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Materials Processing in Space

Program Tasks

Compiled by Elizabeth Pentecost
Space Science Laboratory

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16. ABSTRACT

This report is a compilation of the active research tasks as of the end of the fiscal year 1983 of the Materials Processing in Space Program, NASA-Office of Space and Terrestrial Applications, involving several NASA centers and other organizations. The purpose of this document is to provide an overview of the program scope for managers and scientists in industry, university, and government communities. The report is structured to include an introductory description of the program, its history, strategy and overall goal; identification of the organizational structures and people involved; and a description of each research task, together with a list of recent publications.

The tasks are grouped into four categories: Crystal Growth; Solidification of Metals, Alloys, and Composites; Fluids, Transports, and Chemical Processes; and Ultrahigh Vacuum and Containerless Processing Technologies

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NASA TECHNICAL MEMORANDUM

I. INTRODUCTION

The Materials Processing in Space program is directed toward research in the science and technology of processing materials under conditions of low gravity to provide a detailed examination of the constraints imposed by gravitational forces on Earth. The program is expected to lead, ultimately, to the development of new materials and processes in commercial applications adding to this nation's technological base. The research studies emphasize the selected materials and processes that will best elucidate the limitations due to gravity and demonstrate the enhanced sensitivity of control of processes that may be provided by the weightless environment in space. Primary effort will be devoted to a comprehensive study of the specific areas of research which revealed potential value in the initial investigations of the previous decade. 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 characteristics associated with an orbiting wake shield, minimal knowledge of terrestrial processing methods.

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

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

The current program emphasis on fundamental processing science and technology in selected areas will continue as the Materials Processing in Space program addresses problems of interest to the public and private commercial sectors which can be resolved by recourse to the space environment. During this phase of the program, the development and demonstration of current space technology for materials processing will be transferred, as appropriate, to non-NASA users. In order to assist this process, a Commercial Space Processing

Task Team has been formed to resolve institutional constraints serving as disincentives to cooperative involvement. In addition, this team will serve as a single point of contact for interested parties and represent their interests within NASA.

Emphasis will be placed on the expansion of currently funded activities for ground-based and spaceflight 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 spaceflight investigations by 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.

I. TASKS

1. CRYSTAL GROWTH

TASK NUMBER	PRINCIPAL INVESTIGATOR	SHORT TITLE
CG-001	Broerman	Advance Methods for IR Materials
CG-002	Bourret	Solutal Convection and Its Effects
CG-003	Brown	Analysis of the Float Zone Process
CG-004	Coriell	Solutal Convection during DS
CG-005	Crouch	Semiconductor Materials Growth
CG-006	Foster	The Control of Float Zone Interfaces
CG-007	Gatos	Crystal Growth in Device Quality GaAs
CG-008	Kern	Microgravity Silicon Zoning Investigation
CG-009	Lal	Solution Growth of Crystals
CG-010	Lehoczky	Growth of Solid Solution Crystals
CG-011	Rosenberger	Fluid Dynamics of Crystallization
CG-012	Schnepple	HgI_2 Growth for Nuclear Detectors
CG-013	Verhoeven	Float Zone Experiments in Space
CG-014	Wiedemeier	Fluid Dynamics and Thermodynamics
CG-015	Wiedemeier	Vapor Growth of Alloy-Type Semiconductors
CG-016	Witt	Heat Flow and Segregation
CG-017	Zoutendyk	Vapor Phase of PbSnTe

INTRODUCTION

Melt growth is the most widely used technique for production of high technology, single-crystal materials for semiconductor chips used in large scale integrated circuits for communications and computers. The MPS program emphasis is concentrated on achieving chemical homogeneity, hence, maximum elextrical performance, in HgCdTe and lead-tin-telluride (PbSnTe) semiconductors. These crystals are among the most sensitive and important infrared senors and most difficult to grow materials on Earth. The materials bridge the spectrum of growth conditions. In the case of PbSnTe, one component is less dense than the bulk melt, hence the system is subject to solute instabilities. The HgCdTe, on the other hand, has the opposite problem. One component is more dense than the bulk melt. Therefore, it is subject to solidifying interface-shape instabilties. Low-g experiments will determine how such systems can be grown in the absence of gravity.

Float zone growth is a variation of melt growth in which the material can be melted without the deleterious contact with any container wall. Floating zone techniques are widely used to produce crystals such as doped silicon for semiconductors and solar cells. The MPS program emphasis is on establishing uniform growth conditions in commericially important materials such as indium-doped silicon and CdTe which is a semiconductor with a very high theoretical maximum energy conversion efficiency.

Solution growth is an important alternative to melt growth for materials that are unstable at their melting point because the crystals can be processed at much lower temperatures. The MPS program emphasis is directed toward triglycine sulphate (TGS) a room temperature, infrared detector material and gallium-arsenide (GaAs) one of the most important semiconductors for a wide range of applications from microwave devices, to computers, and solid state lasers.

Vapor growth does not compete favorably with other growth techniques on Earth where large crystals are required because gravity disrupts the vapor transport mechanism; it is a useful process for growing "whiskers" or thin noncrystalline films and for materials that do not lend themselves to other convenient techniques. The absence of gravity opens new possibilities for the growth of large, flat, pure crystals by the vapor technique; therefore, the MPS program includes the investigation of HgI_2 nuclear detector crystals and HgCdTe and copper-indium-antimony (CuInSb) solid solution semiconductor crystals.

CG-001

Advanced Methods for Preparation and Characterization of Infrared-Detector Materials

McDonnell Douglas Research Laboratories
Dr. J. G. Broerman
NAS8-33107 $80K/year
December 1978 - December 1982

The objectives of this research program are to quantitatively establish the characteristics of $Hg_{1-x}Cd_xTe$ as grown only Earth (1-g) as a basis for subsequent evaluation of the material processed in space and to develop experimental, theoretical, and analytical methods required for such evaluation.

A series of differential thermal-arrest (DTA) measurements were performed for $Hg_{1-x}Cd_xTe$ alloy composition x = 0. 0.1, 0.2 0.3, 0.4, 0.6, 0.7, 0.8, 0.9, 1.0. The solidus and liquidus temperatures deduced from the DTA data were used to establish the pseudobinary HgTe-CdTe constitutional phase diagram and the x and interface temperature dependencies of the Cd segregation coefficient, k. Iterative phase-equilibria calculations, based on a regular associated solution (RAS) theory, were performed to establish the solution parameters required to calculate the phase boundaries for the Hg-Cd-Te alloy system.

The DTA measurements were extended to the ternary $(Hg_{1-x}Cd_x)Te_{1-y}$ system for the mercury rich range $0.5 < y < 1$. Liquidus temperatures were obtained for x = 0.1, y = 0.55, 0.6, 0.65, 0.75, 0.85; x = 0.2, y = 0.5, 0.55, 0.6, 0.7, 0.8; x = 0.3, y = 0.55, 0.6, 0.7, 0.8, 0.9; x = 0.4, y = 0.55, 0.75, 0.85. Thus, nearly complete coverage of the mercury rich portions of the ternary phase diagram was obtained.

$Hg_{1-x}Cd_xTe$ crystals (x = 0.2, 0.5, 0.7) were grown by the vertical Bridgman method at growth rates between 0.7 and 5.6 μm/s and characterized for longitudinal and radial homogeneity and by measurements of carrier concentration and Hall mobility. Theoretical models of computer programs specific to $Hg_{1-x}Cd_xTe$ were developed for calculations of charge-carrier concentrations, Hall coefficient, Fermi energy, and conduction electron mobility as function of x, temperature, and ionized-defect and neutral-defect concentrations. A comparision of calculated results with available experimental data indicated that longitudinal optical-phonon and charged and neutral defect scattering are the dominant mobility limiting mechanisms.

CG-002

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

Massachusetts Institute of Technology
Dr. Edith D. Bourret
NSG-7645

This research project is concerned with a theoretical and experimental study of the effects of solutal convection on segregation in binary and pseudo-binary systems with large liquidus-solidus separation (i.e., Ge-Si, $Hg_{1-x}Cd_xTe$, $Pb_xSn_{1-x}Te$). This study is being carried out in collaboration with the Metallurgy Division of the CENG (France) and the Materials Processing Center for MIT. The studies are aimed at advancing the theoretical framework for solidification and at optimizing crystal growth experiments to be conducted in a reduced gravity environment.

Research at Grenoble has centered on growth of Ge-Si in a Bridgman system which is designed so as to provide the planarity of interface throughout the temperature change from the liquidus temperature to the solidus temperature. Research at MIT is concerned with: (1) theoretical studies of directional melting; this work has so far yielded new insights into the first stages of seeded crystal growth of binary compounds; (2) using a one-dimensional model of directional solidification, transients in non-dilute systems have been examined. This model couples heat and mass transfer and includes all the growth parameters for conditions of diffusion-controlled mass transfer (as in a microgravity environment). Growth rate transients as well as segregation transients are predicted; (3) a finite-element method is being developed for simultaneously calculating thermal- and solutal-driven convection, thermal and concentration fields and the melt/solid interface shape in vertical Bridgman growth. This work constitutes an extension of a previous theoretical treatment on temperature-driven convection and; (4) experiments on the melt growth of $Pb_xSn_{1-x}Te$; growth has been carried out successfully in the Czochralski and vertical Bridgman configurations. The relationship between interface shape and radial segregation for different aspect ratios of the melt is being investigated in more detail.

Publications

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Growth.

Bourret, E. D. and Witt, A. F., "A Novel Approach to Czochralski Growth of Volatile Compounds," J. Cryst. Growth (submitted).

Derby, J. J., Bourret, E. D., Witt, A. F., and Brown, R. A., "Dynamics of Bridgman Growth of Pseudo-binary Crystals with Large Liquidus-Solidus Separation," in preparation for J. Crystal Growth.

Adornato, P. M. and Brown, R. A., "Solute-Driven Convection in Vertical Bridgman Growth in a Non-Dilute Binary Crystal," in preparation for J. Crystal Growth.

CG-003

Analysis of the Float Zone Process

Massachusetts Institute of Technology
Professor R. A. Brown
NSG-7645/Supplement 1

This research program is directed toward a fundamental understanding of the interaction of heat, mass, and momentum transfer in the floating zone method for growing single crystals from the melt.

Significant progress has been made on studies of the interaction between heat and mass transfer and melt/solid interface shape in melt growth processes, on the analysis of the fluid mechanics of the floating-zone process, and on the modelling of buoyancy-driven convection in crystal growth from the melt. To do this, new computer-aided methods based on finite-element techniques have been developed for analyzing the interaction between heat and mass transfer and melt/solid interface shape in melt growth processes. These methods are finding wide application in studies of both macroscopic and microscopic problems in melt growth.

The finite element methods have been extended to account for natural convection in the melt and its influence on melt/solid interface shape and solute segregation. A study of a prototype of vertical Bridgman system has been completed and application of the methods to the floating zone process are underway and results have been submitted for presentation. In these systems, mathematical nonuniqueness of the steady state flows and interface shapes complicates the analysis. Newly developed methods have been employed for tracking these multiple solutions and have lead to the first detailed understanding of the evolution between laminar and oscillatory finite-amplitude convection and its dependence on geometry.

Asymptotic methods and finite element analysis have been used to study forced convection caused by crystal and seed rotation in a floating zone system with nearly cylindrical melt volume. The dependence of the flow structure has been mapped as a function of the zone length and on the relative rotation rates of the two rods. Again, multiple flows have been found at low rotation rates and their relative stability has been predicted. The techniques for analyzing the rotation-induced flows are being extended to study the combined effects of rotation and surface-tension-convection flows, a situation that is likely to exist in low-g experiments.

The finite element results are being extended to molten zones that are very deformed by gravity and so model earthbound floating zone experiments. The limits of static stability for these systems has been rigorously analyzed and a full-scale numerical simulation is underway that includes natural and forced convection, as well as the curvatures of the melt/gas and melt/solid interfaces.

Publications

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Harriott, G. M. and Brown, R. A., "Flows in a Differentially Rotated Cylindrical Drop at Low Reynolds Number," submitted to J. Fluid Mech., 1982.

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Chang, C. J. and Brown, R. A., "Effect of Steady Buoyancy-Driven Convection on Melt/Solid Interface Shape and Radial Segregation in Vertical Bridgman Growth," submitted to J. Crystal Growth, 1982.

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Yamaguchi, Y., Chang, C. J., and Brown, R. A., "Axisymmetric Buoyancy-Driven Convection in a Vertical Cylinder Heated from Below," to be submitted to Proc. R. Soc. Lond., 1982.

Chang, C. J. and Brown, R. A., "Multiple Steady Solutions to a Buoyancy-Driven Convection Problem Including a Melt/Solid Phase Boundary," to be submitted to Int. J. Num. Methods Fluids.

Ungar, L. H. and Brown, R. A., "The Effect of Grain Boundaries on the Morphological Stability of Crystals Grown from the Melt," in preparation.

A macroscopic depression in a solid-liquid interface of succinontrile containing 2×10^{-3} wt.% ethanol. Such depressions form only in samples containing solute, when thermally induced convection is present. The depression is a transient feature in the development of a cellular structure, which here is seen just starting.

CG-004

Solutal Convection During Directional Solidification

National Bureau of Standards
Dr. S. R. Coriell
R. J. Schaeffer
R. G. Rehm
G. B. McFadden
H-27954B $190K/year
April 1980 - continuing task

The objective of this task is to calculate and measure effects of convection caused by simultaneous temperature and concentration gradients on directional solidification, including determination of segregation effects in experiments done on Earth and estimation of the effect of microgravity and magnetic fields in avoiding such convection.

Linear stability theory has established criteria for the onset of convective and interfacial instabilities during the vertical directional solidification of a binary alloy. Numerical methods to solve the time-dependent fluid flow, heat flow, and diffusion equations in two spatial dimensions are being developed for fluids with low Prandtl numbers (metals and semiconductors) and moderately large Schmidt numbers (semiconductors). The inhomogeneity of the solute distribution in the solidified crystal due to fluid flow is obtained by numerical computation.

The interaction of fluid flow with a crystal-melt interface is being studied by linear stability techniques. For a buoyancy driven parallel flow in the vertical direction, the results of linear stability analysis are in agreement with experimental studies by Glicksman and colleagues at Rensselaer Polytechnic Institute. The calculations are being extended to a wider range of Prandtl numbers and flow profiles.

Experimental studies of instabilities are being carried out in succinonitrile doped with small additions of ethanol. The concentration and unidirectional solidification velocities have been selected to be close to the values for which the theory predicts transitions from stability to instability. Convection is detected by observations of small neutrally buoyant particles in the liquid. Interactions between thermally induced convection and macroscopic or microscopic solute-induced interfacial instabilities are observed.

Publications

Coriell, S. R., Boisvert, R. F., Mickalonis, J. I., and Glicksman, M. E., "Morphological and Convective Instabilities During Solidification," Adv. Space Res. (Proceedings of XXIV COSPAR Plenary Meeting, Ottawa, Canada, 1982).

CG-005

Semiconductor Material Growth in Low-G Environment

Langley Research Center
Dr. R. K. Crouch
Dr. A. L. Fripp
In-Center Total Cost: $420K
February 1978 - February 1983

The principal purpose of this experiment is to utilize the microgravity environment of space to investigate the effect of convection on the homogeneity and perfection of compound semiconductor crystals. In a gravity field, the specific material $Pb_{1-x}Sn_xTe$ being investigated has unstable solutal gradients or unstable thermal gradients depending on growth orientation.

Three different growth processes will be considered: (1) a vapor phase sublimation for seeded growth, (2) a modified Bridgman growth in which polycrystalline aggregate is necked down to encourage growth of a single crystal, and (3) a modified Bridgman melt back and regrowth.

Research in preparation for the space flight consists of both theoretical and experimental efforts. Numerical analysis of the mass and heat transfer will predict the furnace profile needed to obtain a planar isotherm at the melt-solid interface. Crystals grown on Earth will be used for comparison with those grown in space.

Publications

Clark, I. O., Fripp, A. L., Debnam, W. J., Crouch, R. K., and Brewer, W. D., "Solutal Diffusion Coefficient for Liquid PbTe-SnTe," J. Electrochem. Soc. 130, 104 (January 1983).

Crouch, R. K., et al., "Optimization Studies for Growth of PbSnTe in Space," accepted for publication in Proc. of Am. Ceram. Soc.

Chin, L-Y. and Carlson, F. M., "Finite Element Analysis of the Control of Interface Shape in Bridgman Crystal Growth," accepted by publication in Journal of Crystal Growth.

Fripp, A. L., Crouch, R. K., Debnam, W. J., Clark, I. O., and Wagner, J. B., "Composition Anomalies in Initial Solidification of Binary PbTe-SnTe," to be submitted to Journal of Electrochemical Society.

Carlson, F. M., Fripp, A. L., and Crouch, R. K., "Thermal Convection in Stabally Oriented Furnaces," to be submitted to Journal of Crystal Growth.

Ejim, T. I., Jessen, W. A., and Fripp, A. L., "Melt-Solid Interface Position Determination During Bridgman Stockbarger Crystal Growth," presented at Virginia Academy of Science, Blacksburg, Virginia, April 1982.

Debnam, W. J., Fripp, A. L., and Crouch, R. K., "Reusable Thermal Cycling Clamp," patent application filed.

Debnam, W. J. and Clark, I. O., "Ampoule Sealing Apparatus and Process," NASA Case No. LAR-12847-1, (1982).

CG-006

<u>The Control of Float Zone Interfaces by the Use of Selected Boundary Conditions</u>

Science Applications, Inc.
Dr. Larry M. Foster
NAS8-35108 $22K/year
November 5, 1982 - November 5, 1983

One of the main goals of the Float Zone (FZ) growth project of NASA's Materials Processing in Space Program is to thoroughly understand the molten zone/freezing crystal system and all the mechanisms that govern this system. In addition, more optimal crystal growth conditions at g=1 and possible improvements made by processing in near zero-g environments need to be investigated. To accomplish this, the melt and interface properties, the heat and mass flows, and the dependencies of these on each other and on growth rate and g levels must be studied.

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

The purpose of this effort is to study and compute the surface boundary conditions required to give flat FZ solid-melt interfaces. The successful completion of such a study will result in a better understanding of the FZ diffusion and growth mechanisms and hopefully will provide FZ furnace designers with better methods for controlling solid-melt interface shapes and for computing thermal profiles and gradients.

This study is being undertaken in two phases. The first phase will investigate the solid zones surface boundary conditions required for flat solid-melt interfaces when given the melt zone surface boundary conditions. The second phase complements the first and will investigate the melt zone surface boundary conditions required for flat solid-melt interfaces if given the solid zones surface boundary conditions. Dual integral transform methods are being used in both phases; in addition, the second phase is requiring the use of various numerical methods for elliptic partial differential equations.

The sensitivity of the required surface boundary conditions and computed thermal profiles to variations in the material and process parameters and a priori known melt or solid surface boundary conditions is being examined for each of the twoproposed phases. Since the material parameters of many FZ materials are only crudely known, such a sensitivity study should assist NASA's Space Processing Division in calculating the accuracy required in current and proposed material parameter experiments.

Publications

Foster, L. M., "Materials and Process Constraints for a Flat Interface in the Bridgman-Stockbarger Technique," NASA CR-161511, Section VIII, October 1980.

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Foster, L. M., "Surface Control Temperatures for the Bridgman-Stockbarger Technique," in Developments in Theoretical and Applied Mechanics, SECTAM XI Conference Proceedings (T. J. Chung and G. R. Karr, eds.), April 1982, pp. 603-612.

CG-007

Crystal Growth of Device Quality GaAs in Space

Massachusetts Institute of Technology
Professor H. C. Gatos
Dr. Jacek Lagowski
NSG-7331 $300K/year
April 1, 1977 - continuing task

The experimental and theoretical efforts are aimed at the establishment of relationships among crystal growth parameters, materials properties, electronic properties and device applications of GaAs. Toward this goal steps have been undertaken for the development of new approaches to the preparation and characterization of GaAs. This extensive ground-based program constitutes a necessary step toward insuring successful processing of GaAs under zero gravity conditions. Due to its unique scope combining crystal growth characterization and device-related properties and phenomena, this program bears directly upon exploitation of the potential of GaAs in device applications.

The research task includes the detailed study of the mechanisms of GaAs crystal growth from the melt and from solution and of the development of techniques for the characterization of materials and electronic properties on a microscale, e.g., composition, carrier concentration, mobility, diffusion length, and lifetime. Relationships between electronic properties and device performance are a part of the research task.

Publications

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Presentations

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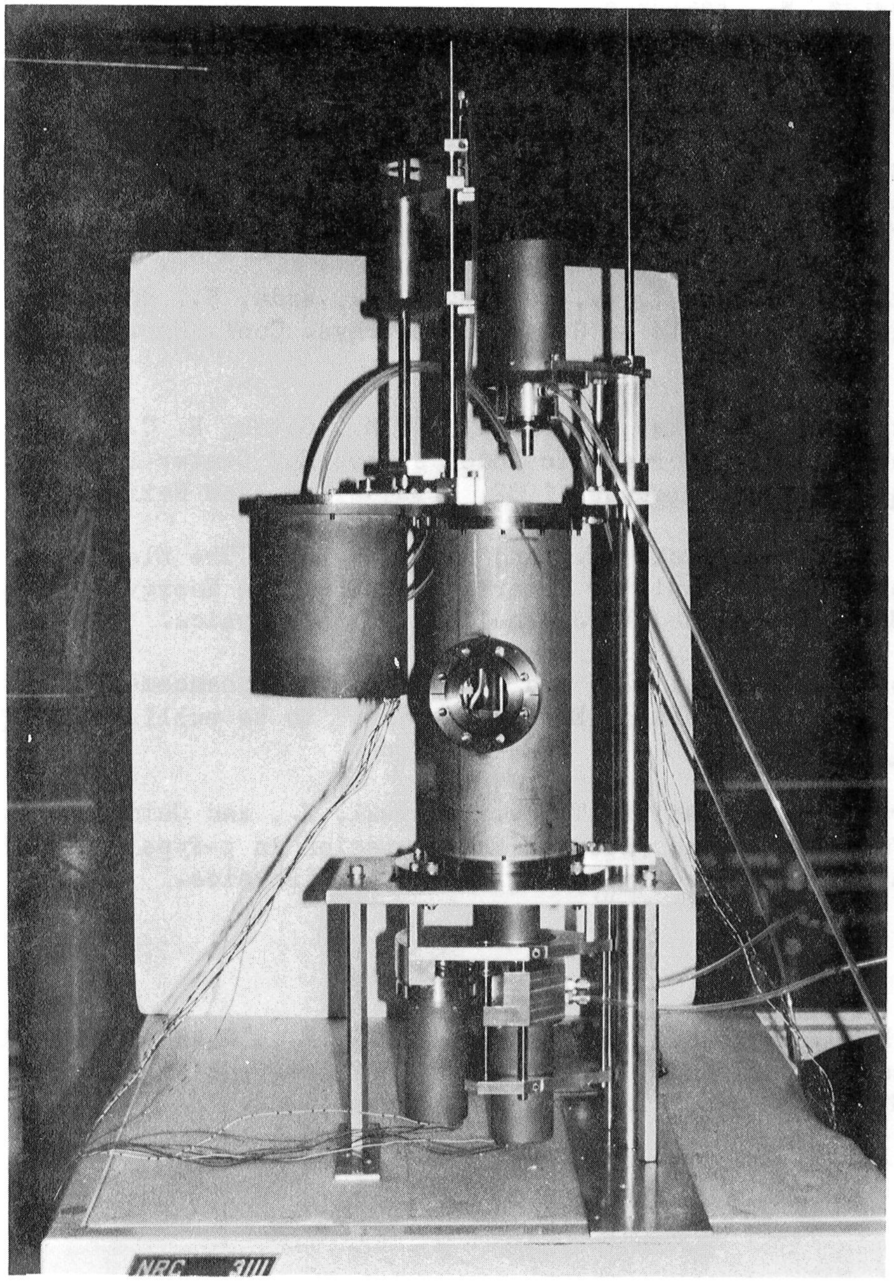

Thin Rod Zoner - for hot wall zoning of 5 mm diameter silicon crystals

CG-008

Microgravity Silicon Zoning Investigation

Westech Systems, Inc.
Dr. E. L. Kern, Consultant
G. L. Gill
NAS8-34920 Total Cost: $218K (approx.)
July 1982 - July 1983

The objective of the silicon floating-zone experimentation program is the growth of uniform silicon crystals through the use of microgravity conditions. It is predicted that the lack of significant gravity will eliminate both steady state and aperiodic melt flows and will allow for a long zone. These in turn, will eliminate resistivity and swirl striations and, with a growth interface of less curvature being possible, will reduce vacancies. Thus, the resistivity uniformity and lower crystallographic defect density needed for a variety of critical devices and semiconductor standards can be provided by space processing. In addition, the understanding of the silicon melt/growing crystal system resulting from this investigation will be a valuable input to improving the ground-base zoning of standard silicon crystals, used in large volume in power and integrated circuit devices, especially as larger diameters are needed.

A second objective is to gain an understanding of all of the component forces on and flows of the silicon melt. These include the effects of buoyancy (gravity), Marangoni (surface tension driven) and r.f. heating fields. Slices of gallium doped silicon will be melted, resolidified and analyzed for the relative effects of buoyancy and Marangoni forces at g=1. A small research furnace, a modification of the ADSF (Advanced Directional Solidification Furnace), using resistance heating (hot wall) and a hot zone tailored to zoning silicon, will be built and tested. Ground-based research on this furnace at small crystal diameters (5mm) will characterize hot wall zoning, and will be correlated to modeling of the zone and modeling of the furnace profile for reducing gradients, which are being done on separate NASA-sponsored programs. It will also specify the experimental conditions for float zoning in space. In the second year, a flight furnace will be specified and designed for MEA (Materials Experiment Assembly) based flights. These flight experiments will further characterize the Marangoni effect on float zoning in microgravity.

CG-009

Solution Growth of Crystals in Zero-Gravity

Alabama A&M University
Dr. R. B. Lal
Dr. R. L. Kroes, MSFC
NAS8-32945 Total Cost: $557K
June 28, 1978 - August 1983

In a low-g environment, buoyancy-driven convection effects in solution crystal growth are greatly reduced and, thus, one can study diffusion mass transport which in 1-g is masked by convective phenomena. Also, triglycine sulfate (TGS) crystals have technological importance for infrared detectors. The objectives of the experiment are (1) to grow TGS crystals from aqueous solution in low-gravity, (2) to investigate mass transport and heat flow in a diffusion-controlled growth system, and (3) to evaluate the feasibility, possible advantages and technical potential of producing solution growth crystals in space.

Single crystals of TGS have been grown using conventional low-temperature solution growth method and the growth process has been extensively characterized. Also, a unique technique of growing solution growth crystals by extracting heat at a programmed rate from the crystal through a semi-insulating sting has been developed. TGS crystals will be grown by this technique during the Spacelab 3 mission. Data on heat and mass transport in a diffusion-controlled system will be obtained using a laser holography technique. Analytical studies are under way to estimate growth rates in low-g conditions.

Publications

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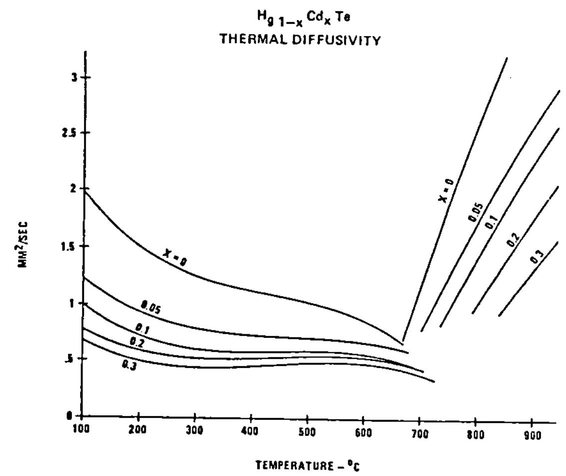
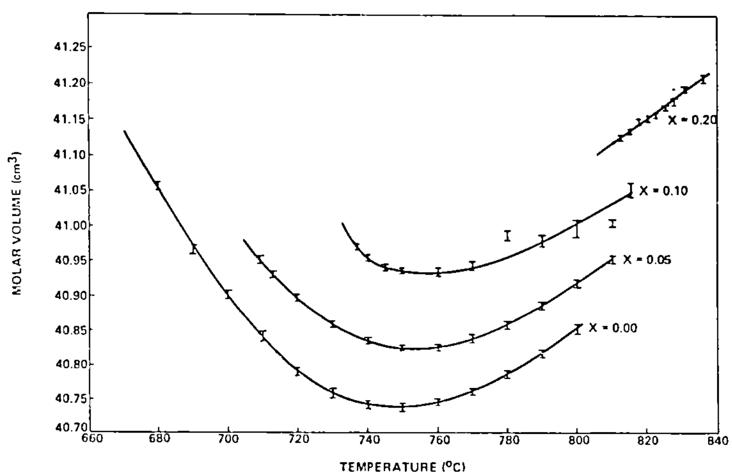


Measured Thermal Diffusivity of $Hg_{1-x}Cd_xTe$ Solids and Melts



Measured Molar Volumes of Liquid $Hg_{1-x}Cd_xTe$

CG-010

Growth of Solid Solution Crystals

Marshall Space Flight Center
Dr. S. L. Lehoczky
Dr. F. R. Szofran
Dr. L. R. Holland, UAH
Dr. J. C. Clayton, Semtec
Dr. D. C. Gillies, Semtec
In-House Total Cost: $540K
October 1977 - October 1983

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

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

A theoretical model has been developed for the axial compositional redistribution during the directional solidification of the alloys and the model have been applied to the initial and final solute segregation transients in order to estimate an effective diffusion constant. The one-dimensional, planar interface model incorporates aspects of the HgCdTe system that are not encountered in the classic doped semiconductor system. The model assumes diffusion-controlled solidification and treats the variation of interface temperature with composition, the variation of k with composition, and a growth rate determined by the thermal field. This treatment removes the assumptions that have made previous one-dimensional models inappropriate for the HgCdTe system.

Other tasks currently under study include: (1) theoretical modeling of the radial solute redistribution, (2) theoretical modeling of the ternary phase diagram, (3) ternary phase equilibrium measurements, (4) the measurement of the temperature and composition dependences of HgCdTe thermal diffusivities and coefficients of thermal expansions in the liquid and solid phases, and (5) experimental characterization and theoretical modeling of the effects of processing conditions on the electrical properties of the alloys.

Publications

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Lehoczky, S. L., Szofran, F. R., and Naumann, R. J., "Crystal Growth of Homogeneous $Hg_{1-x}Cd_xTe$ Crystals from Pseudobinary Melts," Bull. Am. Phys. Soc. 28, 294 (1982).

Gillies, D. C., "Analysis of Mercury Cadmium Telluride by Energy Dispersive Analysis," Journal of Electronic Materials, (in press).

Naumann, R. J. and Lehoczky, S. L., "Effect of Variable Thermal Conductivity on Isotherms in Bridgman Growth," J. Cryst. Growth (in press).

Szofran, F. R. and Lehoczky, S. L., "Liquidus Temperatures of Hg-Rich Hg-Cd-Te Alloys," J. Elect. Mat. (in press).

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Holland, L. R. and Taylor, R. E., "Measured Thermal Diffusivity of $Hg_{1-x}Cd_xTe$ Solids and Melts," J. Vac. Sci. Tech., 1983 (submitted).

Chandra, D. and Holland, L. R., "Density of Liquid $Hg_{1-x}Cd_xTe$," J. Vac. Sci. Tech., 1983 (submitted).

Presentations

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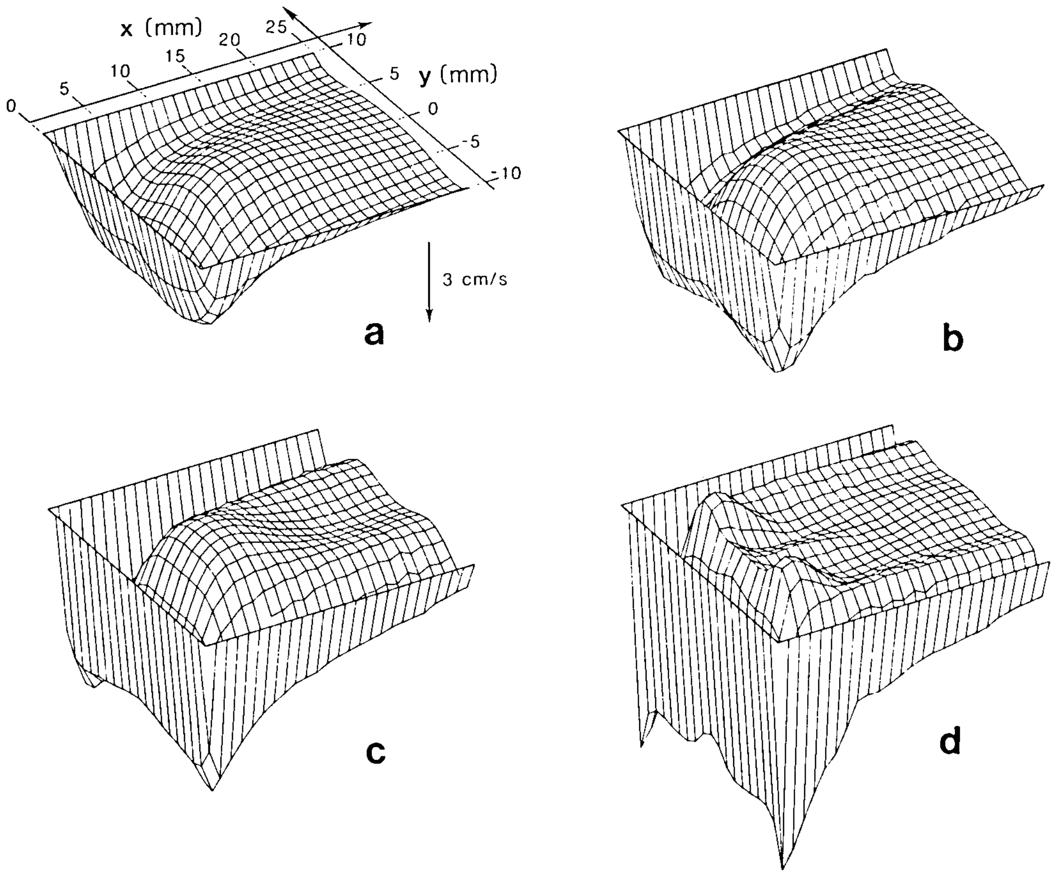
Holland, L. R. and Taylor, R. E., "Measured Thermal Diffusivity of $Hg_{1-x}Cd_xTe$ Solids and Melts," presented at U.S. Workshop on Physics and Chemistry of Mercury Cadmium Telluride, February 8-10, 1983, Dallas, Texas.



Distribution of the velocity component parallel to a crystal on a vertical endwall of a horizontal vapor growth ampoule, measured at midheight for various Rayleigh numbers. (a) Ra = 3,580, (b) Ra = 8,860, (c) Ra = 18,700, (d) Ra = 342,100.

CG-011

Fluid Dynamics of Crystallization from Vapors

University of Utah - Salt Lake City
Dr. F. Rosenberger
NSG-1534

This program is aimed at obtaining fundamental insight into the complex physiochemical fluid dynamics of closed ampoule vapor crystal growth processes to the extent that a desired set of crystal growth conditions can be designed in advance. A more directly applied part of the program is concerned with the synthesis of ultrapure mercuric iodide and the vapor composition (stoichiometry) required for the growth of mercuric iodide high resolution radiation detector crystals.

Numerical modeling of vapor transport in vertical ampoules has shown that diffusion fluxes, in viscous interaction with the wall, establish density gradients normal to the main transport direction. These density gradients act convectively destablizing even in ampoule orientations which, classically, were considered convectionfree (e.g., "heating from top"). Also, it has been demonstrated that the convection behavior in crystal growth ampoules can not be extrapolated from known solutions to fluid dynamically "similar" monocomponent (pure) systems. The net transport across the vapor space causes drastic changes as compared to convection patterns in cylinders with impermeable end faces.

It has been found experimentally that thermal diffusion in ampoules acts convectively more destabilizing than in laterally unbound (Benard) geometries. The vapor composition of transport systems was used aboard Skylab 3 and 4 and that still defy a quantitative description of the transport rates obtained, is being characterized mass spectroscopically.

Rigorous modeling of convection-enhanced vapor transport across a horizontal cavity has shown that at low Rayleigh numbers, earlier, simplifying treatments (Klosse-Ullersma, KU), because of fortuitous cancellation of errors, give reasonable results. At high Rayleigh numbers, however, the KU treatment grossly underestimates the transport rate. Furthermore transport rates strongly depend on the Prandtl number of the vapor, whereas the KU model is based on a vanishingly small Prandtl number. Also, laser Doppler anemometry studies of the convective velocity fields in inclined and horizontal ampoules revealed three-dimensional features of the flow, that have generally not been accounted for in modeling.

For meruric iodide, titrometric and vapor pressure studies have shown that deviations in stoichiometry of mercuric iodide (HgI_{2+x}) can extend to $x = -3 \times 10^{-3}$. No excess in iodine, i.e. $x > 0$, could be detected in vapor- and solution-grown samples obtained from various sources.

Publications

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HgI$_2$ Crystal Growth for Nuclear Detectors

EG&G, Inc.
W. F. Schnepple
Dr. L. Van den Berg
H-34318B Total Cost: $646K
April 28, 1978 - April 28, 1983

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

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

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

Publications

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Levi, A., Roth, M., and Schieber, M. M., "The Development of Mercuric Iodide Gamma Radiation Detectors for Application in Nuclear Medicine," IEEE Trans. Nuc. Sci. NS-29, 457 (1982).

Ortendahl, D., Kaufman, L., Hosier, K., Padgett, L., and Ortale, C., "Operating Characteristics of Small Position-Sensitive Mercuric Iodide Detectors," IEEE Trans. Nuc. Sci. NS-29, 784 (1982).

Presentations

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Lamonds, H., "Review of HgI_2 Development at EG&G Santa Barbara Operations," presented at the Fifth International Workshop on Mercuric Iodide Nuclear Radiation Detectors, Jerusalem, Israel, June 6-8, 1982. To be published in Nuclear Instruments and Methods.

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Ortale, C., Padgett, L., and Schnepple, W. F., "Imaging Devices Made from HgI_2 Nuclear and Z-Ray Multi-element Arrays Using Photolithographic Techniques," presented at the Fifth International Workshop on Mercuric Iodide Nuclear Radiation Detectors, Jerusalem, Israel, June 6-8, 1982. To be published in Nuclear Instruments and Methods.

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Becchetti, F. D., Raymond, R. S., Ristenen, R. A., Schnepple, W. F., and Ortale, C., "Mercuric Iodide Semiconductor Devices as Charged Particle Detectors," presented at the Fifth International Workshop on Mercuric Iodide Nuclear Radiation Detectors, Jerusalem, Israel, June 6-8, 1982. To be published in Nuclear Instruments and Methods.

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CG-013

Float Zone Experiments in Space

Ames Laboratory, Iowa State University
Dr. J. D. Verhoeven
H-34328B $82K/year
October 1982 - October 1983

The objectives of the program are: (1) to determine if surface tension-driven convection in a float zone can be controlled or eliminated by means of surface films and (2) to investigate solute distribution and measure liquid diffusion coefficients in floating zones. If surface tension-driven convection can be effectively controlled or eliminated, it should be possible to obtain homogeneous solute distributions in space, which is not possible on Earth. Also, it should be possible to obtain accurate liquid diffusion coefficients for systems too reactive to be contained in capillary tubes.

Ground-based experimental work involves building an experimental float zone apparatus which will allow high vacuum capability plus control and measurement of temperature gradient, growth rate, and oxygen potential. Oxide layer thicknesses will be evaluated by depth profiling plus ELS-Auger analysis. For objective No. 1, experiments will consist of measuring the effect of oxide layer thicknesses upon interface shapes, the radial and longitudinal composition profiles, temperature profiles and the possible onset of ascillatory temperatures. For objective No. 2, solute profiles will be evaluated in the initial transient zones and the quenched zones.

Publications

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CG-014

Fluid Dynamics and Thermodynamics of Vapor Phase Crystal Growth

Rensselaer Polytechnic Institute
Dr. Heribert Wiedemeier
NAS8-33562 Total Cost: $254K
January 1, 1980 - December 31, 1982

The primary objective of this program is to provide basic mass transport and crystal growth data which, combined with a thorough knowledge of the thermodynamics, will improve the fluid dynamic characterization of vapor transport systems.

The program is concerned with the investigation of the effect of systematic variations (1) of the relative importance of buoyancy-driven convection and (2) of diffusion and viscosity conditions on mass transport and crystal growth. These investigations will be performed in evacuated, closed ampoules for selected temperature gradients and partial pressures of the transport species. The specific experimental tasks include mass transport rate studies as a function of ampoule orientation, geometry, and the effects of inert gas additions. These will be correlated with crystal growth rate and morphology studies. In addition, the analysis of the vapor phase is an essential aspect of this project. The experimental efforts are supported by theoretical studies, including the thermodynamic and fluid dynamic characterization of the gas phase and the estimation of mass transport rates for different diffusive and diffusive-convective conditions. The materials investigated under this program include selected group IV elements and IV-VI compounds and halogens as transport agents. This choice is based on increasing complexity, known thermochemical and structural propertie of these materials, and the existence of microgravity results for some of these systems investigated in previous Skylab and ASTP experiments.

Publications

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Wiedemeier, H. and Pultz, G., "Equilibrium Sublimation and Thermodynamic Properties of SnSe and $SnSe_2$," Z. Anorg. Allg. Chem., in press.

CG-015

<u>Vapor Growth of Alloy-Type Semiconductor Crystals</u>

Rensselaer Polytechnic Institute
Dr. Heribert Wiedemeier
NAS8-32936 Total Cost: $460K
March 1978 - March 1983

The objectives of this program are to investigate through systematic ground-based studies the effects of gravity-driven convection on the growth of single crystals of alloy-type semiconductors, to define optimum conditions for the growth of these materials in a microgravity environment, and to perform crystal growth studies in space. For this purpose, the systems $Hg_{1-x}Cd_xTe$ and $CuInS_2$ have been selected.

The ground-based research is concerned with the quantitative investigation of the chemical transport properties of the preceding materials in evacuated, closed ampoules of fused silica employing elemental iodine or metal-iodides as transport agents. The mass transport rates and crystal morphology of these systems are investigated as a function of total pressure for different temperature gradients and under horizontal and vertical stabilizing conditions. The inherent partial pressure and density gradients of the system and the presence of gravitational forces on Earth cause convective interferences with the transport and condensation process. Present results reveal that the surface morphology and chemical homogeneity of $Hg_{.8}Cd_{.2}Te$ crystals obtained under vertical stabilizing conditions are improved relative to crystals grown under horizontal conditions. The crystal quality of $CuInS_2$ shows similar improvements for the horizontal ampoule configuration with decreasing pressure (decreasing convective interference) of the system. The combined results of ground-based studies will lead to the definition of optimum growth conditions for the actual space experiments.

Publications

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CG-016

Heat Flow and Segregation in Directional Solidification

Massachusetts Institute of Technology
Professor A. F. Witt
NSG-7645

The research task is directed toward the optimization of crystal growth and segregation during solidification in Bridgman-type configurations. The first phase of this study was concerned with a determination of the effects of thermal boundary conditions on growth and segregation of doped Germanium in a conventional system. Making use of interface demarcation and spreading resistance analyses, it was found for conventional thermal geometries that at constant ampoule lowering rates, both growth and segregation remain non-steady state for growth lengths of up to 6 cm. The rate of growth is significantly less than the lowering rate under high axial thermal gradient conditions but exceeds the lowering rate by a factor of two at low applied thermal gradients. Upon temporary arrest of ampoule lowering uncontrolled growth or back melting takes place depending on the magnitude of the existing axial thermal gradient. The experimental evidence obtained suggests that conventional vertical Bridgman configurations cannot provide a thermal environment in which steady state crystal growth and radially uniform dopant segregation is achievable.

To arrive at an improved Bridgman-type configuration suitable for growth on earth and in reduced gravity environment, it was decided to base the systems design on one- and two-dimensional heat transfer analyses. These calculations suggested the use of aligned heat pipes separated by a gradient zone region with variable heat transfer characteristics. Such a system has now been constructed and is in the process of being characterized for thermal and growth characteristics.

With the establishment of thermally stabilized growth conditions in vertical Bridgman configuration, it became possible to study dopant segregation at solidification rates ranging from 0.5 to 15μ m/s. This study revealed that the basis for all generally accepted segregation theories, at constant and rate dependent interface distribution coefficient which is identical with the equilibrium distribution coefficient, does not apply to the system germanium-gallium. It is found that during both faceted and nonfaceted growth the interface distribution coefficient differs from k_0 and in the growth range from 0-2 μm/s exhibits a pronounced rate dependence. This finding is of fundamental importance and, moreover, has implications on space processing since this particular system has been and is extensively used for the characterization of growth in reduced gravity environment.

In a parallel study, directional melting of binary systems (with narrow and wide liquidus-solidus separation) as encountered during seeding in melt growth, was analyzed for concurrent compositional changes at the seed-melt interface. It could be shown that steady state composition conditions cannot normally be reached during seeding and that the growth interface temperature at the initial stages of seeded growth is a function of backmelt conditions. The theoretical treatment was numerically applied to HgCdTe and Ga-doped germanium.

Publications

Wang, C. A., Carruthers, J. R., and Witt, A. F., "End-Effects on Crystal Growth and Segregation Behavior in Vertical Bridgman Configuration," J. Cryst. Growth 60, 144 (1982).

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CG-017

Vapor Phase of PbSnTe

Jet Propulsion Laboratory
Dr. J. A. Zoutendyk
NAS7-100 $130K/year
March 1, 1981 - March 1, 1982

This ground based research is for the experimental study of gravity-driven convection effects in the growth of PbTe and CdTe crystals by physical vapor transport. These binary compound semiconductors are important as substrate material for the epitaxial growth of PbSnTe and HgCdTe layers, respectively, for infrared detector fabrication.

2. METALS, ALLOYS, AND COMPOSITES

TASK NUMBER	PRINCIPAL INVESTIGATOR	SHORT TITLE
ME-001	Frazier	Studies of Model Immiscible Systems
ME-002	Gelles	Liquid Phase Miscibility Gap Materials
ME-003	Glicksman	Dendritic Solidification
ME-004	Hellawell	The Influence of Gravity on Solidification
ME-005	Johnston	Comparative Alloy Solidification
ME-006	Larson	Aligned Magnetic Composites
ME-007	Malmejac	Interfacial Destabilization
ME-008	Pirich	Directional Solidification of Bi/MnBi
ME-009	Potard	Directional Solidification of Monotectics
ME-010	Schmid	Binary Miscibility-Gap Systems
ME-011	Wilcox	Modeling Directional Solidification
ME-012	Wilcox	Study of Eutectic Formation

INTRODUCTION

Directional solidification is a casting process used to produce single crystals and two-phase composite materials wherein the microstructure is aligned in a particular direction such that the mechanical and physical properties differ among various axes, or wherein fine, homogeneous dispersions are achieved. Common example of two(or multi) phase composites might be fiberglass wherein glass filaments are suspended (either unidirectionally or randomly) in a plastic matrix to increase strength and provide anisotropic properties and dispersion hardened steeel wherein small carbide particles are included in ther steel matrix to improve strength. The MPS interest in directionally aligned composites is built upon the extraordinary high magnetic coercivity measured in space-grown composites of Mn-Bi/Bi. Additional interest is based on the potential of approaching the theoretical maximum magnetic strength of materials such as samarium-cobalt ($SmCo_5$) which is 10 times higher than currently realized on Earth.

The second aspect of directional solidification finds application in miscibility gap alloys that defy preparation in one-g in bulk quantities because gravity-driven effects cause the materials to segregate upon solidification. If producible, such materials might have such diverse applications as electrical contacts (as replacements for silver and gold) and self-lubricating bearings. Experiments in low-g have successfully produced finely dispersed, homogeneous mixtures of Ga-Bi and Al-In. Other materials, such as Cu-Pb, Cd-Ga, Ag-Ni, Al-In-Sn, Cu-Pb-Al, Cd·Ga-Al, and transparent model materials, are being studied in the MPS to define nongravity segregation phenomena and to establish the technqiues to produce these unique materials for property evaluation.

Undercooled solidification is the rapid quenching of molten materials at temperatures well below their freezing points. This process is valuable in the preparation of amorphous (glass or glass-like) materials as well as pure single crystal and metastable phases. The NASA MPS program has developed unique groundbased free fall facilities at MSFC in which extreme undercooling (hundreds of degrees centigrade in excess of existent theory) has been achieved in the production of bulk quantities of pure single crystals and superconducting metastable phases; these materials have not been made in bulk quantities by other methods. The emphasis in undercooled solidification centers on the formation of pure Nb and the superconducting phase Nb_3Ge, which has a high superconducting transition and temperature and offers great promise for electrical transmission and electrical devices.

The MPS program is using the zero-g environment to study the formation and resultant properties of various cast materials (both simple model materials and commercial alloys) to establish process controls and techniques that might be adapted on Earth.

ME-001

Studies of Model Immiscible Systems

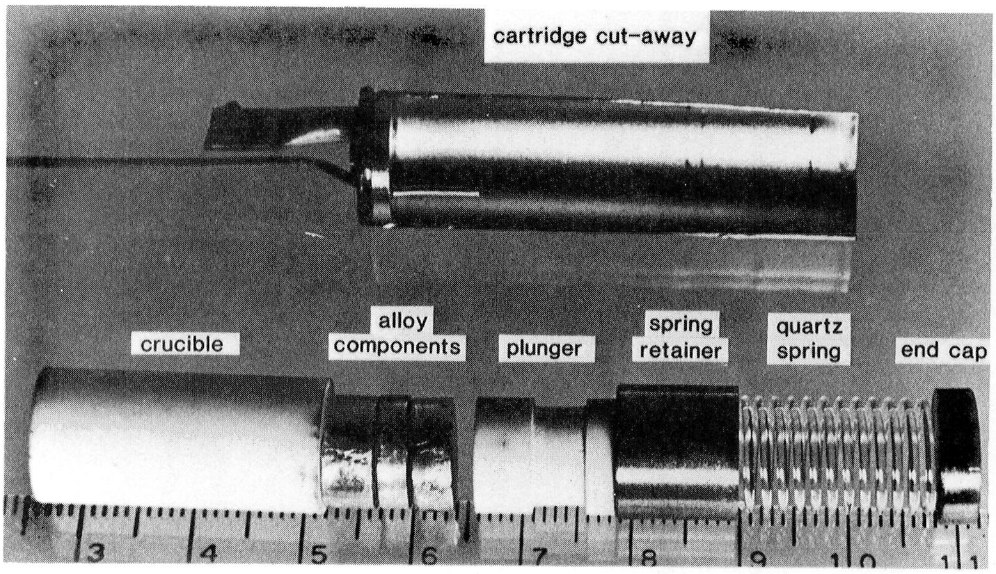
Marshall Space Flight Center
Dr. D. O. Frazier
B. R. Facemire
W. K. Witherow
Dr. W. F. Kaukler, USRA
In-House $50K/year
October 1, 1982 - October 1, 1986

The objectives of this program task are to use model organic immiscible systems to obtain fundamental information applicable to two-phase systems in general and to apply this understanding to materials of interest in the Materials Processing in Space program in order to interpret results of flight experiments involving monotectic alloys.

A number of model immiscible systems are being used to study various aspects of two-phase behavior within the miscibility gap and during solidification. Particle growth, coalescence, and particle motions are being investigated using a holographic microscopy system. The system is capable of working with particle densities up to 10^7 particles/cm^3 through a 100 μm depth and can resolve particles of the order of 2 to 3 μm in diameter throughout the entire cell volume. Particle size distribution changes with respect to time and temperature are determined from sequential holgrams. Initial experiments using diethylene glycol/ethyl salicylate (DEG/ES) have demonstrated the usefulness of the technique. The thermal system for cell temperature control has been refined to give control to at least $\pm$ 0.001°K over the course of an experiment.

Data with respect to solidification of succinonitrile/water solutions are thus far consistent with critical point wetting behavior and Marangoni effects. There is experimental evidence that wetting phenomena are observable by holographic photography. Solid-liquid interfacial free energy differences are, in principle accessible by film pressure (via ellipsometry) measurements. Holographic interfacial free energy measurements. and nucleation studies via differential techniques are underway. These studies, in light of concentration profiles of model solidified ingots, should yield valuable verification of operational limits. Wherever feasible, data in all areas of study will include components for analyzing surface effects (modification of pyrex or quartz surfaces to reverse wetting properties).

A temperature gradient stage with high thermal stability has been assembled to study solidification phenomena <u>in situ</u> using model organic systems. Laser interferometry will be performed with this study in order to measure concentration gradients in the liquid ahead of an advancing solid/liquid interface. The model organic systems for study include monotectic and eutectic phase reactions. Many of the experiments will be performed in the lab and in the KC-135 to observe the effects, if any, on the fine scale concentration and density gradients in the liquid by the gravity vector.



Al-90 WT. PCT. In PLUNGER CARTRIDGE OPENED TO EXPOSE COMPONENTS OF CONSTRUCTION
This cartridge is designed to eliminate the free surface at the melt/gas interface.
Plans call for utilization of this design in MEA-Al Experiments.

ME-002

Liquid Phase Miscibility Gap Materials

S. H. Gelles Associates
Dr. S. H. Gelles
Dr. A. J. Markworth - Battelle Columbus Laboratories
NAS8-32952 Total Cost: $675K
April 1978 - December 1983

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

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

Present plans call for four experiments to be conducted in the Materials Experiments Assembly (MEA) in mid-1983. Two of these involve Al-In alloys which will be processed isothermally in a configuration that avoids the presence of a free melt surface and thus eliminates a major source of convection, namely that arising from gradients in surface tension. In a third experiment, an Al-In alloy will be directionally cooled in order to study thermocapillary-driven droplet migration and solidification under controlled conditions. The fourth experiment will be concerned with the melting and solidification of two Te-Tl alloys in order to understand the influence low interfacial energy and droplet concentration have on the phase separation process. Other experiments are being planned for later Shuttle flights.

ME-003

Dendritic Solidification at Small Supercoolings

Rensselaer Polytechnic Institute
Dr. M. E. Glicksman
NAS8-32425 Total Cost: $313K
March 1, 1977 - December 31, 1982

The objectives of this research task are to obtain information relating to the kinetic and morphological behavior of systems solidifying at small supercoolings, especially regarding the role of convective and diffusive transport and the influence of gravity. These studies provide important data on the fundamentals of solidification at normal terrestrial and reduced gravitational levels. Morphological features of interest include dendritic tip radii, sidebranch evolution, dynamic and isothermal coarsening. Kinetic features encompass axial growth rate as functions of spatial orientation, supercooling, and solute concentration.

The large data base now established for high-purity succinonitrile (SCN) had permitted the most comprehensive check of diffusional dendritic growth theory and the development of "scaling laws" permitting the extension of these theories to many other material systems. The current thrust of this program is on alloys based on SCN as the solvent (melt) system. Current efforts are focussed on SCN-argon, which models a dilute binary alloy. Other binary systems will be explored to establish the generality of these findings and their independence from any specificity inherent to many binary alloys. Finally, techniques are being developed and evaluated for measuring melt flow velocities adjacent to simple (cylindrical) and complex (dendritic) solid-liquid interfaces.

Publications

Huang, S. C. and Glicksman, M. E., "The Influence of Natural Convection on Dendritic Growth," submitted to Journal of Crystal Growth, 1981.

Glicksman, M. E., "Morphological and Convective Instabilities During Solidification," NBSIR 82-2560, 1982.

Glicksman, M. E., Voorhees, P. W., and Setzko, R., "The Triple-Point Equilibria of Succinonitrile-Its Assessment as a Temperature Standard" in Temperature, Its Measurements and Control in Science and Industry, Volume 5 (J. F. Schooley, ed.), American Institute of Physics, 1982, pp. 321-326.

Glicksman, M. E., "The Role of Diffusive and Convective Transport in Solidification," Extended abstract to be published in a Special Annal of the New York Academy of Sciences.

Glicksman, M. E. and Mickalonis, J., "Convective Coupling of a Solid-Liquid Interface in an Internally Heated Vertical Cylinder," to be published in the Proceedings of the 16th Southeastern Seminar on Thermal Sciences, April 1982.

Glicksman, M. E. and Sokolowski, R. S., "Gravitational Influence on Binary Alloy Melt Equilibria," to be published in the Proceedings of the XXIV COSPAR meeting, Ottawa, Canada, May 1982.

Coriell, S. F., Boisvert, R. F., Mickalonis, J., and Glicksman, M. E., "Morphological and Convective Instabilities During Solidification," to be published in the Proceedings of the XXIV COSPAR meeting, Ottawa, Canada, May 1982.

Presentations

Glicksman, M. E., "The Triple-Point Equilibria of Succinontrile-Its Assessment as a Temperature Standard," presented at 6th Symposium on Temperature, Its Measurement and Control in Science and Industry, American Institute of Physics, Washington, D.C., March 17, 1982.

Glicksman, M. E., "Convective Coupling of a Solid-Liquid Interface in an Internally Heated Vertical Cylinder," presented at 16th Southeastern Seminar on Thermal Sciences, Miami, Florida, April 20, 1982.

Glicksman, M. E., "Gravitational Influence on Binary Alloy Melt Equilibria," presented at the XXIV COSPAR Meeting, Ottawa, Canada, May 16-29, 1982.

Glicksman, M. E., "The Role of Diffusive and Convective Transport in Solidification," presented at 4th International Conference on PhysicoChemical Hydrodynamics, New York, June 14-17, 1982.

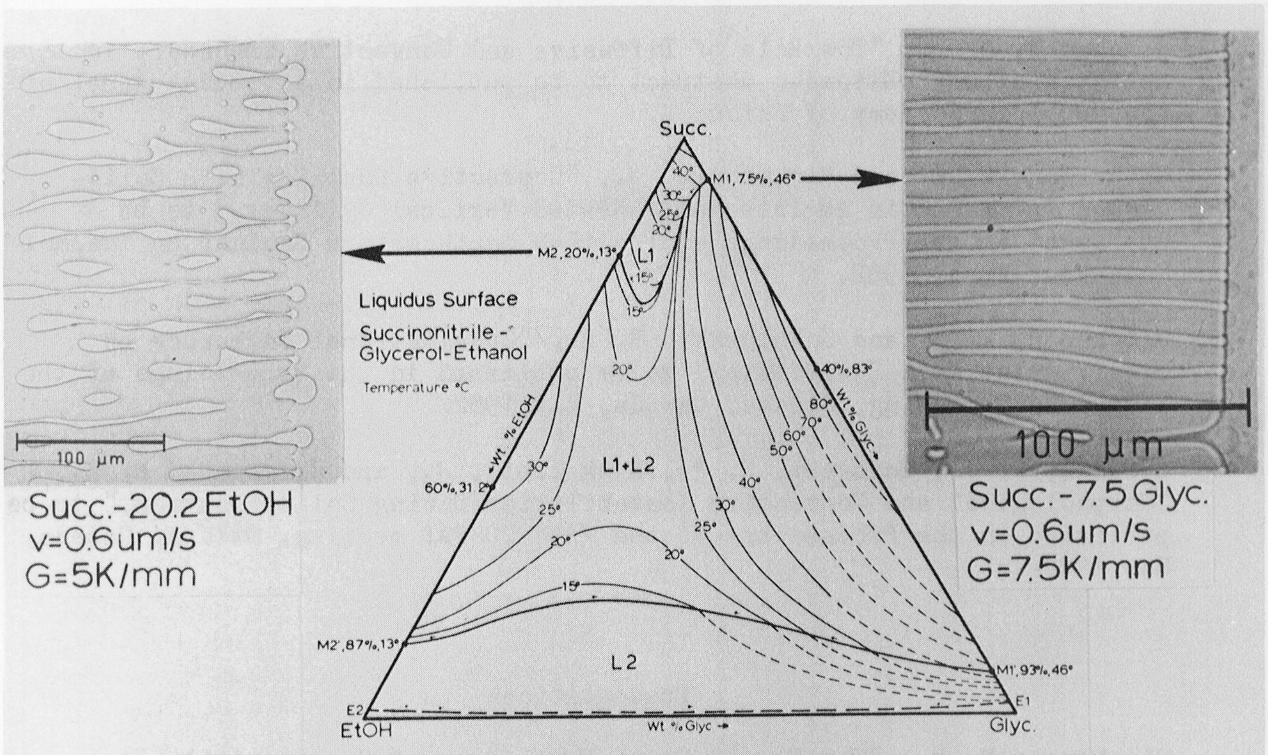

Succ.-20.2 EtOH
v=0.6um/s
G=5K/mm
100 μm
Succ.
M1, 7.5%, 46°
M2, 20%, 13°
L1
Liquidus Surface
Succinonitrile -
Glycerol-Ethanol
Temperature °C
Wt. % EtOH
Wt. % Glyc.
40%, 83°
L1+L2
60%, 31.2°
M2', 87%, 13°
L2
M1', 93%, 46°
E2
E1
EtOH
Wt % Glyc
Glyc.
100 μm
Succ.-7.5 Glyc.
v=0.6um/s
G=7.5K/mm

Fig. 3

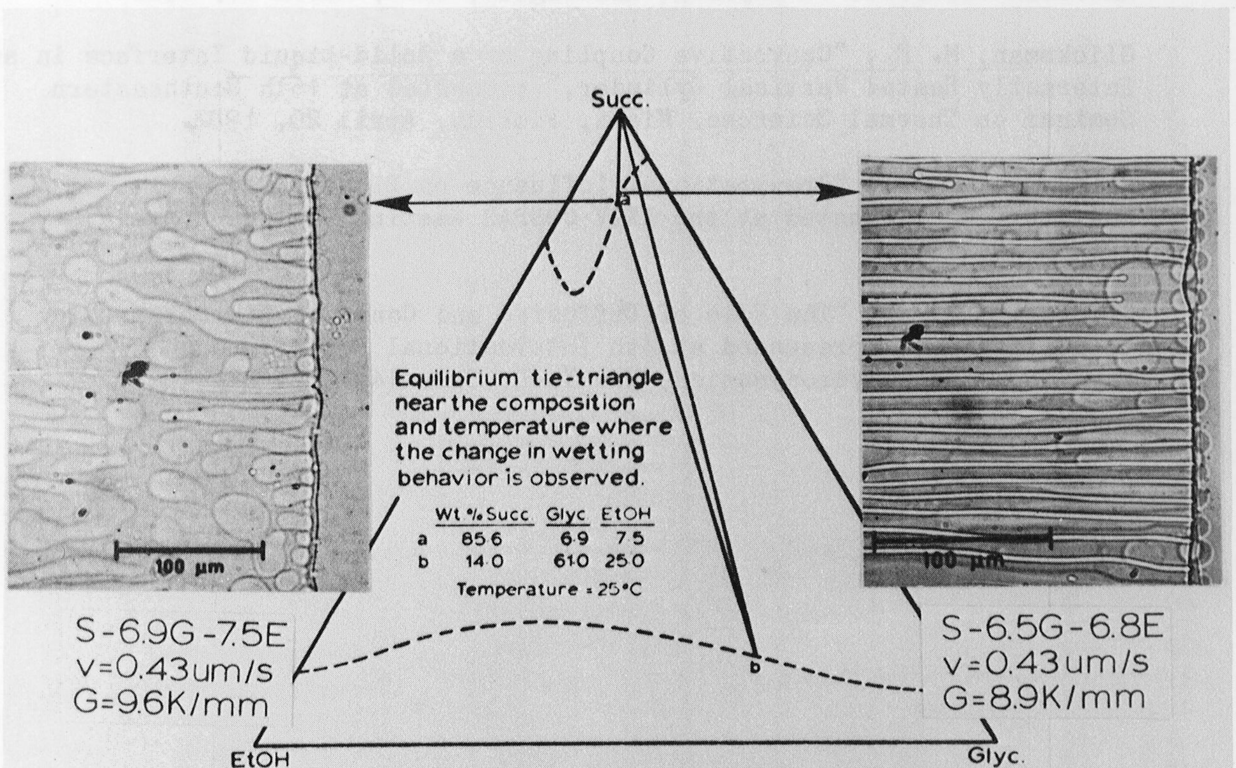

Succ.
a
Equilibrium tie-triangle near the composition and temperature where the change in wetting behavior is observed.
Wt % Succ. Glyc EtOH
a 85.6 6.9 7.5
b 14.0 61.0 25.0
Temperature = 25°C
b
100 μm
100 μm
S-6.9G-7.5E
v=0.43um/s
G=9.6K/mm
S-6.5G-6.8E
v=0.43um/s
G=8.9K/mm
EtOH
Glyc.

Fig. 5

ME-004

The Influence of Gravity on the Solidification of Monotectic Alloys

Michigan Technological University
Dr. A. Hellawell
NAS8-33727 Total Cost: $126K
September 1980 - September 1983

The objective of this research is to examine the monotectic reaction using directional solidification methods in order to obtain aligned composite structures. One aspect of the problem is the separation of two liquids below a miscibility gap and their incorporation within a duplex growth front. Both surface tensions and relative densities influence this process, and the objective of this research program is to identify the gravitational influence.

The systems under examination include Al-In, Cu-Pb, Al-Bi, Cd-Ga, as well as the ternary systems Al-In-Sn, Cu-Pb-Al and Cd-Ga-Al. The transparent analogue system, succinnonitrile-ethanol-glycerol has been determined and the form of the liquidus surface and monovariant monotectic reaction ascertained. Using a temperature gradient stage upon an optical microscope, a transition in growth behavior has been demonstrated in this system which is analogous to that produced in ternary metallic alloys.

Publications

Grugel, R. N. and Hellawell, A., "Monotectic Solidification - The System Succinnonitrile-Ethanol-Glycerol," Materials Research Society, Boston, 1982, to be published.

ME-005

Comparative Alloy Solidification

Marshall Space Flight Center
Dr. M. H. Johnston
In-House

This effort is an extension of the earlier SPAR experiments, which used transparent model systems to investigate the gravitational influence on the solidification process, to actual metallic systems. Effects such as macro and micro segregation, grain size, shape, orientation, and physical properties of ingots cast in low-g will be compared to identical castings in one and high-g environments. A striking decrease in grain size with increasing g-field has already been demonstrated, confirming earlier predictions that dendrite multiplication was influenced by gravity-driven convective flows.

In the low-gravity solidification of the Sn-15wt%Pb alloy, the grain orientations were found to be completely isotropic, indicating probable nucleation in the center of the molten liquid. A significant increase in dendrite arm spacing was noted for the low-g metal sample, thus substantiating earlier results from the metal model systems.

A tin-3wt%Bi alloys was solidified on a second flight. This had a few very large grains in contrast to the very fine grained ground based samples. Further studies of this phenomena were carried out on the KC-135. A sample was partially solidified in low-gravity and then solidification was completed during the high-g pullout. The transition in the grain structure was rapid, as shown in the accompanying photograph.

Ground-based tests are now being run on the Al-4.5Cu alloy. Centrifuge runs have been completed and were chemically analyzed. To obtain additional low-gravity data, samples were solidified during KC-135 flights. These will be compared with microstructures from the upcoming launch experiment on the Al-4.5 Cu alloy.

ME-006

Aligned Magnetic Composites

Grumman Aerospace Corporation
Dr. D. J. Larson, Jr.
NAS8-32948 Total Cost: $652K
July 1978 - July 1983

The objectives of this program are to contribute to understanding the role of convection on plane front solidification of eutectic and peritectic composites and the relationships between morphology and magnetic properties. In addition, assessment will be made of the commercial potential for processing binary composites in low-g.

The aim of this program is to evaluate the impact of convection (thermal and/or solutal) or coupled convective/diffusive transport on the plane front solidification of contained binary magnetic composites. Eutectic, off-eutectic, and peritectic solidification are under investigation. The low-g orbital environment will be utilized to study diffusion controlled solidification for experimental regimes that would be described as convective/diffusive regimes terrestrially. In addition, the relationships between solidification processing parameters, solidification microstructure, microchemistry, and magnetic properties are being studied.

ME-007

<u>Interfacial Destabilization in Metal Alloys</u>

Laboratoire d'Etudes de la Solidification
Centre d'Etudes Nucleaires de Grenoble
Y. Malmejac
J. J. Favier
No Funds Exchanged
January 1980 - continuing task

The overall objectives of this investigation are to study the destabilizing mechanisms that affect a crystal growth interface, to obtain information on destabilized morphologies in the steady and transient states, and on growth kinetics behavior, and to attempt to separate the influences of liquid phase instabilities from the interface instability. These effects will be studied by directional solidification experiments on metal alloys with moderate melting temperatures under three generic types of conditions: (1) solidification at various rates with a given value of the temperature gradient in the melt next to the freezing interface, (2) solidification at a constant rate with the thermal gradient in the liquid next to the interface varying linearly with time; and (3) solidification with a constant thermal gradient in the liquid and solidification rates that vary linearly with time.

Ground-based research and experiment technique development will be performed to prepare for subsequent similar space experiments if the decision is made to proceed with the space phase of the investigation. As presently conceived, the space experiments will directionally solidify Sn-Bi (0.01 to 10 At.% Bi) and Bi-Sn (.01 to 10 At% Sn) alloy samples at rates of 0.0001 to 0.1 cm/sec with liquid temperature gradients of 10 to 500°C/cm under weightless conditions. Samples will be solidified in simultaneous groups with one sample serving as a stationary reference interface so that a differential thermoelectric voltage generated by a moving interface may be continuously monitored under the quiescent conditions of space and related to the solidification morphology and velocity. Steady state and transient growth kinetics will be studied in relation to the thermal, compositional, and fluid flow effects. Complete technical and scientific feasibility of the project including the hardware will be achieved in 1982.

ME-008

Directional Solidification of Magnetic Composites

Grumman Aerospace Corporation
Dr. R. G. Pirich
NAS8-32219   Total Cost: $295K
February 1, 1977 - January 1983

Following the intriguing results obtained on the ASTP experiment, in which Mn-Bi eutectic directionally solidified in space was observed to have a finer microstructure and enhanced magnetic properties, an extensive ground-based and flight investigation has been initiated. This has resulted in several significant findings.

Morphological analyses on eutectic Bi/MnBi samples that were directionally solidified during the 240-s low-g interval of the SPAR VI flight experiment show statistically smaller interrod spacings and rod diameters with respect to samples grown under identical solidification furnace conditions, in the same apparatus, in 1-g. The magnetic property measurements indicate that the flight samples contain 7 v/o less dispersed MnBi than similarly processed 1-g samples for the same starting composition. Convectively driven temperature fluctuations in the melt, which result in unsteady liquid-solid interface movement in 1-g, are suggested to explain the morphological change between low-g and 1-g solidification. As a result of these fluctuations, an adjustment between the interrod spacing, growth velocity, and total undercooling at the solidification interface is proposed to account for the observed change in volume fraction of dispersed MnBi. Future low-g experiments involving both eutectic (SPAR IX) and off-eutectic (SPAR X) compositions are planned to quantify these unusual low-g effects.

ME-009

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

Centre d'Etudes Nucleaires de Grenoble
Dr. C. Potard
No Funds Exchanged
September 1976 - present

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

Solidification of 10 and 50 atomic percent In samples will be carried out under a directional gradient in a SPAR rocket. Previous isothermal experiments with this system resulted in almost complete phase separation in low-g within the In-rich material surrounding a core of Al-rich material. The mechanisms responsible for this separation are not understood, but because In preferentially wetted the alumina crucible, it is believed that capillarity may play an important role in the phase separation. The present experiment will use a SiC crucible which is preferentially wetted by the Al. The directional gradient will be used to investigate the effects of the solidification on droplet distribution. Four experiments are required to obtain minimum information to sort out the various effects.

The supporting ground-based research consists of: (1) Al- In phase diagram re-determination in the experiment composition range, (2) wetting properties of the two-phase liquid against silicon-carbide, (3) Earth gravity orientation effect on solid structures, (4) preliminary observations on capillary forces and coalescence, and (5) liquid-liquid and liquid-solid phase transformation studies (undercooling, kinetics, volume change, monotectic solidification structures).

ME-010

Binary Miscibility-Gap Systems

National Bureau of Standards
Dr. L. A. Schmid
H-27954B
April 1981 - continuing task

Immiscible droplets embedded in a host fluid in which a temperature gradient exists migrate toward the hot end of the host fluid because of the temperature dependence of the interfacial energy of the droplet. This thermocapillary migration effect has been exploited in the design of a controllable heat valve which is the thermal analog of an electronic vacuum triode. Further theoretical studies are planned that take into account the effect on thermocapillary migration of a gradient in chemical composition of the host fluid.

Publications

Schmid, L. A., "Use of Thermocapillary Migration in a Controllable Heat Valve," J. Appl. Phys. 53, 9204 (December 1982).

ME-011

Modeling Directional Solidification

Clarkson College of Technology
Dr. W. R. Wilcox
Dr. T. Papatheodorou
NAS8-34891 Total Cost: $240K(approx.)
May 1982 - May 1985

The objective of the research is to develop tools of use in explaining results of directional solidification in space. These tools will both be mathematical models and experimental models. Secondarily we want to design and to fly experiment at low g.

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

Low melting transparent analogs will be found for the following: One that does not wet the glass ampoule to see exactly what happens to cause ingot diameters to be less than ampoule diameters when solidification is performed in space; A eutectic that forms fibers similar to those of the MnBi-Bi eutectic to help explain why the fibers are smaller than space processing; Off-eutectic melts will be used to see if cooperative solidification really will occur more readily in space; A complete solid solubility binary system will be used to see why fewer grain boundaries are formed in space; A twin-forming material will be used to see why twin structures are so different in space-processed ingots. Thus far we are experimenting on possible analogs for the MnBi-Bi eutectic and for twin forming.

A numerical model is being developed for the Bridgman-Stockbarger technique. It will include buoyancy convective heat transfer for large Prandtl number melts and prediction of the solid-liquid interface shape. To refine this theoretical model, experiments are being performed on naphthalene. Temperatures, interface shapes, and convective velocities are being measured.

ME-012

Study of Eutectic Formation

Clarkson College
Dr. W. R. Wilcox
NAS8-34887 Total Cost: $65K
May 1982 - May 1984

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

A computer program has been developed which computes the concentration field in the melt in front of a plane-front lamellar eutectic with a linear velocity field present in the melt. Increasing the convection increases the extremum value of the lamellar spacing. Results are being compared with experiment and with prior theoretical predictions.

A literature survey has been performed on methods for revealing longitudinal microstructure of MnBi-Bi eutectic. Preferential etching of the Bu does not appear possible. Certainly all prior attempts have failed. Therefore we are planning to try two techniques: slow dissolution, either chemical or electrochemical, under the microscope with time-lapse video recording; fracture at elevated temperature, which hopefully will pull the MnBi fibers from the softer Bi matrix.

3. FLUIDS, TRANSPORT, AND CHEMICAL PROCESSES

TASK NUMBER	PRINCIPAL INVESTIGATOR	SHORT TITLE
FL-001	Bier	Hormone Purification
FL-002	Brooks	Countercurrent Distribution of Cells
FL-003	Cokelet	Blood Flow in Small Vessels
FL-004	Davis	Thermocapillary Flow and Their Stability
FL-005	Dintenfass	Aggregation of Red Cells
FL-006	Dressler	Transient Thermal Convection in Low-g
FL-007	Giarratano	Transient Heat Transfer in Zero Gravity
FL-008	Greenspan	Fluid Dynamics of Crystal Melts
FL-009	Hardy	Surface Tension and Their Variations
FL-010	Harris	New Polymers for Low-Gravity Purification
FL-011	Hymer	Purification and Cultivation of Pituitary
FL-012	Moldover	Experimental & Theoretical Studies in Wetting
FL-013	Morrison	Biosynthesis/Separation Laboratory
FL-014	Riley	Mass Transfer in Electrolyte Systems
FL-015	Saville	Mathematical Models of CF Electrophoresis
FL-016	Snyder	Electrophoresis Technology
FL-017	Spradley	Fluid Dynamics Numerical Analysis
FL-018	Stroud	Theoretical Studies of Surface Tension
FL-019	Subramanian	Physical Phenomena in Containerless Glass
FL-020	Todd	Kidney Cell Electrophoresis
FL-021	Vanderhoff	Production of Monodisperse Latexes

INTRODUCTION

Fluid mechanics are critical to nearly all material processes since at some point in the process, the materials exist in either the liquid or gaseous state and are, therefore, subject to gravitational disturbances. The MPS Program has undertaken to analyze the processes and to develop appropriate theoretical and mathematical models for both the one-g and low-g aspects once such understanding is imperative to understanding the Earth-based property limits and the viability of low-g experimentation. The development of adequate mathematical models (at least for simple materials) is especially important since many, if not most, commercial material processes have been developed empirically over long periods of time and often involve such complex mixtures and combinations of materials that they defy analysis of the reactions and interactions taking place. Low-g offers an opportunity to isolate one of the major variables in understanding these processes.

Chemical processes are being studied to elucidate the effects of gravity in processes where particle size and geometry may affect the chemical reaction kinetics. Currently, the MPS program is investigating the reaction kinetics of polymers to understand and, perhaps, overcome the current commerical size limitations in producing uniform, microscopic particles for applications such as blood cells counter and electron microscopic calibration, calibration of pore sizes in living or other membranes, and for tagging biological materials. Under one-g, as particle size is increased, they tend to aggregate and sediment. An early low-g flight experiment may provide valuable information on chemical process controls applicable to the field of polymer chemistry.

Bioseparation technology is being addressed because Earth-based techniques for producing high purity materials in significant quantities from complex biological mixtures are adversely affected by gravity. In the gravity-free environment of space, separation techniques that are based on electric fields and biological material surface characteristics become highly efficient. Furthermore, such separation techniques are inherently gentle and do not damage or destroy living cells or material. The focus of the MPS program is in developing the technology for separation techniques such as electrophoresis, isoelectric focusing, and phase partitioning.

FL-001

Hormone Purification by Isoelectric Focusing in Space

University of Arizona - Tucson
Dr. Milan Bier
NAS8-32950 $85K/year
March 1978 - March 1982

The objectives of this research are to study the effects of gravity on the isoelectric focusing process, define and produce a definite isoelectric focusing experiment, and to refine future isoelectric focusing technology.

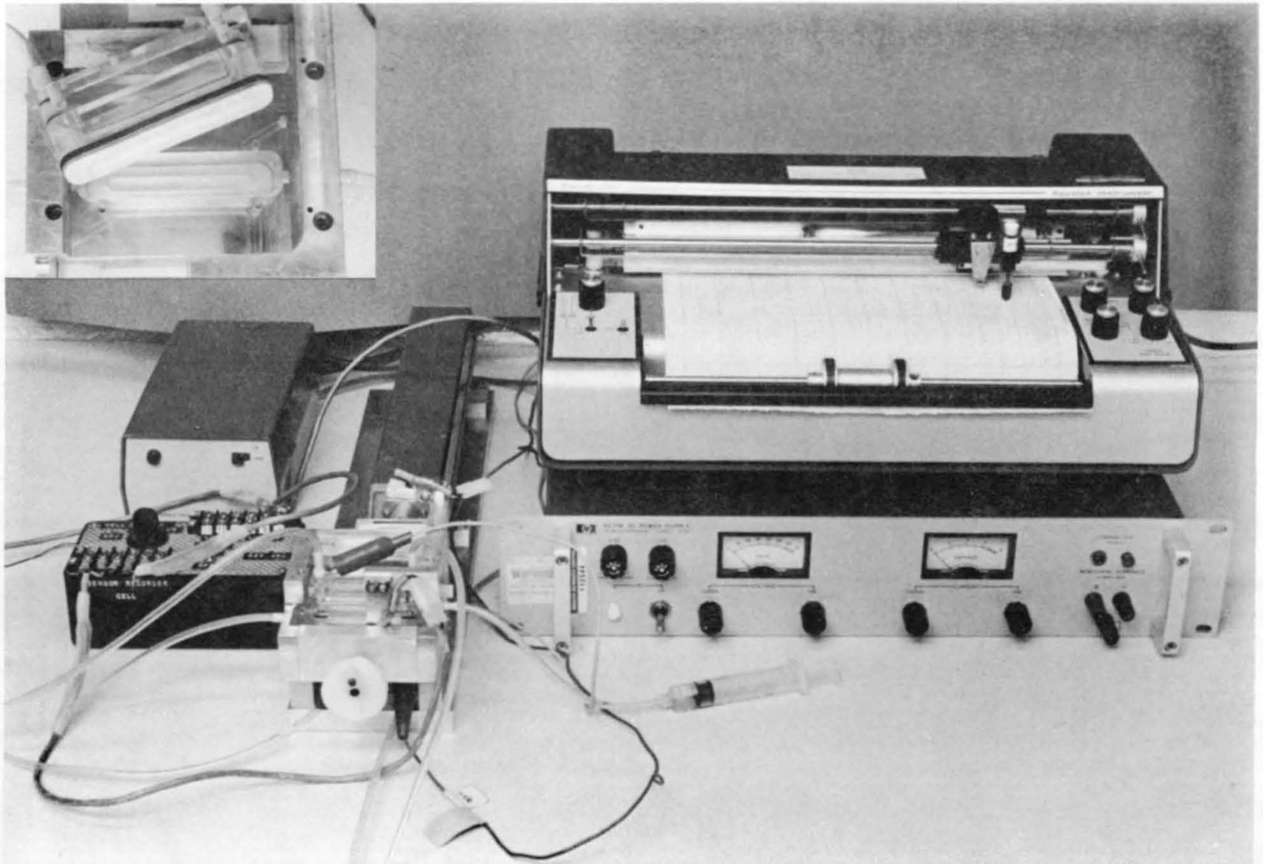
Various ground-based research approaches will be applied to a more definitive evaluation of the natures and degrees of electroosmosis effects on the separation capabilities of the IEF process. A primary instrumental system for this work will involve rotationally stabilized, horizontal electrophoretic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

Utilizing the results from the previous performance period, the pertinent activities toward developing the science samples and the payload hardware for a middeck experiment will be performed in coordination with the Marshall Space Flight Center. The objective of this flight experiment is the characterization of the effects of wall electroosmosis on the IEF process in low-gravity. These will be compared against the outputs of the computer simulations to verify and/or improve the computer simulation capabilities. Specifically, this task involves the complete definition, design, fabrication, assembly, testing, and delivery project activity sequence for the engineering model IEF cells (or columns) for the flight payload IEF cells, and the photographic breadboard subassembly of the flight payload hardware. In addition, this task covers participation in the integrated systems development and verification testing area. Finally, this task covers the full responsibility for the development and delivery of the ground-based control and flight materials samples for the first mission accommodation.

Another task will continue the optimization of the existing computer model of IEF processes to automatically adjust concentration of all of all components so as to produce linear pH gradients. The pH gradient in a given buffer system is a function of the electrochemical parameters of all components (e.g., their disassociation constants and mobilities), their concentrations, and the applied currents. A strategy for the optimization of the concentrations, based on the use of a pattern search technique implemented in the logic block of the Dare P Program (for steady state focusing), will be expanded and emphasized. Other potential improvements will also be evaluated as appropriate.

Publications

Palusinski, O. A., Bier, M., and Saville, D. A., "Mathematical Model for Transient Isoelectric Focusing of Simple Ampholytes," ready for submission.



Electric field-driven phase separation apparatus, showing laser source of beam used to follow phase separation turbidimetrically. Chamber is shown disassembled in inset.

FL-002

<u>Countercurrent Distribution of Biological Cells</u>

University of Oregon Health Sciences Center
Dr. D. E. Brooks
NAS8-33575 Total Cost: $226K
November 16, 1979 - April 1983

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

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

Direct studies on field-induced phase separation are also being carried out using the apparatus in the photo. We have been following phase separation in an electric field turbidimetrically. It has been found that phase separation, as indicated by optical clearing, occurs rapidly under the influence of a modest electric field, but that turbidity then reappears after a few minutes. By isolating the upper and lower halves of the sample chamber at different times after mixing, in the presence or absence of an electric field, we have found that most of the phase separation occurs before a large change in turbidity is detected, however, implying that the optical signal is dominated by the haze of small drops left behind after the bulk of the phase volumes have separated. The direct sampling experiments have demonstrated unequivocally, however, that low electric fields (~ 0.5 v cm^{-1}) enhance the rate at which the phases separate, even in the presence of unit gravity.

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

Publications

Bamberger, S., Seaman, G.V.F., Brown, J. A., and Brooks, D. E., "The Partition of Sodium Phosphate and Sodium Chlorate in Aqueous Dextran Poly(ethylene glycol) Two Phase Systems," submitted for publication.

Bamberger, S., Seaman, G.V.F., Sharp, K. A., and Brooks, D. E., "The Effects of Salt on the Interfacial Tension of Aqueous Dextran Poly (ethylene glycol) Phase Systems," submitted for publication.

Brooks, D. E., Van Alstine, J., Sharp, K. A., and Bamberger, S., "Thermodynamic and Non-thermodynamic Factors in the Partition of Cells in Two Polymer Phase Systems," submitted for publication.

Brooks, D. E., Bamberger, S., Sharp, K. A., Tamblyn, C. H., Seaman, G.V.F., and Walter, H., "Electrostatic and Electrokinetic Potentials in Two Polymer Aqueous Phase Systems," submitted for publication.

FL-003

Blood Flow in Small Vessels

University of Rochester
Dr. G. R. Cokelet
Dr. H. Meiselman (USC)
Dr. H. Goldsmith (Montreal General Hospital)
NAS8-34892 Total Cost: $360K
October 1982 - July 1985

Blood, a typical near-colloidal suspension, consists of particles (cells) dispersed in an aqueous solution (plasma). During flow under low shear stresses the red cells can aggregate, resulting in increaseds blood viscosity, increased red cell sedimentation and red cell syneresis. In a living body, the increased viscosity and red cell syneresis may be significant, affecting the heart work needed for blood circulation, the distribution of red cell fluxes in tissue, etc. Attempts to study these phenomena with blood from healthy and unhealthy individual in *in vitro* experiments under unit gravity are confounded by problems due to cell sedimentation in the long flow channels required in the experimental studies (but not generally found *in vivo*). Performance of these experiments under zero gravity would remove this confounding effect and permit acquisition of meaningful data. The objective of this program is to obtain ground-based data for establishment of need for flight experiments to determine potential flight experiment variable ranges. Suitable flow sections, cell suspensions and methods for obtaining the necessary data will be developed.

From quantitative data of pressures, flow rates, cell concentrations and vessel variables, as well as video recordings of microscopic views of the flow of selected cell suspensions through single small tubes, vessel bifurcations and small networks of small vessels, it will be possible to determine the effect of red cell aggregation (and changes in the tendency for aggregation) on the flow of blood in the microcirculation. Mathematical models of the flow can be tested. The results will be of general applicability to the understanding of flow of colloidal suspensions.

The task is divided into three parts: (1) preparation and characterization of red cell suspensions (including properties of the red cell such as deformability, shape and tendency for aggregation). The time-stability of the cells in suspension must be optimized, (2) development of aggregable red cell ghost suspensions and experimental development of the methods for measuring microrheological parameters of blood cell suspension flow, and (3) production of test flow sections and the testing of potential flight experiment components.

FL-004

<u>Thermocapillary Flows and Their Stability: Effects of Surface Layers and Contamination</u>

Northwestern University
Dr. S. H. Davis
Dr. G. M. Homsy, Stanford University
NAS8-33881 Total Cost: $400K
June 1980 - June 1983

The proposed research concerns the theoretical analysis of the fluid mechanics and heat transfer of motions driven by surface-tension gradients. The object is an understanding of the convection accompanying the process of growing high-quality crystals in a 1-g environment. The geometries considered included thin films, deep films and float-zone configurations. The particular aspects addressed are (1) the effects on steady Marangoni flow of contamination and the placement of third-phase films on the melt-gas interface, (2) the prediction of possible instabilities of Marangoni flows of pure melts, and (3) the effects on such instability criteria of contamination and surface films.

Work has been completed on several uncontaminated thin-film flows. These include the steady flow due to differential heating of a cavity and the instability characteristics of such flows. It is found that for small Prandtl numbers purely mechanical instabilities occur while for large Prandtl numbers, thermal instabilities dominate. In all the above analysis, the flows, the heat transfer, and the free surface shapes are simultaneously obtained.

Publications

Sen, A. K. and Davis, S. H., "Steady Thermocapillary Flows in Two-Dimensional Slots," <u>J. Fluid Mech</u>. <u>121</u>, 163-186 (1982).

Smith, M. K. and Davis, S. H., "Instability of Sheared Liquid Layers," <u>J. Fluid Mech.</u> <u>121</u>, 187-206 (1982).

Smith, M. K. and Davis, S. H., "Instabilities of Dynamic Thermocapillary Liquid Layers. Part I. Convective Instabilities," to appear in <u>Journal of Fluid Mechanics</u>, 1983.

Smith, M. K. and Davis, S. H., "Instabilities of Dynamic Thermocapillary Liquid Layers. Part 2. Surface-wave Instabilities," to appear in <u>Journal of Fluid Mechanics</u>, 1983.

Xu, J-J. and Davis, S. H., "Liquid Bridges with Thermocapillarity," 1983 (pending publication).

Cowley, S. J. and Davis, S. H., "Viscous Thermocapillary Convection at High Marangoni Number," 1983 (pending publication).

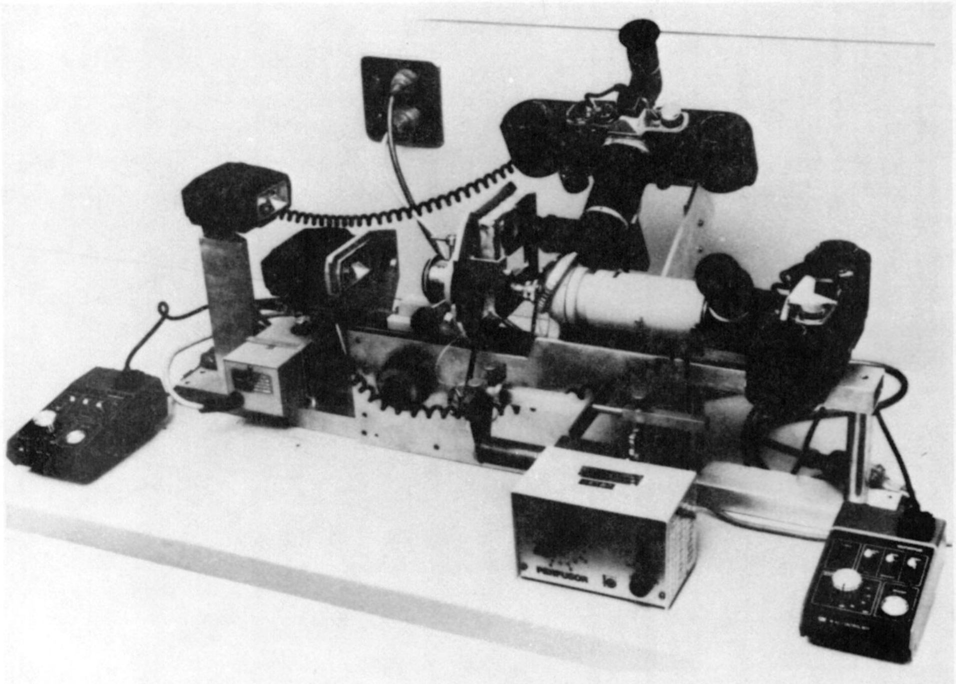
Homsy, G. M. and Meiberg, E., "The Effect of Surface Contamination on Thermocapillary Flow in a Two-dimensional Slot," 1983 (pending publication).

Presentations

Smith, M. K. and Davis, S. H., "The Convective Instabilities of Thermocapillary Shear Layers," Annual Meeting AIChE, Los Angeles, 1982.

Smith, M. K. and Davis, S. H., "The Convective Instabilities of Thermocapillary Shear Layers," Annual Meeting Division of Fluid Dynamics, American Physical Society, Rutgers University, 1982.

Xu, J-J. and Davis, S. H., "The Convective Instability of Cylindrical Liquid Bridges with Thermocapillarity," Annual Meeting Division of Fluid Dynamics, American Physical Society, Rutgers University, 1982.



The Parallel-Plate Slit-Capillary Viscometer, equipped with two Olympus cameras, two flash guns, and Zeiss Microscope. In front is Braun motorized syringe; left and right, two Olympus MAC controllers; the very centre of instrument is occupied by a holder containing glass plates (polished to one-quarter of light wavelengths accuracy, and made of Crown glass). This is the laboratory model used in Sydney. Each plate contains a semicircular channel of 0.2 cm diameter for distribution or collection of blood which flows like a sheet between the plates. Photography of the blood is carried out automatically and sequentially by means of photocameras. The objective is detached from the microscope and is permanently set into a well cut in one of the glass plates. Sequential exposures and sequential flow, no flow conditions are controlled by two microprocessors. Real time, temperature and pressure drop are recorded on the solid phase memory.

FL-005

Aggregation of Red Cells

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

The objectives of this program are: (1) to determine whether the size of red cell aggregates, kinetics and morphology of these aggregates are influenced by near-zero gravity, (2) whether viscosity, especially at low shear rate, is afflicted by near-zero gravity (the latter preventing sedimentation of red cells), (3) whether the actual shape of red cells changes, and (4) whether blood samples obtained from different donors (normal and patients suffering from different disorders) react in the same manner to near-zero gravity. These are objectives for the first Spacelab mission. Subsequent orbital flights intend to elaborate this data and introduce effects of plasma proteins, lipids, drugs, and various agents, in order to develop new diagnostic techniques and to obtain better insight into molecular aspects of blood rheology. These subsequent missions will depend in their construction on information obtained from the first flight.

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

It should be particularly noted that the studies described above are not intended as qualitative or descriptive studies solely; by application of stereological methods, and consequently possible statistical methods, we can define kinetics and morphology of aggregation of red cells in a quantitative manner; that, we can ascribe numbers ('d(Heyn)', 'Lamda', etc.) to specific aggregates at any stage of developmental organization. It is possible to differentiate between kinetics of different blood samples using statistical methods based on stereological evaluations; highly significant differences are observed using either students t-test or comparison of slopes or elevation of linear regression of stereological parameters plotted against time of stasis.

Publications

Dintenfass, L., "Haemorheology of Raynaud's Phenomenon," Adv. in Microcirculation 10, 60-72 (1982).

Dintenfass, L., "Haemorheology of Cancer Metastases: An Example of Malignant Melanoma, Survival Times and Abnormality of Blood Viscosity Factors," Clinical Hemorheology 2, 259-271 (1982).

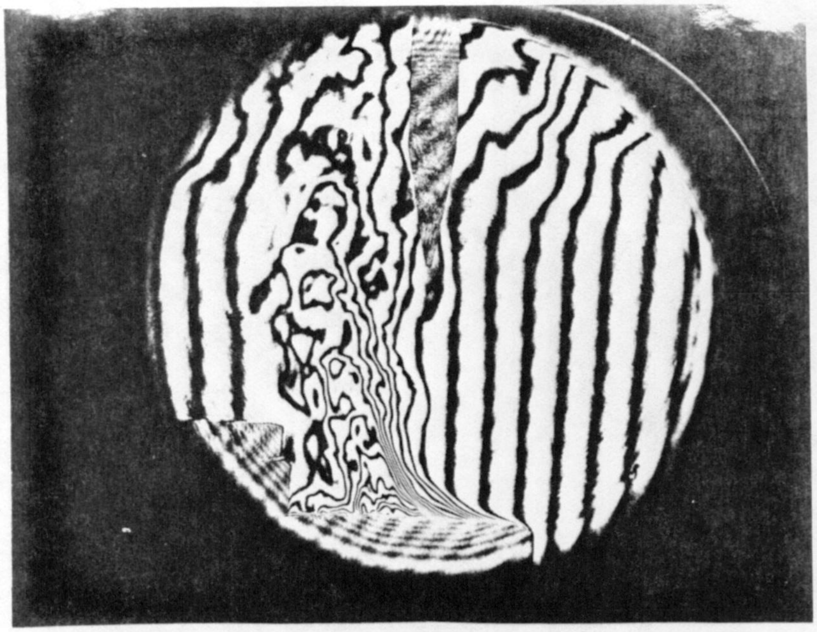
Dintenfass, L., Jedrzejczyk, H., and Willard, A., "Photographic, Stereological and Statistical Methods in Evaluation of Aggregation of Red Cells in Disease: Part I: Kinetics of Aggregation," Biorheology 19, 567-577 (1982).

FL-006

Transient Thermal Convection in Low-g

NASA Headquarters
Dr. R. F. Dressler
In-House
January 1980 - continuing task

The purpose of this research program is to obtain analytical solutions for transient and periodic convection flows for arbitrary low-g excitations with imposed thermal gradient in cylinders and cubes, for both 2-D and 3-D flows.



Typical interferogram during or subsequent to a heat pulse to Freon 13 near its thermodynamic critical point.

FL-007

Transient Heat Transfer in Zero Gravity Environment

National Bureau of Standards - Boulder, Colorado
Dr. P. J. Giarrantano
H-27954B

In order to develop improved models and correlations for transient heat transfer to compressible fluids, it is important to understand the mechanisms of heat transfer throughout the various stages of the transient. Subsequent to the initial pure conduction period of heat transfer, convective motions come into play. Thermally induced motions normal to the heater surface (due to expansion of the heated layer of fluid next to the surface) may be masked by the natural convective motion induced by the buoyancy (gravity) forces acting on fluid particles of variable density. In zero-g environment, the influence of buoyancy forces is eliminated and the effect of thermally-induced motions may be more readily studied. Therefore, experimental measurements are being made in earth gravity and the measurements will be repeated during KC-135 jet airplane parabolic flight, which simulate zero-g environment. Optical techniques will be employed in the measurements (Mach-Zender interferometer) and the resulting interferograms recorded with a high-speed, 16-mm movie camera.

The time-dependent, two-dimensional temperature field in the test fluid above the platinum film heater is measured during a heat pulse of known duration. A signal generator provides the square wave voltage signal which is amplified by a differential operational power supply and fed to the electrodes of the platinum film. As previously noted, the data are recorded both interferometrically and electrically. Subsequent analysis of the interferograms provides the temperature field as well as the heat flux to the fluid. The heat flux is determined by the temperature gradient at the heater surface and the thermal conductivity of Freon 13. The mechanism of pure conduction is presumed to prevail in the immediate vicinity of the heater surface throughout the heat pulse.

Time-dependent temperature fields in the earth's gravity will be measured for a range of applied power levels and the interferograms recorded on 16-mm film. It is anticipated that the experments will be flown on KC-135 flights, scheduled for March, July, and September, and the measurements will be repeated in the low-gravity environment.

FL-008

Fluid Dynamics of Crystal Melts Under Reduced Gravity

Massachusetts Institute of Technology
Dr. Harvey P. Greenspan
Dr. Moshe Israeli
NAS8-35412 $36K/yr.
December 3, 1982 - December 2, 1984

The successful growth and purification of crystals from melts under reduced gravity will require knowledge about the convective flows produced by small coupled forces. This research proposal will focus on a few of the problems in which the effects of rotation are of consequence. The primary problem to be examined concerns the nature and magnitude of the convective flow in the melt under practical operating conditions.

The objectives of theory are to analyze the various, coupled mechanisms and their effects on: (1) the shape of the melt domain, i.e., the solid boundaries and free surfaces, (2) the magnitude of the convective velocity induced by variable surface tension, and as modified by rotation and/or the deposition and desorption of surfactants and, (3) the overall temperature distribution.

FL-009

Surface Tensions and Their Variations with Temperature and Impurities

National Bureau of Standards
S. C. Hardy
H-27954B $190K/year
April 1981 - continuing task

Surface tension gradients at free surfaces of liquid due to temperature or concentration variations can generate fluid flows. In low gravity these can be significant and can influence the temperature and solute fields in the bulk liquid. Consequently, in materials processing in space, knowledge of these surface tension gradients can be essential to the understanding of the observed phenomena. The temperature and chemical concentration dependences of the surface tensions of most materials are poorly known and many basic questions about processes such as segregation at liquid surfaces are as yet unanswered. In this program, traditional sessile drop surface tension measurements are being used in conjunction with Auger spectroscopy and other modern surface analytcial techniques to study the thermodynamics and chemistry of liquid metal and semiconductor surfaces.

At present we are measuring the surface tension of liquid silicon as a function of temperature. Liquid silicon is extremely active chemically and has been found to dissolve or react somewhat with all solids. For this reason the sessile drop samples may acquire surface active impurities from the substrate which will affect the measured surface tension values. In addition, very low partial pressure of oxygen are known to strongly depress the surface tension of silicon. Consequently extensive measurements using various substrate materials must be made both in vacuum and in atmospheres containing known amounts of oxygen and hydrogen to study these dependences. Auger spectroscopy will be used to determine the composition of the samples after solidification.

Publications

Hardy, S. C. and Fine, J., "Surface Segregation in Liquid Ga-Sn Alloys by AES," J. Vac. Sci. Tech., in press.

FL-010

New Polymers for Low-Gravity Purification of Cells by Phase Partitioning

University of Alabama in Huntsville
Dr. J. Milton Harris
NAS8-33978 Total Cost: $102K
September 1, 1980 - June 1, 1983

A potentially powerful technique for separating different biological cell types is based on the partitioning of these cells between the immiscible aqueous phases formed by solution of certain polymers in water. This process is gravity-limited because cells sediment rather than associate with the phase most favored on the basis of cell-phase interactions. We are presently involved in the synthesis of new polymers both to aid in understanding the partitioning process and to improve the quality of separations. The prime driving force behind the design of these polymers is to produce materials which will aid in space experiments to separate important cell types and to study the partitioning process in the absence of gravity (i.e., in an equilibrium state).

Several new polymer derivatives have been prepared: (1) polyethylene glycols with attached crown ethers; (2) polyethylene glycols (PEG's) with attached long-chain hydrocarbons; (3) PEG's with attached proteins (lectins in particular); (4) PEG attached to glass surfaces; and (5) dextrans with attached long-chain hydrocarbons. All of these materials are of interest because of their ability to interact with cell surfaces. In addition, #5 should be useful in separating the immiscible phases formed upon solution of PEG and dextran in water. This property will be of use in phase-partitioning experiments in a reduced-gravity environment. An interesting spin-off of this synthetic work has been the observation of catalytic activity for the crown polymers. Work has also continued with testing of the Ito apparatus for automated phase partitioning.

Publications

Harris, J. M., Hundley, N. H., Shannon, T. G., and Struck, E. C., "Substituted Polyethylene Glycols as Soluble, Recoverable Phase-Transfer Catalysts," J. Org. Chem. 47, 4789 (1982).

Harris, J. M., Hundley, N. H., Shannon, T. G., and Struck, E. C., "Substituted Polyethylene Glycols as Soluable, Recoverable Phase-Transfer Catalysts," Polymer Preprints 23, 193 (1982).

Harris, J. M., Hundley, N. H., Shannon, T. G., and Struck, E. C., "Polyethylene Glycols as Phase-Transfer Catalysts. Synthesis of Derivatives," in Crown Ethers and Phase Transfer Catalysts in Polymer Science (C. E. Carreher and L. Mathias, eds.), Plenum, in press.

Presentations

Harris, J. M. and Chenault, A. A., "Countercurrent Partitioning of Mammalian Cells in the Toroidal Coil Chromatograph," presented at Second International Conference on Partitioning, Sheffield, England, 1981.

Harris, J. M. and Hipps, J. M., "Synthesis of New Derivatives of Polyethylene Glycol," presented at Southeastern Regional ACS Meeting, Birmingham, Alabama, 1982.

Harris, J. M. and Case, M. C., "Octadecyl Ethers of Polyethylene Glycols," presented at Southeastern Regional ACS Meeting, Birmingham, Alabama, 1982.

FL-011

Purification and Cultivation of Human Pituitary Growth Hormone-Secreting Cells

Pennsylvania State University
Dr. W. C. Hymer
NAS9-15566
June 1982 - June 1983

Human growth hormone (hGH) is in demand for treatment of pituitary disease, osteoporosis, etc. While hGH produced by recombinant DNA technology will be widely used in future therapeutic protocols, accumulating evidence shows that the GH molecule exists in many forms within pituitary cells. Our program addresses the problem of (a) electrophoretic separation of various GH producing cell types, (b) electrophoretic separation of human post-mortem secretory granules containing hGH, and (c) analysis of the biological and immunological activities of the stored/secreted hormone.

Results from many experiments show that somatotrophs can be enriched (purified) by continuous flow electrophoresis and density gradient electrophoresis. Other data show that some of these GH cells release hormone which has 5X more activity as measured by bioassay than by immunoassay. Attempts are being made to separate the multiple forms of GH by HPLC. Finally, methods have been developed to assess activity of these separated GH cells in the hypophysectomized rat. These cells release hormone which is capable of restoring muscle mass of the hypophysectomized rat.

Publications

Hymer, W. C., et al., "Hollow Fibers: Their Application to the Study of Mammalian Cell Function," in Regulation of Target Cell Responsiveness (K. McKerns, ed.), Plenum Press, 1983, in press.

Hymer, W. C. and Hatfield, J., "Separation of Pituitary Cells," in Cell Separation: Methods and Selected Applications (T. G. Pretlow and T. Pretlow, eds.), Academic Press, 1983, in press.

Hymer, W. C. and Hatfield, J., "Purification of Cells from the Anterior Pituitary," in Methods in Enzymology (Colowick and Kaplan, eds.), Academic Press, 1983, in press.

Plank, L. , Kunze, E., Lanham, P., Todd, P., and Hymer, W. C., "Pituitary Cell Separations by Electrophoresis," 1983, in preparation.

Angeline, M. and Hymer, W. C., "Development of an Enzyme Linked Immunoadsorbent Assay for Growth Hormone," 1983, in preparation.

FL-012

Experimental and Theoretical Studies in Wetting and Multilayer Adsorption

National Bureau of Standards
Dr. M. R. Moldover
Dr. J. W. Schmidt
Dr. J. W. Cahn
H-27954B
April 1981 - continuing task

The aim of this task is to measure the thickness of the liquid-vapor interface above single phase mixtures of isopropanol-perfluoro-methylcyclohexane. The temperature of the wetting transition T_w and the critical mixing temperature T_c have already been located for two-phase mixtures of these components. We expect to find a percursor to the wetting transition in the single phase mixtures. In preliminary measurements we have characterized this mixture very well. The densities of both phases are known at 23°C; the indices of refraction of both phases were measured as well as their temperature derivatives; and the capillary rises of both the liquid-vapor and liquid-liquid interfaces were measured above and below T_w. At the present time our highest priority is to measure the film thickness between T_w and T_c and to determine its temperature dependence.

An automatic ellipsometer has been assembled together with sample cells and a thermostat capable of precisely monitoring the ellipsometric parameters of the liquid-vapor interface above binary liquid mixtures. A computer model of the ellipsometer has been made in order to optimize its sensitivity to the appearance of the predicted high adsorption layer. A method of measuring and recording the index of refraction of the binary mixture under study simultaneously with the recording of the ellipsometric parameters has been tested. The index of refraction measurement is sensitive to the appearance of macroscopic amounts of a second liquid phase; thus it will serve to locate the adsorption transition with respect to the miscibility gap in the specific liquid sample under study. It is expected that the strategy of simultaneously measuring the index of refraction and the ellipsometric parameters will obviate the necssity of carrying out detailed exacting chemical analyses of each sample of each mixture to be studied.

Publications

Schmidt, J. W. and Moldover, M. R., "First-Order Wetting Transition at a Liquid-Vapor Interface," submitted to Journal of Chemical Physics, 1983.

FL-013

<u>Biosynthesis/Separations Laboratory-Development of a Space Biosynthesis System and Biological Studies for Electrophoresis in Space</u>

Johnson Space Center
Dr. Dennis R. Morrison
Mr. Bernard J. Mieszkuc
In-Center $120K/year
January 1981 - continuing task

The objectives of this program are to: (1) obtain data on the performance of cell culture vessel system elements and to define the biological oxidation process--the transfer of oxygen from gas to liquid and from liquid to oxidant, and (2) determine the limits of ground-based technology using a preprototype reactor for studying enzymatic reactions and suspension cell cultures.

The Biosynthesis/Separations Laboratory supports the Materials Processing in Space studies on biosynthesis and cell separations for investigations into the production of high value pharmaceuticals which are very difficult or impossible to obtain on Earth with currently available technology. This laboratory is responsible for the biological science supporting the Electrophoresis Equipment Verification Test scheduled to fly on STS-3 and subsequent electrophoresis experiments under microgravity conditions.

The laboratory has both monolayer and suspension cell culture capabilities. Current research includes procedures for the obtaining of cell cultures, and the freezing and storage of cells. Procedures for growing cell cultures in suspension are being investigated. A continuous line of baby hamster kidney cells has been grown in suspension, and the growth of cells on microcarriers is being pursued. A variety of beads were used as substrates for the attachment of cells. Procedures for the analysis of biochemicals produced by cell cultures have been established. Fibrinolytic and colormetric methods are being used routinely for the assay of urokinase. The production of urokinase in monolayers of human embryonic kidney cells has been demonstrated. Biochemical purification of secreted products on affinity columns is being developed. Procedures for the chromosome analysis of cell cultures (counting and karyotyping) have been established. Continuing efforts include: (1) the design, construction, verification testing, and flight test of a small space bioreactor to demonstrate the advantages of these new techniques using mammalian cells in culture, (2) the identification of requirements and hardware design concepts for small cell incubators needed to maintain living cells on board Shuttle or Spacelab before and after continuous flow electrophoresis experiments in conjunction with the NASA Joint Endeavor Agreement with McDonnell Douglas Astronautics Corporation, and (3) the screening and selection of living cells to be separated in future space flight experiments.

FL-014

<u>Mass Transfer in Electrolytic Systems Under Low Gravity Conditions</u>

University of Alabama in Huntsville
Dr. C. Riley
Dr. H. D. Coble
Dr. R. B. Owens, MSFC
Gordon Fisher, INCO
NAS8-33542 $33K (with a no-cost extension)
September 1979 - June 1983

Electrodeposition involves mass transfer from one phase to another. In particular one is concerned with the deposition of materials into the solid phase out of the liquid (solution or suspension) phase. Diffusive and convective flow coupled with deposition result in density gradients that are gravity dependent. We desire to characterize this gravity dependence associated with electrodeposition. When a better understanding of the gravity influence is acquired, it should lead to improved control of variables during electroformation of materials. The overall objectives will be the electroformation of materials with improved or more desirable properties and a better understanding of the transport of inert suspensions during the electrodeposition process.

Electrodeposition cells are being utilized to study simple metal-in metal-out reactions using cobalt and copper. The density flow patterns between electrodes with both a vertical and horizontal configuration are being bench characterized using interferometry detection. These results are being compared to those determined for the same cells under reduced gravity conditions ($\sim 10^{-2}$g) produced during parabolic, free-fall flights of a KC-135 aircraft. A special vibration free interferometer has been developed to monitor flow during these flights. Studies with neutral buoyancy particles will be used to model the transport of neutrals under low gravity conditions.

FL-015

Mathematical Models of Continuous Flow Electrophoresis

Princeton University
Dr. D. A. Saville
Dr. R. S. Snyder, MSFC
NAS8-32614 Total Cost: $270K (approx.)
August 1977 - February 1983

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

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

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

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

Publications

Saville, D. A., "The Sedimentation Potential in Dilute Suspensions," Adv. Colloid Interface Sci. 16, 267-279 (1982).

Saville, D. A., "The Electrical Conductivity of Suspensions of Charged Particles in Ionic Solutions: The Role of Counterions Arising from the Dissociation of Surface Groups," J. Colloid Interface Science, in press.

Saville, D. A., "Electrohydrodynamics and other Hydrodynamic Phenomena in Electrophoresis," in Proc. Ninth U.S. National Congress of Applied Mechanics, New York: American Society of Mechanical Engineers, 1982, pp. 395-400.

Presentations

Saville, D. A. and Zukowski, C. F., "The Electrical Conductivity of Dilute Suspensions," 184th National Meeting, ACS, Kansas City, Missouri, September 1982.

Saville, D. A. and Zukoski, C. F., "Studies in Electrohydrodynamics: Electro-osmotic Flow in a Rectangle," 1982 Annual Meeting, AIChE, Los Angeles, November 1982.

FL-016

Electrophoresis Technology

Marshall Space Flight Center
Dr. R. S. Snyder
In-House $81K/year

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, and (3) separate biological cells using apparatus that has been characterized or modified to perform in a predictable manner and according to procedures that have been developed to yield improved separation.

The following results have been accomplished: (1) experiments have been designed to decouple or minimize the fluid effects due to the flow process, electrokinetic effects, and temperature gradients; (2) transparent electrophoresis chambers have been built allowing measurement of internal and wall temperature while observing flow perturbations; (3) techniques have been developed to map the temperature and flow fields in the chamber with small disturbance to the process; (4) the sensitivity of these chambers to lateral temperature gradients has been measured and a new, all-metal chamber has been designed to incorporate the improvements suggested by these experiments; (5) analysis has yielded results that reproduce flow distortions observed in experimental chambers; (6) the DESAGA FF48 and Beckman continuous flow electrophoresis chambers have been compared using standard particles (fixed red blood cells) under various operating conditions and optimum operating parameters for resolution and throughput have been established; (7) experiments have been done in the McDonnell Douglas Continuous Flow Electrophoresis Systems (CFES) to identify the importance of the primary operating parameters of concentration, conductivity and electroosmosis; and (8) ground-based experiments are continuing on the impact of buoyancy-induced convection on electroosmosis.

Publications

Dunning, J. D., Herren, B. J., Tipps, R. W., and Snyder, R. S., "Fractionation of Mineral Species by Electrophoresis," J. Geophys. Res. 87 (B13), 10,781-10,788 (1982).

Omenyi, S. N. and Snyder, R. S., "Settling of Fixed Erythrocyte Suspension Droplets," accepted for publication in Biorheology, 1983.

FL-017

Fluid Dynamics Numerical Analysis

Lockheed Missiles & Space Company
Dr. S. J. Robertson
Dr. L. W. Spradley
NASW-3281 Total Cost: $300K
August 1979 - December 1983

The purpose of this research program is to compute transient thermal convection for cases of importance to Materials Processing in Space. This includes problems too difficult for analytical solutions and also includes verification of ranges of validity of theory developed by Dr. R. F. Dressler.

Lockheed's previously derived GIM code has been modified and adapted for these tasks. Work was completed on two-dimensional transients for stepfunctions for circles and squares. Ranges of validity of Rayleigh number for the Dressler theory have been determined. Axisymmetric transient convection in a sphere idealizing the Lal experiment has been completed. A study has been completed of the effect of Rayleigh accelerations applied to an initially moving fluid.

A numerical analysis was performed to compare natural convection velocities in two-dimensional enclosures of various shapes. The following shapes were investigated: circle, square, horizontal and upright 2 x 1 aspect ratio rectangles, horizontal and upright half-circles, diamond (square oriented with diagonal vertical) and triangle (equilateral with horizontal base). In all cases, the length scale in the various dimensionless parameters, such as Rayleigh number, is defined as the diameter of the equal area circle. Natural convection velocities were calculated for Rayleigh numbers of 1000 and 5000 with the temperature difference taken to be across (a) the maximum horizontal dimension, (b) the median horizontal line (line through centroid) and (c) the horizontal distance such that the temperature gradient is the same for shapes of equal area. For the class of shapes including the square, upright half-circle and upright rectangle, the computed velocities were found to agree very closely with that of the equal area circle when the temperature difference is taken to be condition a. The velocities for the horizontal rectangle and half circle were found to be approximately one-half that if the equal area circle for the same condition.

Publications

Robertson, S. J. and Nicholson, L. A., "Effect of Enclosure Shape on Natural Convection Velocities," Technical Report LMSC-HREC TR D784759, September 1982.

FL-018

Theoretical Studies of the Surface Tension of Liquid Metals

The Ohio State University
Professor D. G. Stroud
Dr. D. M. Wood
Total Cost: $70K
February 1982 - February 1984

The aim of this program is to develop a theoretical understanding of the surface tensions of liquid metals, and of their temperature and concentration derivatives. The motivation for this work is twofold: the problem is of basic interest, since a fundamental theory of surface properties of liquid metals is lacking; and it is also of much practical importance, since surface tension gradients are responsible for substantial convective flows parallel to the surface (Marangoni convection) which may predominate in a low-gravity environment.

Our method is to generalize the relatively well-established theory of bulk liquid metals to surfaces. The approach is first-principles: starting from the pseudopotential which characterizes the conduction-electron ion interaction in a metal, we use elements of the electronic theory of metals, and of classical statistical mechanics of liquids, to calculate surface tensions of pure liquid metals as a function of temperature, and the widths of the surface density profiles. So far, a simplified version of the theory has led to very good agreement with experiment for both surface tensions and surface widths of nine liquid metals. The approach has been extended to the interfacial tension between immiscible liquid metals, with an application to Li_xNa_{1-x}; and a more complete application of the theory to liquid Na is yielding encouraging results for both the surface tension and its temperature derivative. Further plans include study of other metals, possibly including liquid Ga for which detailed experimental results are available. Extension of binary systems is also planned, with an aim of understanding the influence of impurities on surface tension. It is also intended to use the results of our first principles studies to develop empirical rules which may help predict as yet unmeasured surface tensions and their derivatives.

Publications

Mon, K. K. and Stroud, D. G., "Theory of the Interfacial Tension Between Liquid Metals," Phys. Rev. B., in press.

FL-019

Physical Phenomena in Containerless Glass Processing

Clarkson College of Technology
Dr. R. S. Subramanian
Dr. Robert Cole
NAS8-32944 Total Cost: $554K (approx.)
December 1977 - December 1985

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

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

Publications

Jucha, R. B., Cole, R., Powers, D., McNeil, T., and Subramanian, R. S. "Bubble Rise in Molten Glasses," J. Amer. Ceram. Soc. 65, 437-442 (1982).

Annamalai, P., Subramanian, R. S., and Cole, R., "Bubble Migration in a Rotating Liquid-Filled Sphere," Phys. of Fluids 25, 1121-1126 (1982).

Shankar, N. and Subramanian, R. S., "The Slow Axisymmetric Thermocapillary Migration of an Eccentrically Placed Bubble Inside a Drop in a Space Laboratory," J. Colloid & Interface Sci., in press, 1983.

Meyyappan, M., Wilcox, W. R., and Subramanian, R. S., "The Slow Axisymmetric Motion of Two Bubbles in a Thermal Gradient," J. Colloid & Interface Sci., in press, 1983.

Subramanian, R. S., "Thermocapillary Migration of Bubbles and Droplets," in Space Research, Ed. Y. Malmejac, Proceedings of the Symposium on Fundamental Aspects of Material Sciences in Space, COSPAR XXIV, Ottawa, Canada, 1982.

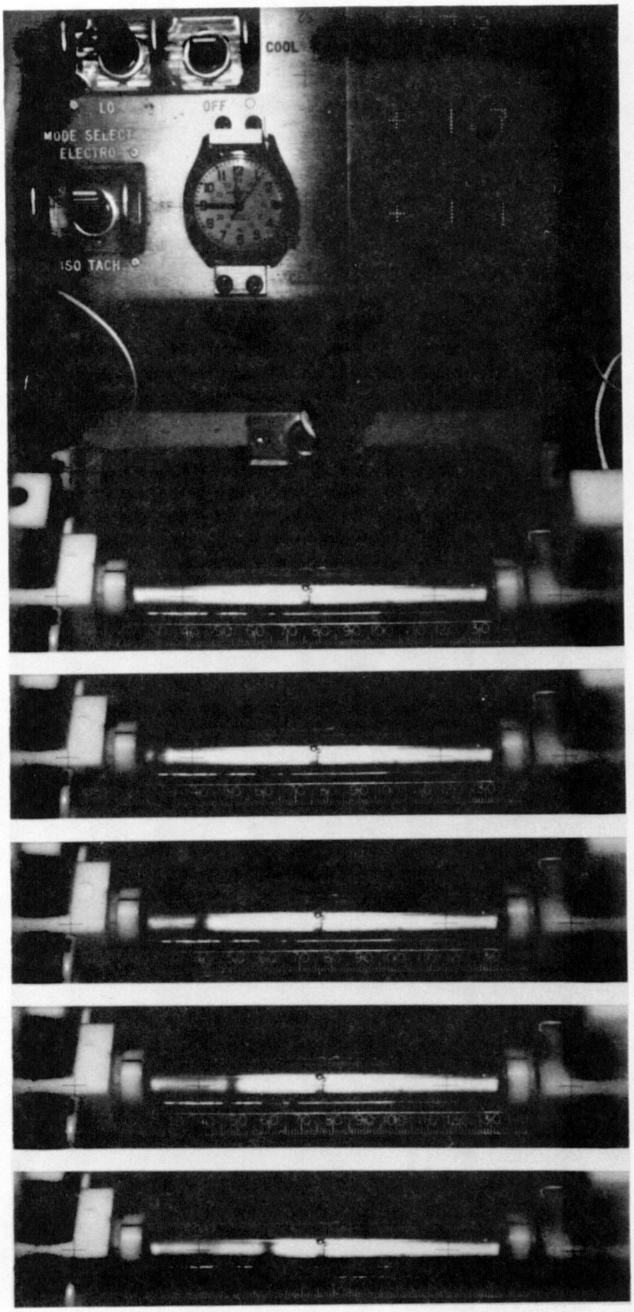
Annamalai, P. and Cole, R., "Drop Motion in a Rotating Immiscible Liquid Body," in Space Research, Ed. Y. Malmejac, Proceedings of the Symposium on Fundamental Aspects of Material Sciences in Space, COSPAR XXIV, Ottawa, Canada, 1982.

Presentations

Kondos, P. and Cole, R., "Volatilzation of Sodium Borate Melts," presented at American Ceramic Society Glass Division, Fall Meeting, Bedford Springs, Pennsylvania, October 1982.

Wiltshire, T., Shankar, N., and Subramanian, R. S., "Dissolution or Growth of a Sphere," presented at AIChE 75th Annual Meeting, Los Angeles, November 1982.

McNeil, T. J., Cole, R., and Subramanian, R. S., "Surface Tension Driven FLow in Glass Melts and Model Fluids," presented at AIChE 75th Annual Meeting, Los Angeles, November 1982.



Sequence of photographs taken at 11 minute intervals on Shuttle flight STS-3 during electrophoresis of fixed human and rabbit red blood cells as test particles. The band of human red cells can be seen moving from left to right as time progresses.

FL-020

Kidney Cell Electrophoresis

Pennsylvania State University
Dr. Paul Todd
NAS9-15584 $60K/year(approx.)
June 1980 - continuing task

The objective of this investigation is to repeat the MA-011 experiment under conditions which are optimum for the viability of human kidney cells and most favorable for the best possible electrophoretic separation of those few (about 5%) cells which produce urokinase or human granulocyte conditioning factor (HGCF), and erythropoietin.

This study effort will perform the ground-based research necessary to establish all of the optimum experimental conditions required to accomplish the best possible electrophoretic separation of human kidney cell fractions, which produce urokinase, granulocyte stimulating factor, or erythropoietin. This overall effort will include: (1) development of optimum buffer systems, (2) viability tests, (3) ground-based research on electrophoretic mobilities, (4) development of standard cells, standard cell culture methods, and standard urokinase assay procedures, (5) acquisition of the ground control data to be compared with results using cells returned from the electrophoretic separations carried out in microgravity, and (6) ground-based research on the electrophoretic mobilities of suspended pituitary cells (last task added in 1982).

Cells from cultures obtained from 32 commercially-prepared explants have been studied with respect to electrophoretic mobility distribution, growth in culture, and urokinase production. The testing of various electrophoresis buffers, which were also used as the medium for freezing viable cells in Shuttle flight STS-3, indicated that their low ionic strength compromised cell viability somewhat, and the buffer, "D-1," which contains EDTA and DMSO, was used in the microgravity experiments involving electrophoresis of human kidney cells and test particles (fixed human and rabbit red blood cells). Ground-based electrophoretic characterization of human kidney cell line "HEK-8514" was accomplished prior to the STS-3 flight, as was the characterization of the test particles. Procedures for post-flight analysis has been established and evaluated prior to the flight. These were not implemented, owing to the well-known post-flight accidental loss of the samples.

Post-flight analysis of the test-particle electrophoresis data recorded on film during the STS-3 flight has been accomplished. A time sequence photograph (Figure 1) shows the progression of the human red blood cell band along the column during electrophoresis in flight.

Optical density scans of the negatives of these phorographs, provided by Johnson Space Center, were analyzed by computer, and ground-based laboratory studies were carried out to simulate the conditions of flight. These studies indicated that the migration rate of cells in space is predictable on the basis of temperature and ionic strength of the "D-1" buffer that was used.

Collaboration with Johnson Space Center and Michael Reese Research Foundation in Chicago continue. Their purpose is the preparation of human kidney cell electrophoresis experiments that will utilize the McDonnell-Douglas continuous-flow electrophoretic separator (CFES) aboard STS flights under the MDAC-NASA Joint Endeavor Agreement. Media and buffers suitable for this purpose are under investigation, and a very-low-ionic-strength triethanolamine electrophoresis buffer is being evaluated as is a serum-free or low-serum medium in which cells can both multiply and produce urokinase.

Publications

Kunze, M. E. and Todd, P., "Evaluation of Econazole as an Antifungal Agent in Quantitative Cell Culture Experiments," In Vitro (in press, 1983).

FL-021

Production of Large-Particle-Size Monodisperse Latexes in Microgravity

Lehigh University
Dr. J. W. Vanderhoff
Dr. F. J. Micale
Dr. M. S. El-Aasser
NAS8-32951 Total Cost: $418K
February 22, 1978 - February 22, 1983

The purpose of this project is to explore the possibility of preparing large-particle-size monodisperse latexes in microgravity to avoid the problems of coagulum formation, as well as creaming and sedimentation, as the particles grow in size and change density. If successful, these experiments could provide a process for producing monodisperse latexes in a size range that is difficult to obtain on the ground. A second purpose is to develop a model for a heterogeneous chemical reaction in space.

Ground-based experiments have been carried out to obtain kinetic data to evaluate alternative methods for preparing these latexes. The flight hardware has been prepared, the first series of four experiments was carried out on the STS-3 mission of the Orbiter. Three of these experiments used a 2.5μ m-size seed latex and produced monodisperse latexes of 3.4, 4.1, and 5.0μ m size. The fourth experiments was a control which used a 0.19 μm-size seed latex. Further series of experiments are planned for the STS-4, STS-5, and STS-6 missions

The laboratory experiments in progress include the development of a method to achieve high monomer-polymer swelling ratios and the development of new agitator forms as well as the evaluation of the results of the STS-3 experiments and the development of recipes for the STS-4, STS-5, and STS-6 missions.

4. ULTRAHIGH VACUUM AND CONTAINERLESS TECHNOLOGIES

TASK NUMBER	PRINCIPAL INVESTIGATOR	SHORT TITLE
CP-001	Bonnell	Measurement of High Temperature
CP-002	Cezairliyan	Dynamic Thermophysical Measurements
CP-003	Chu	Effects of Undercooling of Modified
CP-004	Collings	Influence of Containerless Undercooling
CP-005	Day	Containerless Processing of Glass
CP-006	Downs	Gel Precursors as Glass and Ceramic
CP-007	Dunn	The Upgrading of Glass Microballoons
CP-008	Elleman	Electrostatic Control and Manipulation
CP-009	Ethridge	Homogeneous Crystallization of Glass
CP-010	Frost	Rework of SPAR EML for MEA
CP-011	Margrave	Measurement of the Properties of Tungsten
CP-012	Mukherjee	Ultrapure Glass Optical Waveguide
CP-013	Nordine	Containerless High Temperature Property
CP-014	Robinson	Undercooling in Metastable Peritectics
CP-015	Schmid	Free Cooling at High Temperatures
CP-016	Szekely	Convection in Grain Refining
CP-017	Turnbull	Crystal Nucleation in Glass-Forming Alloy
CP-018	Wang	Fusion Target Technology
CP-019	Wang	Advanced Containerless Processing

INTRODUCTION

Levitation technology is being pursued to develop devices for positioning, melting, manipulating, and resolidifying materials in space without the constraint of containers or crucibles. In space, liquid materials will remain in a stable, spherical drop without containers; thus, small restraining forces are sufficient to keep the drop where desired. The processing of materials without the necessity of containers is an exciting and unique capability of the space environment and permits the formation of pure materials without contamination from the container, permits the formation of amorphous (glass) materials that cannot be made on Earth, and permits the measurement of physical properties of molten materials at temperatures that exceed the melting point of crucibles needed on Earth. The MPS program technology is directed toward the development of high temperature acoustic levitators (or positioning devices) for use with materials with electrically conductive materials, in either gaseous or vacuum processing environments, and electrostatic levitators for use with dielectric materials that need to be processed in vacuum environments. Low-g flight experiments have been conducted successfully with both acoustic and electromagnetic devices, and the practical application of this technology to a vast spectrum of both scientific and commerical processes can be realized through the elimination of detrimental gravitational effects.

CP-001

Measurement of High Temperature Thermophysical Properties

National Bureau of Standards
Dr. D. W. Bonnell
H-27954B

The primary aim of this task has been an ongoing evaluation of the experimental procedures used in the collaborative interaction between General Electric Advanced Application Laboratory (GE) and the Rice University Department of High Temperature Group (RICE) to measure the high temperature enthalpy increments of liquid and solid tungsten, Cp, heat of fusion of liquid and solid refractory materials. Initial concentration has been on tungsten due both to the importance of tungsten as the highest melting element, and because GE has developed a unique apparatus employing electromagnetic levitation and electron beam melting now capable of levitating molten tungsten. RICE has wide experience in high temperature enthalpy increment measurements, with particularly emphasis on drop-type isoperibol calorimetry. The NBS involvement provides the background in coupling the two techniques.

The results of this research are of great theoretical and engineering interest. From the scientific viewpoint, the unqiue location of tungsten at the upper end of the metal and element melting point scale should provide a key part in any extrapolation/ interpolation procedure. Models, such as the well-known Tamman rule, or correlations using chemical periodicity are the current basis for property estimation in many cases. A direct definitive measurement of the heat of fusion of tungsten (current estimates and indirect measurements vary from 8.5 kcal/mole to $\sim$ 14 kcal/mole) will provide a valuable test of periodic table correlation mdoels, probably indicating significant deviations from predictions based on lower melting refractory metals. Questions concerning the liquid state heat capacity function, (i.e., whether it is constant as is suggested by studies of lower melting discontinuity in Cp(s) to Cp(1) (from 14.8 to 8.5 cal/mole. deg.) are even approximately correct) are crucial in testing theoretical models. Accurate data should provide clues to improve parameters in existing models, or suggest the need for new models. Models for predicting alloy properties, for example, require good thermodynamic data to obtain even approximate predictions.

This effort presses the state of the art in such ground-based experimentation. Many of the necessary techniques test solutions to requirements necessary for experiments in a micro-gravity environment. Major efforts have included the development of techniques for imaging pyrometry for temperatures above 3500 K, a semiautomatic calorimeter system for liquid tungsten, and a scheme for a completely automated high temperature calorimetric system. Studies planned following tungsten include liquid tantalum, and a selection of conductive inorganic refractories such as Group IVb carbides.

Publications

Bonnell, D. W., "Measurement of High Temperature Thermophysical Properties of Tungsten Liquid and Solid," in <u>NBS: Materials Measurements</u> (J. R. Manning, ed.), NBSIR 82-2560, 1982, p. 83.

CP-002

Dynamic Thermophysical Measurements in Space

National Bureau of Standards
Dr. A. Cezairliyan
H-27954B
April 1981 - continuing task

The objective of this task is to develop techniques for the dynamic (subsecond) measurement of selected thermophysical properties (such as, heat capacity, heat of fusion, electrial resistivity) of solids and liquids at temperatures above 2000 K in experiments to be performed near-zero-gravity environment. Under the near-zero-gravity conditions, it might be possible to sustain a liquid column (specimen) for the duration of the brief experiment and thereby obtain, for the first time, accurate thermophysical properties data above the melting point of high melting subtances. The construction of a preliminary system for testing the stability of the liquid specimen in a near-zero-gravity condition is completed. At the present time, the system is undergoing ground-based testing in preparation for flight testing in 1984. The ground-based testing includes checking of the overall operation of thr system including the electrical pulse circuit, control and measuring circuits, and diagnostic instrumentations such as the high-speed framing camera and the fast radiance detector. Theoretical and experimental work is also being performed on the stability of the liquid specimen under various forces such as surface tension and electromagnetic forces.

CP-003

Effects of Undercooling of Modified Microstructure on the Physical Properties of Materials Synthesized in the Drop-Tube and Drop Tower

University of Houston
Dr. C. W. Chu
NAS8-35161 $82,684/year
December 16, 1982 - December 15, 1984

It has been reported that large undercooling and modified microstrucure can be achived by compounds and alloys prepared in a low gravity and/or containerless environment. Preliminary studies indicated that samples so synthesized exhibited interesting physical properties, implying the possible stabilization of metastable phases and the possible existence of novel physical mechanisms. In this study, we plan to determine if the suggested metastable phase is stabilized and novel physical mechanisms exist by investigating the microstructural, the electronic, the magnetic and the thermal properties at various pressures and temperatures of solids prepared in the drop-tube and drop-tower facilities at Marshall Space Flight Center. Through the establishment and understanding of the effects of solidification in the drop-tube and drop-tower on the physical properties of solids, we hope that the zero-gravity environment in space can eventually be used for the synthesis of new materials with desired characteristics.

The specific material systems (to be synthesized if not yet available) to be examined are: (1) Au-Ge immiscible alloys, (2) Nb-Ge A15 compounds, (3) Nb-Si A15 compounds (if successfully made), and (4) Ga-Bi immiscible alloys. The reported superconductivity in Au-Ge disordered alloys (prepared in Skylab but not earth bound) will be tested and its cause determined. Should any compound phase be unambiguously observed not to exist in the Au-Ge System, Au-Ge system will be the best candidate for a novel superconducting mechanism to occur. A sharp superconducting transition has been observed in Nb-Ge prepared in a drop-tube at a temperature about that for the equilibrium A15 phase of Nb_3Ge, suggesting the possible stabilization of a metastable phase. The correlation of the superconductivity with a metastable phase will be determined. Attempt will also be made to synthesize the metastable A15 Nb_3Si. The reported unusual resistivity bump of the Ga-Bi immiscible alloys at low temperature prepared in the drop-tower will be evaluated and its cause determined. The electron scattering mechanisms known can hardly account for the observations.

CP-004

<u>Influence of Containerless Undercooling and Rapid Solid-State Quenching on the Superconductive and Magnetic Properties of Some Clustering Alloy Systems</u>

Battelle Columbus Laboratories
Dr. E. W. Collings
NAS8-35145 Total Cost: $42,773
November 1982 - November 1983

Alloys not usually homogeneous on an atomic scale; in fact they may be divided into two categoeies depending on whether the equilibrium phase diagram, in the composition range considered, contains an intermetallic compound or a pair of solid-solution phases. The first class of alloy is referred to as "short-range ordering" and the second is a "clustering" alloy. The proposed research has to do with the properties of three clustering alloy systems and the manner in which they are influenced by rapid quenching from a containerless undercooled melt. It is postulated that rapid quenching under such conditions will result in a more highly disordered alloy than can be obtained under normal solidification conditions, and that the disorder in turn will be reflected in characteristically different physical properties--superconductive or magnetic, as the case may be.

The scope of the program is essentially to gauge the influence of containerless undercooling on the sub-microstructures of clusteringtype alloys, using as diagnostic tools the measurement of the superconducting transition temperature in the case of those alloys which are superconducting above, say, 4.2K, and the measurement of magnetization as a function of applied magnetic field strength in the case of alloys containing "magnetic" elements. Microstructures and macrostructures are to be examined using optical and scanning-electron microscopy.

The technical approach consists, first of all, of the preparation or acquisition of the starting alloys. They will then be containerlessly undercooled. For this purpose, one of the drop-tubes located at the Marshall Space Flight Center will be needed; the alloys will be levitation remelted at the top of a drop-tube, and collected at the bottom in the form of liquid-quenched spheres--ideal shapes, incidentally, for the performance of magnetic measurements. The <u>superconducting</u> alloy is to be Ti-Mo; its superconducting transition temperature will be measured using a magnetic technique. The alloys with potentially interesting <u>magnetic</u> properties are to be Cu-Ni and a commercial austenitic stainless steel; their properties will be measured using a vibrating-sample magnetometer. Samples of all three materials will be sectioned and etched for microstructural study before and after dropping using an optical metallograph and a scanning-electron microscrope.

CP-005

<u>Containerless Processing of Glass Forming Melts in Space</u>

University of Missouri-Rolla
Dr. D. E. Day
NAS8-34758 $80K
February 1982 - March 1984

The principal objectives are to (a) obtain quantitative evidence for the suppression of heterogeneous nucleation/crystallization in containerless melts in micro-g, (b) develop the procedures for preparing precursor speciments that will yield bubble-free, high purity, chemically homogeneous melts in micro-g, (c) perform a comparative property analysis of glasses melted on earth and in micro-g and (d) assess the suitability of the single axis acoustic levitator/ furnace apparatus for processing multicomponent, glass forming melts in micro-g.

Glass formation in containerless melts is being investigated to verify the expected suppression of heterogeneous nucleation/ crystallization, the result of which will be an expansion of the compositional limits for glass formation. The critical cooling rate on earth (R_c-earth) for glass formation of selected calciagallia and alkali silicate compositions if being measured. Compositions of the desired R_c-earth will be remelted in micro-g and cooled at a rate less than R_c-earth. For samples returning as glass, the ratio $R_c$earth (cooling rate-micro-g) will provide a quantitative measure of the degree to which glass formation is enhanced, or conversely, heterogeneous nucleation is suppressed, in containerless melts. A wide range of physical, optical, thermal, and mechanical properties will be measured for glasses made in micro-g for comparision with the properties of glasses made on earth.

The first major task was to prepare precursor specimens for use in the containerless glass melting experiments planned for the MEA-1 apparatus-single axis acoustic levitator. Specimens premelted on earth will be used for the glass formation studies mentioned above. Specimens containing known chemical and optical inhomogeneities are being used to investigate the degree of homogeneity achieved in micro-g where melt homogenization will be primarily by bulk diffusion rather than by gravity driven convection. Special attention is being given to evaluating the degree of initial homogeneity that sintered, hot pressed precursor samples must possess in order to yield chemically homogeneous, multicomponent melts in micro-g within reasonable time/temperature constraints.

CP-006

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

KMS Fusion, Inc.
Dr. Raymond L. Downs
W. J. Miller
No. 956403 (NAS7-100) Total Cost: 160K

The overall objective of the proposed work is to determine experimental procedures to produce gel starting materials for investigations of containerless processing in space. This containerless processing is directed at producing ultra high purity and/or amorphous materials such as glasses or ceramics whose production under terrestrial conditions are extremely difficult or improbable.

Containerless processing means not only the eliminations of melting crucibles, but also mechanical stirring to achieve compositional homogeneity. Melt-organic derived gels will be investigated as a source of compositionally homogeneous starting materials which will not require mechanical mixing to produce a final refined glass or ceramic product.

There are some problems in using gel starting materials. The most serious are prevention of precipitation of oxide components prior to gelation, segregation of components in the gel during drying, removal of residual carbon, and excessive evolution of gases during melting. These problems can be minimized by proper choice of starting materials and careful heating and firing of the products. Addressing those potential problems in a systematic manner is a major objective of the proposed research. The compositions chosen for investigation are the alkali tungstate and calcium tantalate systems.

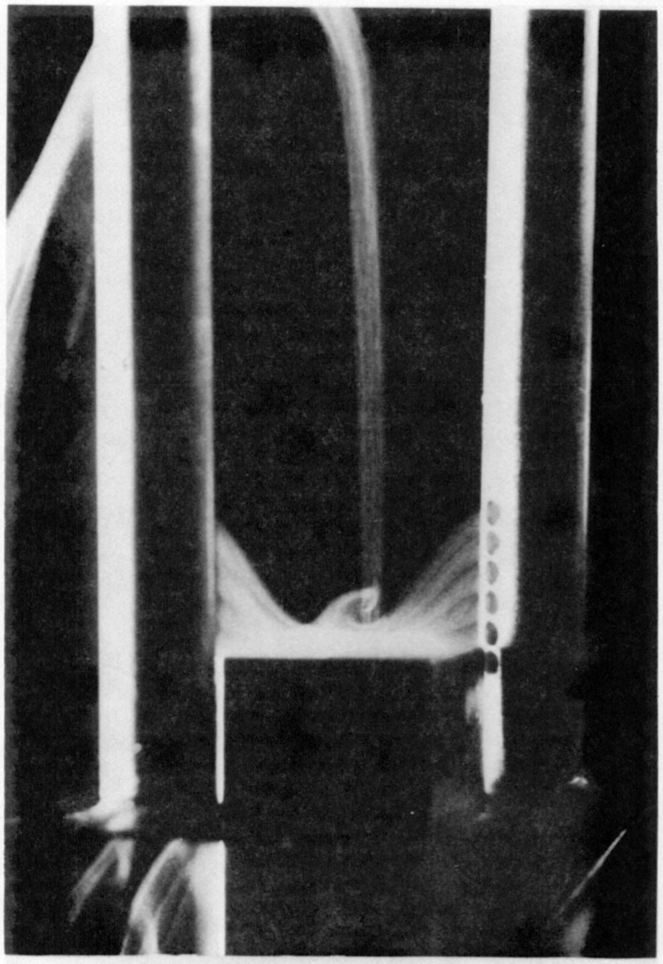


Figure 1. Sonic Pump Flow Patterns (Dry Ice-Water Fog).

CP-007

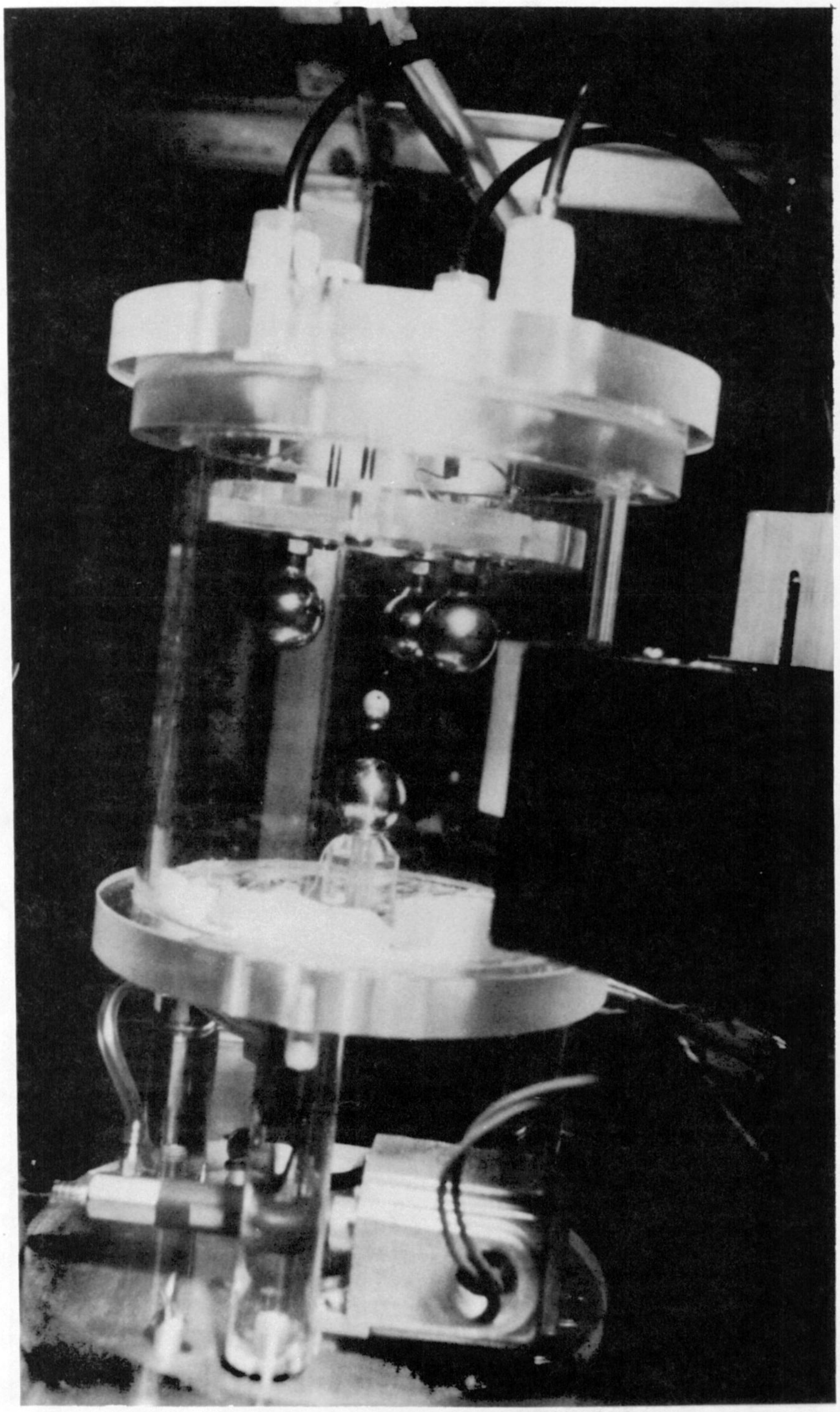
The Upgrading of Glass Microballoons

Bjorksten Research Laboratories
Dr. Stanley A. Dunn
Robert T. Nagler
Elmer G. Paquette
A. Pomplun
Dr. E. J. Crosby
NAS8-33513 $50K/year(approx.)
August 28, 1978 - July 31, 1983

The objective of this program is to study extensively the processes and mechanisms involved in producing glass microballoons of acceptable quality for laser fusion by gas jet levitation and manipulation in the molten condition.

The levitation microfurnace (LMF) is being adopted to variable and microgravity through computerized optical feedback control of multiple CHSs appropriately located on orthogonal triaxial coordinates. A pressure transducer for driving the CHSs was developed, capable of sufficiently rapid response to keep up with the computer generated corrective signals. The transducer, termed Sonic Pump, is comprised of an audio speaker with the output side of the cone sealed to a plate perforated at the center with a tubular duct leading to the plenum of the CHS. Sound generation by the speaker results in a corresponding sinuously reversing flow in the duct. At the mouth of the CHS two entirely different flow patterns result for fluxes into and out of the CHS. The influx vortex flow pattern is nearly isotropic; the eflux though pulsating is in one direction only and in other respects similar to the desired collimated flow resulting from a constant pressure supply of gas to the CHS plenum. (see Figure 1)

An important technique was developed for producing CHSs of virtually any degree of fineness and aspect ratio (hole length to effective hole diameter). Whereas optimum levitating stability requires larger aspect ratios of the order of 10 and above, most small hole drilling techniques are limited to values of 3 or 2 or below. The essence of the subject technique rests upon the discovery that holes of noncircular cross sections may perform quite as satisfactorily as circular. The CHS by this technique consists of the interstices within a symmetrically packed and confined bundle of uniformly sized wires.



Electrostatic Position Feed-back Control Module. Positioning a solid sample of 3.5 gm/cc density and 1 cm diameter in the reduced gravity of the KC-135 aircraft. This same module has also position and manipulated liquid samples in a similar environment.

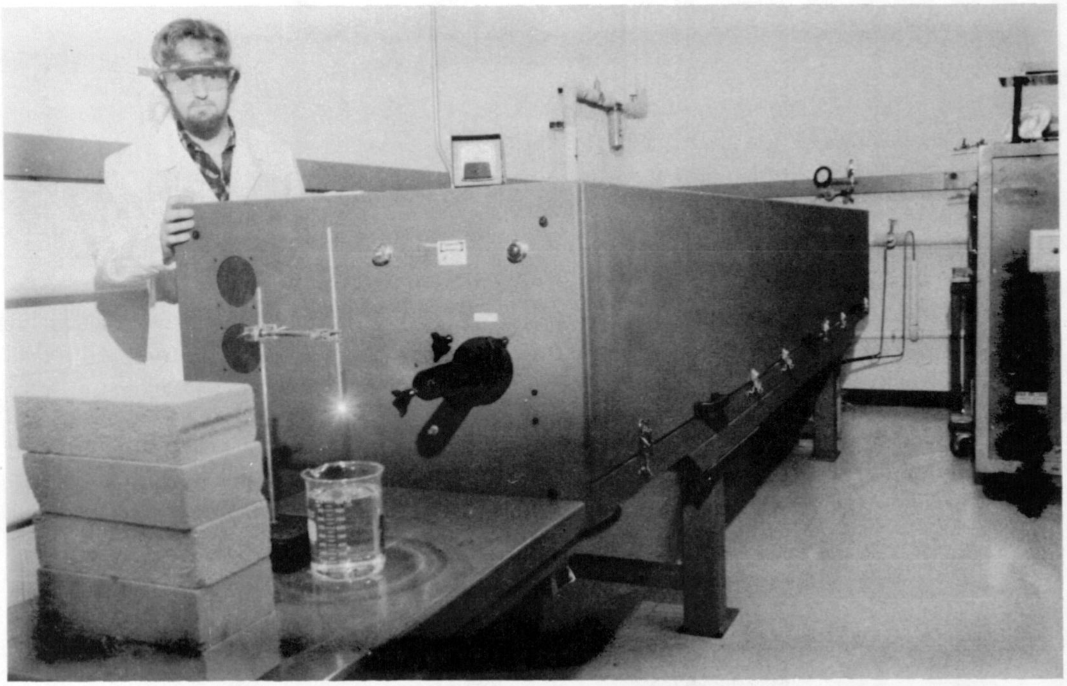
CP-008

Electrostatic Control and Manipulation of Materials for Containerless Processing

Jet Propulsion Laboratory
Dr. D. D. Elleman
Dr. W. K. Rhim
In-Center $400K/year
October 1978 - continuing task

The primary long-range objectives of this task are to develop and advance the technology of contactless positioning and manipulation of an electrostatic positioning module. The technology developed in ground-based experiments and low gravity tests in a KC-135 aircraft will be employed in the design of flight versions of electrostatic modules for material science studies in a zero gravity environment provided by rockets and the Space Shuttle.

In FY'82 several models of the electrostatic positioning module were constructed and tested in ground-based operations and in the 25 sec. low gravity environment provided by the KC-135 aircraft. In the ground-based operations, a three-axis feed back control electrostatic levitator with a tetrahedral arrangement of the electrodes was able to levitate and position low density samples of 0.1 gmocc and 1 cm diameter in a one-g environment in excess of 5 hours of uninterrupted operation. This same module, when tested in the low gravity of the KC-135 aircraft was able to position samples of density in excess of 3.5 gm/cc and 1 cm in diameter for the full 25 sec. of reduced gravity provided by the aircraft. Control of thr sample was maintained even during 0.1 g jitter perturbations. In addition, liquid samples were tested at room temperature to simulate molten materials. The electric fields employed ($<$ 5k volts) produced a distortion from sphericity of the liquid sample of less than 5%. During FY'83, levitation of samples at elevated temperatures will be initiated.



Ceramic Processing CO_2 Laser

CP-009

Homogeneous Crystallization Studies of Borderline Glass Forming Systems

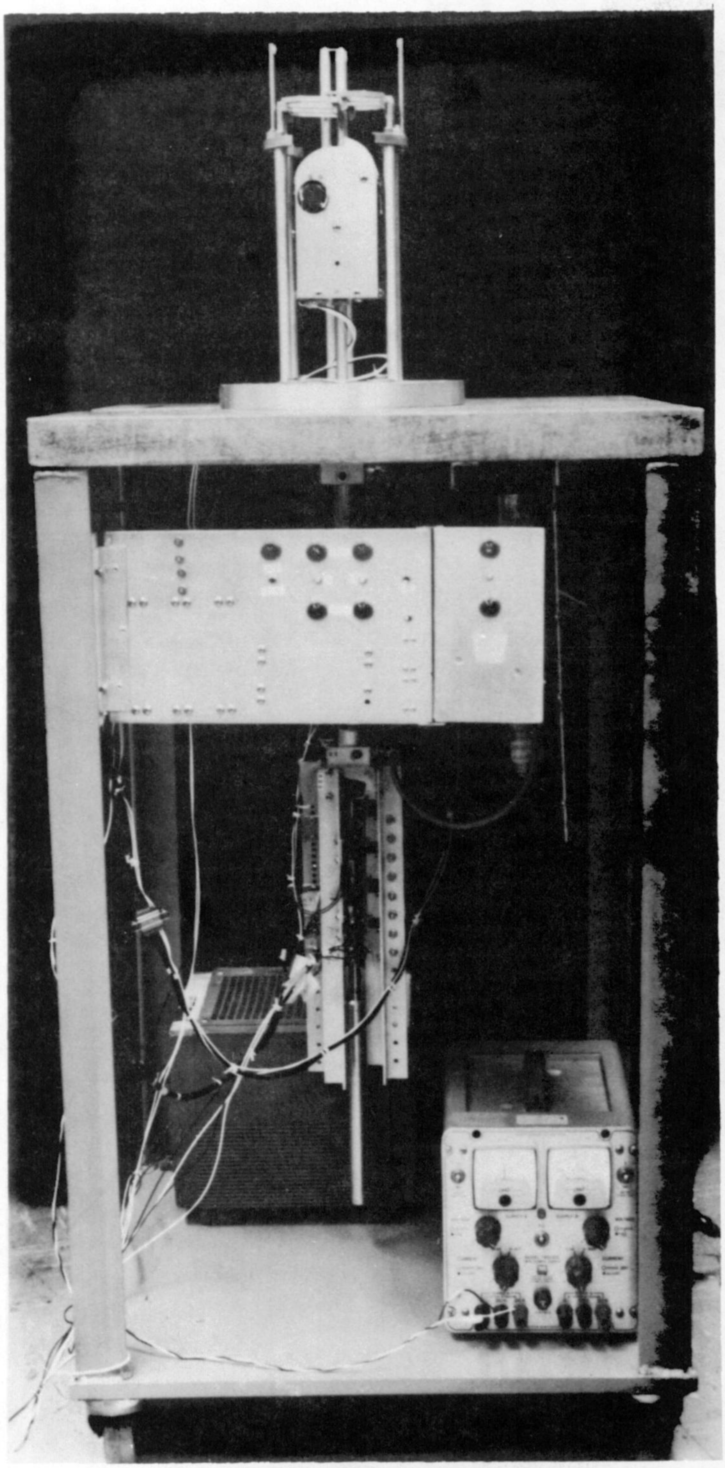
Marshall Space Flight Center
Dr. E. C. Ethridge
Dr. P. Curreri
In-House $60K/year
April 1, 1981 - April 1, 1984

The primary objective of this study is to use containerless as well as pseudocontainerless processing techniques to melt and resolidify borderline glassformers in such a way as to obtain critical cooling rates to avoid homogeneous crystallization. A secondary objective is to develop new techniques for supercooling oxide melts to produce bulk samples of candidate materials for ground-based screening tests of potential flight compositions.

The research plan is to melt and resolidify samples in containerless and pseudocontainerless fashion. For rapid cooling rates, pendant drop melting and rapid solidification in free fall will be utilized. For slower cooling rates, vitreous supports during sample heating and cooling will be used. A 400 Watt CO_2 laser will be used for heating and controlled cooling. Critical cooling rates and crystallization rates will be measured.

Publications

Ethridge, E. C. and Curreri, P., "Containerless Electromagnetic Levitation Melting in Microgravity Conditions," in Electromagnetic Moldless Casting (J. P. Wallace, ed.), North Holland, 1983.



Accelerating calorimeter is pneumatically driven 30 cm and captures levitated molten specimen within 0.1 sec. Will be used for ground-based measurement of enthalpy for high temperature molten refractory materials to reduce enthalpy loss during capture. Device, without instrumentation extension shown below control panel, can be adapted for drop calorimetry in space where calorimeter must engulf motionless levitated specimen.

CP-010

Rework of the SPAR Electromagnetic Levitator (EML) for Materials Experiment Assembly (MEA) Accommodations

General Electric Company
Dr. R. T. Frost
NAS8-34231 $116K/year
October 1981 - March 1983

The goals of this project are: (1) to study the upgrade requirements and approaches needed for incorporation of an Electromagnetic Levitator (EML) in the Materials Experiment Assembly carrier, (2) to work with members of the Electromagnetic Containerless Processing science working group (SWG) to define future experiment for the EML, and (3) to assist these investigators in further development of ground based experiment techniques to the limits possible in the terrestrial gravitational environment. An automated drop calorimeter was designed, built and utilized to make the first drop calorimeter measurement of heat of fusion for tungsten. An accelerating calorimeter was developed which will be required to extend these techniques in a microgravity environment and which will also reduce calorimeter capture time. A specimen exchanger for EML underwent preliminary design. Experiment functional requirement documents were written for several space experiment conceived by members of the SWG.

Present work is directed toward development of an imaging two-color pyrometer for application to measurement of thermophysical properties of materials at high temperatures. This instrument, based on a random access charge injection device (CID) will first be used for temperature measurements in the GE lectron beam heat drop calorimetry facility. A chopped radiometer using a pyroelectric detector will also be tested with the facility.

Publications

Chang, C. W., Das, D. K., Frost, R. T., and Kumar, K., "Electromagnetic Containerless Reaction of Samarium with Cobalt for the Formation of Samarium-Cobalt Alloys," Met. Trans. A 13A, 1868 (October 1982).

CP-011

Measurement of the Properties of Tungsten at High Temperatures

Rice University
Dr. J. Margrave
NAS8-33199 $15K/year
November 1978 - March 1, 1985

This research is directed toward the measurement of the thermophysical properties of tungsten and tantalum using containerless techniques. The properties of tungsten and tantalum are of interest because they lie at the extreme of metal melting points and are key data in any extrapolation or interpolation process. In addition, difficulties in handling molten tungsten and tantalum may establish the limitations of ground-based containerless systems in processing materials at high temperatures.

Samples are suspended containerlessly by an electromagnetic levitator. Additional heat is supplied by electron bombardment. Temperatures are measured by pyrometers. Heat capacities are determined from cooling curves, and/or by dropping the molten metals in a drop calorimeter. Enthalpy increments and heat capacities and emissivities are being measured.

CP-012

Ultrapure Glass Optical Waveguide Development in Microgravity by the Sol-Gel Process

Battelle Columbus Laboratories
Dr. S. P. Mukherjee
NAS8-34894 Total Cost: $150K
June 1982 - December 1983

The objectives of the program are: (1) to study the homogeneity of gels and gel-derived glasses in the oxide systems which are potentially important in the field of optical waveguide applications, (2) to study the glass formation ability of certain compositions in the selected oxide systems by the containerless melting of homogeneous multicomponent noncrystalline gels, (3) to study the influence of impurities obtained from the containers on the glass formation ability of selected compositions in the chosen systems, and (4) to perform containerless melting of ultrapure multicomponent gels and evaluation of purity and crystallinity.

The oxide systems SiO_2-GeO_2, SiO_2-TiO_2, and GeO_2-PbO/Bi_2O_3 will be used. Gels and gel-monoliths will be prepared by the chemical polymerization of alkoxysilane and metal alkoxides and/or suitable metal salts. The gels and/or gel-monoliths will be transformed into glasses by melting in a container and also by the containerless melting in a levitating system. Glasses prepared by the melting of gels in a container and by the containerless melting in a levitation system will be studied in terms of homogeneity, crystallinity and stability towards devitrification, and molecular structures. The structure and property of the gel-derived glasses will be influenced by the processing factors. Hence, homogeneity and structure of gels before melting, the melting history (time, temperature, and cooling rate), and contamination during melting will be investigated. Results of the studies on the three systems will be critically analyzed for the selection of one particular system for the containerless processing of ultrapure gels in the microgravity environment of space.

CP-013

Containerless High Temperature Property Measurements by Atomic Fluorescence

Midwest Research Institute
Dr. P. C. Nordine
Professor C. A. Walker, Yale University
Dr. R. A. Schiffman, Yale University
NAS8-34383 $90K/year(approx.)
June 1, 1980 - May 31, 1983

The objective of this program is to measure high temperature properties in containerless experiments using laser excited atomic fluorescence. Its purposes include: (1) the development of new techniques for Earth-based study of candidate Spacelab high temperature experiments and material processing applications and (2) the measurement of high temperature thermodynamic and transport properties. The method is to obtain absolute temperature and concentration profiles in the gaseous boundary layer surrounding a hot solid or liquid by laser excited atomic fluorescence measurements.

The basic idea is that a laser beam, whose bandwidth includes a doppler and pressure broadened atomic absorption line will, in the absence of quenching processes, produce fluorescence from that species at an intensity proportional to its concentration. Therefore, the spatial variation in fluorescence intensity from ambient or vaporizing species can be used to measure boundary layer temperature and concentration profiles. The experimental parameters which may be controlled (e.g., specimen dimensions and temperature, total pressure, uniform or nonuniform boundary layer temperature, etc.) allow experiments in which thermodynamic, transport and/or gas phase kinetics control the observable gradients. Thus, specimen vapor pressure, temperature, or evaporation rate, gas phase transport properties, or gas phase reaction rate constants may be determined.

Experiments performed include measurements of (1) Al atom evaporation from CW CO_2 laser heated and aerodynamically levitated sapphire and alumina spheres, and self-support sapphire filaments, (2) Al stom reaction with ambient oxygen in the wake of a levitated specimen, (3) Hg atom concentrations in the wake of levitated alumina and sapphire spheres, relative to the ambient Hg atom concentration, (4) Hg atom concentrations and velocities in supersonic levitation jets, (5) ground state and metastable, electronically excited W atom concentrations produced by evaporation of an electrically heated tungsten filament, and (6) Mo-atom evaporation from laser heated and electromagnetically levitated molybdenum specimens.

Publications

Nordine, P. C. and Schiffman, R. A., "Laser Induced $Hg(^3P_1)$ Fluorescence Study of a Supersonic Free Jet," (in preparation).

Schiffman, R. A. and Nordine, P. C., "Laser Induced Fluorescence Study of Al_2O_3 Evaporation and High Temperature Gas Thermometry," (in preparation).

CP-014

Undercooling Studies in Metastable Peritectic Compounds

Marshall Space Flight Center
M. B. Robinson
In-House

The objective of this program task is to investigate undercooling and containerless solidification of metastable superconducting alloys Nb_3Ge and Nb_3Al and pure metal melts such as Nb; specifically, to investigate the structure and superconducting properties of undercooled Nb-Ge alloys and to determine the feasibility of forming metastable Nb_3Ge in bulk form.

Pure Nb droplets have been undercooled in excess of 500 K in free fall using the MSFC drop tube. The droplets form single crystals with no shrink cavity in the interior. The outer surface is rough, indicating the shrinkage associated with solidification was taken up by the interdendritic fluid. NbGe droplets have also been deeply undercooled and rapidly solidified in the drop tube. The undercooling has been measured for the NbGe alloy drops with results showing that the Nb 18 a/o Ge drops undercooled 500 K, where the Nb 22 a/o Ge drops undercooled 300 K. These undercoolings do not represent the maximum extent possible since these drops undercool the complete length of the drop and nucleated only after reaching the catcher. An increase in the transition tempera ture of the heavily undercooled NbGe drops have a measured transition temperature of $\sim$10K which is 4K above the as cast materials. The increase indicates that at least some of the metastable AI5 structure has been formed. The presence of the metastable AI5 phase has been confirmed by x-ray diffraction, compositional analysis using EDAX and further microstructural analysis. Further work will be to improve the transition temperature of the produced AI5 phase through annealing and even larger degrees of undercooling which should be possible in the new 100m drop tube. This work may also be extended to include NbSn, NbGa, and NbSi.

CP-015

Free Cooling at High Temperatures

National Bureau of Standards
Dr. L. A. Schmid
H-27954B
April 1981 - continuing task

The specific heat and thermal diffusivity of hot spherical sample of refractory materials can be deduced from radiometric observation of the sample as it cools by free radiation into a cold vacuum. Analytical formulas have been derived that express the temperature-dependent specific heat and diffusivity as functions of the observed time-dependent surface temperature and rate of energy loss.

Publications

Schmid, L. A., "Mathematical Analysis for Radiometric Calorimetry of a Radiating Sphere," Journal of Research of the National Bureau of Standards, (in press).

Schmid, L. A., "High-Temperature Radiometric Calorimetry for Freely Cooling Spherical Specimens: II. Mathematical Analysis," submitted to International Journal of Thermophysics, 1982.

CP-016

Convection in Grain Refining

Massachusetts Institute of Technology
Professor J. Szekely
Professor M. C. Flemings
NSG-7645

The purpose of this program is to obtain a better understanding of the relationship between fluid flow phenomena, nucleation, and grain refinement in solidifying metals both in the presence and in the absence of a gravitational field. An ultimate technical aim is to determine ways to achieve significant grain size reductions in hard-to-process melts and significant property refinement by obtaining solidification under highly nonequilibrium conditions.

The research has two principal components: (1) a dominantly experiment component aimed at the study of undercooling phenomena and the structures thus produced, with the ultimate objective of utilizing these concepts in space experiments, and (2) a dominantly mathematical modelling component aimed at the study of heat flow and convection in electromagnetically driven (or positioned) systems to interface with both the interpretation of the undercooling work and to facilitate the rational planning of the space experiments. A brief summary of results obtained to date is the following: (1) nickel base alloys samples of approximately 1 gram have been successfully levitated in inert atmospheres and undercooled by amounts up to 270°C, and a wide range of grain sizes and solidification structures obtained, depending on amount of undercooling and cooling rate; (2) two important innovative techniques have been developed to obtain large amounts of undercooling in high temperature (iron, nickel, and cobalt) alloys. In one of these, the metal is melted and then "emulsified" (stirred into fine droplets) in a molten oxide or salt. In the second, small pre-alloyed metal droplets are interspersed at room temperature with finely crushed oxide or salt. The admixture is then melted; (3) extremely large undercoolings have been obtained in the above two methods because of the fine particle size and cleansing action of the slag; (4) emphasis of the experimental work at present time is on increasing amounts of undercooling obtainable, and therefore the types of structures obtainable through (a) use of alternative emulsification media, (b) increasing rate of heat extraction, and (c) process variations; (5) a computational capability has been developed to determine the electromagnetic force field, the fluid flow field and the temperature field in induction stirred systems, including contained cylindrical metals and levitated spherical melts; (6) calculations were carried out for a variety of conditions, including heat and fluid flow in a metal held in an inductively stirred cylindrical crucible and levitation melted specimens both on the ground and in a zero gravity environments;

(7) calculations have shown that the fluid flow field is markedly different for ground based and for zero gravity conditions: and, (8) the techniques developed for solving MHD type problems in molten metal and glass systems and the results generated are thought to have made an important contribution to this overall field. The foregoing results of this research have implications both for study of convection at zero-g and for potential engineering application, both at 1-g and at zero-g. As noted before, enormous undercoolings have been obtained at 1-g and greater undercoolings were anticipated under microgravity conditions.

Some relevant space experiments would include the following: (1) room temperature mixing of metal droplets in emulsification media on earth and melting and cooling in microgravity, (2) emulsification in space with subsequent dampening of convection before cooling and solidification begins, (3) thermal analysis of rapidly recalescing metal droplets, and (4) fundamental convection in space.

Publications

Abbaschian, G. J. and Flemings, M. C., "Levitation and Supercooling of Fe-25% Ni Alloys," to be published in Met. Trans.

El-Kaddah, N. and Szekely, J., "The Turbulent Recirculating Flow Field in a Coreless Induction Furnace," to be published in Journal of Fluid Mechanics.

El-Kaddah, N. and Szekley, J., "The Temperature Field, Electromagnetic Force Field and the Velocity Field in Levitated Metal Droplets," submitted for publication.

CP-017

<u>Crystal Nucleation in Glass-Forming Alloy and Pure Metal Melts Under Containerless and Vibrationless Conditions</u>

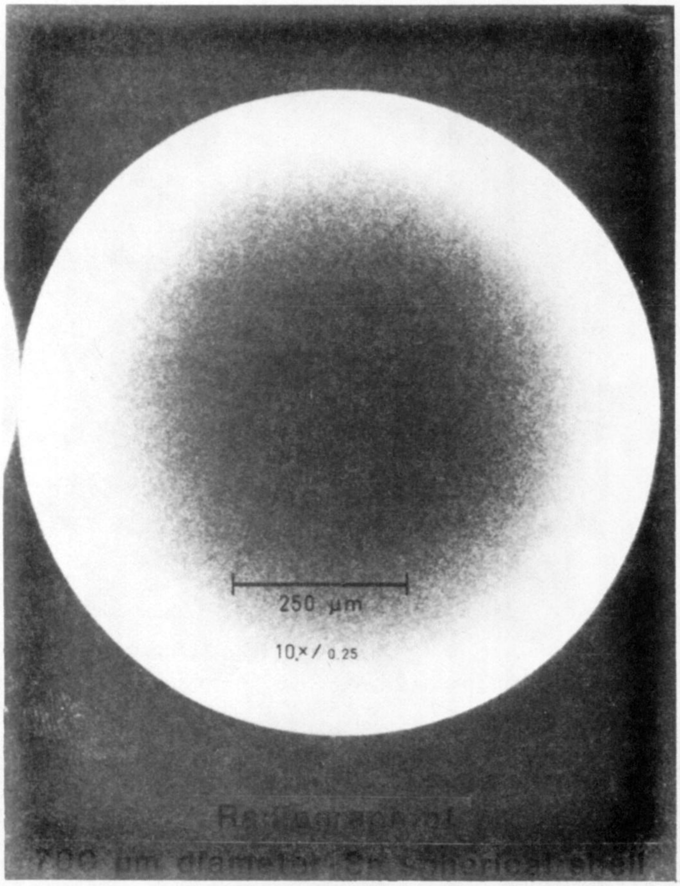
Harvard University
Professor David Turnbull
NAS8-32691 Total Cost: \$160K (approx.)
June 1978 - January 22, 1983

The main objective of this research is to characterize nucleation behavior in glass-forming alloy melts. Such experiments should indicate if formation of alloy glasses in bulk form is possible and, if so, what are the necessary conditions. The most favorable conditions would be those in containerless, vibrationless experiments in high vacua or inert atmospheres.

The crystal nucleation behavior of Au_4Si and Pd_4Si glassforming melts and of pure Ni by the droplet technique is being investigated. It has been found that the onset undercooling, ΔT_0, for copious nucleation in molten Au_4Si droplets varies widely with thermal treatments which alter the nature of the SiO_2 film on the droplet surface. However, T_0 as large as 1/3 of the liquidus temperature for some droplets was observed. Glass and crystallization temperatures of Au_4Si based alloys are sharply increased ($\sim$1° per atom %) when Cu replaces some of the Au. The transient period for crystal nucleation has been shown to be important for glass formation in alloys, such as these, with low reduced glass transition temperatures.

Drop tube experiments are being performed with droplets of Pd-Si and some Fe-based glass forming alloys. Analysis of the crystallization probably as a function of droplet size shows that crystal nucleation occurs in the droplet surface and is influenced by the atmosphere in the drop tube (especially moisture).

In the theory of crystal nucleation, we have considered various experimental factors (small impurity concentrations, stress effects) and theoretical estimates (free energy change on nucleation in pure metals and alloys, solid-liquid surface tension) that can influence the analysis.


250 µm
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CP-018

Fusion Target Technology

Jet Propulsion Laboratory
Dr. T. G. Wang
In-Center
October 1979 - continuing task

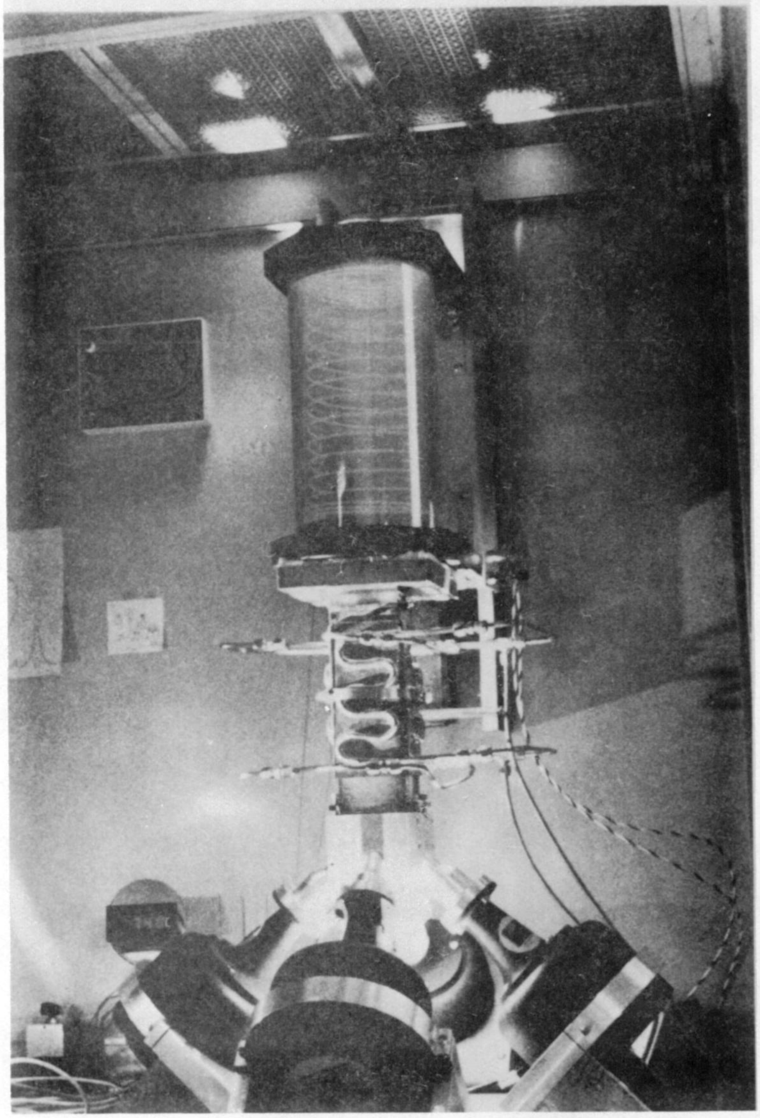
The objectives of this task are to (1) study the dynamics of liquid bubbles and of the gravitational effects relevant to the production of spherical shells both in the laboratory and in a weightless environment, (2) develop the technology that is pertinent to the production of metallic and metallic glass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

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

Publications

Lee, M. C., Feng, I-an, and Wang, T. G., "A Technique for Thick Polymer Coating of Inertial-Confinement-Fusion Targets," to be published in Journal of American Vacuum Society.

HIGH TEMPERATURE LEVITATION SYSTEM



CP-019

Advanced Containerless Processing Technology

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

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

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

The subjects to be addressed in FY-83 are experimental and theoretical studies of: (1) acoustic positioning and manipulation capabilities in a high temperature gradient environment (from 25°C to 900°C), (2) acoustic waveforms, harmonic contents, power transfer, sample transport and stability associated with high temperature gradient system, (3) high temperature ground based levitation systems which will allow us to melt, process, and solidify samples without crucibles in the laboratory, (4) KC-135 and laboratory tests of various acoustic geometries which may have spherical applications in the Materials Processing in Space Program, and (5) provide technical information to ACES engineering team and establish the operation conditions for ACES.

Publications

Trinh, E., Robey, J., and Wang, T. G., "A Dual Temperature Acoustic Levitation Chamber for Materials Study," to be published in Proceedings of 11th International Congress of Acoustics, 1983.

APPENDIX A
MPS ORGANIZATIONS

Alabama A&M University
Huntsville, AL

Battelle Columbus Laboratories
Columbus, OH

Bjorksten Research Laboratories
Madison, WI

Clarkson College of Technology
Potsdam, NY

EG&G Corporation
Santa Barbara, CA

French Atomic Energy Commission
Nuclear Research Center of Grenoble
Grenoble Cedex, France

S. H. Gelles Associates
Columbus, OH

General Electric Company
Space Sciences Laboratories
Valley Forge, PA

Grumman Aerospace Corporation
Bethpage, NY

Harvard University
Cambridge, MA

NASA
Headquarters
Washington, D. C.

Iowa State University
Ames Laboratory, ERDA
Ames, IA

Jet Propulsion Laboratory
California Institute of Technology
Pasadena, CA

NASA
Johnson Space Center (JSC)

KMS Fusion, Inc.
Ann Arbor, MI

Science Applications, Inc. (SAI)
Huntsville, AL

Semtec, Inc.
Huntsville, AL

University of Alabama, Huntsville (UAH)

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Tucson, AZ

University of Houston
Houston, TX

University of Missouri-Rolla
Rolla, MO

University of Oregon
Health Sciences Center
Portland, OE

University of Rochester Medical Center
Rochester, NY

University of Sydney
Sydney, AUSTRALIA

University of Utah
Salt Lake City, UT

University of Wisconsin - Milwaukee

Westech Systems, Inc.
Phoenix, AZ

NASA
Langley Research Center (LaRC)
Hampton, VA

Lehigh University
Bethlehem, PA

Lockheed Corporation
Huntsville Research & Engineering Center
Huntsville, AL

Massachusetts Institute of Technology
Cambridge, MA

Michigan Technological University
Houghton, MI

Midwest Research Institute
Kansas City, MO

NASA
George C. Marshall Space Flight Center (MSFC)
Marshall Space Flight Center, AL

McDonnell Douglas Corporation-East
St. Louis, MO

National Bureau of Standards
U. S. Department of Commerce
Washington, D.C.

National Bureau of Standards
Boulder Laboratories
Boulder, CO

Northwestern University
Evanston, IL

Ohio State University
Columbus, OH

Pennsylvania State University
University Park, PA

Princeton University
Princeton, NJ

Rensselaer Polytechnic Institute (RPI)
Troy, NY

Rice University
Houston, TX

APPENDIX B
INDEX OF PRINCIPAL INVESTIGATORS

INDEX OF PRINCIPAL INVESTIGATORS

APPROVAL

MATERIALS PROCESSING IN SPACE PROGRAM TASKS

Compiled by Elizabeth Pentecost

The information in this report has been reviewed for technical content. Review of any information concerning Department of Defense or nuclear energy activities or programs has been made by the MSFC Security Classification Officer. This report, in its entirety, has been determined to be unclassified.

A. J. Dessler
Director, Space Science Laboratory

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