work to expand upon this analysis is being pursued currently.

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Role of Gravity on Macrosegregation in Alloys

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NAG3-723 (NASA Contact: A. Chait, LeRC)
April 1986 - April 1989

The major objective is to develop a comprehensive convection/solidification computer code to model macrosegregation in alloys that freeze in a dendritic mode. The finished code could be used to design experiments to study the effect of a low gravity environment on macrosegregation in binary alloys. It is also anticipated that the code would be used to assist engineers in designing or controlling commercial casting processes in which convection is driven by gravity.

In order to model macrosegregation phenomena in alloys which freeze dendritically, a quantitative analysis of solute redistribution is absolutely necessary. Hence appropriate forms of the mass, momentum and energy equations must be selected to predict each of these transport processes in the all-solid, liquid-plus-solid, and all-liquid zones of a solidifying casting. In addition to predicting macrosegregation variations across a casting or from its bottom to top, major emphasis is on modeling the intricate convective phenomena responsible for localized defects, often called "freckles", which are particularly troublesome to producers of ingots or castings. Here, multi-diffusive convection is thought to be responsible for the "freckles". When combined with thermodynamic data for gas-forming reactions, the basic solidification can be extended to predict the conditions when interdendritic porosity forms or, indeed, to predict the avoidance of such a defect. Because the overall program deals with defect-avoidance, it is expected that practitioners should derive significant benefit from the research.

A part of the early effort in the program has been in collecting and evaluating physical and thermal properties. Such data must be quantitatively analyzed so that extrapolations to the solidification temperature range can be made with confidence.

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Microgravity Solidification Processing of Monotectic Alloy Matrix Composites

Massachusetts Institute of Technology
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(NASA Contact: P.A. Curreri, MSFC)

Microgravity processing has great promise in the production of advanced metal matrix composite materials. In particular, elimination of gravity driven convection currents and instabilities may make it possible to fully utilize the unique wetting characteristics of monotectic alloys in the production of metal-matrix, non-metallic fiber-reinforced composites.

A coordinated project between MIT and NASA is proposed. The monotectic solidification in the presence of SiC, Al₂O₃, and graphite preformed fibers will be investigated for Al-In, Al-Bi, and Al-Pb alloys unidirectionally solidified at different growth rates and temperature gradients under microgravity and normal gravity conditions. The effect of the presence of non-metallic fibers on wetting conditions will be examined. The size and morphology of L₂ and solid α will also be determined under the constraints imposed by the scale of the interstices of non-metallic fibers preforms.

Containerless High Temperature Property Measurements

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January 1987 - January 1988

The objective of the research is to do advanced containerless processing and materials research at high temperatures in space. In this way, the production and processing of very pure and high quality forms of important ceramic, superconducting, semiconducting, very hard, very strong, and other useful kinds of materials may be achieved. New techniques adaptable to in-space work have been developed in earth-based research and the limits of earth-based containerless experiments are advanced and defined so that good choices for in-space R&D can be made. Methods of experimentation include gas jet and electromagnetic (EM) levitation, laser heating with or without EM heating, and laser induced fluorescence or mass spectrometric measurements of vapor and ambient gas concentrations. Non-contact temperature measurement is achieved by optical pyrometry. A new, absolute method for liquid specimen emittance measurement is under development.

Research to date has employed a combination of techniques for containerless experiments, including gas jet, and electromagnetic levitation, EM and CW CO₂ laser heating, laser induced fluorescence (LIF measurements of ambient and vapor atom concentrations and temperature measurements at high temperatures on materials of high purity and materials that react with containers.

Some of the results provide promising directions for continued earth based research. For example, optical properties of very pure single crystal sapphire were obtained at temperatures up to the melting point of Al₂O₃ (2327K) and accurate vapor pressures were measured for LaB₆, a material for which no non-reactive container material exists at the experimental temperatures (up to 2500K). Although there are other materials for which similar experiments would provide important results, such experiments would not further develop capabilities for space-based R&D because they can be completed entirely on earth. Stable gas jet levitation of laser heated liquids has not been achieved and it appears that acoustic positioning is the preferred method for containerless in-space R&D on liquids that are poor electrical conductors. For electrical conductors, EM levitation and heating is possible on earth and in space. In a low-gravity environment, the combination of radiant heating with EM positioning techniques promises very wide application. Low power, very high frequency levitators may be developed to extend in-space EM positioning of relatively poor conductors of electricity.

Electromagnetic levitation was used to achieve containerless conditions. CW CO₂ laser heating was used to heat the specimen above the minimum temperature achieved by EM heating. This improved levitation stability by allowing independent control of the levitation force and temperature. Vapor analysis is by laser induced fluorescence and, for experiments carried out in a vacuum, by mass spectrometry. An optical pyrometer is used to determine apparent specimen temperature, which can be corrected to the true temperature if the specimen's spectral emittance is known.

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Graphite Formation in Cast Iron

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NAG8-469 (NASA Contact: R. Mixon, MSFC)
June 1986 - May 1988

The objectives of this research are: (1) to better understand the solidification mechanics of cast-iron and similar type alloys using directional solidification experiments in microgravity; (2) to determine the contribution of gravity dependent effects on the final microstructure and properties of the alloys; (3) to investigate unique microstructures that may be obtainable by processing of alloys in a microgravity environment; and (4) to make the results of the study available for application in improving terrestrial casting techniques.

Results of experiments performed in the first part of the project are as follows. For Fe-C-Si alloys solidifying with stable eutectic (with either lamellar or spheroidal graphite), it was concluded that solidification under low-g results in a decreased number of eutectic grains, which could be attributed to a decrease in nucleation because of the change in the wetting properties of substrates occurring in low-g processing. Also, low-g processing resulted in an increase in the secondary dendrite arm spacing, with a subsequent decrease during high-g zones. Further it has been shown that buoyancy-driven graphite phase segregation can be prevented during low-g processing. In the metastable Fe-C eutectic alloys, a refinement of interlamellar spacing has been observed during low-g processing.

In the second part of the research the results are as follows. It was found for two different systems, Fe-C-Si and Fe-C-V, that primary particles (spheroidal graphite and vanadium carbide) tend to reach larger sizes when solidifying in the low-g zone as compared with the high-g zone, during parabolic flights. Calculations have shown that under the described experimental conditions, particles were either entrapped or have floated, which explains the rather complex microstructures obtained.

In the third phase of the research the results are as follows. Solidification of Fe-VC type in-situ composite under low-g and in a directional parallel to the gravity vector seems to be conducive to uniform dispersions of VC particles in the matrix and thereby to uniform microstructures. The best microstructures (spherical VC + α matrix), which will be conducive to superior mechanical properties, are obtained when the sample is solidified parallel to the gravity vector at very low growth rates or it is the same as solidifying it under very low growth rates in the space shuttle. The flotation characteristics of the VC particles appear to be well defined by Stokes law, however, solidification antiparallel to the gravity vector at rates higher than Stokes flotation velocity leads to change in the shape of the carbides, thereby complicating the situation. The physio-chemical interaction between the VC particles nucleating ahead of the solid-liquid interface perhaps also affects the particle distribution in the matrix as indicated in the samples solidified under variable gravity levels at rates higher than Stokes velocities (≥ 10 mm/min). In general, VC particle size is finer in portions of samples solidified in low gravity as compared to those solidified in high-g and the observation is in agreement with an earlier study.

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Microsegregation in Directionally Solidified Pb-8.4 At. Pct Au Alloy

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NCC-360 (NASA Contact: Dr. Hugh Gray, LeRC)

June 1986 - August 1987

The dependence of microsegregation behavior on growth rate and thermal gradient has been examined in a Pb-8.4 at. pct Au alloy material partially directionally solidified and quenched. The composition of the quenched "liquid" at the dendrite tip (C_t), that of the eutectic-like solid phase freezing from the interdendritic liquid at the base of dendrite (C_{se}), and the volume fraction of this eutectic-like region (f_e), and solute profiles in the interdendritic quenched liquid and ahead of the dendrite have been measured. Two dendritic growth models for solidification of a binary alloy melt in a positive thermal gradient at the liquid-solid interface, one for dendrites with "minimum undercooled dendrite tip" and the other for an Ivantsov type of dendrite with "marginally stable tip," have been examined for a quantitative comparison with measured values of c_t , C_{se} , and f_e . Convection in the melt, possibly due to horizontal density gradients, is found to be a serious limitation for theoretical understanding of the observed experimental behavior and meaningful comparison of theories.

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Cellular/Dendritic Solidification of Binary Alloys in a Positive Thermal Gradient

Cleveland State University

Dr. S. N. Tewari

Dr. A. Chopra

NCC-395 (NASA Contact: Dr. Hugh Gray, LeRC)

September 1987 - September 1989

An experimental ground based program is planned to study the development of cellular/dendritic microstructures during directional solidification of binary metallic model alloys in a positive thermal gradient. Cell to dendrite transition behavior will be investigated in Pb-Sn, Pb-Au and Pb-Tl alloys. Important microstructural features, the cell/dendrite tip radius, tip temperature, liquid composition at dendrite tip and primary arm spacing will be measured as a function of processing variables, such as, growth speed, thermal gradient and solute partitioning coefficient. The experimentally observed behaviors will be examined against theoretical predictions from cellular/dendritic growth models.

Containerless Studies of Nucleation and Undercooling

Jet Propulsion Laboratory
Dr. Eugene H. Trinh
Dr. T. G. Wang
January 1987-January 1988

The long term research goals are to perform experiments to determine the achievable limits of undercooling using acoustic levitation, to study the characteristics of heterogeneous nucleation of levitated samples, and to measure the physical properties of significantly undercooled melts. Specially designed ultrasonic levitators operating in ground based laboratories as well as in the KC-135 NASA aircraft are to be used to investigate 0.1 to 3 mm specimens of pure metals and alloys (Ga, In, Sn, Al-In,...) as well as glass-forming organic compounds (0-Terphenyl, low melting glasses). Non-invasive measurement techniques for the surface tension, viscosity, density, sound velocity, and perhaps specific heat, are to be developed and refined to probe the physical state of undercooled levitated melts.

Additional data on the surface tension of undercooled liquid Indium have been obtained, confirming previous results. The density of substantially undercooled 0-Terphenyl has been determined down to -15 C. A ground-based study using levitated samples of undercooled Water, 0-Terphenyl, and indium has been initiated to quantitatively determine the effects of the high intensity acoustic field. No dependence on the acoustic frequency between 20 and 40 kHz has been detected, and no consistent dependence on the acoustic pressure level range permissible in 1-g has been found. The investigation of the effects of sample rotational and vibrational motion on the onset of solid phase nucleation is being carried out at the same time as the experimental study of the crystal growth phenomena in levitated melts. Also of primary interest at the present time is the convective flow fields induced within the melt by the acoustic streaming in the host gas; the principal effort is now to study methods to minimize any fluid convection inside the liquid sample.

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Ostwald Ripening of Solid-Liquid Mixtures

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The objective of this program is to use the unique conditions provided by space flight to study the kinetics of Ostwald ripening. The data derived from this experimental work will provide baseline data for the field and thus permit the refinement of existing theories of the kinetics of first-order phase transformations. In addition, as the Ostwald ripening process has a major impact on the properties of materials, the experimental results will yield information which can be used to improve the properties of materials containing dispersed phases.

A particularly ideal system to use in these experiments is a mixture consisting of solid particles in a liquid. Since the coarsening rate in such a system is comparatively fast, and in a properly chosen system the solid particles can be spherical, the experiments can serve as a careful test of theory. However, experiments performed using a low volume fraction solid, where the theory is most accurate under terrestrial conditions, shows that buoyancy driven convection of the solid particles is prevalent and thus the experiments do not satisfy the theoretical requirements of fixed spatial locations of the particles. To eliminate this problem the experiments will be performed in the reduced gravity environment of space.

We have located a solid-liquid mixture in which the materials parameters necessary to compare the experimental results to the theoretical predictions are known and developed an experimental protocol necessary to produce a dispersion of solid particles in a liquid. We have examined the coarsening kinetics of solid particles in a liquid in the volume fraction solid range above 0.6 where the development of a solid skeletal structure inhibits particle sedimentation. The experimentally measured coarsening rate constants are found to exceed those calculated from theory by factors ranging from 2 to 5. Possible causes for the disagreement between theory and experiment are the movement of particles within the skeletal structure due to density differences between the solid and liquid phases or convection of the liquid matrix. Only experiments in a microgravity environment will eliminate conclusively these possibilities. Numerical calculations of the morphologies of solid particles in high volume fraction solid-liquid mixtures were performed in an effort to explain the experimentally observed particle morphologies. These calculations show that the experimentally observed particle morphologies are due to strong diffusional interactions between the coarsening particles.

Publications

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Influence of Convection on Microstructure

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NAG8-480 (NASA Contact: P. Curreri, MSFC)
June 1984 - June 1987

The objective of this research is to gain an understanding of the influence of microgravity on the microstructure of the MnBi-Bi eutectic.

David Larson and Ron Pirich of Grumman have shown that directional solidification of the MnBi-Bi eutectic in space results in a fiber spacing 1/2 of that obtained by solidification on earth under otherwise identical conditions. We had shown previously that the microstructure is unaffected by temperature gradient and that the microstructure responds more quickly to a change in freezing rate than the freezing rate changes in response to a change in ampoule translation rate.

Computer computations have been carried out with a planar interface for the influence of convection on the compositional field in front of lamellar and fibrous eutectics, and the resulting effect on microstructure. Experimental results with spin-up/spin-down (Accelerated Crucible Rotation Techniques) gave good agreement with predictions for lamellar eutectics. However buoyancy-driven convection is calculated to be too weak to noticeably influence the microstructure.

Recent electrochemical experiments have shown that spin-up/spin-down causes large fluctuations in mass transfer, and therefore in heat transfer and freezing rate during solidification. Decantation experiments have shown that the MnBi fibers project for large distance into the melt during solidification, and that they infrequently branch. Elevated temperature fracture of samples have also showed little branching. Temperature measurements showed large oscillations in the melt without ampoule rotation, with the amplitude decreasing as the solid-liquid interface is approached.

Spin-up/spin-down experiments were also performed on the lead-tin eutectic. While the lamellar spacing was unaffected under the conditions used, spiralling of the microstructure depended strongly on rotation rate. Rotation also caused one end of the ingot to be lead rich while the other was tin rich.

Publications

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Modelling Directional Solidification

Clarkson University
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NAG8-541 (NASA Contact: F. Szofran, MSFC)
January 1, 1987 - January 1988

The objective of this research is to develop an improved understanding of some phenomena of importance to directional solidification, to enable us to explain and predict differences in behavior between solidification on earth and solidification in space.

Experiments on organic compounds showed that in contrast to recent computer models, the convection in a vertical Bridgman-Stockbarger ampoule is usually not axisymmetric and may vary with time. If the temperature in the furnace increases with height the convection may be greatly suppressed. On the other hand, if the temperature decreases with height the convection may be vigorous. Theoretical models have built into their initial equations steady state, axisymmetric flow, and a constant heater temperature. Experiments are underway to determine the influence of convection on compositional homogeneity of directionally solidified organic compounds.

KC-135 experiments showed that liquid in a non-wetted cylindrical ampoule does not pull away from the ampoule walls in low g, as has been proposed to explain the observation that ingots solidified in space often have diameters smaller than their containing ampoules. Rather the liquid separated into separate columns or bubbles formed along the walls. Theory predicted that bubbles are unstable beyond a critical size. This prediction agreed with experiment. Likewise experiment and theory agree on the fraction of the flat walls in a triangular ampoule that are in contact with the melt (the liquid pulls away from the corners.) A new transparent solidification apparatus being built by MSFC should help resolve the mystery of the reduced diameter ingots obtained in space.

Theoretical analyses were done on the diffusional decay of compositional variations in a crystal during the period it is cooling to room temperature. Decay of striations is favored by a slow freezing rate, a small period for the composition variations, a small temperature gradient and a large diffusion coefficient in the solid. Under some realistic conditions striations decay within a few wavelengths, while under other conditions they may persist for some distance. Thus the compositional inhomogeneities observed in a grown crystal are not necessarily indicative of those produced at the growth front.

Apparatus and procedures were developed to determine the influence of spin-up/spin-down and of freezing rate fluctuation on the perfection of directionally solidified InSb-GaSb alloys. (Mullard Laboratories in Southampton, England have shown both increased homogeneity and much larger grain size caused by spin-up/spin-down during solidification of HgTe-CdTe alloys. The mechanism responsible for increased grain size is unknown.)

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3. FLUID DYNAMICS AND TRANSPORT PHENOMENA

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Experimental and Theoretical Studies of Wetting and Multilayer Adsorption

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Dr. R. F. Kayser

Dr. M. R. Moldover

Dr. J. W. Schmidt

April 1977 - continuing task

The Structure of a Fluid Interface

The structure of the liquid-liquid interface in three mixtures (carbon disulfide + methanol, methanol + cyclohexane + deuterated cyclohexane and nitrobenzene + n-decane) has been studied using ellipsometry in the reduced temperature range $0.0009 < t < 0.042$. Although the ellipticity data varies by a factor of 10 between mixtures all three mixtures can be scaled to the same universal constant by a combined mean field plus capillary wave model of the interface.

Systematics of Wetting

Five first-order wetting transitions have been located at the vapor-liquid interface for a series of alcohol + fluorocarbon mixtures. Contact angles of fluorocarbon-rich pendant drops (suspended at the vapor-liquid interface) were measured for the series. In addition surface tension for the fluorocarbon-vapor, alcohol-vapor, and liquid-liquid interfaces were measured using a modified Du Nouy ring technique.

Interfacial Tension in the Critical Region

Methods for accurately predicting the interfacial tension of binary mixtures near their liquid-vapor critical lines were developed in collaboration with Dr. J. Rainwater (NBS Boulder). The general method is applicable to miscibility gaps encountered in many systems considered for materials processing in space.

Wetting Layers on Solid Substrates

A new derivation of wetting layer thickness has been obtained for wetting layers consisting of two fluid phases coexisting near a substrate. In cases in which high dielectric fluids are in contact with ionizable substrates, dispersion forces in competition with gravity cannot account for the thicknesses of the observed wetting layers. The present derivation differs from that of dispersion forces and arises when a solid surface can become electrically charged.

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Thermo-Diffuso Capillary Phenomena

Lewis Research Center
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Dr. C.L. Lai
Dr. R. Balasubramanian
In-House

The objective of this program is to conduct fundamental microgravity research on fluid motion generated by temperature and/or concentration gradients due to surface tension and/or buoyancy.

The research being conducted involves three areas of interest: (1) thermocapillary convection and oscillation; where progress has been made in studying the effect of thermal conductivity of the end walls. No oscillatory behavior has been observed to date; (2) thermocapillary motions of bubbles and droplets in a thermal gradient in a host fluid. In this area analytical studies have been performed including effects of inertia and convection. Numerical and experimental investigations and the effects of concentration gradients are planned; and (3) thermal and double-diffusive convection due to presence of temperature and/or concentration gradients; where analytical studies are underway to obtain detailed understanding of the flow with both inertia and convection present.

Publications

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Convective and Morphological Stability during Directional Solidification

National Bureau of Standards

Dr. S. R. Coriell

Dr. J. R. Manning

Dr. G. B. McFadden

Dr. R. J. Schaefer

W-16, 171 (NASA Contact: Roger Crouch, NASA HQ)

December 1987 - November 1988

The general aim of this task is the theoretical and experimental study of the fluid flow, solute segregation, and interface morphology which occur during directional solidification, including effects of gravity and microgravity. Space flight experiments, designed to determine cellular wavelengths as a function of growth conditions, are planned in collaboration with J. J. Favier and D. Camel of the Centre d'Etudes Nucleaires de Grenoble utilizing the directional solidification furnace being developed by the MEPHISTO project.

During solidification of an alloy at constant velocity, thermosolutal convection can occur. The effects of this convection on the solute segregation in crystals grown by vertical directional solidification of binary metallic alloys or semiconductors has been calculated using finite differences in a two-dimensional, time-dependent model that assumes a planar crystal-melt interface and small Prandtl number. As the solutal Rayleigh number is varied, multiple steady-states, time-periodic states, and quasi-periodic states may occur. Numerical calculations of the solute, temperature, and flow fields are being carried out for a variety of conditions, including time dependent gravitational accelerations (g-jitter) and both stress-free and rigid lateral boundaries.

Three-dimensional steady-state solutions for nonplanar interface morphologies are computed numerically by using finite differences. A linear temperature field is assumed; the solute field in the melt and the crystal-melt interface position are computed self-consistently. For a model of an aluminum-chromium alloy with distribution coefficient greater than unity, steady-state solution corresponding to two-dimensional bands and three-dimensional hexagonal nodes are obtained, as well as solutions with rectangular interface platforms. Near the onset of instability, the calculations predict hexagonal nodes, which is consistent with weakly nonlinear theory. In collaboration with R. F. Sekerka of Carnegie-Mellon University, the weakly nonlinear theory has been extended to take account of nonlinear temperature fields and anisotropic surface tension.

Linear stability analyses are being applied to a number of problems associated with the morphology of the crystal-melt interface. For example, in collaboration with A. A. Wheeler of the University of Bristol and D. T. J. Hurle of the Royal Signals and Radar Establishment, the effect of an electrical current on the morphological stability of planar interface during directional solidification of a binary alloy at constant velocity has been investigated. Electromigration of solute and the perturbation of the electric field by the perturbed crystal-melt interface modify the conditions for morphological stability. The model is being extended to take account of joule heating and thermoelectric phenomena, namely, the Peltier, Seebeck, and Thomson effects. Investigation of the effect of time-dependent electric currents on the solute distribution at a planar crystal-melt interface is planned.

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Theory of Solidification

NASA Lewis Research Center

Prof. Stephen H. Davis

NAG 3-747

October 14, 1986 - October 13, 1989

The research concerns the effort to understand on a quantitative level how various factors affect the morphology of a solidification front of binary materials. These factors include buoyancy-driven convection with and without Soret diffusion, phase-change convection, crystal and kinetic anisotropies and effects of bounding surfaces. The central theme is the understanding of the phenomena through the study of the instability behavior of the appropriate coupled systems.

The research entails the study of instabilities in coupled systems that describe the directional solidification of a binary material from the melt. The study encompasses double-diffusive convection, crystal growth, and the understanding of the phenomena through the study of the instability behavior of the appropriate coupled systems.

The research entails the study of instabilities in coupled systems that describe the directional solidification of a binary material from the melt. The study encompasses double-diffusive convection and forced flows coupled to the phase-change process. It includes effects of bounding surfaces and material anisotropies. It utilizes nonlinear stability theories, asymptotic and numerical methods.

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Computer Simulations and Experimental Observations

Princeton University
Dr. P. G. Debenedetti
Dr. W. B. Russel

At equilibrium a concentrated colloidal suspension will assume an ordered state if the volume fraction exceeds a certain value, whose magnitude depends exclusively upon the nature of the reversible interparticle forces. The dynamics of the transition, however, are governed by the irreversible interactions between the particles and the surrounding fluid, and are of fundamental importance in determining the ultimate morphology of many densely packed systems formed in processes of technological relevance (sedimentation, ultrafiltration, slip casting).

The proposed work will address this problem through computer simulations and experiments. The former would represent the first three-dimensional study of the dynamics of concentrated colloidal suspensions with realistic descriptions of hydrodynamics, interparticle potentials, and Brownian motion. These simulations will address problems which cannot be studied within the framework of equilibrium statistical mechanics, including the evolution of the morphology and the properties and stability boundaries of metastable states.

Experiments with well characterized particles under conditions similar to those being simulated will be conducted in order to test the influence of gravitational forces upon the phase transition. This will allow us either to proceed with more quantitative (light scattering) work on Earth or to design experiments to be performed in a microgravity environment.

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. A.T. Chai, LeRC)
November 15, 1983 - January 14, 1987

The long term objective of the experiment is to observe the dissolution of isolated immobile bubbles of specified size and composition in a solvent liquid of known concentration in the reduced gravity environment of earth orbit. Preliminary bubble dissolution experiments conducted both in the NASA Lewis 2.2 sec. drop-tower and in normal gravity using the SO₂-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 SO₂ in Toluene had 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 verify the concept of having a bubble with a critical radius in a state of unstable equilibrium. The method of bubble injection is continuing to be that of syringe injection, which is acceptable at this stage of the feasibility study. The critical radius concept is of major importance 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 local background concentration of the dissolved gas in the liquid has been attained. This background concentration is not uniform throughout the liquid, which makes subsequent bubble dissolution data of less value than desired. The critical radius concept is ready to be tested using the CO₂-Toluene system in normal gravity. An improved prototype experiment package has been designed and constructed for this purpose at NASA Lewis. Bubble rise will be prevented by the use of very fine fibers. After establishment of a bubble in critical equilibrium is achieved in normal gravity, Lear Jet tests (23 sec. of free-fall time) will be conducted in order to refine the injection system and to determine whether a critical bubble can be stabilized in this time period. Finally, injection hardware will be further examined in the NASA Lewis drop-towers, and work will begin in the conceptual design of the middeck experiment.

The total experiment will involve the injection of a single bubble of gas of approximately a prescribed size and composition into a quantity of thermostated 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.

Suppression of Marangoni Convection in Float Zones

George Washington University
Dr. Robert F. Dressler
NAG1-325 (NASA Contact: A. Fripp, LaRC)
January 1987 - January 1988

The objective of this research is to demonstrate, by means of a 1-g experiment, that the idea of space processing to use tangential gas jets for suppressing the unwanted thermal-capillary (Marangoni) convection always present in a float zone, is valid and efficacious.

For proposed processing of highly reactive semiconductor materials, e.g. silicon, in microgravity (g), although the thermal-buoyant convection will be suppressed in g , this will not reduce the Marangoni convection since there must always be a temperature gradient, hence a surfacetension gradient, in a float zone. Our idea for space processing is to blow jets of a non-contaminating gas, e.g. argon or xenon, tangentially over its free molten surface to establish a shear stress to counterbalance the surface-tension shear which excites the Marangoni convection. Since the principle involved is identical, our earth-based experiment uses an air jet and a transparent silicone oil in a half-float zone configuration to demonstrate that the Marangoni convection can be significantly reduced by our method. There are three major difficulties due to 1-g in our experiment, but in spite of these, we have attained an average reduction of 66 to 75% in the Marangoni velocities, showing our idea is workable. We are now engaged in the initial planning for a new project, using a middeck Shuttle experiment, in which all three 1-g problems will be eliminated. Therefore, we expect our anticipated g experiment will attain reductions better than about 98%. This will then indicate use of our method for commercial fabrication of semiconductors in the Space Station.

Heat and Mass Transfer in Zero Gravity

National Bureau of Standards - Boulder Laboratories

Dr. Patricia J. Giarratano

Dr. Vincent D. Arp

W-16, 170

January 1987 - January 1988

The objective of this work is to provide predictive techniques in the form of computer codes and correlations for applications in the design of heat and mass transfer equipment, especially in systems in which transients occur. Our existing mathematical computer model describes transient heat transfer prior to the onset of gravity-driven fluid motion. The model includes the effect of motion induced by the thermal expansion of the fluid adjacent to a flat geometry heater surface and predicts the temperature profile in the fluid during a transient heat pulse. A near-zero-gravity environment is necessary to study this thermally induced motion because in earth gravity the effect is masked by buoyancy-driven convection in the fluid. Mach-Zehnder interferometry was the measuring technique used to study the temperature field in experiments in the laboratory and during two series of flights on the KC-135. Reference (1) contains a description and preliminary zero-g data of this work. This measurement technique proved inadequate for measurement of the temperature fields in the very thin boundary layers developed during the heat pulse. Therefore the research this year has focused on exploring the suitability of a special holographic technique which employs a diffuse light source instead of a collimated beam. Preliminary ground tests using holography has allowed considerable more of the boundary layer to be optically probed. This fiscal year the holographic technique has been tested during one series of flights on the KC-135. Complete evaluation of the equipment and the resulting holograms has been completed although preliminary results are encouraging (cursory examinations of the holograms show a discernible fringe pattern adjacent to the heater surface). Reference (2) summarizes the results of the project to date. Ground tests on the holographic technique and equipment are continuing.

Publications

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Combined Buoyancy-Thermocapillary Convection: An Experimental Study

Stanford University
George M. Homsy

Thermocapillary convection is now well-recognized as one of the major sources of convective mixing of fluids in a microgravity environment. It is furthermore apparent that the environment of a spacecraft is not completely free of either residual or transient accelerations that may also cause convection. Thus convection caused by the combined mechanisms of buoyancy and thermocapillary is of interest. In spite of its importance, very few experimental investigations of this class of motions exist, and where they do, the range of parameters studied is often limited. Although the nature of convection is fairly well understood when each mechanism acts separately, very little is known about the structure of the flow and the convective transport in the combined case. Even less is known about possible instability phenomena and instability modes when the relevant parameters become large. Accordingly, we have undertaken an experimental study of the problem in a well-characterized and controlled geometry, namely a rectangular channel.

We are considering the simple prototype problem of convection in a rectangular channel with a free surface, heated from one side and cooled from the other. The strength and nature of the convection is determined by the aspect ratios of the container, and the magnitudes of the Rayleigh, Ra , Marangoni, Ma , and Prandtl, Pr , numbers. We are generally interested in large Ra and Ma , as convection dominated over conduction in that case. The objectives of the research are to: (1) establish the conditions under which the motion is two-dimensional and steady; (2) characterize the convection by a combination of flow visualization and laser-speckle velocimetry; (3) identify the qualitative nature of the flow as a function of the ratio, Ma/Ra ; and (4) observe any instabilities, measure the critical parameters for onset of instability, and characterize them with respect to their temporal and spatial variation.

We have chosen a low viscosity silicone oil as the working fluid. Theoretical considerations indicate that the observed phenomena may be most easily interpreted in the case of moderate Pr : our fluid has $Pr = 8.5$. The apparatus consists of a cell of nominal dimension 1 cm x 1 cm x 5 cm. The combination of fluid properties and physical dimensions allows experiments in which both mechanisms contribute to the observed flows. An optical technique is used whereby a chopped laser sheet is used to illuminate the flow in a vertical plane. The fluid is seeded with small tracer particles, the scattering from which is used for both qualitative visualization and quantitative laser speckle velocimetry.

There have been some preliminary results which still await further experiments and a careful analysis. The flow structure consists of a relatively large thermocapillary eddy near the free surface, which penetrates deeply into the fluid on the cold wall, but less deeply as the circulation approaches the hot wall. It has also been seen that quantitative measurements of the velocity vector is possible from analysis of the spacing and orientation of fringes. As Ma and Ra are increased, the flow takes on a boundary layer character, with a strong thermocapillary boundary layer near the free surface, and thin buoyancy layers near the vertical side walls. Thermocapillarity drives a central vortex, while buoyancy produces a stable vertical stratification, limiting the vertical velocity. Serial sectioning of the flow shows that for $Ma < 3 \times 10^4$ and $Ra < 4 \times 10^4$, the flow is two-dimensional. For parameter values substantially in excess of these, there

is strong evidence for a three-dimensional flow pattern. We are currently studying the nature of this pattern, which is indicative of a new instability mode.

Center for Microgravity Fluid Mechanics and Transport Phenomena

University of Colorado
Professor D.R. Kassoy
Professor R. Sani
NAGW-951 (NASA Contact: R.K. Crouch, HQ)
September 1, 1986 - March 31, 1989

The Center has developed an integrated program of research and education in low-gravity science and technology. Participants in the program, now numbering twenty faculty, students and research associates, focus on the role of fluid mechanics, heat and mass transfer in materials processing, fluid handling, thermal management and combustion. The research projects are interdisciplinary in character. Investigators interact directly with engineers and scientists in laboratories and industries concerned with specific processes and technologies.

The Center has initiated research activity in the following areas:

- (1) *Modeling and Experiments on Fluid Systems with G-jitter*, P.D. Weidman and S. Biringen

Computational methods are used to model jitter effects in a thermospan heat loop and on buoyancy-driven convection in rectangular cavities and three-dimensional boxes. Ground-based jitter experiments are designed to measure effects on convection in fluid-filled boxes as well as those with a free surface.

- (2) *Gradient Induced Convection in Materials Processing*, W.B. Krantz

Mathematical modeling is used to describe convection induced by the presence of very large thermal or concentration gradients at an interface, in the absence of gravity. This newly discovered effect arises from localized molecular interactions occurring near an interface between two dissimilar fluids.

- (3) *Low Gravity Effects on Thermoacoustic Convection in Helium*, D.R. Kassoy

Modeling methods are used to ascertain the magnitude of thermoacoustic convection induced in confined gaseous helium by heat transfer from the confining boundary. The competition between thermoacoustic, and buoyancy-induced convection at reduced gravity levels is analyzed.

- (4) *Formation of Immiscible Alloys in Low Gravity Conditions*, R.H. Davis

A theoretical framework is developed for mechanisms responsible for phase segregation in immiscible materials processed at reduced gravity levels. Predictions are made for processing conditions necessary to produce desired microstructure in a given material mixture.

- (5) *Computer Aided Analysis of Floating Zone Processing*, R.L. Sani

Finite element numerical algorithms are being developed to model the flow, transport and stability of floating zone processing configurations. Interactions between convection, heat and mass transport, as well as deformable free surface effects are emphasized.

(6) *Manufacturing Spherical Shells under Microgravity Conditions*, C.Y. Chow

Electromagnetic-capillary instabilities on liquid metal cylinders are studied in order to determine the feasibility of manufacturing spherical shells from thin-walled metal tubing. Magnetohydrodynamic pinch effects are found to be an effective means for dividing the cylinder into elements of uniform size. Surface tension then generates spherical shells.

(7) *Thermal Instabilities in Low-Prandtl Number Liquids*, J.E. Hart

Experiments in an annular mercury bath, heated on one vertical wall and cooled on the other, are used to discover an assortment of novel convective instabilities. Finite element computations are employed to resolve internal motion not accessible to measurement.

In addition to these University-based research projects, another Center participant, H. Snyder, has developed an expertise in fluid handling problems, particularly superfluid helium transfer, through an interaction with Ball Aerospace in Boulder, Colorado.

The Center provides graduate level courses in low-gravity sciences and in materials processing in space. In addition, the University of Colorado provides financial support for the Center-sponsored Science Seminar. This weekly series gives both U.S. and international experts an opportunity to report on the latest development in low-gravity science and technology. Approximately 22 students participate in these educational programs.

The Center fosters extensive interactions with scientists and engineers in other universities, in government laboratories, and in industry. Cooperative research projects are being developed and long-term visits have been arranged. The Center seeks the broadest possible participation by members of the low-gravity community.

Publications

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Experimental Investigation of Surface-Tension-Driven Convection as a Feasibility Study
for a Microgravity Experiment

University of Texas
Professor E. L. Koschmieder
NAG3-393 (NASA Contract: J.A. Salzman, LeRC)
July 1988 - July 1989

This effort is a follow-on study of ground-based laboratory experiments on surface-tension-driven Benard convection (Pearson's problem) generated in a fluid layer heated uniformly from below and cooled from above by an air layer. 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 adaptation 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 greater than 2mm 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. If then the temperature difference is increased, a second instability occurs which transforms the first weak pattern into conventional strong hexagonal Benard cells. The second instability is in agreement with the critical temperature gradients predicted by Nield.

The motivation for pursuing thin fluid layer experiments was to attempt to test Pearson's theory in an earth-based laboratory. The presence of unforeseen subcritical instabilities which were encountered show that this clearly cannot be accomplished. If we want to verify Pearson's theory we will have to conduct an experiment in a low-gravity environment where the Rayleigh numbers are vanishing because g is so small and where we therefore can make experiments with deeper fluid layers which are neither affected by subcritical motions nor by buoyancy.

The objective of the current effort is to establish the feasibility of designing a space-based experiment to clearly test existing theories on Benard convection. Specific issues which will be addressed center on the time line of the experiment (i.e., can the heating rates be increased), a better understanding of the subcritical motion under steady conditions, and the impact of non-zero gravitational levels, or g -jitter, on the integrity of the experiment operation and results.

Publications

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Fundamental Study of Nucleate Pool Boiling under Microgravity

University of Michigan

Professor Herman Merte, Jr.

NAG3-663 (NASA Contact: F. Chairamonte, LeRC)

October 1985 - February 1989

This research is part of a program for the study of the fundamentals of nucleate pool boiling heat transfer under the microgravity conditions of space, seeking to improve the understanding of the basic processes that constitute boiling by removing the buoyancy effects which mask other phenomena, and which will be part of the development of data base for space applications of boiling.

Freon 113 is the initial fluid being used in a closed vessel with the pressure being maintained constant. The independent variables are subcooling and heat flux, with a step increase from zero to a prescribed power input. Measurements of space temperature are made simultaneously with motion photography during the transient heating process, including the onset of boiling, until terminal condition is reached appropriate to the particular circumstances present. Two heating surfaces are being developed: a semitransparent layer of gold vacuum deposited on a quartz substrate, which acts simultaneously as a well-defined electrical heater and resistance thermometer, and which permits viewing simultaneously from the side and beneath the boiling surface, and a copper surface indirectly heated electrically, gold coated so as to present the same surface energy conditions to the boiling fluid. Testing will be conducted in the laboratory at $a/g = \pm 1$, and in a drop tower for short term microgravity. Plans are being developed for subsequent orbital flight in the shuttle to provide the longer time periods necessary.

Influence of Time-Dependent Gravitational Acceleration in the Presence of Magnetic Fields on the Fluid Dynamics and Heat Transfer in Solidification Processes

Massachusetts Institute of Technology
Professor S. Motakef
NSG-7645 (NASA Contact: R.K. Crouch, NASA HQ)
October 1, 1986 - September 30, 1988

The goal of this research is to determine how effectively various low duration low-g vehicles achieve microgravity conditions during bulk growth of semiconductor crystals.

This study numerically analyzes the transient response of buoyancy-driven flows on low prandtl number melts to variations in g-level. Using recorded g-level data from KC-135, space processing applications rockets (SPAR) and TEXUS flights, the results of this analysis are used to establish the duration of low-g periods on board these vehicles.

The transient behavior of natural convection in unidirectional solidification processes has been thoroughly investigated both from fundamental fluid mechanic considerations as well as calculations related to NASA KC-135 aircraft and TEXUS and SPAR sounding rockets. The time constants of convection in the melt of semiconductors to step increases and decreases in g have been calculated in the range of 0-1.5g for the MIT Bridgman-Stockbarger system. The time constant for step increases in g is controlled by the relative dominance of inertial and viscous forces. For step reductions in g , the system response is controlled by the momentum diffusive time scale in the melt. Transient analysis has been extended to study the solidification processes on board KC-135 and sounding rockets using the recorded g-level data. The effectiveness of these vehicles is controlled for a given material by the charge size and is, to first order, independent of furnace design. The relationship between the duration of low-g growth and the charge size for various semiconductors on board the low duration low-g vehicles is calculated. The present KC-135 furnaces do not appear to provide the necessary low-g period for meaningful experimentation, whereas TEXUS rocket furnaces do provide sufficiently long low-g growth periods of up to 4 minutes.

The response of convection in the melt to periodic variations in the g-level has also been investigated by conducting a frequency response analysis. The system response is controlled by the momentum diffusive time scale; at oscillating frequencies less than the diffusive time scale the convection in the melt follows the periodic variations in the g-level, and at higher frequencies the amplitude of the oscillating low velocities decrease linearly with the oscillating frequency. Using the recorded g-level data, it is shown that g-jitter on board KC-135 and sounding rockets, as well as the shuttle, do not significantly interfere with convection in the melt at low-g levels.

Publications

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

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Professor Daniel F. Jankowski
NAG3-568 (NASA Contact: Dr. A.T. Chai, LeRC)
September 1987 - September 1988

Finite-element and finite-difference methods have been used to calculate the basic state in a model of the float-zone, crystal-growth process. A temperature gradient along a nondeformable free-surface generates thermocapillary convection. The energy-stability limit (Ma_E , a value of the Marangoni number below which stability is guaranteed) for this basic state has been calculated from discrete versions of the quadratic functional that defines the limit. The numerical procedures have been verified by their application to the basic state in a cylinder heated from below.

The problem has provided to not only a significant undertaking from the standpoint of fluid mechanics and stability theory, but also requires state-of-the-art computational techniques and computer resources. In particular, the stability calculation requires the treatment of large, sparse, generalized, algebraic eigenvalue problem without the property of positive-definite matrices. In addition, the problem has many parameters which hinder an orderly presentation of the results. However, for a certain meaningful range of these parameters, there is reasonable agreement between the finite-difference and finite-element stability results. Both methods show the expected increase in Ma_E with increasing buoyancy. Both methods show that the "linking parameter" can have a significant effect on the stability results, with Ma_E increasing as this parameter decreases. In spite of these positive comparisons, there is a difficulty associated with varying Prandtl number that is currently being investigated.

Publications

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Modeling of the Thermoacoustic Convection Heat Transfer Phenomenon

University of Tennessee, Knoxville
Professor M. Parang

The objectives of the research are to: (1) formulate and appropriately scale the governing equations for the three dimensional, unsteady, compressible flow in a confined geometry with various thermal boundary conditions of interest; (2) develop and formulate a numerical scheme for the solution of a special case of the governing equations and boundary conditions applicable to the experimental conditions; (3) solve the governing equations and obtain numerical solutions for various special cases of interest; and (4) compare and verify the numerical solutions with experimental results. The investigation will focus on the development of a numerical model of the thermoacoustic convection (TAC) phenomena with special emphasis on the verification of the model by means of the experimental evidence obtained by the principal investigator.

A recently completed investigation of thermoacoustic convection (TAC) heat transfer phenomena establishes the importance of this convective process in space manufacturing, materials processing, and fluid handling and storage in the space environment. That study suggests that TAC heat transfer can be an important mode of heat transfer in microgravity conditions for reducing the transient time in heating of fluids and can produce up to two orders of magnitude higher heat transfer rates relative to a pure conduction heat transfer mode. However, numerically-computed solutions obtained based on the available TAC heat transfer models in the literature fail to agree with the results of that experimental investigation. In the proposed project a study of TAC heat transfer process will be undertaken to resolve the important discrepancy between the numerical and the experimental results.

Breakdown of the Non-Slip Condition in Low-Gravity

Los Alamos National Laboratory

Dr. Donald R. Petitt

C-32005-K (NASA Contact: R.A. Wilkinson, LeRC)

May 1988 - May 1989

This endeavor is a follow-on study of experiments that indicated anomalous slip of liquids at a solid wall in the low gravity environment of a KC-135 parabolic trajectory. If the effect is real, important consequences obtain for space based fluid systems. As a consequence, the critical output of this effort is to determine the validity of the established no-slip boundary condition in low-gravity.

The purpose of the experiment is to unambiguously determine if the established no-slip boundary condition of continuum Fluid Mechanics breaks down (by some degree of slip) in the low-gravity environment of KC-135 parabolic trajectory flights. The factors considered will be shear rate dependence, viscosity dependence, surface adhesion dependence, and the effects of orientation of couette flow rotation axis with respect to the gravity vector in earth gravity and low gravity. The activity requires building up a couette flow torsional viscometer with several sleeve walls, performing earth-gravity experiments, executing nine KC-135 flights for different fluids and sleeves, and finally analyzing/documenting results in a report or journal publication.

Morphological Stability and Fluid Dynamics of Vapor Crystal Growth

University of Alabama in Huntsville
Professor Franz Rosenberger
NSG-1534/NAG1-733 (NASA Contact: Dr. A.L. Fripp, LaRC)
January 1987 - January 1988

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 the experimental and numerical work performed under this grant 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. Concentration fields in the vapor are studied by thermal deflection spectroscopy. Concurrently the evolution of the macroscopic morphology of the crystals are recorded by Moiré reflectometry.

The interfacial kinetics aspects of MS in vapor growth are being addressed through macroscopic studies of growth 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 transition. In addition we have expanded the traditional statistical treatment of atomic surface roughness to include the variation of bond strength at surfaces. The resulting model predictions agree well with experimental observation, in contrast to the constant bond 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 bench mark experiment in space.

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Hydrodynamic Instability as the Cause of Morphological Breakdown during Electrodeposition

Massachusetts Institute of Technology
Professor D. R. Sadoway
Professor R. A. Brown
NSG-7645 (NASA Contact: R.K. Crouch, NASA HQ)
October 1, 1987 - September 30, 1988

The objective of this work is to understand the origins of surface roughening during the course of electroplating. Specifically, the central question is the role of hydrodynamic instability in the electrolyte in initiating and promoting morphological breakdown during the process.

The work combines experiments to measure flow characteristics and resulting surface structures with calculations of critical physical parameters. The study is unique in several respects. The causes of morphological breakdown during electrodeposition have never been the subject of a systematic study that seeks to investigate the problem under conditions where the kinetic processes are clearly defined. Specifically, this study is conducted in a physical model system that will permit the observation of simple electrocrystallization under conditions of strict mass transfer control. Furthermore, this study is unique in its attempt to combine experimental observations with mathematical models of complex fluid flow behavior originally developed for the analogous solidification problem in crystal growth. For these reasons, this research has the potential to be a landmark in the field of electrodeposition and may have significance in other fields of materials processing as well.

The experimental program measures the time for the onset of buoyancy driven convection by laser interferometry. As well as the determination of the time constant, this technique reveals the characteristic spatial dimension of the electrolyte circulation cells. These data are compared with scanning electron micrographs of the surface of the electrodeposit in order to test the hypothesis that in the absence of hydrodynamic instability morphological breakdown is completely avoided. Furthermore, the development of techniques for direct *in situ* measurements of surface quality is being pursued.

Because the experiments focus on mass transfer controlled electrocrystallization, microgravity has an important role to play here. It has been shown in the field of solidification that gravity greatly influences the critical concentration for the onset of convective and morphological instability. It is expected that the experiments in this electrocrystallization study will proceed so as to prepare for in-flight testing of the theory and calculations.

The first major accomplishment has been the discovery of a physical model system in which simple electrocrystallization occurs under conditions of strict mass transfer control. It was extremely difficult to identify such a system because the database is almost bare in two respects: (1) the physical properties of appropriate aqueous electrolytes and (2) mass transfer controlled electroplating. The physical model system is the electrodeposition of silver onto silver in acid nitrate solutions. This chemistry was chosen because silver is not complexed in these solutions and the deposition of silver onto silver is known to have a relatively high exchange current density.

Studies in Electrohydrodynamics

Princeton University
Professor Dudley A. Saville
NAG3-259 (NASA Contact: Dr. A.T. Chai, LeRC)
February 1982 - January 1986

The purpose of this investigation is to develop and test (in a limited sense) models of electrodynamic processes involving liquids with poorly ionized solutes at high (applied) field strengths.

Extant theories which account for the details of the physicochemical processes associated with charged interfaces deal with 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 faithfully, it fails to give a comprehensive picture.

The research involves several tasks: (1) construction of a mathematical model for low field strength electrokinetics for rigid particles with poorly ionized solutes, (2) construction of a similar model for fluid globules wherein the interface is permeable to ions, and (3) extension of the model described in (2) to high field strengths.

Fluid Dynamics

Institute for Theoretical Physics (ITP), University of California, Santa Barbara
J. R. Schrieffer
D. M. Eardley
J. S. Langer

Fluid Dynamics as a broad subfield of physics has seen explosive growth in recent years. Research in this field is being vigorously pursued at the Institute for Theoretical Physics (ITP) in a number of directions including fundamental questions concerning dynamical systems in general, applications to material science, astrophysics and magnetohydrodynamics as well as advanced computational techniques relevant to these fields and to meteorology, oceanography and aerodynamics.

The ITP is structured around six month research programs involving 25-30 participants and focussed on specific topics, in addition, to continuing research activities carried on by the permanent members and post doctoral members. As an example, a research program entitled, *Active Galactic Nuclei*, was held at ITP January - June 1987, with hydrodynamics playing a central role. Several of the key participants and their research focus are as follows:

1. Professor Robert Abramowitz, Trieste; *Hydrodynamics of Thick Accretion Discs*
2. Professor Roger D. Blanford, Caltech; *Magnetic and Plasma Processes Near Black Holes, Winds from Black Holes*
3. Professor Peter Goldreich, Caltech; *Hydrodynamic Instabilities of Thick Accretion Discs; Generation of Hydrodynamic Oscillations by Convection in the Sun*
4. Professor Shoji Kato, Univ. of Kyoto; *Hydrodynamic Instability of Thick Accretion Discs*
5. Professor Arie Kovig, Univ. Chicago; *Hydrodynamics and Magnetohydrodynamic Jets at High Mach Number*
6. Dr. Charles Evans, Caltech; *Accretion onto Black Holes; Numerical Hydrodynamics*
7. Dr. John Hooley, Caltech; *Numerical Magnetohydrodynamics*

Numerous papers were published from this program dealing with hydrodynamics.

In addition, a program concerning fundamental problems in statistical mechanics was held September-December 1987, coordinated by Robert Griffiths (Carnegie Mellon Univ.), J. S. Langer (ITP) and Joel Lebowitz (Rutgers Univ.) The relation of hydrodynamics to dynamical systems theory was explored in depth as well as specific problems in nonlinear growth kinetics and pattern formation. This work is closely related to the ongoing research activities of Prof. J.S. Langer and his group at ITP.

A third program, *Computational Fluids Dynamics*, will be held at ITP September-December 1988. This program is being coordinated by Prof. P. Marcus (Berkeley), and S. Teukolsky (Cornell Univ.), with strong input by Prof. D. Eardly (ITP).

The program will have a balance of "pure" fluid dynamicists and scientists from fields such as astrophysics, general relativity, etc. The program will include hydrodynamics magnetohydrodynamics, plasma physics, and novel numerical techniques. Attention will be given to communicating the most recent concepts and techniques between the disciplines. Some of the key participants in the program, in addition to the coordinators, will be Drs. T. Piran (Hebrew Univ.), R. Wilson (LLL), P. Wirta (Georgia State Univ.), J. Hawley (Univ. VA), S. Finn (Cornell Univ.), D. Marion (Caltech), M. Nauenberg (Santa Cruz), M. Choptuik (Cornell Univ.), W. Press (Harvard), and H. Zel'dovich (Univ.). Many of these topics are related to ongoing research of Prof. D. Eardley and his group at ITP. A parallel program on Cosmology and Microphysics coordinated by J. Hartle (Santa Barbara), M. Turner (Univ. Chicago) and F. Wilczek (ITP) will no doubt interact effectively with this program.

The ITP plans to continue vigorous research in hydrodynamics and related problems in the future.

The Institute for Theoretical Physics produces a total of 160-180 technical articles per year partially supported by NASA.

Influence of Hydrodynamics on Capillary Containment of Liquids in a Microgravity Environment

Cornell University

Dr. P. H. Steen

NAG3-801 (NASA Contact: Dr. S.A. Kassemi, LeRC)

June 1, 1987 - May 31, 1989

The objective of this research is to determine whether small-to-moderate shear stresses promote or inhibit the static capillary instability of a liquid bridge. Cylindrical and noncylindrical shapes are considered in the ground-based experiments and associated mathematical analyses. Hydrodynamic stability theory in conjunction with perturbation techniques are used and we take account of finite-bridge length. In the experiments shear stresses are induced by several means including thermocapillary induced surface-tension gradients. The broad aim is to develop an understanding of the influence of hydrodynamics on shape stability; the possibility of a dramatic stabilization is explored.

Containment of liquids by capillary forces can be used to advantage in the processing of materials in a space laboratory environment, as is well known. The molten metal region of the float-zone refining process is one example. Wherever surface tension is used to advantage, however, the shape of the contained liquid is limited by the geometric capillary instability. Although most modifications of a dominant capillary force by other forces destabilize the configuration, both experimental and theoretical evidence suggests that certain shear stresses and pressure distributions induced at an interface can stabilize the capillary break-up.

The Study of Electromagnetically Driven Flows in Molten Salts Using a Laser in a Microgravity Environment

Massachusetts Institute of Technology
Professor Julian Szekely
David Forrest
NAS3-24642 (NASA Contact: Fred Harf, LeRC)

The objective of this research is to develop the feasibility of studying electromagnetically driven flow in molten salts, using laser velocimetry, in a microgravity environment. The main motivation for doing this in microgravity is due to the fact that under earthbound conditions the joule heating in the poorly conducting salts would give rise to buoyancy driven flows, which would become dominant.

Computational work has been carried out which has proved the feasibility of the experimental concept. A ground based apparatus is now being built and a proposal has been submitted for parabolic flight experiments. These parabolic flight experiments will provide a full testing of the concept and will provide a sound basis for designing space shuttle or space station experiments.

Collision and Coalescence Studies

Jet Propulsion Laboratory
Dr. Taylor G. Wang
In-House

The objective of this investigation is to study three aspects of the collision and coalescence of uncharged free drops: time dependent deformation of a drop upon collision, dynamics of air-film drainage, and drop stability after coalescence.

Spacelab provides a unique environment of near-weightlessness for conducting science in space, and thus will offer a unique opportunity to obtain quantitative data on the dynamics of collision and coalescence of liquid drops. The proposed experiments will utilize a modified version of the Drop Physics Module (DPM) provided by NASA to obtain accurate quantitative data on the behavior of collision and coalescence of liquid drops in microgravity conditions. These investigations will be free from the constraints imposed by the influence of Earth's gravity (including insufficient droplet sizes for accurate observation), and will extend for periods of time unattainable even in the longest rocket flight tests.

The results of the investigations will be used to verify existing linear theory, and to provide the necessary insight for further theoretical development of the subject. The deficiencies of the existing theory, which disregards nonuniformity of the air-film drainage, irregularity in the surface of separation, rotational energy, and oscillation energy, are exemplified by inconsistent results appearing in the literature.

In FY 1987 we have performed preliminary collision and coalescence experiments in an immiscible system with acoustic as the driver force. The static deformation of two impinging drops compare favorably with the calculation, but the final rupture shows a resonance effect. Whether this is an art effect due to the experimental set up or something significant will be investigated in the following year.

Transport Processes in Solution Crystal Growth

NASA Lewis Research Center
Dr. R. A. Wilkinson
In-House

The objective of this task is to conduct fundamental research on non-intrusive direct measurement techniques useful in quantifying transport properties of solution crystal growth systems. The utility of this effort is to enable study of diffusion limited transport in low gravity solution crystal growth.

This activity is being conducted to evaluate the science feasibility of utilizing Raman spectral scattering signals to provide 3-dimensional point measurements of concentration profiles near a crystal interface during growth or dissolution of KH_2PO_4 (KDP). Optical multichannel detection of a solute vibrational band provides direct quantification of solute concentration with band intensity. Orthogonal incident laser and Raman collection optics provide 3-dimensionally selective point measurements of the solution concentration field. Crystal growth is induced by cooling the crystal substrate such that the solution near the crystal surface is supersaturated. The Raman band intensity is not sensitive to the few degree centigrade temperature variations induced but is responsive to concentration differences. The spectroscopic sample volume and crystal face growth rate are documented with optical microscopy.

Precision calibration of Raman intensity vs. KDP concentration at sub 0.5% standard deviation error levels has been demonstrated. A fiber optic, sampling incident laser intensity, piping light to unused optical multichannel analyser (OMA) channels, had to be implemented to guarantee data quality. It provided a fully synchronized monitor of fluctuations in laser power to correlate with observed Raman signals. With 1 W of laser power at the sample (transparent) good data statistics required 8 repeated data sets at approximately 2.5 minutes per set. The roughly 20 minutes accumulated represents the time to measure concentration at one spatial location. 35mm photomicroscopy was implemented to document the 30 micron diameter by 200 micron long laser Raman scattering region in the solution with respect to the crystal surface. The laser beam was able to approach up to 25 microns from the crystal surface. During solution crystal growth scattering of nucleated microcrystals in solution caused some intensity noise. These microcrystals convect right up to the crystal surface indicating no quiet diffusion region under normal gravity conditions.

Fluid Simulation on Molecular Basis (Molecular Dynamics)

NASA Lewis Research Center
Dr. R. A. Wilkinson
In-House

The objective is to establish a molecular level first principles equilibrium and non-equilibrium fluid modeling capability simulating presence or absence of gravity and its effects on transport and surface phenomena. Molecular Dynamics (MD) simulation provides a means to get low gravity information on fluid behavior that can complement and optimize microgravity in space and ground based experiments. The activity will develop the algorithm and apply it to a variety of problems; for example, surface tension, surface configuration, and to momentum transport near a wall.

The activity entails, in addition to developing the main program that numerically integrates molecular equations of motion, developing algorithms to compute continuum fluid local density, velocity, pressure tensor, heat flux, surface tension, and surface excess quantities. A desktop PC based MD algorithm was developed for molecules in the liquid state flowing near a non-wetting wall. A Lennard-Jones 6-12 potential with xenon parameters is being used to model the liquid. Several possible tests for equilibrium or steady state are included. The code developed is transportable from a PC, for small number systems, to the Cray, for large systems. The PC expedites code debugging. All continuum equilibrium and non-equilibrium fluid properties can be calculated from the main algorithm output. To that end supporting algorithms have been added: 1) to periodically propagate the initial conditions for a larger system, 2) to calculate the instantaneous mass density in a selected set of subregions, 3) calculate the velocity profile in a selected set of subregions, 4) calculate the self-diffusion coefficient, and 5) calculate the local pressure tensor throughout the fluid. Algorithm development at this point is the main activity.

4. BIOTECHNOLOGY

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Center for Separation Science

University of Arizona

Dr. M. Bier

NAGW-693

January 1, 1985 - December 21, 1987

The Center for Separation Science has been organized to serve as a center of excellence for NASA and the U.S. biotechnology industry in matters relating to microgravity science and applications. The Center's primary expertise is in electrophoresis, covering a broad range of its subspecialties, including (1) the preparative techniques of electrophoresis, isoelectric focusing and isotachopheresis; (2) the mathematical modelling and computer simulation of electrophoretic transport processes, and (3) the analytical techniques of two-dimensional and capillary electrophoresis.

This expertise has been assembled at little cost to NASA, through the generous contribution of instruments, by their manufacturers from Sweden, Germany and England, as well as the U.S. Thus, the Center is presently recognized by industry and scientists as a unique resource in electrophoresis. This is attested to by numerous visits from industry and its financial support, the presence of postdoctoral fellows supported by German and Swiss science grants, and a graduate student supported by the French pharmaceutical industry.

In addition, scientists from the Center have been asked to collaborate in microgravity research projects by the German and French national space organizations, which will lead directly to space experimentation.

The Center is also exploring other novel techniques which may benefit from operation in microgravity. These include techniques based on cell fusion, supercritical fluids and phase partitioning methods.

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Research in Biological Separations and Cell Culture

University of Texas Health Science Center at Houston
Dr. R. William Butcher, BRCH
Dr. Baldwin H. Tom, BRCH
Dr. L. Scott Rodkey, U.T. Medical School
NAS9-17403 (NASA Contact: D.R. Morrison, JSC)
June 1985 - September 1988

The objectives for the research at the Bioprocessing Research Center at Houston (BRCH) center around development of bioseparation science and technology in partial support of NASA's efforts in bioprocessing. The research involves three interrelated objectives: (1) New research applications of the McDonnell Douglas, Continuous Flow Electrophoresis Systems (CFES); (2) selection of cell candidates for spaceflight separation experiments; and (3) Comparing CFES and Recycling Isoelectric Focusing (RIEF) for purification of cell products direct from culture medium.

CELL SEPARATIONS LABORATORY

A laboratory area has been developed to support continuous flow electrophoresis studies, using not only the CFES from McDonnell Douglas, but other instrumentation which complements the CFES. Included in the bioseparations studies will be comparative studies using HPLC, and Hirschmann-Ace-710 free flow electrophoresis system. The most effective separation conditions will be determined for biologicals in group-based studies that may be translated into use on the CFES on the Space Shuttle. Target materials for separation include soluble hormones and proteins, and whole cell and subcellular particles.

EVALUATIONS OF CELL CANDIDATES FOR NEW FLIGHT PROPOSALS

Screening of new cell candidates for microgravity separation is complicated. Cell stability in culture and target cell function must be well characterized. Studies on cell attachment and growth, chromosome karyotype, culture requirements sensitivity of assays for target functions, effects of handling and experiment procedures must be thorough. Comparisons of CFES, other free-fluid electrophoresis methods, laser-activated flow cytometry and sorting, and isoelectric focusing are used to determine the best method for separating target cells on Earth and to predict the efficiency of potential microgravity experiments. Current studies include CFES experiments separating SKHEP liver and CALU-3 lung cell lines which secrete urokinase. Other candidates include hybridoma cells which make both IgG & IgM, T-lymphocyte clones selected for antigenic determinants for myoglobin and apo-myoglobin, and colon cancer cells which produce carcinoembryonic Antigen (CEA).

PROTEIN SEPARATIONS FROM CONDITIONED CULTURE MEDIUM

Microgravity CFES experiments have demonstrated that proteins can be purified directly for medium 125X more concentrated than that on Earth. Recirculating Isoelectric Focusing (RIEF) experiments in space demonstrated unique applications for purification of selected candidates in spite of some limitations of fluid stability which can occur as a result of undamped solute driven convection. However, CFES protein purification on five shuttle flights were proprietary and not published in the open literature. Comparisons of CFES RIEF, and solid state IEF are determining the most efficient techniques for separating hormones, enzymes, and other protein products.

Proteins suitable for CFES must be tolerant to initial concentrating steps since the carrier buffer dilutes the sample stream. In contrast, proteins which normally must be purified in high concentrations of urea or guanidine (to prevent precipitation at the isoelectric point) are likely candidates for RIEF in microgravity. Recent results have shown unexpected interactions between target proteins and ampholytes used to establish the pH gradient. Definition of potential flight experiments include comparisons of CFES with RIEF and HPLC purification of interferon and monoclonal antibodies.

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Protein Crystal Growth in Low Gravity

Stanford University, Center for Materials Research
Professor Robert S. Feigelson
NAG8-489 (NASA Contact: Marc Pusey)
August 8, 1984 - December 31, 1987

The objectives of this research task are: (1) to understand the mechanisms of protein crystal growth and (2) to design a space flight experiment to compare ground base data with that obtained in microgravity.

A program to study the mechanisms of protein crystal growth and what parameter influence that growth. Canavalin will be used as a model protein. It will be grown by changing the pH of the protein solution by concentrations and temperatures. Concentration-induced flows will be studied using Schlieren techniques. Measurements will be made of the concentration of both the protein and the hydrogen ion over time. The same type of measurements will be made with lysozyme to investigate the interaction between the precipitant and the protein. Techniques of local supersaturation control will be investigated as a means of controlling nucleation. Growth by supersaturation control will be studied. The data gathered in earth-based experiments will be used to design a space flight experiment to gather the same type of data in a low-gravity environment.

Cell Partition in Two Aqueous Phase System

University of Alabama in Huntsville

Dr. J. Milton Harris

NAS8-35362 (NASA Contact: R. J. Snyder, MSFC)

July 1987 - July 1990

The goals of this work are to understand and control low-g demixing of two-phase systems formed by solution of pairs of polymers (generally dextran and polyethylene glycol) in water and then to use these systems to purify cells by partitioning. The immediate objective has been to control demixing by controlling wall wetting with covalently-bound polymer coatings. Ground-base experiments have now been completed which show that we can achieve essentially any contact angle between the phase interface and the container wall by using dextran coatings of different molecular weights. The next step will be to use the Shuttle to determine the effect of wall wetting on the rate of demixing in the absence of density-driven sedimentation.

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Biological Separations in Microgravity

Johnson Space Center
Dr. Dennis R. Morrison
In-Center
January 1985 - Present

This research includes ground-based research and flight experiments involving purification of mammalian cells, medically important hormones and enzymes, and development of micro-analytical assays for secreted cell products. This research is conducted in collaboration with the University of Texas Health Sciences Center, Baylor College of Medicine, and Penn State University.

GROUND CONTROL RESEARCH

Current ground-support research includes: 1) comparative separations with commercially available Continuous Flow Electrophoresis (CFE) devices and other free-fluid electrokinetic separations methods; and (2) CFE control studies and analytical electrophoresis measurements in support of cell separation flight experiments using electrophoresis systems designed specifically for enhanced resolution (and throughput) under microgravity conditions. Collaborations between NASA centers and academic institutions involve purification of secreted cell products and development of improved product assays. Results include new ultra-sensitive ELISA and chromatographic assays and new methods to detect different bioactive vs. immunoreactive hormones secreted by human cells.

Urokinase and other plasminogen activators are used to activate the body's fibrinolysis mechanism which dissolves blood clots after they are formed. We have shown that cultured subpopulations of human kidney cells separated by CFE will produce two different molecular forms of urokinase (uPA): single-chain proenzyme (scuPA); PA inhibitors; and tissue-plasminogen activator (t-PA). Several experiments were conducted to compare CFE results with RIEF (recycling isoelectric focusing) separations on various culture media containing urokinase. The CFES was able to resolve different urokinase molecular forms better than RIEF; however, the RIEF was able to separate total plasminogen activators from other proteins better than CFES.

FLIGHT SEPARATION EXPERIMENTS

The flight projects are focused on completion of the analysis of flight samples from cell separation experiments on STS-8 which were compromised by the pre-flight contamination and operational problems during the mission. In two experiments on STS-8 different concentrations of human kidney cells were separated into more than 33 fractions. Microscopic studies of cells cultured after separation showed a unique distribution of four morphological types (believed to have different functions) and an unexpected distribution of cell size according to EPM. Surprisingly, all cultures of separated cells produced some uPA and tPA. Five to six fractions produced significantly more uPA than did the other fractions. All flight cells were needed for postflight cultures to identify the highest producing cell fractions, so follow-on further mechanism studies were not done. However, analyses of the remaining 3000 medium samples from the cultured flight cells continues.

Injecting high concentrations of cells into CFE devices reduces the bandspread and resolution. The mobility distribution of kidney cells is reduced by 50% at sample

input concentrations greater than $2 \times 10^{+6}$ cells/ml. Pituitary cells also appear to encounter the same problem at concentrations greater than $2 \times 10^{+7}$ cells/ml. Comparisons of CFE experiments on STS-8 indicate that the effect is absent up to $8 \times 10^{+6}$ cells/ml. Similar concentration effects have been demonstrated in the EPM distribution of cultured SK-HEP liver cells. A four-fold increase in sample concentration reduced the EPM distribution bandwidth and the mean EPM by more than 45%. Studies are underway to elucidate mechanisms and predict the practical upper limit for cell sample concentrations under microgravity conditions. Three possibilities are under investigation: (1) effects of buffer formulations and mismatches in local ionic concentrations between the sample and carrier buffers; (2) effects of Ca^{++} and Mg^{++} ions and new methods to avoid cell clumping; and (3) CFE experiments on different cell types to see if the 1-g limitation holds for all cells. Preliminary results from comparisons of CFES and ACE 710 separations of the same cell lots indicate that cell clumping also is a major problem at high cell concentrations in the ACE 710.

The results of STS-8 clearly demonstrated some advantages of cell separations in microgravity; however, the experiments must be repeated under optimum conditions to determine the full potential of CFES for live cell separations. Enough viable cells in each supopulation must be returned for the functional assays to identify the target fractions which then can be subcultured for additional studies. Current work involves redefinition of micro-g separations of human kidney cells and mammalian pituitary cells using the next generation of CFE or other flight hardware. This research includes: (a) development of next flight protocols; (b) improvements in sample handling and pre-launch support; and (c) conduct of new control experiments to compare with the microgravity separations.

EFFECTS OF FLUID SHEAR ON CULTURED CELLS

In 1985 Stathopoulos and Hellums at Rice University showed increased secretion of urokinase from primary cultures of human embryonic kidney cells subjected to low shear stress. A similar result was found for the secretion of prostacyclins by cultured human endothelial cells (Frangos et. al., 1985). Our experiments at JSC in collaboration with investigators at the University of Houston (Goochee) indicate that low level shear (13 dynes/cm^2) causes intracellular synthesis of some twelve new "shear stress" proteins which have not been reported before.

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Cell Maintenance Systems and Inflight Biological Sampling Handling

NASA Johnson Space Center
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In-Center
April 1985 - continuing task

Mammalian cells are difficult to maintain alive outside of an incubator for more than a few hours. Cells require control of temperature, pH, dissolved oxygen, and pressure in the immediate environment. These requirements must be considered when cells are taken into space. Methods are being investigated to measure cell tolerance to less than optimum environment conditions and to develop appropriate small scale hardware and techniques for maintenance culture, harvesting, fixation and storage of living cell specimens used in microgravity bioprocessing research.

Recent studies have demonstrated that a particular set of "stress response proteins" are synthesized by mammalian cells in response to temperature stress, dissolved oxygen deprivation, and subnormal glucose levels. Experiments have been conducted using 2D-PAGE to determine the patterns of intracellular protein synthesis for human embryonic kidney (HEK) cells exposed to three different experimental conditions: (1) normal growth conditions in quiescent medium (37°C), (2) two hours at 42°C (temperature stress), and (3) exposure to shear stress (12 dynes per square centimeter) in a laminar flow chamber for two hours. The patterns of intracellular protein synthesis were very different under each of these three conditions. For temperature-stressed cells, at least eleven intracellular proteins were observed in 2D-PAGE gels that were not evident in the control cells. These are well-documented "heat-shock" proteins. For the shear-stressed cells, at least fifteen intracellular proteins were observed in 2D-PAGE that were not in the control cells. There was little overlap between the eleven "heat-shock" proteins and the fifteen "shear-stress" proteins indicating that these cells have a specific functional response to low level shear. Techniques are now being developed for inflight radioisotope labeling of cells to use the technique to verify stress levels during the experiments. A method of accurately measuring the exact synthesis rate for specific proteins is being developed as a technique to test the performance of cell transport culture and harvesting apparatus.

The Cell Transport Assembly (CTA) flown on STS-8 is being redesigned to maintain live cells longer than 48 hours. Methods and small scale apparatus are being developed for harvesting cells and concentrating the cell suspension and removing cell-free medium for inflight assays. Sterile containers, transfer apparatus and procedures are being developed in conjunction with an advanced CTA which can be used to support living cells for electrophoresis and cell biology experiments.

The Skylab Biological Specimen Test Apparatus (BSTA) also is being modified to support cultures of both plant and animal cells in the Shuttle middeck.

Containerless Polymeric Microsphere Production/Biotechnology

Jet Propulsion Laboratory
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Dr. Michael T. Hyson
Dr. Manchum Chang
NAS7-918
September 1987 - September 1988

The objective of this task is to investigate containerless production of monodisperse polymeric microspheres for biomedical applications. Microspheres of precise sizes and specific properties are needed for medical diagnostic tests, chromatography, cell sorting, and cell labelling. The uses and economies involved in the mass production of biomedically valuable microspheres in space will be investigated.

Using a ground-based electrostatic levitator, various monomer or polymer droplets will be injected, levitated, polymerized, and collected. Conditions needed for polymerization, monodispersity, the physical and chemical properties and their utilities for various biomedical purposes will be determined. Information derived from these experiments will be used to assess the feasibility of mass producing microspheres in the space microgravity environment. In addition, using monodisperse HEMA particles we produced, antibody binding tests will be carried out followed by affinity separations of bone marrow cells, separations of natural killer lymphocytes, and cell labeling by fluorescent particles.

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Research and Technology for Isoelectric Focusing

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NAS9-17403 (NASA Contact: C. Sams, JSC)
January 1987 - January 1988

This research is designed to develop and optimize both analytical and preparative isoelectric focusing technologies. This involves development of analytical techniques for ultimate use in analyzing separated proteins which are separated by other techniques. Additionally, we are undertaking comparative studies of continuous flow electrophoresis and recycling isoelectric focusing to determine relative characteristics of throughput, resolution, denaturation, and other characteristics involved with the different techniques. Work related to both preparative and analytical isoelectric focusing technologies which involves optimization and chemistry for synthesizing inexpensive ampholytes are underway. Related to this, are studies of ampholyte-protein interaction and studies to eliminate such interactions.

Initial studies were undertaken in order to optimize analytical isoelectric focusing techniques. These studies involve development of techniques for transblotting of proteins from analytical isoelectric focusing gels onto nitrocellulose membranes. These studies were successful in that techniques were developed which were highly sensitive. Using the analytical techniques which were developed, studies were undertaken to characterize hybridoma products. Studies of standard hybridoma antisera which are standards used by CDC (Center for Disease Control) in Atlanta, were studied by this technique. These studies pointed to this analytical technique as being the method of choice for quality control of hybridoma synthesis.

Additionally, studies have been done to optimize the chemistry involved with synthesizing inexpensive ampholytes. These studies are focused on attempts to make high quality, high resolution ampholytes. These studies have been very successful, in that several new chemistries have been evaluated and incorporated into methodology for synthesis of inexpensive carrier ampholytes for large scale isoelectric focusing. Closely related to this, are studies of ampholyte-protein interaction. These studies have successfully shown that ampholytes interact with proteins in an ionic manner. This is to be distinguished from ampholyte interactions by other means, such as hydrophobic interactions, which we have shown do not occur. Further, these studies have been successful in showing methods for removal of ampholyte from proteins following isoelectric focusing. These studies are highly significant, in that it gives a methodological approach to removing ampholytes from proteins following large scale protein purification by preparative isoelectric focusing.

Other studies have been initiated which attempt to compare various properties of the McDonnell Douglas continuous flow electrophoresis system with that of the recycling isoelectric focusing system. Numerous parameters have been studied in attempts to understand the positive and negative features of both techniques. Studies relating to questions of resolution, capacity, and other characteristics have been carried out. Clearly, the recycling isoelectric focusing has been shown to have much higher resolution than continuous flow electrophoresis. The question of capacity of each technique remains somewhat open, in that there are clearly limitations on each technique, but the limitations seem to be unique in each case, which makes direct comparison of this parameter difficult.

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Cell Separation and Characterization

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January 1987 - January 1988

The research objectives for this project are to establish a continuous flow electrophoresis separation laboratory, complete with characterization facilities for studying separated cells and cell products. Additionally, this center has as a principal goal, studies of various types of cell populations, and studies of feasibilities of cell separations for various types of cells and cell lines.

The research tasks involved in this project are principally separation and characterization of cell populations and secondarily, characterization of cell products (hormones, peptides, enzymes, etc.). Studies are underway to characterize subsets of human kidney cells (HEK-871) and human hepatoma cells (SK-HEP). Other types of cells which are under study include CALU-3, cells derived from human lung carcinomas, human bone marrow cells, NIH 3T3 tumor cells, and the AS3 T-cells which are NIH 3T3 cells which have been transfected with DNA from human skin tumor cells. All cell lines have been, or will be, separated on the CFES and subpopulations will be characterized for different biochemical and functional characteristics. A second area which is being studied is that of use of the continuous flow electrophoresis equipment for separation of cell products. Principal among these is carcinoembryonic antigen and monoclonal antibodies. Results to date indicate poor recovery for carcinoembryonic antigen and excellent recovery for monoclonal antibodies. The continuous flow electrophoresis technology appears to be quite useful for first step purification of monoclonal antibodies from ascitic fluid. Another area of work involves comparison of the Coulter ACE 710 instrument with the McDonnell Douglas continuous flow electrophoresis system in order to determine if the ACE 710 can function as an appropriate screening technology for cells to be separated in the continuous flow electrophoresis system. Results to date indicate that, for some cell populations, the ACE 70 appears to be a reliable method for analysis of cell populations for their behavior in continuous flow electrophoresis.

Enhanced Hybridoma Production Using Electrofusion under Microgravity

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NAG8-716 (NASA Contact: R.S. Snyder, MSFC)
May 1, 1988 - April 30, 1993

Hybridoma technology and the production of monoclonal antibodies (MABs) has revolutionized biomedical research and medical diagnostics. MABs present new opportunities for the treatment of cancer, for the production of vaccines, and are essential for defining second-generation tests for the detection of life-threatening viruses such as Human Immunodeficiency Virus Types 1 and 2.

Most MABs are derived from antibody-forming B-cells (usually the spleen of rodents) immunized with relatively large amounts of antigen and are immortalized by chemical fusion to suitable myeloma cells using polyethylene glycol (PEG). Antigen-specific hybridomas are selected from the many irrelevant hybrids that result from this fusion process by a variety of screening methods including immunoassay, western blotting and tissue staining. It is then necessary to sub-clone hybridomas previously selected to ensure their clonality and stability by any variety of labor-intensive methods and to repeat the screening prior to the "scale-up" for the production of the desired MAB. Using conventional methods, a period of six months may be required for a suitable hybridoma to be generated, selected, screened for specificity and stability, and for usable quantities to be made.

To date, overall efficiency and cost of production has proven adequate for most applications. Nevertheless, new applications (e.g., production of immunovaccines which are effective against rapidly mutating and poorly immunogenic viruses and immunotherapy of constantly changing malignant cells) will require large increases in the efficiency of antibody production and a substantial shortening of the time required for generation, selection and manufacture of suitable MABs. Moreover, the problems inherent in the use of xenographic MABs for "in vivo" applications (including short half-life, sub-optimal biological activity and the potential for life-threatening anaphylactic reactions) dictate the increasing need for human MABs. The production of useful human MABs is likely to be difficult for the following reasons: (1) suitable human B-cells are available in small numbers, (2) "in vitro" immunization are required for the preparation of fusible human B-cells, (3) antigens to mutating viruses and changing malignant cells are likely to be limited and expensive to obtain, (4) as for murine hybridoma, today's time scale for production of usable quantities of human MABs will be excessively long, particularly for the purposes of immunovaccines production and anti-tumor therapy.

To meet the challenges of future application of MABs, considerable innovation is necessary to realize the time and cost reduction required in the hybridoma technology. Therefore, for these reasons, we are developing an extensive ground-based and microgravity program to modify methods and to implement emerging technologies in order to substantially improve the efficiency of and to expedite hybridoma production. Although our approach will exploit murine systems, we believe that this advanced technology will be readily transferable to the production of human hybridomas.

The long range objective of this cell fusion program is to attain high fusion frequencies and hybridoma yields with methods that are rapid and efficient, and which are directly adaptable for future applications of the monoclonal antibodies. The primary

mission of our effort is to perform ground based and microgravity research to improve electrofusion methodology and utilize the advantages offered by new technologies in B-cell selection and isolation which are required for the accomplishment of our long range goal. Our strategy is to pre-select antibody-producing B-cells prior to fusion (thus avoiding the need to select post-fusion selections), utilize in vitro immunization using anti-T-cell receptor complex MABs, utilize electrofusion instead of PEG, and conduct experiments under microgravity to advance our knowledge about the fundamental principles and processes involved.

The prospect that the frequency of fusion and the yield of viable hybridomas is greatly enhanced under microgravity has been demonstrated with short duration flights of the TEXUS sounding rockets. We will be participating in the KC-135 and sounding rocket flights to extend previous findings under microgravity and to test new protocols and hardware that are being developed during our ground based research. The work is directed toward optimizing electrofusion conditions and will utilize microgravity experiments (to be performed without sedimentation and convection forces) to advance our program goals. This research will culminate in a series of shuttle-based experiments designed to fully exploit the microgravity effects on cell fusion in the D-2 and subsequent missions.

To provide well-designed flight opportunities, the ground based research must address a number of defined and undefined cellular and physical limitations of the myeloma cell, the B-cell lymphocyte, the fused hybrid, growth and maintenance conditions, separation and evaluation methodologies and electric field conditions. Because of the multi-faceted nature of our cell fusion program, we are proceeding along several investigative lines in a simultaneous fashion. The current research tasks are as follows: (1) discover and develop optimal fusion partners for electrofusion, (2) develop optimal growth and maintenance conditions for parental and hybrid cells under "in vivo" conditions, (3) analyze cell populations and single cells for accurately monitoring cellular effects of the electric field conditions on the fusion process, (4) separate and recover cell hybrids from the parent cells for determination of fusion frequencies and hybridoma yields, (5) recover cell organelles, membranes, and macromolecules of fused cells to determine the underlying changes that may be responsible for irreversible cell damage during execution of the fusion procedures, and (6) improve and innovate existing hardware for fusion of small numbers of cells under both ground based and microgravity conditions by taking into account the ground based and microgravity data.

This work is being performed in collaboration with Drs. Normal Klinman, Garry Neil and Howard Urnovitz, and in conjunction with Scripps Research Institute, University of Iowa, Calypte Biomedical Company (United States), and Drs. Kurt Hannig and Ulrich Zimmermann of the Max Planck Institute and the University of Wuerzburg (West Germany).

Fluid Mechanics of Continuous Flow Electrophoresis

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NAG 8-759 (NASA Contact: Dr. Robert Snyder, MSFC)
August 1986 - July 1988

A definitive set of micro-g experiments were proposed to establish the hydrodynamic characteristics of continuous flow electrophoretic separation devices operated with homogeneous buffers. These results will help establish the ultimate resolving power of the process. The program is a joint effort between Princeton and MSFC. Efforts at Princeton are devoted to: (i) improving the extent mathematical models to predict flow and particle trajectories in the apparatus as conceived at MSFC and (ii) a study of the effects of particle concentration on sample conductivity and dielectric constant. The latter have been identified as playing crucial roles in the behavior of the sample and, thus, on the resolving power and throughput of continuous flow devices.

Earlier work at Princeton (under contract NAS8-32614) showed that particle concentration has a strong influence on sample conductivity (more specifically, on the "real" or "dc" conductivity). Recent work has shown that the dielectric constant (the "imaginary" part of the complex conductivity conductivity) is similarly affected. Concurrently, experimental studies by P. H. Rhodes at MSFC showed that sample conductivity influences the spreading of the sample. A large conductivity mis-match between sample and the buffer causes the sample to spread rapidly from the front to rear walls of the channel. Rhodes developed an electrohydrodynamic theory of this spreading which shows that in addition to the conductivity, the dielectric constant should also affect spreading behavior. To optimize performance of a continuous flow device it will be necessary to understand the spreading process and, therefore, how it is influenced by the conductivity and dielectric constant. Accordingly we need to be able to measure the dielectric behavior of the sample as well as the DC conductivity. The Princeton group is working on development of a device to measure the conductivity and dielectric constant (the complex impedance of the sample) over a range of frequencies and relate them to the properties of the sample particles and the buffer. Progress has been excellent thus far and we can measure these properties of a given suspension at frequencies between 500 Hz and 200 KHz. A complete frequency scan takes about 25 minutes, far faster than existing techniques.

Publications

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Electrophoresis Technology

Marshall Space Flight Center
Dr. Robert S. Snyder
Percy H. Rhodes
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) analyze the electrophoretic process 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.

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

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

A moving wall concept is being developed for laboratory testing which will eliminate and/or control all of the above-mentioned disturbances. The moving wall will entrain the fluid to move as a rigid body and hence produce a constant residence time for all sample distributed across the chamber thickness. By aligning the moving wall at an angle to the chamber axis, a component of the moving wall motion can be made to oppose and hence cancel the electroosmotic flow. In absence of electrokinetic effects, i. e., electroosmosis, the electrohydrodynamical effect manifests itself as a ribbon, being either vertical (perpendicular to the electric field) or horizontal (aligned with the electric field) depending on the ratio of conductivity of the sample to that of the buffer. Therefore, by using low conductivity sample solutions to provide a vertical ribbon, the moving wall concept should produce distortion-free separations.

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Growth of DNA Crystals in a Microgravity Environment

University of Pennsylvania

Dr. Donald Voet

NAG8-611 (NASA Contact: K.D. Sowell, MSFC)

During the first nine months of this project, we have successfully synthesized several asymmetric species of double stranded DNA, developed methods for their high purification, and worked out procedures for their crystallization resulting, recently, in the formulation of what initial analysis indicates are diffraction-quality crystals in a 12 base pair DNA. The difficulty of DNA crystallization and the importance of knowing its structure makes it a promising candidate for the development of microgravity-based crystallization techniques.

The objective of the research is to determine the x-ray structure of several large segments of double stranded DNA so as to elucidate how the conformation of DNA varies with its base sequence. To this end, we have synthesized, in tens of mg quantities, fragments of the *E. coli lac* operator. These DNA's all consist of two strands of different sequences rather than being formed from one type of self-complementary sequence as are all the DNA's whose x-ray structures have yet been reported. The core sequences of these DNA's are identical. Their various lengths and overhanging nucleotides or lack of them were all designed in attempts to discover more readily crystallizable sequences. The DNA syntheses were carried out by the solid state phosphite-triester method with particular attention paid to minimizing the impurities in these large-scale procedures.

Experience has shown that macromolecular crystallization and crystal quality are highly dependent on the purity of the macromolecule. This seems particularly true of DNA. We have therefore taken great pains in developing HPLC techniques for obtaining DNA's of the highest purity. Synthetic DNA's are subject to length inhomogeneities, incomplete deblocking, and a great variety of minor side products and degradation products. Consequently, several purification steps are necessary to obtain DNA's of the required high purity. Moreover, the relatively low loading capacities of most available HPLC columns dictates that each purification step be repeated several times in order to obtain the required quantities (tens of mg) of DNA. Thus, DNA purification has proven to be the most consuming and labor-intensive part of the DNA crystallization project.

Once a DNA single strand has been purified, it is combined with a equimolar quantity of its purified complimentary mate thereby forming double stranded DNA. Excess single strand is then removed, again by HPLC. The final products, according to HPLC analyses, show only a few traces of impurities. These are, nevertheless, worrisome and we are therefore continuing our efforts to remove even these residual impurities.

Initial crystallization conditions were selected by a randomization method so as to produce a full spectrum of unbiased starting conditions. Promising conditions, that is, those appearing to form crystals of some sort, were then followed up by slight variations in these conditions. The results of many such experiments suggested that the blunt-ended 20-mer crystallized more readily than any of the 19 or 20 base pair segments with overhanging nucleotides or the blunt-ended 18-mer. However, the 12 base pair segment crystallized significantly more readily than any of these larger segments. Consequently, a large quantity of the 12-mer (90 mgs) was synthesized and over 500 crystallization experiments were conducted.

Recently, we have obtained small (0.5 mm x 0.4 mm x 0.05 mm) birefringent crystals that diffract x-rays to a resolution limit of at least 3.4 Å. Preliminary x-ray photographs indicate that the unit cell has orthorhombic symmetry and dimensions of approximately 30 Å x 80 Å x 120 Å. These diffraction photographs exhibit strong bands along the 80 Å axis at 3.4 Å resolution which is indicative of B-DNA. These crystals, as is usual for macromolecules, decayed in the x-ray beam during their characterization. We have therefore set up numerous crystallization experiments similar to those which produced the crystalline 12-mer and expect to complete crystal characterization and commence intensity data collection once these crystals have grown to sufficient size.

5. GLASSES AND CERAMICS

Multimode Acoustic Research

Jet Propulsion Laboratory

Dr. Martin Barmatz

NAS7-918

October 1, 1981 - September 30, 1988

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 the development of theories in uniform and temperature gradient environments for the acoustic forces and torques associated with sample translational and rotational stability using a variety of acoustic positioning modes (multimodes). These calculations are used to (1) determine those acoustic modes that produce stable levitation, (2) determine operating conditions to avoid translational and rotational instabilities, (3) determine the shape and position of levitated liquid drops, (4) isolate the levitation and rotation capabilities to produce more than one axis of rotation, and (5) develop methods to translate samples down long tube chambers. Experimental levitators will then be constructed to verify the stable levitation, rotation and sample translation.

Theoretical analyses carried out under this task have predicted stable acoustic levitation in rectangular, cylindrical and spherical resonators using one acoustic mode of excitation. This theory is now being used to design cylindrical single mode levitators for use at very high temperatures ($>1500^{\circ}\text{C}$). Stable levitation in a single mode cylindrical levitator has already been demonstrated in a ground-based laboratory up to 950°C .

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Study of Powder Agglomeration in a Microgravity Experiment

Ohio State University
Professor James D. Cawley
NAG3-755 (NASA Contact: Dennis Fox, LeRC)
November 1986 - November 1988

The population of agglomerates in the powder suspension plays an important role in the behavior of the system during shape forming processes. The work is designed to extend our understanding of the mechanistics of the agglomeration process through both experimentation and numerical modelling. In particular, we intend to exploit a microgravity environment to isolate convection from diffusion in the growth of agglomerates within dilute suspensions.

Our efforts to date have concentrated on the use of optical microscopy to monitor the agglomeration of 0.4 and 4.0 μm alumina particles constrained to the surface of an aqueous suspension. Through the analysis of video tapes we have been able to identify that the relative contribution of convection versus diffusion in the particle motion is dependent on local geometry and that at various locations convection dominates while at others the motion is almost purely diffusive, i.e. Brownian. In a convective field, small agglomerates are deposited onto larger ones due to differing Stoke's diameters. The trajectory of the small agglomerates is such that they preferentially deposit on the periphery. This contributes to an unstable interface which leads to qualitatively the same structures that appeared to require pure diffusive transport when studied by the computer models.

Pure diffusional agglomeration in three dimensions will be carried out in a microgravity environment and the agglomeration process will be monitored using light scattering. A computer model based on molecular dynamics is being developed which will allow both diffusion and convection to be taken into account.

Glass Formation in Reluctant Glass Formers

Marshall Space Flight Center
Dr. Edwin C. Ethridge
In-House

The objective of this research 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.

An 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. In the absence of suitable 1-g levitation melting techniques, a thermocouple was used for sample support. Recently the effects of various processing conditions on the heterogeneous nucleation on an eutectic composition within the gallia-calcia system were reported. The experiments utilized an ellipsoidal heater to melt the samples which were contained on a type S thermocouple positioned at one foci of the ellipsoid. The sample thermal history was recorded directly by a strip chart recorder and the experiment was visually recorded and imaged and recorded with a video camera/recorder. Under most experimental conditions the cooling rate is approximately linear. Many hundreds of experiments were performed. As a result of the research it was found that processing conditions affect sample nucleation in various ways. Some of the more important processing conditions are the extent of superheating above the thermodynamic melting temperature and the mechanism of quenching the sample.

The apparatus for nucleation experiments is being automated. Obtaining data utilizing current techniques is very time consuming and labor intensive, but automation of the experiments will greatly increase experiment efficiency. Experiment automation combined with telepresence and telescience are future directions for some of the materials processing space experiments. Schmidt et (1987) presented arguments for the promotion of telescience as the operational philosophy of future space experimentation. Based upon prior experience with Spacelab, there are typically about three (3) hours of astronaut crew time available for Space Shuttle based manned operations of each experiment rack per day. On Space Station, however it is projected that there will be much less time for crew tending of experiments being on the order of 20 minutes per experiment rack per day. This low level of astronaut availability may require that many experiments be highly automated. Also, it is likely that flight opportunities may exist for experiments on free flying man-tended satellites such as the Industrial Space Facility (ISF). Ideally each experiment could be operated from ground based laboratories in which the principal investigator could select processing conditions, initiate the experiment, and view the experiment (both data and video) in a near real time mode. Nucleation and crystallization experiments are well suited to automation and the development of these telepresence and telescience concepts on earth. Aspects of telescience are being incorporated into the automated apparatus.

Publications

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Study of Foaming in Glass Melts Under Microgravity

Case Western Reserve University
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Dr. Ali Ilhan
NAG3-740 (NASA Contract: M. Jaskowiak, LeRC)
October 1, 1987 - October 1, 1988

The research has three objectives; a fundamental understanding of the effect of gravity on foam, understanding the mechanism of foaming in glass melts, and coping with foam generation in glass under microgravity conditions. Fundamental understanding of the effect of gravity on generation and collapse of gravity sensitive foam is both interesting from the science point of view, and of practical importance. On the one hand, the effect of surface and gravity forces can be separated if gravity can be controlled, and thus the mechanism of their actions can be clarified. On the other hand formation or destruction of foam is a part of many industrial processes, including the manufacture of glass.

Gravity and surface forces in foams are approximately the same order of magnitude, and neither of them can be effectively controlled on earth unless gravity is reduced. Making a liquid less sensitive towards gravity by increasing viscosity, or by increasing the rate of bubble generation, cannot be used as an alternative to microgravity, because it would shift the balance of forces towards that between the viscosity forces and the gas pressure forces, thus making the liquid less sensitive to surface forces as well.

This study is oriented towards foaming in glass melts because the experience with molten glass is important for future glass preparation under microgravity conditions and understanding of foaming in melts is visual for glass manufacturing and nuclear waste vitrification. Apart from molten glass, room temperature liquids are used as model systems. Since our proposed experimental tool in this stage is the drop tube, the selection of liquids and melts is limited to those which generate transient foams undergoing an appreciable change during the experimental time available.

Both theoretical considerations and preliminary experiments show that gravity affects evolution of certain foams. It can be expected that under microgravity the absence of bubble motion due to external forces will prevent formation of surface foam, and so only bulk foam can be generated. Also, the absence of gravity drainage will affect the foam collapse rate and mode, even if a surface foam has been generated before it is exposed to microgravity.

The preliminary experiments show that it is possible to generate transient room temperature and high temperature foams suitable for microgravity experiments and to record their behavior by a video or still camera in a simple experimental set up. It was established that sodium sulfate generates in soda lime glass a transient cellular foam at 1425-1500 C, the behavior of which is easy to control and record.

At the present stage, drop tube experiments are prepared, to measure foam generations and foam collapse rate in a room temperature liquid and in molten glass, and to record foam collapse mode of a pendant foam drop.

Study of Phase Separation in Glass under Microgravity

NASA Lewis Research Center

Mark J. Hyatt

January 1, 1987 - January 1, 1988

The objective of this research is to define an experiment which will use a low gravity environment to aide in the study of phase separation in glass systems. In a reduced gravity environment, surface forces will dominate the separation of phases in a glass melt. This will allow study of the fundamental aspects of the phase separation process. The low gravity environment will eliminate the effects of thermal and gravity driven convection in the glass. Convection in the glass would interfere with the early stages of the phase separation and would affect the final structure of the glass.

The approach to this research is outlined in the following steps:

- 1) Measure important physical property data of a model immiscible glass system. Property data includes phase equilibria, density, and interfacial properties. A relatively unknown system is preferred which will yield important new physical data and also has potential for unique applications.
- 2) Develop model of phase separation using immiscible organic systems. Dynamic light scattering is being evaluated as an experimental technique in this model development. Immiscible organic systems are favored because they can be studied at low temperatures. Also, gravity level can be varied artificially by deuteration of one component in the system. Suitable systems with known physical properties will be employed to minimize the experimental effort required in development of the model.
- 3) Use the model to predict phase separation behavior in the glass system. Physical data obtained in (1) will be used in the model to predict phase separation [predictions based on current theory as well.
- 4) Define microgravity experiment on model glass system to verify model of phase separation. Experimental results will be used to correct the model if necessary.

Glass Research

Jet Propulsion Laboratory
Dr. George F. Neilson
In-Center

Research efforts span three general areas of glass science: nucleation and crystallization of glasses, amorphous phase separation in glasses, and gel-derived glasses.

The ability to prepare many inorganic oxide glasses is limited by the tendency of their melts to heterogeneously nucleate and crystallize from the container walls. Also, the ability to study internal homogeneous crystal nucleation and free surface nucleation 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 sought simple oxide systems which exhibit internal crystal nucleation by which tend to also crystallize at the melt-container interface and are studying their ground-base crystallization behavior. Through theoretical modelling procedures, these results will be compared with the crystallization behavior anticipated for the different crystallization conditions appropriate for a containerless environment.

Spontaneous amorphous phase separation can occur during the supercooling of many glass melts that might be prepared in microgravity, thereby preventing the formation of homogeneous glasses even though crystallization effects are avoided. However, the kinetics and morphology of phase separation under microgravity conditions may be quite different from that observed on earth due to the absence of gravity-driven sedimentation and convection effects. To obtain a baseline for interpretation of phase separation effects in microgravity, we are conducting phase separation studies in simple binary oxide glasses prepared by both conventional and sol-gel procedures.

Gel precursors can be used to prepare unique metastable glasses and glass ceramics. They offer several advantages for glass preparation in a containerless environment in that they can be prepared initially in homogeneous and highly purified states that can be converted to homogeneous glasses at temperatures below the liquidus. These characteristics make gels attractive precursors for containerless glass forming experiments. However, unsintered gels are very porous and often their chemical components are not uniformly distributed on a molecular level. This could lead to the formation of non-uniform glasses if the gels were employed in microgravity. These questions concerning gel structure are being addressed in the ground-based portion of this program.

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Spherical Shell Technology

Jet Propulsion Laboratory
Dr. Taylor G. Wang
J. M. Kendall
M. Chang
C. P. Lee
M. C. Pee
M. Zak
In House
January 1987 - January 1988

The primary objective of this task is to investigate the science and technology of spherical shells and to study the effects of gravitation on the formation of spherical shells both in the laboratory and in a weightless environment. The technology base being developed includes, but is not limited to, the fluid dynamics of viscous media, metallic, and amorphous materials. This technology is being applied to the process of shell fabrication; for rendering the shell spherically symmetric; to the technology of applying multi-layer coatings to the interior and exterior surfaces; to the sintering of the shell into a composite matrix material; to the development of the production of a novel high-strength, low-weight material for bonding of the spheres; and to the development of techniques needed for the encapsulation of various materials within the spherical shell such as phase-change materials for heat regeneration.

The science and technology of a new method for the production of spherical shells are developed under a coordinated experimental and theoretical program. The underlying production process is fluid-dynamic in nature and requires low or zero gravity. Foreseeable applications pertain to lightweight structural materials, to biotechnology, and to a variety of technical or industrial products.

The gravitational effects and the hydrodynamic instability with inclusion of the effects of viscosity, rotation, etc., are being studied. One emphasis of the work is encapsulation of biological materials for human implantation. Here cells or other biological materials are encapsulated within 200-400 μ m shells of specified permeability. Nutrients are free to pass into the shells, reactive products are free to diffuse outward, but the cell within each is protected against the large molecular-weight compounds of the immune system borne by the host. Accomplishments include the production of shells of uniform size of various polymers, a demonstration permeability to small and medium-sized molecules, and a demonstration of impermeability to large molecules. Also, encapsulation of cell-sized inert particles has been accomplished.

Theoretical model for the annular jet instability has been developed in which the liquid layer enclosing the gaseous stream in the jet is modeled as a membrane with no thickness but finite mass/area which moves under the influences of its own surface tension, inertia, and the gaseous pressure. It should be noted that the hollow jet instability is a phenomenon completely different from the more familiar Rayleigh instability of a simple jet. The methodology of converting a free surface flow problem into a one-dimensional one using a thin sheet model that can be handled more easily is an innovative contribution to the field of theoretical nonlinear physics.

We have derived a microscopic model of first-order phase changes in a one-component system with spherically symmetric, two-body potentials. Included is a microscopic model for the driving forces inducing the phase change, cooperative model for homogeneous nucleation, and a possible solution to the "fcc-hcp" problem in rare gases.

The basis of the production method involves the extrusion of liquid through suitable nozzles, following which discrete, precision, droplets are formed. These solidify in spherical and concentric form by means of freezing or by chemical reaction without gravitational distortion. The research concerns the hydrodynamic instability of liquid or compound jets, with inclusion of the effects of viscosity, rotation, etc. Both numerical and analytical results are utilized to interpret the experimental findings. Current emphasis of the work is upon encapsulation for biotechnology applicants. Here, cell or other biological materials are encapsulated within 200-400 μ m shells of specified permeability. Nutrients are free to pass into the shells, reactive products are free to diffuse outward, but the cell within each is protected against the large molecular weight compounds of the immune system borne by the host. Accomplishments include the production of shells of uniform size of various polymers, a demonstration of permeability to small- and medium-size molecules, and a demonstration of impermeability to large molecules. Also, encapsulation of cell-sized inert particles has been accomplished.

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Crystallization of Glass

University of Arizona
Dr. M. C. Weinberg
JPL (NASA Contact: G. Neilson)
September 1987 - September 1991

The objectives of this research are: (1) to study bulk (homogeneous) crystal nucleation in lithium diborate and other simple glasses; (2) to assess the viability (and desirability) of performing such nucleation experiments in a containerless facility in space; and (3) to provide accurate methods of computing the volume fraction crystallized in complex systems and/or systems of small particles.

Crystal nucleation may be enhanced by the presence of foreign substances in contact with the melt, leading to heterogeneous nucleation. Since crucible walls and contaminants introduced into the melt from the crucible can serve as heterogeneous nucleation sites, and uncontained melt might be subject solely to homogeneous nucleation. It is this belief which is the basis for the anticipated benefit of containerless processing for the potential production of novel glass compositions.

However, in order to be able to assess the potential advantages of containerless processing two items are required: (1) comparative ground based nucleation and crystallization experiments, (2) a more comprehensive knowledge of the factors which influence crystallization processes and a reliable theory to explain the latter. It is the purpose of this program to provide a framework which will allow for the interpretation, and guide in the judicious selection, of glass flight experiments pertaining to glass crystallization.

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Glass Forming and Crystallization of $\text{PbO-B}_2\text{O}_3$ Compositions in Space

University of Arizona
Dr. M. C. Weinberg
JPL (NASA Contact: G. Neilson, JPL)
September 1987 - September 1991

The objectives of this research are: (1) to investigate the surface characteristics and surface devitrification tendencies of lead borate glasses; (2) to study crystal growth rates in high PbO composition lead borate glasses; and (3) to assess novel optical and electro/optical applications of $\text{PbO-B}_2\text{O}_3$ glasses containing large proportions of lead.

A unique opportunity for studying glass formation and for the possible extension of the normal glass-forming compositional range is offered by a containerless processing facility in a microgravity laboratory. It is important to select appropriate compositions for such studies. It is believed that $\text{PbO-B}_2\text{O}_3$ compositions are well suited for this purpose for the following reasons. Lead glasses have a wide variety of commercial applications, and are of scientific interest, too, since they exemplify one of the unusual cases where large concentrations of a non-traditional glass-forming cation (pb) can be present in a glass-forming composition. Other advantages include: low liquidus temperature, acceptable degree of volatility, and a demonstrated tendency to devitrify at the melt-container interface.

In order to provide a framework for the interpretation of flight experiments, two types of ground-based experiments are planned at the University of Arizona. First, crystal growth studies will be executed as a function of temperature and glass composition. Next, the relative proclivity for free surface vs. container induced surface devitrification will be assessed. These experiments are key for understanding the intrinsic glass-forming limits of these compositions.

6. COMBUSTION SCIENCES

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Design and Evaluation of an Apparatus for Experiments on the Vaporization of Fuel Droplets in a Supercritical Environment at Microgravity Conditions

University of Wisconsin-Madison
Professor Gary Borman
Professor P. V. Farrell
NAG3-718 (NASA Contact: Kurt Sacksteder, LeRC)
April 15, 1986 - April 14, 1988

This program represents a joint venture between the University of Wisconsin-Madison and General Motors Research Laboratories. The study concentrates on the details of fuel spray breakup and vaporization in conditions similar to those of a direct injection diesel engine. The environment into which fuel is typically injected is well above the fuel critical pressure and may be above fuel critical temperature. Near-critical property and surface tension effects are known to vary rapidly as the critical point is approached, possibly leading to unexpected droplet breakup and vaporization effects. In order to study these effects in detail, it is desired to eliminate all convective effects (convective heat and mass transfer). One way to eliminate natural convection in a non-isothermal, multi-component field with diffusion is to reduce acceleration by operating at very small (microgravity) levels.

The program has focussed on two major areas: modelling and experiments. The objective of the modeling effort was to develop a computer code capable of modeling supercritical liquid vaporization in microgravity conditions. The most significant problems in the modeling effort arise in developing equation of state models valid near and above the liquid critical point, as well as at lower temperatures. The model work resulted in a paper which was presented at the 38th IAF Congress in Brighton, England in October 1987, and its being considered for publication in *Acta Astronautica*.

The experimental work has focussed on development of a compression device which can deploy a single, motionless droplet in microgravity, and rapidly increase the surrounding gas temperature and pressure to the desired levels. Diagnostics, including high speed movies, pressure transducer measurements, and Rayleigh scattering for gas density measurements are employed during the subsequent vaporization event.

The compression device has been constructed. Two series of tests have been performed using the device at the NASA Lewis 2.2 second drop tower. Preliminary results indicate that the mechanical performance and durability of the system appear satisfactory. The droplet deployment device, however, still requires some development. The currently installed diagnostics include high speed movies only, but the Rayleigh scattering device is under development.

Publications

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Buoyancy Effects Upon Vapor Flame and Explosion Processes

Science Applications International Corporation

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Dr. M. Yousef Bahadori

NAS3-22882 (NASA Contact: Dennis Stocker, LeRC)

November 1984 - January 1988

The objective of this microgravity project is to gain a better fundamental understanding of the effects of buoyancy on laminar gas jet diffusion flames and to establish the relationship between buoyancy and (1) unsteady phenomena associated with ignition and flame development, (2) steady-state flame structure, (3) soot formation and disposition, (4) radiation, (5) extinction phenomena, and (6) chemical kinetics. The findings will aid in defining the hazards and control strategies of fires in spacecraft environment as well as to improve the understanding of earthbound fires. The specific objectives of the program are to: (a) obtain measurements that include flame-shape development, flame extinction, flame color and luminosity, temperature distributions, species concentration, radiation, pressure, and acceleration, and (b) extend the numerical models developed to date to include transient effects, chemical kinetics, soot formation, and more detailed radiation effects.

The program is structured in terms of closely interrelated ground-based experiments and theoretical modeling. This program has evolved as a result of theoretical analyses and limited experimental observations which have delineated the requirements to gain a more fundamental understanding of the effects of buoyancy on gas jet diffusion flames. The experimental portion of the ground-based program is designed to provide both additional time and quantitative measurements based on the findings of the past 2.2 second drop-tower experiments. This data will be obtained by the combined use of the Lewis 5.18-sec. zero-gravity facility and the KC-135 aircraft. The results will be used as a database for the model development and will clarify the requirements on time for approach toward steady state. This will establish the need for space experiments. Also, inverted-flame studies are underway at SAIC, which will provide measurements of temperature, species concentration and velocity fields, in addition to radiation and quantitative information on flame dimensions. These studies will provide a baseline data set for both normal-gravity and negative-g situations, and help in the model development and validation. The device provides information on the effects of buoyancy outside the range of 1-g and 0-g conditions, and allows variation of all of the parameters while obtaining buoyancy effects based on the unlimited time available.

Since the time of the submission and approval of the Science Requirements Document and review of the Conceptual Design, the Initial Hardware Design Review, Preliminary Design Review, and Final Design Review have been held at NASA Lewis Research Center during 1987. Progress to date includes various analyses and modeling for the support of experiment fabrication. Work in progress includes the development of steady-state and transient models, in addition to the selection and development of submodels for diffusion, gas-phase kinetics, radiation, and soot generation and burn-off for inclusion in the models. A spark ignitor has been developed, and preliminary results of the 2.2-sec. drop tower experiments have shown that hydrocarbon flames can be successfully ignited in microgravity. In addition, fabrication of the experiment package has been initiated. The various findings of the experimental and theoretical efforts have been presented at the PACE Symposium (Washington, DC, March 1986), Discipline Working Group Meeting (NASA-LeRC, October 1986), Modeling Workshop

(NASA-LERC, November 1986), Seminar (UCSD, November 1987), and AIAA 26th Aerospace Sciences Meeting, Reno, Nevada, January 1988).

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Presentations

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A Fundamental Study of the Effect of Buoyancy on the Stability of Premixed Laminar Flows

University of California, Berkeley
Professor A. Carlos Fernandez-Pello
NAG3-861 (NASA Contact: Dennis Stocker, LeRC)
January 1988 - April 1988

The objective of this research is to investigate the effect of buoyancy on the stability of premixed laminar flames. The information obtained will help in the understanding of the mechanisms responsible for instabilities in laminar flames which are often regarded as precursors of turbulent combustion processes. The immediate objective is to perform a set of low-gravity, drop-tower experiments to elucidate the effect of the absence of gravity on the onset and evolution of cellular flame structure.

Experiments are being carried out at normal gravity and at microgravity of the onset of cellular structures on a burner-stabilized premixed flame. Normal gravity experiments have been already made to identify the range of parameters at which cellular flames occur. A few preliminary microgravity experiments have been performed at the 2.2 sec NASA LeRC drop tower. The experiments provided information about the effect of the absence of gravity on the premixed flame stability, combusting plume fluctuations, and soot generation. The results seem to indicate that the formation of cells is reduced under microgravity conditions, which implies that the absence of gravity has a stabilizing effect on the flame. The fluctuating character of the plume vanishes at microgravity, which results in a cylindrical plume surrounded by a weak diffusion flame. Except for the soot distribution in the plume, no major differences are observed in soot formation at normal and microgravity conditions. This work is currently being continued.

Presentations

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Ignition and Subsequent Flame Spread in Cellulosic Materials for Microgravity Applications

National Bureau of Standards, Center for Fire Research
Dr. Takashi Kashiwagi
C-32000-K (NASA Contact: R. Friedman, LeRC)
January 1988 - December 1990

The objective of this work is to determine the ignition and subsequent transition to flame spreading of cellulosic materials (filter-paper specimens) through radiant heating. This study is to lead to applications such as material flammability acceptance standards for spacecraft.

The research involves parallel efforts in theoretical modeling and experimental observations, both performed by the National Bureau of Standards investigator. The theoretical studies cover the prediction of the solid and gaseous-phase thermal behavior and the oxidative reactions, to establish the temperature and mass-flux fields in the reaction zone. The experimental studies cover measurements of mass loss, energy release, flame spread, and combustion-product species as functions of radiant ignition energy, oxidant flow, and other parameters. Experimental information on ignition-delay times, flame shape, and radiative properties of the fuel will contribute to the verification of the theoretical model.

The gas-phase analysis will incorporate governing equations that neglect gravity. Hence, the model will serve as a guide to define a specific experimental system and test procedure for microgravity to demonstrate material flammability and acceptance criteria, in follow-on work.

The Effect of Gravity on Premixed Turbulent Flames

University of California, San Diego
Professor Paul A. Libby
NAG3-654 (NASA Contact: Howard Ross, LeRC)
September 1, 1985 - October 31, 1987

Recent experimental and theoretical research has shown that the interaction of force fields arising from gradients of either mean pressure or Reynolds shear stresses with density fluctuations due to heat release as in reacting flows, leads to new mechanisms of turbulent transport and turbulence production. Gravity would appear to provide an additional force field which may result in significantly different behavior for premixed turbulent flames propagating upward for downward.

The experimental portion of a combined experimental and theoretical effort to study the effect of acceleration on premixed flames was completed in 1986 and submitted for publication as indicated in 8. An extension of the Bray-Moss-Libby theory of premixed turbulent flames to describe the effects of gravity on such flames has been completed and the result manuscript submitted for publication.

Publications

Hamins, A., Heitor, M., and Libby, P. A., "Gravitational Effects on the Structure and Propagation of Premixed Flames," Acta Astron., 1987 (accepted).

P. A. Libby, "Theoretical Analysis of the Effect of Gravity on Premixed Turbulent Flames." Comb. Sci. Techn., 1987 (submitted).

Effect of Low Velocity Forced Flow on Flame Spread over a Thermally-Thin Solid Fuel in the Absence of Buoyancy-Induced Flows

NASA Lewis Research Center
Sandra L. Olson
January 1987 - January 1989

The objective of this study is to determine the effect of low velocity opposed forced flow on flames spreading over thermally-thin fuels. The approach used in this study is to perform a series of experiments in low gravity varying oxidizer concentration and opposed forced flow velocity to determine the extinction limits and steady burning characteristics of flames spreading over solid fuels under these conditions. Tests will be conducted in a low speed combustion tunnel developed for use in all three NASA-Lewis Research Center's low gravity facilities.

Low gravity is required for these experiments because in normal gravity buoyancy-induced gas flows around the spreading flame are on the order of or greater than the range of forced flow velocities to be studied (0-30 cm/sec). These natural convective flows overwhelm or combine with the forced convective flows so that the effect of the forced flow on the flame spread rate cannot be isolated.

The study will be performed in four phases:

- 1) Preliminary normal gravity and low gravity quiescent environment (zero flow) and normal gravity high forced flow velocity tests will be performed to define fuel burning characteristics and to estimate fuel extinction limits as a function of oxygen concentration and flow velocity.
- 2) Simultaneously, flow field characterization of the combustion tunnel with cold flow in normal gravity will be performed and instrumentation will be developed for the low gravity tests.
- 3) Normal gravity tests and low gravity tests in all three Lewis low gravity research facilities will be conducted to determine the extinction limits as a function of oxygen concentration and flow velocity. The flame development and steady burning characteristics of flame spreading over solid fuels will be studied and compared with current modelling work underway at Lewis.
- 4) When the ground-based research is completed, the data will be analyzed and a report published of the results. If it appears that a space experiments is appropriate and feasible, a Science Requirements Document will be drafted. If necessary, further ground-based research will be proposed.

The normal and low gravity quiescent environment flame spread experiments in both the 2.2 and 5 second drop towers were completed during 1987. The results of these experiments are documented in NASA TM 100195. The proposal was approved for funding in September 1987. The remainder of the year was dedicated to building up the 2.2 second combustion tunnel experiment package for the flow experiments.

Publications

Olson, S. L., "The Effect of Microgravity on Flame Spread over a Thin Fuel," NASA TM 100195, 1987.

Time-Dependent Computational Studies of Flames in Microgravity

Naval Research Laboratory
Dr. Elaine S. Oran
Dr. K. Kailasanath
(NASA Contact: H. Ross, LeRC)
January 1987 - December 1987

The focal issues to be addressed are: (1) Will a gas mixture which is not flammable on earth burn in a reduced gravity environment?; (2) Are the minimum ignition energies different for mixtures which are flammable both at normal and reduced gravity?; and (3) Are there differences in the propagation of flames in normal and reduced gravity?

The approach is to use detailed time-dependent, one- and two-dimensional numerical models to calculate flame properties and then to look for a quantitative comparison between the predictions and experiments on earth and in space. These models solve the multispecies coupled partial differential reactive flow equations. These models contain detailed chemical kinetics mechanisms, algorithms for thermal conduction, molecular and thermal diffusion, and convective transport and include the effects of gravity.

In 1987, we completed the development of a detailed two-dimensional flame model based on the implicit BIC-FCT algorithm. The model includes algorithms for describing convection, chemical kinetics, thermal conduction, molecular diffusion, and buoyancy. The algorithm used for each physical process was tested separately, and the combined results were benchmarked against predictions from a different one-dimensional Lagrangian program. Finally, the two-dimensional model was used to study the evolution of perturbed hydrogen flames in both the lean and rich regimes, and the results were presented in terms of current theories of flame stability and the formation of cellular structures.

Publications

Kailasanath, K. and Oran, E. S., "Effects of Curvature and Dilution on Unsteady Flame Propagation, I. Flames and Configurations," Prog. Aero. and Astron. **105** (1986).

Patnaik, G., Boris, J. P., Guirguis, R. H., and Oran, E. S., "A Barely Implicit Correction for Flux-Corrected Transport," J. Comput. Phys., 1987 (in press).

Presentations

Patnaik, G., Boris, J. P., Guirguis, R. H., and Oran, E. S., "An Implicit Flux-Corrected Transport Scheme for Low Speed Flow," presented at AIAA 25th Aerospace Sciences Meeting, 1987, AIAA Paper 87-0482.

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: S. Olson, LeRC)
January 1987 - April 1988

The objective of the overall research program is the design and performance of smolder combustion experiment under microgravity conditions. The experiments will help to understand the mechanisms controlling smoldering, and in turn the prevention and control of smolder originated fires in normal gravity and in space-based experiments. The specific objectives are: to develop theoretical models to predict the controlling mechanisms and leading non-dimensional parameters of smolder combustion; to develop ground-based experiments to determine the effect of gravity on the different modes of smoldering; to provide a data base for verification of the theoretical models. To perform drop-tower tests to obtain data on the smolder transition processes of ignition, flaming and extinction; and to use the experimental and theoretical data to design a space-based smoldering combustion experiment.

The research program includes complementary theoretical and experimental tasks. To date theoretical models have been developed of the forced flow co-current and counter-current smoldering combustion. The models identify the non-dimensional controlling parameters and provide the smolder velocity and temperature distribution as a function of the material and oxidizer gas properties. Experimental tasks based on normal gravity and drop-tower experiments are currently underway. The experiments carried out to date have concentrated on determining the effect of buoyancy on the downward, co-current and counter-current smolder configuration. Since buoyancy is proportional to $g(\rho_i - \rho)$ it can be controlled by varying either g or the density difference. Most of the work performed to date has been following the latter approach. The density difference is varied by means of the gas pressure. Measurements of the co-current smolder velocity as a function of the oxidizer mass flow rate and pressure show that buoyancy only affects the process at low air mass fluxes. At large air mass fluxes the smolder velocity is linearly proportional to the air mass flux and weakly dependent on pressure. For downward counter-current smolder in free convection the smolder velocity is more sensitive to ambient pressure because of the natural convection gas currents induced at the top of the smolder reaction. A few preliminary tests performed in the NASA LeRC, 2.2 second drop tower indicate that the absence of gravity reduces the intensity of the smolder reaction, while the sudden increase in g induces the transition to flaming. Research in the above task is currently being continued.

Publications

Dosanjh, S. S., Peterson, J., Fernandez-Pello, A. C., and Pagni, P. J., "Buoyancy Effects on Smoldering Combustion," Acta Astron. **13**, 689-696 (1986).

Dosanjh, S. S., Pagni, P. J., and Fernandez-Pello, A. C., "Co-current Smoldering Combustion," Comb. & Flame **68**, 121-147 (1987).

Effects of Gravity on Flame Spread Involving Liquid Pool Fuels

NASA Lewis Research Center
Dr. Howard Ross
In-House
October 1987 - September 1990

The object of the proposed study is to increase the fundamental understanding of flame spread involving liquid fuel pools through experiments in a microgravity environment. Gravity affects the liquid fuel and gas phase motions and as such influences the supply of oxidizer and heat transfer ahead of the flame. Its role is sufficiently complex so that it is not clear a priori whether the flame spread rate will be faster or slower in reduced gravity than in normal gravity. To improve the understanding of the role of gravity, a range of liquid Grashof numbers on the order of 0.01 to 1,000,000 will be studied.

Reduced gravity tests will be performed in the 2.2 and 5 second facilities at LeRC. It is planned to study the flame spread rate dependence on gravity, pressure, container material and dimension, and oxidizer concentration for fuels above and below their flash points at a normal room temperature (ethanol, methanol, propanol, and butanol). Some normal gravity tests will be performed to establish baseline and additional scientific data. A comparison of experimental results to existing numerical models will then be made. These models may be modified as needed.

Depending on the results of the proposed efforts, the justification and feasibility of proceeding to a space experiment or of further ground-based microgravity research will be determined. If a space experiment is appropriate, a science requirements document will be drafted.

In 1987, 2.2 and 5 second drop tower rigs were designed and constructed to support initial tests to determine the feasibility of maintaining a quiescent, flat liquid-gas interface in reduced gravity conditions (this facilitates a direct comparison of normal and reduced gravity flame spread results). In 1988, a variety of sharp-edged container shapes will be tested; the containers will be filled completely in normal gravity, and then dropped. The time to reach a quiescent equilibrium shape will be recorded.

Ignition and Flame Spread Above Liquid Fuel Pools

University of California, Irvine
Professor William A. Sirignano
NAG3-627 (NASA Contact: Boyd Bane, LeRC)
January 1, 1987 - March 31, 1988

The objectives of this program are to obtain theoretical and computational results that will guide the development of an experiment on the subject of flame propagation above liquid fuel pools.

A computational study has been made of transient heat transfer and fluid flow in a cylindrical enclosure containing a two-layer-gas-and-liquid system. The geometric configuration and the boundary conditions on the problem are relevant to the analysis of the prevaporation and preignition processes during the fire accident situation involving a pool of liquid fuel in the vicinity of an ignition source. It is demonstrated that the effects of the natural and thermocapillary convection, radiative transfer, and thermal inertia and conduction of the walls bounding the enclosure and the magnitude of the gravity field play important roles in the development of the temperature and velocity fields in the container.

Publications

Abramzon, B., Edwards, D. K., and Sirignano, W. A., "Transient, Stratified, Enclosed Gas and Liquid Behavior with Concentrated Heating from Above," J. Thermophys. & Heat Transf. 1, 355-364 (1987).

7. EXPERIMENTAL TECHNOLOGY AND GENERAL STUDIES

Electrostatic Containerless Processing Technology

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

The long range objective of the task is the development of the science and technology base that is required to conduct contactless positioning and manipulation of high temperature materials using electrostatic and electrophoretic forces.

The primary objectives to be addressed are experimental and theoretical investigations of: (1) the hybrid electrostatic-acoustic positioning module in both the 1-g laboratory environment and in the reduced gravity environment of KC-135 aircraft; (2) the high temperature focused radiator electric quadruple levitator; (3) the melting and solidification of metallic samples in the focused radiator furnace; and (4) the hot furnace using low density samples in one-g environment.

Publications

Rhim, W. K., Chung, S. K., Trinh, E., Hyson, M. T., and Elleman, D. D., "Large Charged Drop Levitation Against Gravity," in *Proceedings of IEEE Industry Application Society*, 1986, p. 1338.

Rhim, W. K., Chung, S. K., Hyson, M. T., and Elleman, D. D., "Charged Drop Levitation and Their Applications," Mat. Res. Soc. Symp. Proc. 87, 103 (1987).

Rhim, W. K., Chung, S. K., Trinh, E., and Elleman, D. D., "Charged Drop Dynamics Experiment using a Electrostatic-Acoustic Hybrid System," Mat. Res. Soc. Symp. Proc. 87, 329 (1987).

Rhim, W. K., Chung, S. K., Trinh, E., Hyson, M. T., and Elleman, D. D., "Large Charged Drop Levitation Against Gravity," IEEE Trans. Indus. Appl. 1A-23, 975 (1987).

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Telepresence for Materials Science Experiments

NASA Lewis Research Center
Dr. James C. Johnston
M. J. Bonner
R. Hahn, RPI
B. Merbach, RPI

Telepresence involves the remote viewing and control of a process. A number of the materials science experiments that are anticipated for the space station would benefit from the application of telepresence. In some cases the ability of the Principal Investigator to observe an experiment, detect a developing problem and correct it can make the difference between success and failure. In others, the application of telepresence can free the station crew from a time-intensive interaction with an experiment and allow them to do other tasks.

This project involves the remote observation and control of three experiments currently available in the Microgravity Materials Science Laboratory (MMSL) at the NASA Lewis Research Center. The objective of these telepresence experiments is to determine the minimum digital communications rate and the minimum video frame rate and resolution necessary for successful performance of the materials science experiment. The experimental apparatus that are being used in the MMSL are the Isothermal Dendrite Growth Apparatus, the Crystal Growth Experiment, and the Single Axis Acoustic Levitator. Each of these experiments presents different problems and places unique demands on the telepresence system. The final outcome of the project will be a knowledge of the minimum communications bandwidth necessary for the performance of materials science experiments in space. This knowledge should prove invaluable in the planning and allocation of communications resources for space station and finally provide some hard numbers to use as a reference in the decision making process. The results of this work will be in the form of critical reviews answering the above questions.

This program represents a cooperative effort between the MMSL and Rensselaer Polytechnic Institute (RPI), and it is part of the telepresence project being undertaken by the Universities Space Research Association (USRA) which is funded through NASA Headquarters.

Presentations

Hahn, R., "Telescience Test Activities," Discipline Working Group (DWG) meeting, Washington, DC, November 1987.

Advanced Containerless Processing Technology

Jet Propulsion Laboratory
Dr. Taylor G. Wang
In-Center
Continuing Task

The objectives of this task are to: (1) develop next generation systems for Space Station containerless experiments such as electromagnetic-acoustic hybrid system, electrostatic-acoustic system, and high temperature acoustic system; (2) advance the knowledge base of high intensity and high temperature acoustics in the area of non-linear, instability, and streaming; (3) investigate the effects of acoustic electrostatic, electromagnetic, and temperature fields on the levitated sample, and vice versa; and (4) provide the assistance to develop a set of high temperature materials experiments for Space Station.

The detailed studies of the characteristics of high frequency (20 to 40 kHz) levitators have been carried out through experimentation at low and high temperatures. The flow visualization of acoustic streaming patterns around a levitated sample has revealed unexpected configurations which are the same at 20 and 40 KHz. These flows dramatically affect the heat transfer process between a heated levitated sample and the environment.

Two 50-watt CO₂ lasers are installed for heating test samples. The use of the laser for heating of a levitated sample has been demonstrated successfully. A holographic interferometer is being integrated into this system. Heat transfer rate of heated samples at temperatures up to 1500°C will be measured.

An automatic scanning laser acoustic pressure probe is under development. This system will perform a non-invasive pressure profile measurement. A general theory of oscillational instabilities of acoustically levitated samples in a resonant cavity has been developed.

The effects of spot heating levitated samples were evaluated for single-mode cylindrical levitators. An Xe arc lamp stably heated a room temperature sample to ~500°C.

Publications

Rhim, W. K., Chung, S. K., Trinh, E. H., Elleman, D. D., "Charged Drop Dynamics Experiments Using and Electrostatic-acoustic Hybrid System," Mat. Res. Soc. Symp. Proc. **87**, 103 (1987).

Trinh, E. H., and Olli, E. H., "Single Axis Acoustic Torque Generation and Control," NASA Tech. Brief, NPO 17086, 1987.

Robey, J. L., Trinh, E. H., Wang, T. G., "Acoustic Force Measurement in a Dual Temperature Resonant Chamber," J. Acoust. Soc. Am., 1987 (in press).

Lee, C. P., and Wang, T. G., "Acoustic Radiation Force on a Heated Sphere Including Effects of Heat Transfer and Acoustic Streaming," J. Acoust. Soc. Am., 1988 (in press).

Presentations

Leung, E. W., Baroth, E., and Wang, T. G., "Thermal-Acoustical Interaction in a Resonant System" American Chemical Society Meeting, New Orleans, August 1987.

B. FLIGHT EXPERIMENTS

1. ELECTRONIC MATERIALS

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Compound Semiconductor Growth in Space

NASA Langley Research Center
Dr. A. L. Fripp
Dr. W. J. Debnam
Mr. I. O. Clark
Dr. Roger K. Crouch, NASA HQ
In-House

This is an in-house effort with a small support contract for numerical analysis.

This research analyzes the effects of convection on the macroscopic and microscopic homogeneity of compound semiconductors grown by the Bridgman technique. The material of primary interest is lead-tin-telluride which is always thermodynamically unstable in a gravity field. Ground based experiments have involved the measurement of thermophysical properties, development of delineating etches to show composition variations, interface measurements and mathematical of the fluid dynamic during growth. Other work involve the direct fluid flow in a temperature gradient and the measurement and characterization of non-steady flow in high gradients.

The first space flight of this effort was on the D-1 mission in October 1985. Hardware anomalies resulted in unexpected temperature profiles. Ground based tests are still being conducted to understand the flight results.

Publications

Crouch, R. K., Fripp, A. L., Debnam, W. J. Woodell, B. A., Clark, I. O., Carlson, F. M., and Simchick, R. T., "Results from a Compound Semiconductor Crystal Growth Experiment in a Low Gravity Environment, *Proceedings of the SAMPLE Electronics Materials and Processes Conference*, June 23-25, 1987. Santa Clara, CA.

Barber, P., Crouch, R.K., Fripp, A. L. Debnam, W. J. and Simchick, R. T., "Image of Liquid Solid Interfaces in Directionally Solidified Crystals," NASA Tech Brief 13, 42 (1988).

Barber, P. Coleman, J., "Electrochemical Growth of Crystals in Gels," NASA Tech Brief 12 48 (1988).

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Crystal Growth of Device Quality GaAs in Space

Massachusetts Institute of Technology
Professor Harry C. Gatos
Dr. Jacek Lagowski
NSG 7331 (NASA Contact: Dr. Roger Crouch HQ)
December 1, 1987 - November 30, 1988

The objectives of the research are to investigate means of achieving GaAs single crystals of theoretical quality by utilizing zero gravity environment.

The research is aimed at developing characterization approaches and techniques for achieving quantitative relationships among crystal growth parameters and electronic properties of GaAs. These types of relationships are expected to point the way to achieving bulk GaAs crystals of theoretical quality.

Publications

Walukiewicz, W., Wang, L., Pawlowicz, L. M., Lagowski, J., and Gatos, H. C., "Effects of Macroscopic Inhomogeneities on Electron Mobility in Semi-insulating GaAs," J. Appl. Phys. **59**, 3144 (1986).

Kang, C. H., Kondo, K., Lagowski, J., and Gatos, H. C., "Arsenic Ambient Conditions Preventing Surface Degradation of GaAs during Capless Annealing at High Temperatures," J. Electrochem. Soc. **134**, 1261 (1987).

Kang, C. H., Lagowski, J., and Gatos, H. C., "Characteristics of GaAs with Inverted Thermal Conversion," J. Appl. Phys. **62**, 3482 (1987).

Lagowski, J., Bugajski, M., Matsui, M., and Gatos, H. C., "Optical Characterization of Semi-Insulating GaAs: Determination of the Fermi Energy, the Concentration of the Midgap EL2 Level and Its Occupancy," Appl. Phys. Lett. **51**, 511 (1987).

Presentations

Skowronski, M., Lagowski, J., Milshtein, M., Kang, C. H., Dabkowski, F., and Gatos, H. C., "The Effects of Plastic Deformation on Electronic Properties of GaAs," *14th International Conference on GaAs and Related Compounds*, Crete, October 1987.

Gatos, H. C. and Lagowski, J., "Bulk Crystal Growth of Semiconductor Compounds - GaAs," *Conference on Compound Semiconductor Growth Processing and Devices for the 1990's: Japan/U.S. Perspectives*, Gainesville, FL, October 1987.

Lagowski, J., Matsui, M., Bugajski, M., Kang, C. H., Skowronski, M., Gatos, H. C., Hoinkis, M., Weber, E. R., and Walukiewicz, W., "Quantitative Correlation Between the EL2 Midgap Donor, the 1.039 eV Zero Phonon Line, and the EPR Arsenic Antisite Signal," *14th International Conference on GaAs and Related Compounds*, Crete, October 1987.

Kobayashi, T., Lagowski, J., and Gatos, H. C., "Thermodynamic Analysis of the Role of Boron in Growing Semi-Insulating GaAs by the Bridgman Method," *5th Conference on Semi-Insulating III-V Materials*, Sweden, 1988.

A Comparative Study of the Influence of Convection on GaAs

GTE Laboratories, Inc.

Dr. James A. Kafalas

Mark Levinson

Ben Yacobi

Alfred Bellows

Dr. John Gustafson

NAS3-24644 (NASA Contact: Dr. R. Lauver, LeRC)

September 6, 1985 - September 5, 1989

The objective of this study is to determine the effects of buoyancy driven fluid flow on the properties of melt grown GaAs crystals.

Baseline GaAs crystals grown in the convection-free environment of the Space Shuttle will be compared to crystals grown on earth under various fluid flow conditions as determined by gradient orientation and the presence and orientation of a magnetic field. The characterization of the GaAs crystals will correlate the degree and nature of the convection with macro- and microsegregation effects, dislocation density distribution and electronic properties. The data will be interpreted based on model calculations of the fluid flow patterns in the melt under the various growth conditions. The improved understanding of the role of convection in the growth of GaAs gained from the proposed research will contribute to the refinement of GaAs growth techniques to produce substrate material with improved homogeneity and lower dislocation densities.

Publications

Kafalas, J. A. and Bellows, A. H., "A Comparative Study of the Influence of Buoyancy Driven Fluid Flow on GaAs Crystal Growth", in *Proceedings of 6th European Symposium on Material Science under Microgravity Conditions*, ESA SP-256, 1987, pp. 525-527.

Bellows, A. H., Duchene, G. A., "A Payload for Investigation the Influence of Convection on GaAs Crystal Growth", in *Proceedings of 1987 Get Away Special Experimenter's Symposium* (N. Barthelme, ed.), NASA CP 2438, 1987, pp. 77-82.

Solution Crystal Growth In Low-g

Alabama A&M University

Dr. R. B. Lal

Dr. W. R. Wilcox

Dr. J. D. Trolinger,

NAS8-36634 (NASA Contact: Mr. Rudolph Ruff, MSFC)

September 25, 1986 - December 31, 1988

This project involves a reflight of an earlier experiment, "Solution growth of crystal in zero-gravity", flown on spacelab-3 mission (NASA Contract NAS8-32945). The objectives of this research project are: 1) to grow crystals of triglycine sulfate (TGS) using polyhedral seeds using modified Fluid Experiment System (FES); 2) to study holographic interferometry tomography of the fluid fields in three dimensions and, 3) to study the fluid holography of tracers, and to estimate the influence of g-jitter on the growth rate.

Single crystal of TGS will be grown in the modified FES using (001) oriented polyhedral seeds. Experiments are underway to determine the proper seed size, so that natural (001) face seeds can be used for the flight experiment. The optical part of the FES system has been mocked up including the FES crystal growth cell and holography system. Some preliminary experiments have been conducted to determine the size, type, and number density of particles that should be used in the FES to monitor convective flow. Holograms for different conditions have been recorded and the reconstruction have been conducted to determine the size, type, and number density of particles that should be used in the FES to monitor convective flow. Holograms for different conditions have been recorded and the reconstruction have been used to see how clearly the flow field is characterized by the particles. Also, test is to design and select a holographic optical element (HOE) that can be attached to the FES windows that will split the incoming beam at the first window into three beams at different angles and the second window will reconverge the three beams so that they all hit the hologram. This will provide three independent views of the fluid field. A preliminary design of HOE is complete.

Publications

Lal, R. B., Aggarwal, M.D., Batra, A.K., Kroes, R. L., Wilcox, W. R., Trolinger, J. D., and Cirino, P., "Growth of Triglycine Sulfate (TGS) Crystals Aboard Spacelab-3", NASA CP-2429, 1987.

Lal, R. B., Aggarwal, M. D., Batra, A. K., and Kroes, R. L., "Solution Growth of Crystals in Zero-Gravity," Final Technical Report, NASA Contract NAS8-32945, July 1987

Presentations

Lal, R. B., Batra, A. K., Aggarwal, M. D., Wilcox, W. R., and Trolinger, J. D., "Solution Growth Experiment on International Microgravity Laboratory (IML)," Symposium of Microgravity Research and Processing, American Chemical Society Meeting, September 1987, New Orleans, LA.

Trolinger, J., Tan., H. and Lal, R. B., "A Spaceflight Holographic System for Flow Diagnostics in Microgravity Experiment," Sixth International Congress on Applications of Lasers and Electron Optics (ICALEO) November 1987, San Diego, CA.

Growth of Solid Solution Crystals

Marshall Space Flight Center
Dr. S. L. Lehoczky
Dr. F. R. Szofran
In-House
Continuing task

The major objective of this research is to establish the limitations imposed by gravity during growth on the quality of bulk solid solution semiconducting crystals. An important goal is to explore the possible advantages of growth in the absence of gravity. The alloy system being investigated is $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$ 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 required to define the optimum experimental parameters for the planned flight experiments. $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$ is representative of several II-VI alloys which have electrical and optical properties that can be compositionally tuned to meet a wide range of technological applications in the areas of sensors and lasers with applications to optical computing and communications as well as the national defense.

A series of $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$ alloy ingots ($0 < x \leq 0.6$) has been grown from pseudobinary melts by a vertical Bridgman-Stockbarger method using a wide range of growth rates and thermal conditions. Precision measurements were performed on the ingots to establish compositional distributions for the ingots. Growth rates and thermal conditions required to obtain the desired growth interface shape have been established for the system.

To assist the interpretation of the results and the selection of optimum in-flight growth parameters, the pseudobinary phase diagram ($0 \leq x \leq 1$), liquid and thermal diffusivities ($0 \leq x \leq 0.3$), and the specific volumes as a function of temperature ($0 \leq x \leq 0.15$) have been measured. From these measurements and other available data, the heat capacity, enthalpy of mixing, and the thermal conductivity of pseudobinary melts have been calculated using a regular associates solution model for the liquid phase. A one-dimensional diffusion model that treats the variation of the interface temperature, interface segregation coefficient, and growth velocity has been used to establish effective diffusion constants for the alloy system. Theoretical models have been developed for the temperature distribution and the axial and radial compositional redistribution during directional solidification of the alloys. These models are sufficiently accurate that they will be used along with the experimental results to select parameters for the first flight experiment.

A microscopic model for the calculation of charge-carrier concentration, Fermi energy, and conduction-electron mobility as functions of x , temperature, and both ionized and neutral defect densities has been developed. For selected samples, measurements were performed of electron concentration and mobility from 10-300K. The experimental data were in reasonably good agreement with theory and were successfully analyzed to obtain donor and acceptor concentrations for various processing conditions.

The crystal growth studies have been extended to include additional Hg-based II-VI alloys. Large crystal ingots of HgZnTe and HgZnSe have been successfully grown by the Bridgman-Stockbarger method and a detailed theoretical analysis of the measured

axial compositional distribution in the ingots was used to establish for the first time effective HgTe-ZnTe and HgSe-ZnSe interdiffusion coefficients for the molten alloys. Both the Te and Se-based alloys showed improvements in lattice strength resulting from the substitution of Zn into the respective Cd lattice sites. In particular, measured electrical and optical properties of the HgZnSe crystals indicated that the addition of Zn to the HgSe system was effective in stabilizing the electrical properties, thus providing the first direct experimental confirmation for predicted improvement in lattice stability against point-defect formation resulting from Zn-additions.

Publications

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Vapor Crystal Growth of Mercuric Iodide

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January 1987 - January 1988

Single crystals of mercuric iodide are used in high-efficiency x-ray and gamma ray detectors operating at ambient temperature. Optimal operation of the devices is determined to a large degree by the density of structural defects in the single crystalline material. Since there are strong indications that the quality of the material is degraded by the effects of gravity during the growth process, a program was initiated to grow one or more crystals of mercuric iodide in the reduced gravity environment of space.

Specifically, there are two reasons to perform the space experiments:

1. Single crystals of mercuric iodide are prone to slippage under the effect of gravity, especially at the elevated growth temperatures, with a concurrent decrease in structural quality.
2. It is not clear what effects convection flows in the vapor phase have on the growth and the homogeneity of the crystals. Growth in reduced gravity would provide information regarding these questions.

The first experiment, performed during the flight of Spacelab 3 (April 29-May 6, 1985), was highly successful in the sense that all scientific objectives were fulfilled. The structure of the space-grown crystal was more homogeneous and the critical electronic properties were increased by a factor of seven compared with the best earth-grown crystals.

Preparations are underway for the next experiment, to be flown on the first flight of the International Microgravity Laboratory (IML). Present ground-based research and experimental development activities concentrate on improving the control system of the flight equipment and increasing the temperature of the growth process so that larger crystals can be obtained in the limited time available during the flight.

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Vapor Growth of Alloy-Type Semiconductor Crystals

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NAS8-32936 (NASA Contact: D. A. Schaefer, MSFC)
March 1978 - March 1988

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 objectives of ground-based studies are 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. An important aspect of the theoretical effort is the further development and improvement of transport models for diffusion limited mass transport of simple and of multi-component, multi-reaction vapor transport systems.

The specific experiments to be performed in a microgravity environment include the investigation of vapor transport and crystal growth phenomena of the $\text{Hg}_{1-x}\text{Cd}_x\text{Te-HgI}_2$ system. Emphasis for this system is on the mass flux, on the unseeded growth of bulk crystals, and on the growth of epitaxial layers. The above experiments are performed in closed, fused silica ampoules.

The objectives of the $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$ experiments are to determine the positive effects of microgravity on vapor phase 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 involve systematic investigations of the growth rate, morphology, homogeneity, and electrical properties 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 goal to observe the effects of convective interferences on layer morphology and properties. The results of on-going ground-based studies are continuously evaluated and are used for the systematic modification of grown parameters with the important goal to define optimum experimental conditions for the microgravity experiments of this system.

In addition to the above experimental efforts, theoretical work on the $\text{Hg}_{1-x}\text{Cd}_x\text{Te-HgI}_2$ system is concerned with the thermodynamic analysis of the solid-vapor equilibria, with the development of a transport model, and with the prediction of diffusion limited mass transport rates of this system.

The above studies are supported by quantitative vapor pressure measurements of $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$ for different compositions and as a function of temperature, employing dynamic microbalance techniques. The major objectives of these experiments are the direct, in situ determination of Hg vacancy concentration of $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$ and the derivation of the heat of vacancy formation for this important material. In combination with the results of electrical measurements, the above studies will make a significant contribution to the further elucidation of the mechanism of vacancy formation of $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$.

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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**2. SOLIDIFICATION OF METALS, ALLOYS
AND COMPOSITES**

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Dynamic Thermophysical Measurements in Space

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January 1, 1987 - January 1, 1988

The objective of this research is to develop techniques for the millisecond-resolution dynamic measurement of selected thermophysical properties (heat of fusion, heat capacity, surface tension, electrical resistivity, etc.) of high-melting-point electrically-conducting solids and liquids at temperatures above 2000 K in a microgravity environment. This will enable, for the first time, extension of the accurate thermophysical measurements to temperatures above the limit (melting point) of the ground-based millisecond-resolution experiments.

The first phase of the research is to establish the geometrical stability of a specimen when heated rapidly to temperatures above its melting point in a microgravity environment. Work in this direction is being continued. A test equipment package has been designed and constructed which permits rapid heating of the specimen to temperatures above its melting point and checking of the geometrical stability of the liquid specimen. This system consists 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. This system has been flown several times on board a KC-135 aircraft. The results suggested several refinements and modifications in the system, which are presently being made. Some additional theoretical work is underway in this direction to understand the behavior (geometrical stability) of the liquid specimen heated rapidly by the passage of a high current pulse through it, and as a result, optimize the specimen geometry and the operating conditions of the overall system.

The second phase of the work is to add new capabilities to the system for the rapid and accurate measurement of current, voltage, temperature, and temperature gradients in the specimen, taking into account the requirements for operation in a microgravity environment for extended periods of time. Significant progress has been made in this direction, including construction of two high-speed pyrometers (multiwavelength and spatial scanning). The system will be used to demonstrate the applicability of the technique to performing definitive measurements of selected thermophysical properties of a refractory metal, such as niobium, at and above its melting point in a microgravity environment. The feasibility of a new technique for measuring the surface tension of liquid metals at high temperatures was demonstrated by preliminary experiments in KC-135 aircraft.

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Alloy Undercooling Experiments in Microgravity Environment

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May 29, 1986 - March 31, 1989

The objectives of the research are: (1) to evaluate containerless melting and solidification of nickel and iron base alloys with and without softened glass coatings; (2) to develop techniques for study of recalescence and growth behaviors during solidification of undercooled melts at zero gravity; (3) to develop an understanding of undercooling phenomena in microgravity environment; and (4) to develop and understanding of microstructures so produced.

The first alloy undercooling experiments was performed in an electromagnetic levitator during the Columbia STS-61C Mission in January 1986. One eutectic nickel-tin (Ni-32.5wt%Sn) alloy specimen was partially processed before an unanticipated equipment failure (clogging of the water cooling line) terminated the experiment. Examination of the specimen and the results of the thermal history measurement showed evidence of undercooling. Detailed results and discussion of the flight experiment are summarized and reported in published papers. The directly related ground base experimental and analytical studies, which have also been published, include dendrite growth rate measurement with initial undercooling, thermal history measurement, metallographic studies, and a summary paper.

Among the results that have been obtained thus far is the fact that maximum recalescence temperatures after some milliseconds in the samples (Ni-25wt%Sn) are such that the liquid and solid must be present in amounts and compositions exactly at that equilibrium predicted by the lever rule and the equilibrium phase diagram. Ostwald ripening or "coarsening" is being shown to be of controlling importance in the later stages of recalescence as well as in the fineness of the structures. By optical and high speed photographic measurements, we are able to observe the growth of dendrites in the early stages of growth and observe these to sweep across the entire sample at relatively early stages of the recalescence. By this method, we are able to carry out in-situ measurements of dendrite growth rates as a function of undercooling. As a result of these experiments we are beginning to develop the first quantitative understanding of solidification and recalescence behavior in undercooled droplets—certainly the first that is based not only on modeling studies by on experimental verification. Experimental results of tip velocity vs. undercooling agree well with Lipton, Kurtz, and Trivedi analysis. No evidence was observed of "solute trapping" over the range of undercoolings studied (30–320 K) for Ni-25wt%Sn and Ni-32.5%Sn alloys.

We will continue to work on Ni-x%Sn alloys as well as Fe-x%Ni alloys with increased emphasis on modeling. In experiment work, thermal history measurements, including recalescence time and solidification time, will be performed in detail. Metallographic work will be contained on samples produced. Optical and scanning electron microscopy (SEM) will be used to observe morphology and fineness of microstructure, including grain size and dendrite arm spacing. EMPA and EDAX will be used to analyze the composition profiles. TEM and STEM will be used for ultra-fine microcrystalline structures.

New experimental results are being used to modify and improve the assumptions

of this solidification model. Consequently, we are now coming to understand much better the significance of and ways to model: (1) kinetic processes occurring at the dendritic tip during the early stage of recalescence; (2) solute diffusion controlled thickening of dendrite arms during the second period of recalescence; and (3) ripening controlled growth and fineness of the structure during the third period of recalescence. This work will be continued and extended to develop a full physical model of the solidification process at high undercoolings encompassing thermodynamics, kinetics, heat flow, solute diffusion, interface velocities, remelting, and coarsening-and correlation of the model with experimental measurements.

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Isothermal Dendrite Growth Experiment

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October 1983 - October 1987

The objective of this flight experiments is to assess the influence of gravity on the growth kinetics and solidification morphology of freely growing dendrites.

The work has focussed on three areas: (1) growth chamber design and development, (2) evaluation of the photographic data collection system, and (3) data analysis and reduction procedures.

Growth Chamber Design

The laboratory growth chamber has been redesigned for the flight experiment. The preliminary design of the flight growth chamber has incorporated a number of features not present in the laboratory model. The chamber design has not been tested and remains unproven; nevertheless we are confident this design will meet the scientific and engineering requirements for flight aboard STS. The flight chamber design has passed a Preliminary Design Review held at NASA Lewis Research Center on April 21, 1987. A discussion of the preliminary design of the flight chamber is presented below.

An exhaustive set of materials compatibility tests were run in order to construction materials with the least interaction with SCN is a subset of stainless steels. To obtain free dendritic growth, driven only by diffusion of heat through the liquid, it is necessary to indicate dendritic growth at the center of the chamber. This is accomplished via a capillary injector (stinger). The stinger consists of a stainless steel tube, with a glass tip seal to the end, in the growth chamber. The glass tip is pulled to a small diameter, approximately one millimeter outside diameter and 200 micron inside diameter. The end of the stinger outside the growth chamber is sealed with a welded stainless steel plug and capped with a copper plate.

The initiation of the growth front in the stinger is accomplished with four small thermoelectric coolers attached to the copper plate on the end of the stinger stem. The coolers are potted in epoxy to provide thermal isolation from the thermostatic bath. These coolers will allow the central microprocessor to initiate growth when all coolers will allow the central microprocessor to initiate growth when all of the experimental conditions have been met. Once the coolers are switched on they drive the temperature of the stinger well below the nucleation temperature of SCN. Once initiated, the growth front then proceeds down the stinger where it emerges in the center of the growth chamber to be photographed. The growth chamber must provide two unobstructed orthogonal viewing axes. Thus, the chamber contains four windows. The windows consist of glass flats sealed to stainless steel bezels which are, in turn, electron-beam welded to the chamber body. Sample volume change compensation, which is a special problem under low gravity, also had to be included in the growth chamber design. The thermal expansion of the solid from 25 C to 58.1 C, the phase-change volume expansion, and the thermal expansion of the liquid from 58.1 to 62 C must be compensated for to prevent stray vapor cavities from forming and to prevent overpressuring the specimen chamber. In addition the system must remain hermetically sealed, prevent free surface formation such as shrinkage pores, and be constructed

entirely from stainless steel and glass. The volume expansion and free surface control are accomplished with a stainless steel bellows electron-beam welded between the top and bottom halves of the growth chamber. The bellows throw and spring rate sufficient to compensate for all volume changes with a large safety factor. The growth chamber is sealed under vacuum and the external pressure transmitted through the bellows from the thermostatic bath is sufficient to prevent any free surface formation. The final component of interest in the growth chamber is an ultra stable thermistor. This thermistor serves two functions. First, when used in conjunction with a laboratory temperature standard during ground based testing, the purity of the SCN in the chamber can be checked. Secondly, during flight, this thermistor may be used to cross-calibrate all temperature sensors located inside the thermostatic bath to the actual measured melting temperature of the sample.

Photographic Data Collection System

Several photographic testing sessions have been conducted over the past year in conjunction with NASA Lewis Research Center. These sessions have attempted to identify and define certain features of the photographic system needed to meet depth of field and resolution requirements necessary for the precise measurement of dendrite tip radii and growth velocities. A modified shadowgraphic technique will be utilized using a conventional flash lamp light source which provides the best compromise between the quality to the image and the exposure time. In addition, flat window glass growth chambers were designed to aid in the photographic testing.

Data Analysis and Reduction

In conjunction with the photographic tests, a data analysis and reduction system has been developed concurrently in order to analyze the negatives from the various photographic systems. The system, consisting of a Ram Optical Instrument (ROI) optical measurement microscope (linear resolution - 1 micron), an oscilloscope and an IBM XT computer, has been assembled to objectively evaluate the quality of an image. The output of the microscope's camera is fed into the oscilloscope enabling the relative intensity of the image to be displayed. Three different types of measurements can be made to assess the quality of a particular dendritic image. Growth velocities are calculated by measuring the distance a dendrite has advanced between successive frames and the time between those frames and compared to ground based data. Secondly, an edge function width is measured with the aid of the oscilloscope. The edge function is defined as the distance over which the intensity of image falls from white (dendrite) to black (background) and may be equated to the uncertainty of the actual position of the edge. Data points around the edge of the dendritic image are measured and fit to a parabola using standard multiple regression routines. Tip radius may then be calculated and compared to the ground based data. The ROI optical microscope has been interfaced to an IBM XT and communication software has been developed allowing the direct input of data from the microscope into the computer. In addition, digital image processing of the images is currently under development to assist further in the objective evaluation of the photographs.

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Orbital Processing of Aligned Magnetic Composites

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November 1987 - October 1989

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

Three flight experiments have been planned in which aligned, two-phase, magnetic composites will be grown by plane front directional solidification. Each experiment sequentially processes four independent samples in Automated Directional Solidification Furnace (ADSF) Systems. 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) under thermal conditions such that the solidification interface is flat (plane front solidification) and at a constant solidification velocity.

The first of these experiments was conducted in the Low Temperature Automated Directional Solidification Furnace System (ADSF-1), which was integrated into the mid-deck of Space Shuttle "Discovery" on Mission 51-G. The second of these experiments flew in the High Temperature Automated Directional Solidification Furnace System (ADSF-2) on the Material Science Laboratory Carrier (MSL-2) in the payload bay of the Space Shuttle "Columbia" on Mission 61-C. The 51-G experiments studied off-eutectic Bi-Mn directional solidification whereas the 61-C experiments studied Co-Sm eutectic solidification.

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 thermal model for the Bridgman-Stockbarger solidification technique including sample, ampoule, and translation, has been developed, and solidification models for eutectic, off-eutectic, and peritectic solidification with partial mixing in the melt, have been derived. The level of natural thermo-solutal convection is varied by employing magnetic field (transverse and longitudinal) damping (MFD). Studies varying the thermosolutal driving force for convection and comparative analyses with magnetically damped and micro-g processed samples will identify the role of gravitationally driven convection in Bridgman-Stockbarger plane front solidification.

Relationships between solidification processing parameters (including gravity vector), microstructure, macrostructure, chemistry as a function of fraction solidified, crystal structures, and magnetic properties are also being developed. Microstructure and macrostructure are being quantitatively analyzed using quantitative metallographic 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.

Micro-g results from Mission 51-G have shown that diffusion controlled growth ($k_{\text{eff}} = 1$) can be achieved in orbit that appear to be unachievable terrestrially, even using MFD. The diffusion-controlled results in micro-g, however, showed a greatly enhanced contribution of Soret diffusion to the chemical macrosegregation. This result was shrouded terrestrially by gravitationally driven convection.

Results from Mission 61-C have shown damping of the thermosolutal convection comparable to that measured in the 51-G experiment, diffusion-controlled growth having been achieved. Surprisingly, in a portion of the sample allowed to free-cool, the morphology noted can only be explained on the basis of significant thermal undercooling of the melt. Attempts to reproduce this morphology terrestrially have only succeeded under rapid solidification conditions. Further attempts employing MFD and deep thermal undercooling techniques are being undertaken.

The next flight experiment will be conducted in the Low Temperature Automated Directional Solidification Furnace (ADSF-1) System, in the mid-deck of Space Shuttle "Discovery" on Mission 26. Bi-Mn eutectic (0.72 w% Mn) and off-eutectic (0.60 and 0.45 w% Mn) samples will be directionally solidified at 1.0 cm/h, with an imposed thermal gradient of approximately 100K/cm. The contribution of Soret diffusion to the macrosegregation in the off-eutectic samples will be quantitatively evaluated and the Soret diffusion coefficient inferred from the flight data will be compared with the value determined terrestrially. Further, the eutectic rod and inter-rod dimensions will be determined and the interface undercooling measured in-situ, in order to determine whether the previously noted microstructural refinement during micro-g processing results from increased interface, undercooling, a decreased transport coefficient, or both.

The one-g experimental studies, in conjunction with theoretical analyses and experimentally determined thermophysical property measurements, 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 damped results will determine the uniqueness of the orbital processing.

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Solidification Fundamentals

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NAG3-417 (NASA Contact: Hugh Gray, LeRC)
March 1983 - continuing task

The objective of this research is to obtain a fundamental understanding of gravitational during solidification of metals and alloys.

Experimental work underway can be divided into three major categories. First, experiments in support of a Space Shuttle experiment on macrosegregation behavior in Pb-Sn alloys. Second, experiments aimed at obtaining a somewhat more fundamental understanding of dendritic and cellular growth, using a directional solidification apparatus. Third, experiments aimed at understanding the influence of undercooling on macro and micro segregation behavior in bulk samples (> 20 grams) of binary Pb-Sn alloys. This experimental work is also being complimented by theoretical work aimed at understanding these fundamental solidifications phenomena. Much of the experimental work described here is being performed in the MMSL, at LeRC.

The Space Shuttle experiment which was originally designed to employ the General Purpose (Rocket) Furnace (GPF) is now being re-defined since the GPF will not be available for future shuttle missions. Current plans are to employ the Multiple Experiment Processing Facility (MEPF) which will have many of the capabilities of the GPF. Analysis of the experimental work directly in support of the shuttle experiment was also the subject of the M.S. thesis work of Mr. Anthony Stider, which is now completed. The significant findings of this work are 1) the isothermally processed samples show a small and gradual increase in fraction eutectic, and hence tin content, from the bottom to the top of the ingot. This is usually referred to as normal macrosegregation and suggests flotation of segregated and lighter, tin enriched, liquid to the tip of the ingot during solidification. 2) there are significant radial variations of eutectic fraction and tin content, with radial variations being most pronounced near the top of the ingot. 3) the average fraction eutectic formed in the sample (although not the detailed variations) can be predicted quite satisfactorily on the basis of simple semi-analytical models which allow for variation of the equilibrium partition ratio during solidification and also, at the same time, account for some back diffusion of the rejected solute into the solid phase. In Pb-15 wt% Sn alloy, K varies from about 0.50 near the liquidus temperature to about 0.30 near the eutectic temperature.

Experiments aimed at obtaining a somewhat more fundamental understanding of dendritic and cellular growth form the subject of the doctoral thesis of Mr. Li Wang. These are aimed at obtaining simultaneous measurements of tip radial, primary and secondary dendrite arm spacings, tip temperature and tip composition, and details of microsegregation within the interdendritic liquid and in the immediate vicinity of the growing tips in the array. During the past year more than 50 experiments were conducted in Pb-Sn alloys, with various compositions to cover the entire phase diagram. Unidirectional solidification experiments with Pb-rich alloys, invariably lead to difficulties with gravitational instabilities. These are being properly classified. It has been possible to measure dendrite tip characteristics in the Sn-rich alloys, and preliminary analysis indicate satisfactory agreement between theory and experiment on

the basis of models proposed by Laxmanan. Another significant finding here has been that the diffusivity of Pb in Sn is a fairly strong function of composition. Diffusivities were measured independently in an apparatus similar to that used by Lommel and Chalmers with Pb-Sn alloys.

Finally, in our bulk undercooling experiments (samples size > 20 grams) with Pb-Sn alloys, in collaboration of Mr. Henry deGroh III, substantial undercoolings have been achieved; up to 40 K in some cases. Gravitational segregation effects are quite apparent in these undercooled samples.

Ground-based research in the last two categories could form the basis for defining some future space shuttle experiments.

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Casting and Solidification Technology (CAST)

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Dr. T. Dwayne McCay
Dr. Robert Owen
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NAS8-37292 (NASA Contact: R.C. Ruff, MSFC)
December 1986 - December 1990

The objectives of research are: (1) to determine the influence of gravity on the fluid flow and nucleation that occurs during casting, and (2) to investigate the solidification and coarsening processes of dendrite arms and their subsequent influence on the grain structure in castings.

The purpose of the investigation is to study the directional solidification of metal-model materials under low-gravity conditions. In particular, the inverted density layer and the thermal and solutal fields ahead of growing interface will be analyzed. This investigation is an extension of previous low-g studies done on Space Processing Applications Rocket (SPAR) and KC-135 flights. To complete these studies, longer periods of low-g are required in order to allow solidification to occur at slower and more controllable rates. Therefore, detailed analysis will be made of fluid motion near the solidification interfaces using the optical techniques (Schlieren, shadowgraph, interferometry and holography) available in the ground based laboratory and in the Fluid Experiment System on IML-1. To aid in separating the thermal and solutal profiles, thermocouples will be placed at intermittent locations along the cuvette wall. The temperature measurements will enable the investigators to mathematically separate the thermal and solutal effects on the interferograms.

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3. FLUID DYNAMICS AND TRANSPORT PHENOMENA

Zeno: Critical Fluid Light Scattering

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NAG3-727 (NASA Contact: Dr. R. Lauver, LeRC)
May 15, 1986 - December 15, 1987
NAG3-849
December 15, 1987 - October 1, 1988

The objective of this research is to measure the decay rates of critical density fluctuations in a simple fluid (xenon) very near its liquid-vapor critical point using laser light scattering and photon correlation spectroscopy. Such experiments are severely limited on earth by the presence of gravity which causes large density gradients in the sample. The goal is to measure fluctuation decay rates at least two decades closer to the critical point than is possible on earth, with a resolution of 3 microKelvin. This will require loading the sample to 0.1% of the critical density and taking data as close as 100 microKelvin to the critical temperature ($T_c = 289.72$ K). The minimum mission time of 100 hours will allow a complete range of temperature points to be covered, limited by the thermal response of the sample. Other technical problems have been 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, a fast optical thermostat with satisfactory microcomputer temperature control and measurement, and accurate sample loading. There remains the important engineering tasks of mounting the experiment to maintain alignment during flight and using vibration isolation to prevent Shuttle motions from distorting the sample.

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

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

During this period the engineering requirements have been developed from the science requirements, various trade-off studies completed for overall design requirements, and several key technologies demonstrated. The current lab version of the experiment has been used to demonstrate the necessary alignment techniques, automated operation of the experiment including a high resolution search for T_c , multiple correlation collection to improve accuracy of decay rate measurements, long term digital integration for high precision transmission measurements. A new optical cell with 100 micron optical path was accurately filled with Xe and used to make lab measurements from critical fluctuations.

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Surface Tension Driven Convection

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Professor Y. Kamotani
NAG3-570 (NASA Contact: T.P. Jacobson, LeRC)
August 1984 - July 1986

The objective of the investigation is to design a thermocapillary experiment to study the transient and steady-state flows in the long-duration low-g environment of the Shuttle.

The experiment consists of a circular container (5 cm dia. and 5 cm deep) filled with silicone oil, heating systems, and a data acquisition system. The fluid free surface will be heated locally by a CO₂ laser or by a submerged circular heater placed at the center. The resultant temperature variation along the free surface will generate thermocapillary flow in the container. The flow field will be studied by a flow visualization technique and the temperature distribution along the free surface, which is important because it determines the driving force of the flow, will be measured by a thermography technique. The surface heat flux distribution, the heating level, and the static free surface shape will be varied to study their effects on the nature and extent of the flows. Two series of experiments are planned. In the first one, the basic thermocapillary flow will be studied and attempts will be made to obtain oscillatory thermocapillary flow. In the second series the oscillation phenomenon will be studied in detail because it is considered to be an important aspect of thermocapillary flow. Ground-based and drop tower experiments together with a numerical analysis have been made to provide base data and to ensure that the operating condition and the configuration will lead to flows that can be reasonably observed and measured. The engineering feasibility study of the experiment is being conducted at NASA Lewis Research Center.

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Mechanics of Granular Materials

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NAS8-35668 (NASA Contact: I. C. Yates, MSFC)
November 2, 1983 - July 31, 1988

This research effort is aimed at understanding the constitutive behavior of granular materials subjected to very low intergranular stress levels.

Current research tasks include assessment of fabric and structure in the granular specimens by means of digital image processing techniques and quantification of degrees of homogeneity and isotropy. The aim is to relate such indices to the constitutive behavior of the specimen. Ground-based experiments have been conducted for several years to broaden the data base, especially at very low stresses, where the self-weight of the material tends to destabilize the specimen. Special attention is given to specimens at high packing density and how they behave at low stresses, where the dilatancy rate is the largest. Various constitutive models have been proposed and are used to predict behavior in the absence of body forces at the materials level. At the global specimen level the system is treated as a full boundary value problem that is solved by finite element analysis techniques. The problem is highly nonlinear, and the rate equations are solved in terms of incremental and iterative implicit schemes. Inverse identification of material constants and parameters are performed both at the local material and global levels.

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The Mathematical and Physical Modelling of Electromagnetically Drive Fluid Flow and Associated Transport Phenomena in Contained and in Containerless Melts

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NAG3-594 (NASA Contact: Fred Harf, LeRC)

The objective of this research is to develop an improved fundamental understanding of electromagnetic, heat flow and fluid flow phenomena in levitation melted specimens under both earthbound and microgravity conditions. The main motivation of this work is twofold:

- a number of fundamental hydrodynamic and electromagnetic issues may be uniquely addressed in this manner;
- levitation melting is a key ingredient of many materials processing experiments in space, thus the present project provides an important support function for this effort.

The current research pursues two complementary directions:

- extensive computational work is being carried out to predict the electromagnetic force field, the velocity field and the temperature fields in electromagnetically stirred (positioned) metallic specimens. An important novel feature of this effort is that an allowance is being made for the behavior of free surfaces and free surfaces deformation.
- experimental work is being carried out to measure and predict electromagnetically driven flows in a molten Woods metal pool due to the passage of current between two electrodes.

Important milestones of the research include the following:

- the development of a general methodology for computing electromagnetic force fields and velocity fields in complex geometries. (These results have important, immediate ground based applications!)
- very accurate measurement of electromagnetically driven flows in molten metal systems.
- new initiatives for carrying out preliminary measurements in sounding rockets.

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Production of Large-Particle-Size Monodisperse Latexes in Microgravity

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Dr. F. J. Micale
Dr. M. S. El-Aasser
Dale Kornfeld, MSFC
NAS8-32951 (NASA Contact: V. Yost, MSFC)
January 1987 - January 1988

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

The following tasks were performed this past year:

- 1) The demonstration that the uniformity of the space particles is better than the most uniform of those made on earth. The coefficients of variation of the 5-30 μm space particles were 1.0-1.4%; those of the best 10-100 μm particles made on earth were 2.0-2.5%, with others ranging up to 5%.
- 2) The acceptance of two 30 μm space latexes by the National Bureau of Standards as Standard Reference Materials (earlier, the 10 μm space particles were accepted as a Standard Reference Material and offered for sale; about 35% of the samples have been sold) and the claim by the National Bureau of Standards that the space particles are more perfect spheres than those made on earth.
- 3) The development of polymerization recipes in preparation for flight that give on earth monodisperse polystyrene particles as large as 100 μm in size with tolerable levels of coagulum.
- 4) The discovery that some seeded emulsion polymerization give uniform nonspherical particle (instead of spheres) as a result of the shrinkage of the monomer-swollen crosslinked spherical network upon heating and the solidification of new domains by polymerization.
- 5) The development of systematic methods to produce uniform non-spherical particles (e.g., ellipsoidal and egg-like singlets, asymmetric and symmetric doublets, and ice cream cone-like and popcorn-like multiplets).
- 6) The systematic determination of the effects of polymerization parameters on the phase separation; the degree of phase separation increased with increasing degree of crosslinking of the seed particles, monomer/polymer swelling ratio, polymerization temperature, and seed particle size; it decreased with increasing divinylbenzene concentration in the swelling monomer.
- 7) The development of a thermodynamic analysis of the swelling and polymerization of latex particles in seeded emulsion polymerization taking into account the elastic-retractile force of the crosslinked network that causes the network to shrink upon heating, the polymer/water interfacial tension force that restricts the swelling of the particles, and the monomer-polymer mixing force that occurs upon swelling of the particles with monomer.

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4. BIOTECHNOLOGY

Cell Partition in Two Polymer Aqueous Phases

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Dr. James Van Alstine, USRA
Dr. J. Milton Harris, UAH

When aqueous solutions of two different polymers are mixed above certain concentration they frequently form immiscible, liquid, two-phase solutions. Each of these phases usually consists of more than 90 percent water and can be buffered and made isotonic by the addition of low molecular weight species. If a cell or particle suspension is added to such a system in 1-g, then shaken, the system demixes rapidly and cells are usually found to have partitioned unequally between one of the phases and the interface. This preferential partition behavior can be used as the basis of a separation procedure for differing cell populations since partition in these systems is determined directly by cell membrane properties. Such systems are being employed in many countries to carry out biotechnical separations and continuous bioconversion extractions.

By manipulating the composition of the phase systems, separation on the basis of a variety of molecular and surface properties have been achieved, including membrane hydrophobic properties, cell surface charge and membrane antigenicity. When the results of these separations are compared with predictions based on thermodynamic measurements made on single cells in the systems, it is found the separation efficiency is orders of magnitude lower than the thermodynamic limit. This may be due in part to cell sedimentation but other factors are undoubtedly also responsible for this discrepancy. Displacement of cells from their location of lowest equilibrium free energy may be due to the chaotic hydrodynamic environment in which the cells are imbedded during convection-driven phase demixing. To test this idea we are aiming at performing cell separations in microgravity where demixing occurs in the absence of convection, creating a more quiescent hydrodynamic environment. In order to carry out such experiments information regarding the determinants of demixing rates and the disposition of demixed phases in the absence of buoyancy effects is required. Studies conducted onboard KC-135 aircraft during parabolic maneuvers and by Senator E.J. Garn onboard Shuttle flight STS-51D have indicated that in low-g aqueous polymer two-phase emulsions demix by a slow coalescence process. Very low fluid shear is present, suggesting that low-g partition may be able to resolve cell subpopulations unobtainable, by any method, on Earth.

In low-g, phase emulsions demix to yield one phase floating like an egg yolk, surrounded by the phase which preferentially wets the container wall. Current research is aimed at controlling the rate of demixing and final disposition of the phases via both passive means (e.g., altered chamber geometry or polymeric wall coatings with different wetting properties) and active means (electrophoresis of the phase whose interfaces exhibit zeta potentials). In addition, variables such as interfacial tension, phase volume ratios and phase viscosity, are being studied to better understand their influence on demixing of the phases on both low-g and 1-g. Many of these variables will be investigated in another passive demixing Phase Partitioning Experiment (PPE) to be flown on STS-26. The demixing processes under study are relevant to a variety of demixing phenomena in materials processing.

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Protein Crystal Growth in a Microgravity Environment

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NAS8-36611 (NASA Contact: John Price, MSFC)
January 1987 - January 1988

The long range objective for this research task is to develop systematic and reliable techniques and hardware for growing protein crystals in space. Studies will be performed to evaluate the potential for enhanced protein crystal growth under microgravity conditions. Fundamental studies of protein crystal growth, both on the ground and in space will be performed in order to identify the major parameters that affect protein crystal growth.

This research program involves a multidisciplinary effort to produce protein crystals in space of sufficient quality and size to permit molecular structural characterization by X-ray crystallography, while simultaneously providing basic ground-based experimental and theoretical supporting research to develop a better understanding of protein crystal growth and to determine if gravity plays a limiting role in the growth process. Beginning with the Apollo program and extending into the Spacelab program, it has been demonstrated that the microgravity environment can provide stable growth conditions than can result in crystals with improved homogeneity and fewer defects. In this program, a variety of proteins will be crystallized on space shuttle flights over a three-year period. Optimum techniques for reliably growing protein crystals under microgravity conditions will be developed. Initially, emphasis will be placed on vapor-diffusion and dialysis techniques, since they can be used with microliter quantities of material, and are two of the most widely used techniques for ground-based growth of protein crystals. Long-range plans include development of new methods for growing protein crystals, based upon the experimental and theoretical studies performed as part of this research program.

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Pituitary Growth Hormone Molecules

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NAS9-17416 (NASA Contact: D.R. Morrison, JSC)
December 1, 1984 - December 1, 1988

The objectives of the research are: (1) development, validation and establishment of sensitive bioassays for rat and human GH; (2) isolation of GH cell subpopulations and subcellular GH containing particles; and (3) isolation of GH variants from the mammalian pituitary which have high biological activities.

In an effort to accomplish the objectives, we are trying to develop a number of new bioassays for GH. These attempts are based on recent literature which report GH directed effects that we believe can be usefully applied to this goal.

- a. CH on bone cells - We are developomg ways to isolate resting cells from the proximal zone to the rat tibial epiphyseal cartilage plate. Current literature suppests that these undifferentiated cells are responsive to GH. Consequencws of stimulation include 1) cell proliferation, 2) clonal expansion via IGF-1 amplification and 3) differentiation. We are therefore testing GH effects on ³HTdR incorporation; release of collagen from single cells; relase of IGF-1 from single cells; and induction of IGF-1 mRNA synthesis. These latter procedures are being done by cell biotting(see publication list) and Western blotting.
- b. GH on liver cells. - We are probing GH effects on IGF-1 expression using the cell blot technique.
- c. 3T3 fibrobiasts. - We are probing Gh effects on IGF-1 expression via Southern blotting.
- d. Macrophages. We are measuring production of oxygen metabolities (singlet oxygen, superoxide radicals) spectrophotometrically. These metobolites are produced when are exposed to GH.

The primary reason for developing these assays is to be able to test samples from both rat and human pituitary tissue generated under objectives 2 and 3 for the purpose of testing their B/I activity ratios.

- e. Standard assays. The immunological (I) assay used for both human and rat GH will be that of enzyme immunoassay (see publication list). The biological (b) test that we have used in past research, viz. the tibial line assay, will serve as our standard for comparisions of the assays described above. The expense and relative insensitivity of the tibial line assay require development of these other bioassays in order to make steady progress.

GH-containing cells present in enzymatically dissociated rat anterior pituitary gland suspensions as well as GH-containing subcellular particles prepared from human postmortem pituitary tissue will be separated by continuous flow electrophoresis on the

McDonnell Douglas instrument. This device is currently (4/88 being installed at Penn State under the auspices of our NASA-sponsored Center for Cell Research (CCDS Program). Purity of GH cells in fractions will be established by flow cytometric immunofluoresce. The B/I activities of GH released from the cells after culture, or contained in subcellular particles, will be done by the assays described under objectives #1.

The aim of these experiments is to isolate, from the rat and human pituitary, a form of the GH molecule which is enriched in biological activity but poor in immunological activity. This is being attempted by HPLC (size exclusion, ion exchange). Antisera to candidate molecules are being generated. A high titer preparation will probably be used for further immunoaffinity purification techniques.

The activity of the pituitary GH "system" is suppressed in microgravity. This system coordinated, in as yet poorly understood ways, activities of bone, muscle and the immune system. Our research is aimed not only at the isolation of GH molecules(s) with high biological activity, but at their location in subcellular particles and in their cells of origin.

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Todd, P., Hymer, W. C., Morrison, D. R., Goolsby, C. L., Hatfield, J. M., Kunze, M. E., and Motter, K., "Cell Bioprocessing in Space: Applications of Analytical Cytology," in *Proceedings of Ninth Annual Meeting IUPS*, Commission on Gravitational Physiology Czechoslovakia, September 28 - October 1987. (in press)

Continuous Flow Electrophoresis System (CFES)

NASA Marshall Space Flight Center
Dr. Robert S. Snyder
Teresa Y. Miller
Percy H. Rhodes
In-house

The objectives of the Biophysics Branch of the Marshall Space Flight Center to use the Continuous Flow Electrophoresis System (CFES) developed and built by the McDonnell Douglas Astronautics Company (MDAC) for use in the mid-deck of the Space Shuttle are: (1) to use model sample materials at a high concentration to evaluate the continuous flow electrophoresis process in the MDAC CFES instrument and compare its separation resolution and sample throughput with related devices on Earth; and (2) to expand our basic knowledge of the limitations imposed by fluid flows and particle concentration effects on the electrophoresis process by careful design and evaluation of the space experiment. Under terms of the Joint Endeavor Agreement (JEA), NASA is provided an opportunity to process samples in CFES on the Shuttle Shuttle. All experiment objectives and operational parameters, such as applied field, sample residence time in the field, and buffer composition have to accommodate the MDAC capabilities and NASA flight constraints. These restrictions, however, are not significant and a series of successful experiments has demonstrated the utility of CFES.

Future experiments will emphasize sample interactions of the type that can only be investigated in a free flowing system in the absence of buoyancy-induced thermal convection and sedimentation. The electrohydrodynamic distortion observed on STS-6 and STS-7 has now been modeled analytically and experiments that separately consider electrical conductivity and dielectric constant discontinuities across the sample/buffer interface must be done. The model sample material will be selected to meet MDAC and NASA priorities as well as the established CFES operating parameters.

Initial Blood Storage Experiment

Center for Blood Research
Dr. Douglas Surgenor
NAS9-17222

The objectives of the research are: (1) to investigate the effects of microgravity on the formed elements of the blood, (2) to evaluate the fundamental cell physiology of erythrocytes, platelets and leukocytes during storage at microgravity in three different polymer/plasticizer formulations, (3) to improve our understanding of basic formed element physiology, and (4) to contribute to improved survival and efficacy of formed elements for transfusion.

IBSE was planned and conducted as a carefully controlled comparison between identical sets of human blood cells which orbited the earth for the duration of the shuttle mission, and which were held on the ground. Both sets of blood cells were kept throughout the experiment under specific conditions of preservative medium, temperature and air flow designed to foster optimum survival for each type of cell.

The IBSE experimental protocol required that all experimental measurements be carried out on coded samples so that the investigators would be blinded as to the conditions of gravitational force to which the samples being studied has been exposed. More than 1500 measurements were made on the samples provided from the experiment.

Some 1500 pieces of data were obtained from the postflight analyses of the blood cell samples from the experiment. The first statistical report was made available to the investigator team on January 15, 1987. This provided analysis of variance findings comparing the main data sets: specific measurements analyzed (a) by gravitational status (μ g vs. 1-g), (b) by plasticizer/polymer composition of the blood bag, without respect to gravitational status, and (c) for interactions between (a) and (b). Subsequent analyses were made to test for suspected effects of clustering; for effects attributable to the positions of the sample bags within the experimental hardware during the experiment; and for effects of gravitational status (μ g vs. 1-g) in single plasticizer/polymer bags.

Laboratory efforts included continued development and documentation of findings validating the so called compression method for platelet preservation. This technology not only made possible the important platelet findings obtained at microgravity, but also holds considerable promise for improving platelet therapy for patients on the ground. Another set of studies were devoted to ground based experiments on platelet storage at 2-g vs. 1-g. These represent a logical extension of the flight experiments. More important, they strengthen the microgravity platelet findings by revealing that 2xg storage of platelets is inferior to 1xg storage. Still other studies were directed at filling in details desired following the process of discovery of the main experiment findings.

Publications

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Kidney Cell Electrophoresis in Microgravity

Pennsylvania State University

Dr. Paul Todd

NAS9-17431 (NASA Contact: D.R. Morrison, JSC)

May 1, 1985 - August 21, 1988

The objectives of this research task are: (1) provide ground-based cell electrophoresis technology and cell culture support for electrophoretic purifications of cultured human embryonic kidney cells in microgravity; (2) develop flow cytometric methods for analyzing and sorting of human embryonic kidney cells; and (3) perform biophysical analyses of purified cultured human embryonic kidney cells returned from space flight.

To accomplish the first objective, cultured human embryonic kidney cells, in early passage will be subjected to continuous flow electrophoresis. The effects of content of divalent cations, antibiotics, and neutral additives will be studied in low conductivity electrophoretic separation experiments. Cells from various lots will be examined and compared with respect to their ability to multiply, to produce plasminogen activators, to differentiate morphologically, and to retain differentiated function in vitro.

In response to this objective it was found that: (1) kidney cells migrate in continuous flow electrophoresis according to their electrophoretic mobility as determined by analytical electrophoresis (laser grating anemometry) before and after separation, (2) calcium ions at moderately high (20 mM) concentrations can neutralize the negative charge in kidney cells, (3) Ficoll, which is neutral, increases electrophoretic mobility, and (4) one cell lot, designated HEK-1593, has a high plating efficiency, high growth rate, high plasminogen activator production, and relatively narrow electrophoretic mobility distribution.

To accomplish objective two, a laser flow cytometer will be used to quantify cells that produce plasminogen activator on a one-by-one basis by staining them with fluorogenic amide substrates that release 4-methoxy-2-naphthylamine which can be precipitated intracellularly by the addition of 5-nitrosalicylaldehyde. The percent plasminogen activator producing cells will be quantified in early-passage cultures and purified cell suspensions by this method. The light scattering signature in flow cytometry of electrophoretically purified cell subpopulations will be determined in order to establish whether or not there exist basic morphological criteria that may also be used as a means of cell purification by viable cell sorting. A flow cytometer will also be used to examine the relationship between cell surface charge and cell function by staining simultaneously with fluorescent poly-L-lysine and plasminogen activator fluorogenic substrates.

In response to this objective, it was found that: (1) about 50% of a population of kidney cells fluoresced brightly in the EPICS V cytometer when stained for intracellular plasminogen activator, (2) about 50% of this fluorescence intensity was lost after cells had spent 3 days in "production medium," which stimulated plasminogen activator secretion, (3) cells from different electrophoretic sub populations have different light-scattering signatures as determined by flow cytometry, and (4) in model cell types, polysine staining of the cell surface is a dynamic process, but staining can be shown to be proportional to sialic acid on the cell surface when cells are fixed.

To accomplish objective three, post-flight biophysical analyses of cells purified in space, the above mentioned methods will be used. The percent cells producing each of the three different types of plasminogen activator will be identified in each fraction using specific markers in flow cytometry. Cell life cycle analysis using DNA staining and flow cytometry of cells from each purified fraction will be used to reveal which fractions retain reproductive potential. The electrophoretic mobility of cells from each purified fraction will be confirmed on the basis of analytical cell electrophoresis using an automated electrokinetic analyzer.

In response to this objective, cell life cycle analysis was performed on the basis of propidium iodide staining of nuclear DNA followed by flow cytometry with no unusual findings, and three electrophoretic fractions were compared with respect to reproductive potential with no obvious differences observed among them. The electrophoretic mobilities of kidney cell populations separated by continuous flow electrophoresis on the ground were measured and found to be consistent with their migration distances. No space experiments could be performed during the project period.

Emphasis during the last calendar year has been placed on the development of biophysical analytical tools for the pre-flight and post-flight analysis of kidney cell populations.

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5. GLASSES AND CERAMICS

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Containerless Processing of Glass Forming Melts in Space: Critical Cooling Rates and Melt Homogenization

University of Missouri, Rolla
Dr. D. E. Day
NAS8-34758 (NASA Contact: V. Fogle, MSFC)
February 1982 - January 1988

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 the cooling rate (R) used in micro-g will serve as a quantitative measure of the degree to which glass formation is enhanced, or heterogeneous nucleation is suppressed. Ternary calcia-gallia-silica compositions possessing different critical cooling rates will be heated, melted, and quenched in an acoustic levitator/furnace for the MEA/A-3 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 and a soda-lime-silica 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

Chakraborty, I. N., Rutz, H., and Day, D. E., "Glass Formation, Properties and Structure of Y_2O_3 - Al_2O_3 - B_2O_3 System," J. Non-cryst. Solids **81**, 173 (1986).

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Fluoride Glasses: Crystallization and Bubbles in Low Gravity

Rensselaer Polytechnic Institute
Dr. Robert H. Doremus
Contract No. 955870
January 1, 1987 - June 30, 1987

To study the influences of surface composition and structure on properties of zirconium fluoride glasses, vaporization, crystallization and chemical reaction.

Publications

Mathew, J., and Doremus, R.H., "Outgassing of ZrF₄-Based Glasses," J. Am. Ceram. Soc. 70, C-86 (1987).

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Physical Phenomena in Containerless Glass Processing

Clarkson University
Dr. R. S. Subramanian
Dr. Robert Cole
NAS8-32944 (NASA Contact: V. Fogle, MFSC)
December 1977 - December 1989

The objective of this investigation is to develop an understanding of fluid motion and bubble and droplet motion and interactions when drops containing bubbles are subjected to stimuli such as surface tension gradients, rotation, expansion, contraction and oscillation.

At this time, all of the research is ground-based. Flight experiments in an acoustic levitator are planned for execution in the future.

The experiments on ground include studies of bubble and drop migrations in a temperature gradient, motion of droplets within compound drops, and motion of bubbles and drops in rotating liquids. Also, appropriate theoretical descriptions of the experiments are under way.

Publications

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6. COMBUSTION SCIENCES

Scientific Support for an Orbiter Middeck Experiment on Solid Surface Combustion

University of Kentucky
Professor Robert A. Altenkirch
Dr. M. Vedha-Nayagam
NAS3-23901 (NASA Contact: S. Olson, LeRC)
December 19, 1984 - December 20, 1992

The overall objectives of the experiment are to: (1) determine the mechanism of gas-phase flame spread over solid fuel surfaces in the absence of any buoyancy induced or externally imposed gas-phase flow; and (2) improve the fire safety aspects of space travel.

The spread of 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 flow, 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 has been designed for the Middeck of the Space Shuttle to aid in understanding the process of flame spreading in the absence of a buoyancy-driven flow. A chamber approximately 0.035 m^3 in volume is to contain either a thin sample of a cellulosic material or a thick sample of polymethylmethacrylate and an oxidizing environment of O_2 and N_2 . Samples will be ignited at one end, and the ensuing flame spread process 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 has been constructed at NASA's Lewis Research Center and tested in the Drop Tower facilities. Current testing is being carried out at the University of Kentucky. Methods of data reduction are being developed as are theoretical analyses of reduced-gravity flame spread problem.

Results to date show that measured spread rates over tin cellulosic fuels are less at microgravity than for downward spread in normal gravity. Theoretically predicted microgravity spread rates not accounting for radiation heat loss from the flames are for infinitely-fast, gas-phase chemistry generally a factor of approximately five times what is measured. For finite-rate, gas-phase chemistry, the predicted values are approximately three times those measured.

Publications

Altenkirch, R. A., "Combustion Studies in Microgravity," in *Progress in Astronautics and Aeronautics* 108, 225-230 (1986).

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Vedha-Nayagam, M., Saito, K., and Altenkirch, R. A., "Edge Effects in Flame Spreading Down Thermally Thin Solid Fuels," presented at Eastern Section/The Combustion Institute Meeting, San Juan, Puerto Rico, December 1986.

Vedha-Nayagam, M. and Altenkirch, R. A., "Flame Extinction during Downward Flame Spread into an Opposing Forced Flow of Oxidizer," presented at Central States Section/The Combustion Institute Technical Meeting, Argonne, IL, May 1987.

Particle Cloud Combustion Experiment

University of California, San Diego
Dr. A. L. Berlad
NAS3-24639 (NASA Contact: Howard Ross, LeRC)
September 1, 1985 - August 31, 1988

The principal objectives of this microgravity experimental program are to obtain flame propagation rate and flame extinction limit data for several important premixed, quiescent particle cloud combustion systems under near zero-gravity conditions. The data resulting from these experiments are needed for utilization with currently available flame propagation and extinction theory. These data are also expected to provide new standards for the evaluation of fire hazards in particle suspensions in both Earth-based and space-based applications. Both terrestrial and space-based fire safety criteria requires the identification of the critical concentrations of particulate fuels and inerts at the flame extinction conditions. The Particle Cloud Combustion Experiment (PCCE) utilizes an array of flame tubes. Within each flame tube a uniform quiescent cloud of particles (of selected stoichiometry) is to be suspended in near zero gravity. Flame propagation and extinction characteristics are then observed. Particulates under study include the fuels lycopodium, cellulose and coal, as well as a number of inert particulates. Ground-based supportive studies include the use of the LeRC drop tower and Learjet research facilities as well as the laboratories at the University of California, San Diego.

Preparation of flight experiment designs is supported by LeRC and UCSD experimental studies of particle mixing processes, optical transmissivities of particle cloud distributions, kinetics of particle-particle and particle-wall interactions, and suppression of agglomerative growth of nonmonomeric particle clusters. Pyrolysis-vaporization kinetics of particulates of interest are made to provide thermokinetic data needed in application of particle cloud flame theory to anticipated flight studies. In preparation for interpretation of microgravity combustion experiments in both airplanes and future STS flights, theoretical studies emphasize comprehensive flame propagation and extinction relations for both freely propagating and stabilized particle cloud flames.

Publications

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Presentations

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Scientific Support for a Space Shuttle Droplet Burning Experiment

Princeton University
Professor F. A. Williams
Professor F. L. Dryer
NAS3-24640 (NASA Contact: John Haggard, LeRC)
November 30, 1987 - November 30, 1988

The general objective of this program is to ascertain how best to make use of reduced gravity to pursue scientific investigations of droplet combustion. The specific objective is to provide scientific support during development of a droplet burning experiments that are to be carried out in the NASA LeRC drop towers and in the Space Shuttle. The planned experiments are intended to improve our understanding of droplet combustion, especially in relationship to time-dependent and extinction phenomena.

The research tasks include theoretical modeling of droplet burning, ground-based experimentation on droplet burning, support to NASA in providing advice on hardware aspects of the flight experiment and analysis of data to be obtained in the experiment. The modeling addresses questions related to burning rates, to soot behavior, to disruption, and to ignition and extinction phenomena. Ground-based experiments are focused on droplet ignition and on impulses imparted to droplets by ignition sparks; spark designs for minimum impulse are addressed. In addition, drop tower experiments are addressing burning rates and mechanisms of soot production and of droplet disruption during combustion. The support activities include advisory participation in planning and in implementation of the flight experiment on droplet combustion.

Publications

Shaw, B. D., Dryer, F. L., Williams, F. A., and Gat, N., "Interactions Between Gaseous Electrical Discharges and Single Liquid Droplets," Comb. & Flame, 1988 (in press).

Shaw, B. D., Dryer, F. L., Williams, F. A., and Haggard, J. B., "Sooting and Disruption in Spherically Symmetrical Combustion of Decane Droplets in Air," Acta Astron., 1988 (in press).

C. FUNDAMENTAL PHENOMENA

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

University of Oregon
Dr. Russell J. Donnelly
Dr. Charles E. Swanson
(NASA Contact: D. D. Elleman, JPL)

The objective of this research is to assess a method of measuring the circulation length of helium II in space.

The superfluid properties of liquid helium are associated with the properties of a complex order parameter ψ , which has amplitude and phase. The distances over which the amplitude and phase are strongly correlated are referred to as correlation or coherence lengths ξ and these lengths are thought to diverge as the temperature T_λ approaches the lambda transition T_λ of liquid helium. The coherence length is fundamental to many properties of liquid helium: (1) it enters the renormalization group calculations of the specific heat of liquid helium; and (2) it determines the scale needed to build He II Josephson junctions, the scale of fluctuations near the lambda transition (which are studied by light scattering), the size of quantized vortex cores, the distance over which superfluidity vanishes near a wall, and the scale of "size effects" which shift the lambda transition in confined geometries.

At the present time the coherence length is known in order of magnitude, but not with precision. The coherence length coefficient ξ_0 is seen to vary by a factor of ~ 4 among different experiments. One reason is the influence of gravity which sets a limit on how close to T_λ one can carry out a precise experiment in a container of given height. Indeed much of the same technology to be used for the specific heat experiment in space is directly useful in coherence length measurement for the JPL space cryostat and the Lipa subnanodegree thermometer.

Tasks will include bibliographic research, consulting with experts in the field, numerical simulation of the experiment, and preliminary experiments.

Publications

Homsy, G. M. and Donnelly, R. J., "Fluid Mechanics and Fluid Physics in a Low-Gravity Environment," in *Opportunities for Academic Research in a Low Gravity Environment*, Volume 108 (M. Summerfield, ed.), AIAA, 1987.

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Cryogenic Equivalence Principle Experiment

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

The objective of this research 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.

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 a 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., "Almost Exactly Zero: The Equivalence Principle," Near Zero, 1986 (in press).

Lambda Point Experiment

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JPL 957448

The objective of the research is to perform a new test of the theory of cooperative or second order phase transitions by making use of the microgravity conditions on the Shuttle, the JPL SL-2 Helium Dewar, and the Stanford High Resolution Thermometer.

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.

The experiment plan calls for the observation of the temperature dependence of the heat capacity of helium very near the lambda point at 2.17K with a resolution of a few times 10^{-10} deg. This experiment is being conducted in conjunction with JPL, who provides the cryogenic facility. By performing the measurements in space the resolution of the experiment can be improved by a factor of about 100 due to the reduction of the hydrostatic pressure head. The measurements will lead to a much stronger confrontation between theory and experiment than has been possible on earth, and perhaps as severe a test as is feasible with current technology. If the experiment does cast doubt on the theory, it could also have a significant impact on other areas of physics. For example, theories of quark confinement in high energy physics draw heavily on the same type of analysis as used for phase transitions. A major revision of the theory could thus have quite a large ripple effect in theoretical physics.

Publications

Lipa, J. A., Chui, T.C.P., and Marek, D., "The Lambda Point Experiment in Microgravity," in *Aerospace Century 21*, Volume 64, Advances in Astronautical Sciences (G.W. Morgenthaler, et al., eds.), 1987, p. 1245.

Lipa, J. A., Li, Q., Chui, T.C.P., and Marek, D., "Testing the Renormalization Group Theory of Cooperative Transitions at the Lambda Point of Helium," in *Proceedings of the 3rd University of California Conference on Statistical Mechanics*, 1988, Nuclear Physics B (in press).

Critical Transport Properties in Liquid Helium under Low Gravity

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The objective of this research task is to measure the shear viscosity of ζ in helium near the liquid-vapor critical point T_c and near the normal-superfluid transition $T_\lambda(x)$ in particular in ^3He - ^4He mixtures near the critical point ($X_t = 0.67$, $T_t = 0.87\text{K}$) where X and T are the ^3He concentration and T the temperature. As the critical point is approached, ζ diverges, but gravity produces a rounding of this divergence. In the absence of gravity, this rounding will be suppressed and the predicted divergence should be observed until frequency effects (from the measuring oscillator) should be observed that give another rounding closer to T_c . Near the tricritical point in mixtures, a similar behavior can be expected but so far there are no measurements and our program is to conduct a systematic study of ζ near T_t . For this purpose, the viscosity of mixtures near $T_\lambda(x)$ (that terminates at the tricritical point) needs to be studied in mixtures with various concentrations $0 < X < X_t$. No such systematic study has ever been made, and it is necessary to understand the trend of the singular behavior, crossing over from the "critical" to the "tricritical" regime. We are in the process of doing this and we are moving systematically towards measurements at lower temperatures.

When ζ measurements near T_t are completed, we will be able to decide whether such measurements are more interesting than those near the liquid-vapor transition, for adaption on cryostats incorporated for a shuttle space slight or on a space platform. And most important, are they feasible given the experimental constraints and requirements (long equilibrium times, temperature stability needs, etc.)? Are the experiments in the end good candidates for a flight under low gravity conditions?

In the last twelve months, we have measured with our torsional oscillator the product $(\delta\zeta)$ where δ is the mass density (at temperatures above the superfluid transition, δ is the total density, while in the superfluid phase it is the portion of the "normal" fluid). Our measurements were made with mixtures with $X = 0.1, 0.2, 0.3, 0.4, 0.5$ and 0.65 . We are in the process of taking more and hopefully better data with $X=0$ and 0.03 . We are slowed down by long equilibrium times, and by the many data points to be taken close to $T_\lambda(x)$, which is very time consuming. A considerable amount of time was spent finding and repairing some electronic problems in our data taking system and in developing new software for the recently acquired MacII computer superseding an old microprocessor.

The experiments is manned by two graduate students: Suwen Wang has started writing his thesis which could be finished in October. Carl Howard is supervising the data acquisition and is developing the software. We are looking for a post-doctoral associate to replace Wang.

We want to complete the measurements with $X=0$ and 0.03 down to 1.2K . Then we need to test a density cell to measure δ in mixtures between 1.2 and 4K . Continuation of the $(\delta\zeta)$ data with those of δ will give $\delta(T)$ for the various mixtures. Then we plan to write a paper on the data, constructing a new oscillator and modifying the cryostat for extending measurements of $(\delta\zeta)$ to 0.8K for mixtures near the tricritical point.

Publications

Agosta, C., Wang, S., and Meyer, H., "The Shear Viscosity of ^3He Near the Liquid Vapor Critical Point," J. Low Temp. Phys. **67**, 237 (1987).

Wang, S. and Meyer, H., "The Shear Viscosity of ^3He - ^4He Mixtures near the Liquid-Vapor Critical Point," J. Low Temp. Phys. **69**, 377 (1987).

Meyer, H., "The Transport Properties of ^3He - ^4He Mixtures near the Lambda Line - a Short Review and Outlook," J. Low Temp. Phys. **70**, 219 (1988).

Presentations

Meyer, H., "The Transport Properties of ^3He - ^4He Mixtures near the Lambda Line," American Physical Society Meeting, March 1988, New Orleans, Invited Paper.

Wang, S., Howald, C., and Meyer, H., "Shear Viscosity of ^3He - ^4He Mixtures near the Superfluid Transition," Bull. Am. Phys. Soc. **33**, 493 (1988), contributed paper.

Precise Viscosity Measurements Very Close to Critical Points

National Bureau of Standards

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Professor R. W. Gammon, University of Maryland

C-86129D (NASA Contact: Dr. R.W. Wilkinson, LeRC)

January 1, 1987 - January 1988

The objective of the research is to measure the viscosity of a pure fluid near its liquid-vapor critical point. The space experiment will be the fourth of a series of tasks which are: (1) theoretical studies, (2) critical viscosity measurements of binary liquid mixtures, (3) critical viscosity measurements of pure fluids in 1-g, and (4) measurements on pure fluids in low gravity. We have developed a torsion oscillator viscometer and used it to study four binary liquid mixtures near their consolute points.

Near the critical temperature T_c the viscosity η diverges as:

$$\eta \sim (T - T_c)^{-\gamma}$$

Analyses of all four binary liquid data sets show that the viscosity exponent is $0.0404 < \gamma < 0.0444$ significantly higher than the theoretically predicted value of 0.032. These results are being submitted for publication.

The low frequency, low shear viscometer is described in the first publication below. The viscometer has also been tested on a near-critical microemulsion and on liquid metal samples cooled to as low as -80 C. We are now preparing for critical viscosity measurements on two pure fluids (CO_2 and xenon) and have built a new thermostat and a disc-shaped cell designed to contain the sample at its critical pressure. The cell's internal height and radius are 1 and 15 mm respectively. These dimensions were chosen to minimize the undesirable effects caused by shear, gravity, thermal diffusivity, and loading inaccuracy near the critical point. We are currently addressing the issues of vibration isolation and automated temperature control.

Publications

Moldover, M. R., "Opportunities for Low-Gravity Experiments in Critical Phenomena," in *Opportunities for Academic Research in a Low-Gravity Environment*, Volume 108 (G.A. Hazelrigg and J.M. Reynolds, eds.), AIAA, 1987, pp. 57-79.

Berg, R. F. and Moldover, M. R., "Viscometer for Low-Frequency Low-Shear Rate Measurements," *Rev. Sci. Instrum.* **57**, 1667 (1986).

Berg, R. F., Moldover, M. R., Rabinovich, S., and Voronel, A., "Viscosity and Density of Two Alkali Metal Mixtures," *J. Phys. F: Metals Phys.* **17**, 1861 (1987).

Berg, R. F., Moldover, M. R., and Huang, J. S., "Quantitative Characterization of the Viscosity of a Microemulsion," *J. Chem. Phys.* **87**, 321 (1987).

D. FACILITIES

Microgravity Materials Science Laboratory

NASA Lewis Research Laboratory
Thomas K. Glasgow
In-House
February 1984 - Continuing Task

The Microgravity Materials Science Laboratory (MMSL) was created to serve as focal point for ground-based experimentation in preparation for or conjunction with flight experiments. It is open to users from industry, academia, and government. The MMSL addresses a broad range of materials including metals, alloys, salts, glasses, ceramics, and polymers.

The laboratory is equipped with a wide variety of apparatus for characterizing the interaction of liquids of gasses with gravity during materials processing. Included are transparent furnaces for observation of salt solidification or physical vapor transport, an electromagnetic levitator for containerless melting of metals) a high pressure acoustic levitator is under development for glasses and ceramics), a bulk undercooling furnace, a magnetically damped directional solidification furnace, a transparent isothermal dendrite growth apparatus, a transparent model directional solidification furnace, a functional duplicate high temperature acoustic levitator, glass melting and characterization equipment, polymers preparation and characterization equipment, a metallography laboratory, and computational facilities for process modelling. Most of the equipment operates under computer control. New equipment is acquired or built in response to specific requests. The staff includes engineers and technicians drawn divers disciplines including mechanical, nuclear, metallurgical, and welding engineering, physics, and chemistry.

The most fruitful uses of the MMSL have involved interaction of researchers from industry with members of the Lewis Materials Divisions. Experiments underway by lab users include directional solidification of metal and salts, vapors growth of anisotropic crystals, phase separation in glasses and polymers, growth of model dendritic materials, bulk undercooling of alloys, and computational modeling. The MMSL is also home to experiments to define telescience requirements, to define an advance laser light scattering apparatus, and to examine advance furnace technology. Potential users of the MMSL should request the brochure "The Microgravity Materials Science Laboratory" for a more complete description and application for use.

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Ground-Base Research Facilities

NASA Lewis Research Center
Jack Lekan
In-House
January 1, 1987 - January 1, 1988

Several ground-base low gravity facilities are available for use at the NASA Lewis Research Center (LeRC). These include the 2.2-Second Drop Tower, the 5-Second Zero-Gravity Facility, and Learjet which provides up to 20 seconds of low-gravity test time. Ground-base facilities are used to obtain the baseline normal gravity data and valuable reduced-gravity data to advance the scientific understanding and concepts and ultimately to test prototypical Shuttle flight hardware.

Due to the delay of Shuttle flights and with the potential longer range limitation on available STS manifest opportunities for experiments, there has been continuing increased interest in expanding the microgravity scientific data return from the ground-based low-gravity facilities at LeRC. While space experiment hardware technology development continues to be an important activity in these facilities, each experiment program has been reexamined to determine how additional ground-based microgravity data might be utilized to enhance the success and value of limited space-based experiment time. Expanded ground-based test programs have been defined and are being initiated for several experimental programs to obtain unique microgravity data which can be used to refine current analytical models of the processes/phenomena under investigation. These data and improved models will then be used to refine the final test matrices and experimental techniques used in the future space-based experiments.

The 2.2-Second Drop Tower is utilized extensively by NASA research scientists and experiment designers as well as principal investigators from the academic community who are conducting research in areas of combustion and fluid physics. The 2.2-Second Drop Tower provides an environment in which the gravitational acceleration acting on an experiment is less than $1^{-5}g$. The experiment falls 30.5 meters unguided in the tower. A drag shield falls along with the experiment, but unattached from the experiment, to provide shielding from the surrounding atmosphere to reduce air drag. The rapid turnaround time (up to 8 drops per day) and low cost make this facility particularly attractive to research scientists.

This facility supported ten programs during 1987 as 229 research drops were performed. Nearly as many normal gravity tests were also executed. An ambitious experiment buildup program was also accomplished as five new experimental drop packages were fabricated and became operational.

The Zero-Gravity Facility with its 145 meter free-fall distance and accompanying vacuum system that permits an experiment to free-fall unguided in an atmosphere with a residual pressure of less than 200 microns of Hg represents a significant expansion in research capabilities and experiment sophistication when compared to the Drop Tower. Low-gravity levels of less than $10^{-5}g$'s are obtained for a time period of 5.18 seconds. Due to the complexity of facility operations only one test is generally performed per day. Seven research programs were supported by this facility in 1987 as 91 experiment drops were performed.

The Learjet Model 25 aircraft provides test intervals of up to 20 seconds at gravitational levels of 10^{-2} by flying parabolic trajectories. Up to six trajectories can be

performed per flight. While the effective gravity levels obtained during these maneuvers is not as low as those of the drop towers, the Learjet does allow investigators to operate, and reconfigure their experiments. During 1987 the Learjet made 22 flights and a total of 74 reduced-gravity trajectories were flown to obtain research data.

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APPENDIX B
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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 1987 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 includes an introductory description of the program, the strategy and overall goal, identification of the organizational structures and people involved, and a description of each task. The report also provides a list of recent publications.</p> <p>The tasks are grouped into six major categories: Electronic Materials; Solidification of Metals, Alloys, and Composites; Fluid Dynamics and Transport Phenomena; Biotechnology; Glasses and Ceramics; and Combustion. Other categories include Experimental Technology, General Studies and Surveys; Foreign Government Affiliations; Industrial Affiliations; and Physics and Chemistry Experiments (PACE). The tasks are divided into ground-based and flight experiments.</p>					
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