

NASA  
Technical  
Memorandum

NASA-TM-82496 19830005875

NASA TM-82496

# Materials Processing in Space

## Program Tasks

FOR REFERENCE

Compiled by Elizabeth Pentecost  
Space Science Laboratory

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National Aeronautics and  
Space Administration

George C. Marshall Space Flight Center



1. REPORT NO. NASA TM-82496	2. GOVERNMENT ACCESSION NO.	3. RECIPIENT'S CATALOG NO.	
4. TITLE AND SUBTITLE  Materials Processing in Space Program Tasks		5. REPORT DATE September 1982	6. PERFORMING ORGANIZATION CODE
		8. PERFORMING ORGANIZATION REPORT #	
7. AUTHOR(S) Compiled by Elizabeth Pentecost *		10. WORK UNIT NO.	
9. PERFORMING ORGANIZATION NAME AND ADDRESS  NASA-George C. Marshall Space Flight Center Marshall Space Flight Center, Alabama 35812		11. CONTRACT OR GRANT NO.	
		13. TYPE OF REPORT & PERIOD COVERED  Technical Memorandum	
12. SPONSORING AGENCY NAME AND ADDRESS  National Aeronautics and Space Administration Washington, DC 20546		14. SPONSORING AGENCY CODE	
		15. SUPPLEMENTARY NOTES  Prepared by Space Science Laboratory, Science and Engineering Directorate.  *USRA	
16. ABSTRACT  This report is a compilation of the active research tasks as of the end of the fiscal year 1982 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.			
17. KEY WORDS  Crystal growth Solidification of Metals, Alloys and Composites Ultrahigh Vacuum and Containerless Processing Technologies		18. DISTRIBUTION STATEMENT  Unclassified-Unlimited	
19. SECURITY CLASSIF. (of this report)  Unclassified	20. SECURITY CLASSIF. (of this page)  Unclassified	21. NO. OF PAGES  200	22. PRICE  NTIS

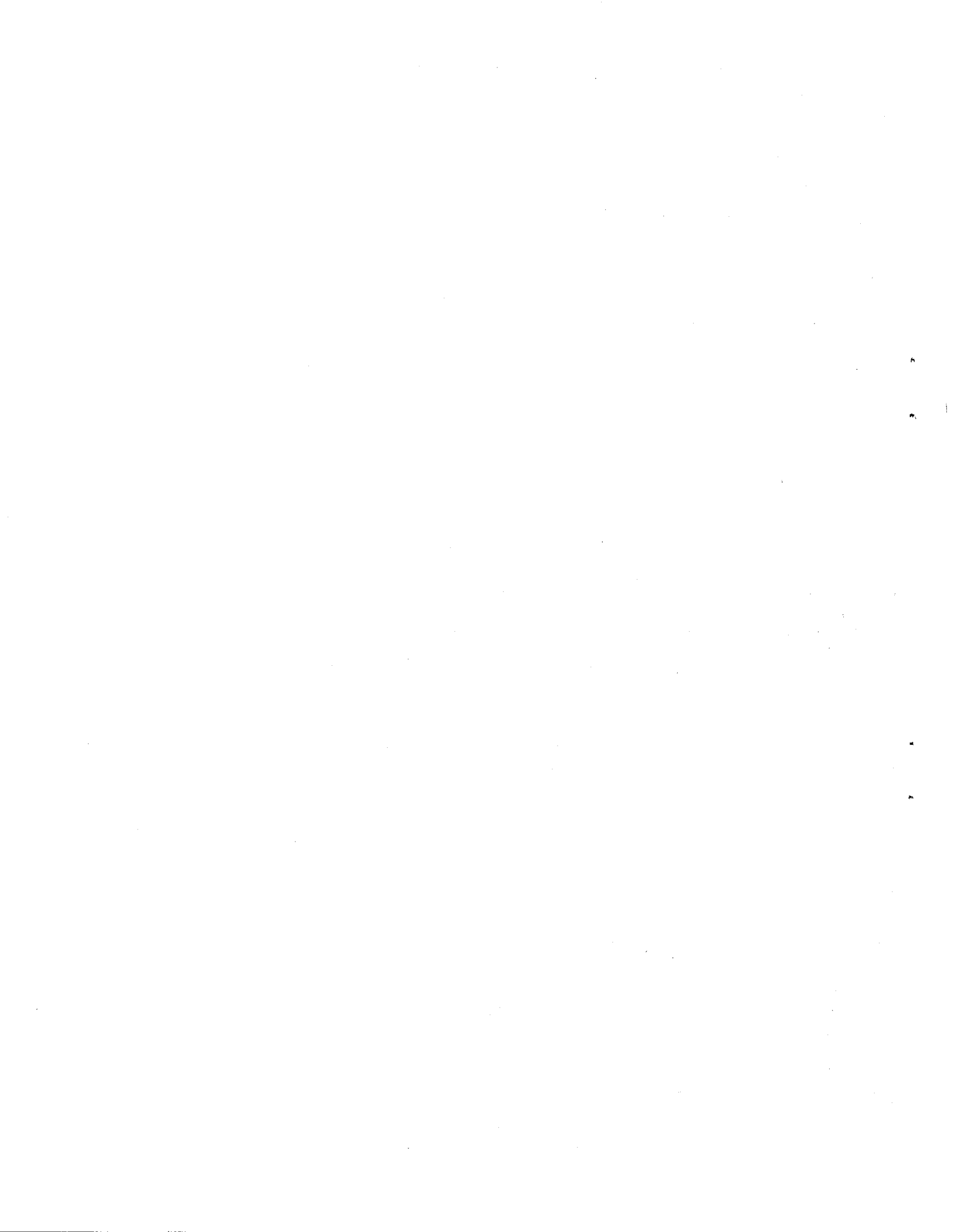




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# NASA TECHNICAL MEMORANDUM

## 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.

## II. TASKS





1. CRYSTAL GROWTH

TASK NUMBER (TN)	PRINCIPAL INVESTIGATOR (PI)	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	Gatos	Crystal Growth in Device Quality GaAs
CG-007	Kern	Microgravity Silicon Zoning Investigation
CG-008	Lal	Solution Growth of Crystals
CG-009	Lehoczky	Growth of Solid Solution Crystals
CG-010	Rosenberger	Fluid Dynamics of Crystallization
CG-011	Schnepple	HgI <sub>2</sub> Growth for Nuclear Detectors
CG-012	Tiller	Direct Observation of Interface Stability
CG-013	Verhoeven	Float Zone Experiments in Space
CG-014	Vydyanath	Defect Chemistry of HgCdTe
CG-015	Wiedemeier	Fluid Dynamics and Thermodynamics
CG-016	Wiedemeier	Vapor Growth of Alloy-Type Semiconductors
CG-017	Witt	Heat Flow and Segregation
CG-018	Zoutendyk	Vapor Phase of PbSnTe



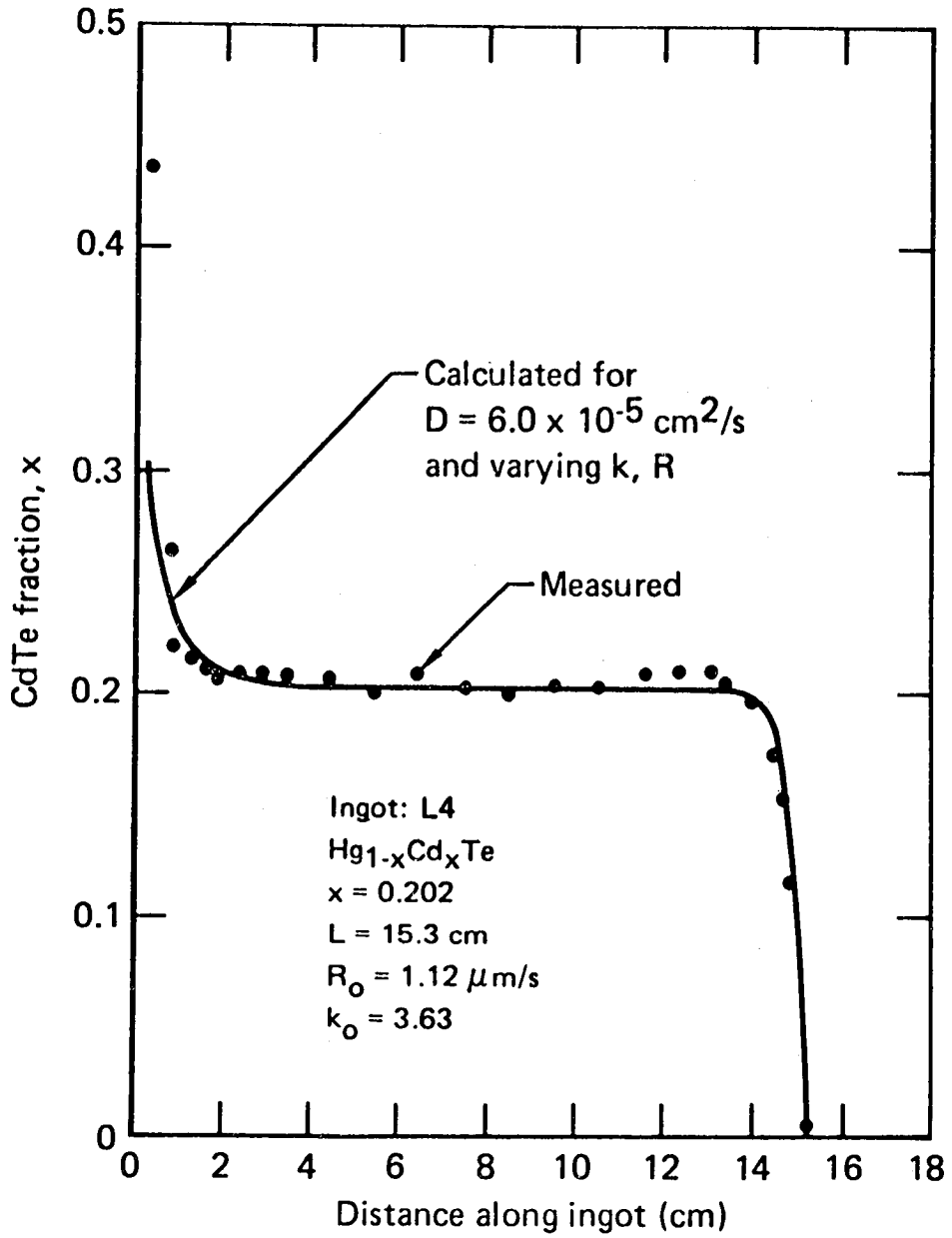
## INTRODUCTION

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 electrical performance, in HgCdTe and lead-tin-telluride (PbSnTe) semiconductors. These crystals are among the most sensitive and important infrared sensors 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 instabilities. 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 commercially 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<sub>2</sub> nuclear detector crystals and HgCdTe and copper-indium-antimony (CuInSb) solid solution semiconductor crystals.



A comparison between the measured and calculated compositional profiles for ingot L4. The calculations assumed a diffusion coefficient of  $6.0 \times 10^{-5} \text{ cm}^2/\text{s}$  and included the variations in  $k$  and  $R$  along the growth axis.

Advanced Methods for Preparation and Characterization of Infrared-Detector Materials

McDonnell Douglas Research Laboratories

Dr. J. G. Broerman

Dr. S. L. Lehoczky\*

Dr. F. R. Szofran\*

NAS8-33107 \$80K/year

December 1978 - December 1982

The objectives of this research program are to quantitatively establish the characteristics of  $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$  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  $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$  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. Further DTA measurements and theoretical analysis are being performed to determine phase equilibria parameters for selected regions of the ternary Hg-Cd-Te phase diagram.

Theoretical models and computer programs specific to  $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$  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 comparison of calculated results with available experimental data indicated that longitudinal optical-phonon and charged and neutral defect scattering are the dominant mobility limiting mechanisms.

Publications

Szofran, F. R. and Lehoczky, S. L., "The Pseudobinary HgTe-CdTe Phase Diagram," J. Elect. Mat. 10, 1131 (1981).

Lehoczky, S. L. and Szofran, F. R., "Directional Solidification and Characterization of  $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$  Alloys," in Materials Research Society Symposia Proceedings, Materials Processing in the Reduced Gravity Environment of Space, Volume 9 (G. E. Rindone), North Holland, 1982, pp. 409-420.

\*presently at Marshall Space Flight Center, Alabama

Lehoczky, S. L., Summers, C. J., Szofran, F. R., and Martin, B. G. "Electrical Characterization of  $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$  Alloys," in Materials Research Society Symposia Proceedings, Materials Processing in the Reduced Gravity Environment of Space, Volume 9 (G. E. Rindone, ed.), North Holland, 1982, pp. 421-432.

Lehoczky, S. L. and Szofran, F. R., "Advanced Methods for Preparation and Characterization of Infrared Detector Materials," Interim Report, NAS8-33107, September 1981.

Kelley, J. D., Martin, B. G., Szofran, F. R., and Lehoczky, S. L., "Application of the Regular Associated Solution Model to the Cd-Te and Hg-Te Binary Systems," J. Electrochem. Soc., in press.

#### Presentations

Lehoczky, S. L., Summers, C. J., and Szofran, F. R., "Directional Solidification and Characterization of  $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$  ( $x \leq 0.25$ )," presented at NATO Cadmium Mercury Telluride (CMT) Workshop, Grenoble, France, April 23-24, 1981.

Lehoczky, S. L. and Szofran, F. R., "Diffusion-Limited Directional Solidification of  $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$ ," presented at Fifth International Conference on Vapor Growth/Fifth American Conference on Crystal Growth, Coronado, CA, July 19-24, 1981.

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,  $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$ ,  $\text{Pb}_x\text{Sn}_{1-x}\text{Te}$ ). 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  $\text{Pb}_x\text{Sn}_{1-x}\text{Te}$ ; 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

Bourret, E. D. and Witt, A. F., "Theoretical Analysis of Directional Melting," presented at American Conference on Crystal Growth, Coronado, CA, July 19-24, 1981; Proceedings of ACCG-5 (1981).

Bourret, E. D., Favier, J. J., and Witt, A. F., "Segregation During Directional Melting and Its Implications on Seeded Crystal Growth: A Theoretical Analysis," accepted for publication in Journal of Crystal Growth.

Bouret, E. D. and Witt, A. F., "Seeded Crystal Growth of Pseudo-Binary Systems with Large Liquidus-Solidus Separation," abstract in Ceramic Bulletin, March 1982; presented at ACS Annual Meeting, Cincinnati, Ohio, May 2-5, 1982.

Bouret, E. D., Adornato, P. M., Brown, R. A., and Witt, A. F., Proceedings of 33rd International Astronautical Congress, Paris, France, September 27-October 2, 1982.



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.

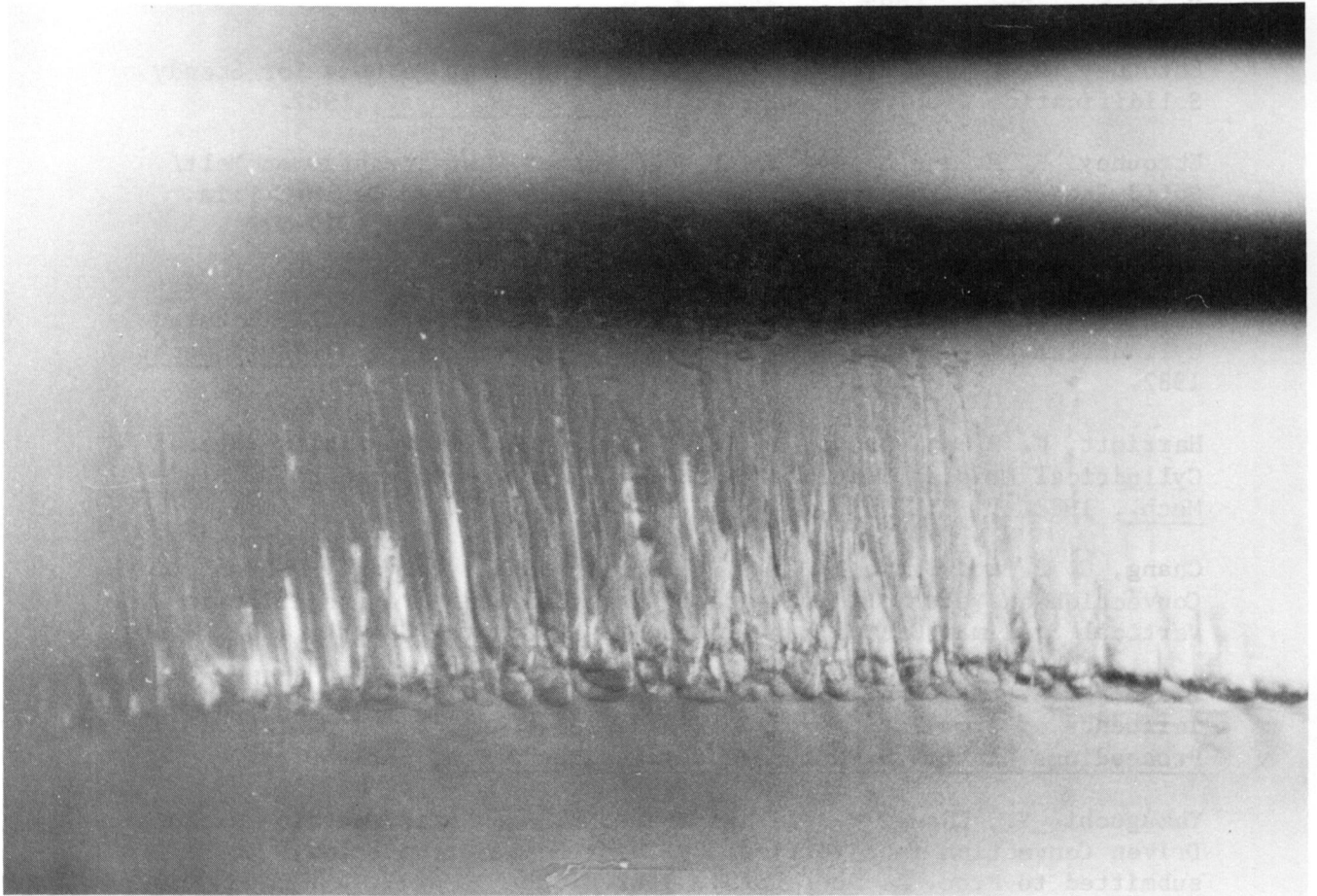
The finite element results are being extended to molten zones that are very deformed by gravity and so model earthbound floating zone experiments. The limits of static stability for these systems has been rigorously analyzed and a full-scale numerical simulation is underway that includes natural and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



## Publications

- Ungar, L. H. and Brown, R. A., "The Dependence of the Shape and Stability of Captive Rotating Drops on Multiple Parameters," Phil. Trans. R. Soc. Lond., in press, 1982.
- Chang, C. J. and Brown, R. A., "Finite Element Calculations Buoyancy-Driven Convection near a Melt/Solid Phase Boundary," Proceedings Second National Symposium on Numerical Methods in Heat Transfer, Hemisphere Press, 1982.
- Ettouney, H. M. and Brown, R. A., "Finite Element Methods for Steady Solidification Problems," submitted to J. Comp. Phys., 1982.
- Ettouney, H. M. and Brown, R. A., "Effect of Heat Transfer on Melt/Solid Interface Shape and Solute Segregation in Edge Defined Film Growth: Finite Element Analysis," J. Cryst. Growth 58, 313-329 (1982).
- Harriott, G. M. and Brown, R. A., "Flows in a Differentially Rotated Cylindrical Drop at Low Reynolds Number," submitted to J. Fluid Mech., 1982.
- Harriott, G. M. and Brown, R. A., "Flows in a Differentially Rotated Cylindrical Drop at Moderate Reynolds Number," submitted to J. Fluid Mech., 1982.
- 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.
- Brown, R. A., "Natural Convection in Melt Crystal Growth: The Influence of Flow Pattern on Solute Segregation," to be published in Proceedings Ninth U.S. Congress Applied Mechanics, 1982.
- 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.

Coriell  
004



Cellular structure formed as a result of interfacial instability in a sample of succinonitrile containing  $2 \times 10^{-3}$  wt. percent ethanol, growing at a velocity of 4 micrometers per second. This cellular structure is concentrated in the central region of the cylindrical crystal growth chamber due to the effects of radial temperature gradients.

Solutal Convection During Directional Solidification

National Bureau of Standards

S. R. Coriell

R. J. Schaeffer

H-27954B \$195K/year

April 1977 - 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.

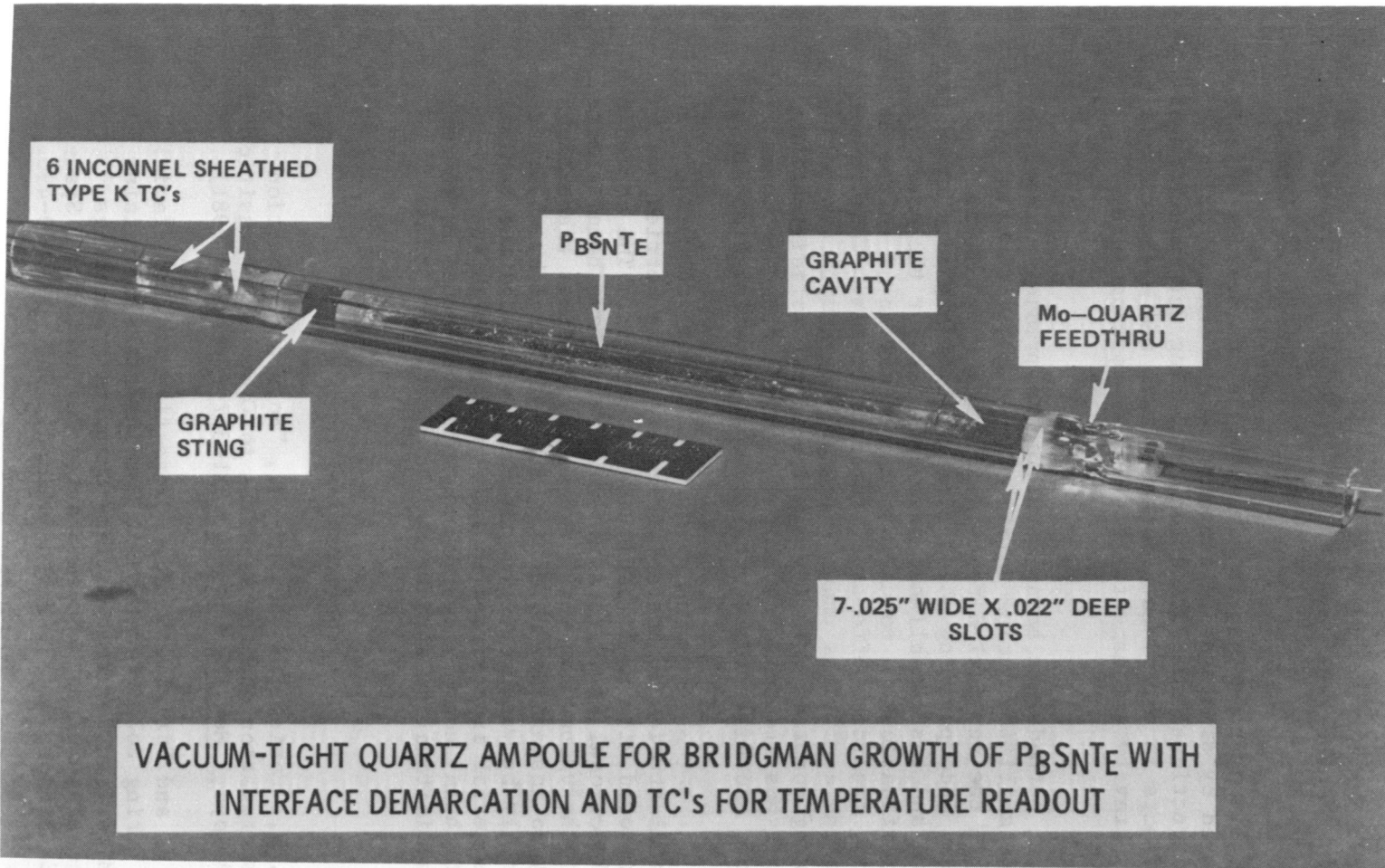
Theoretical methods are being developed to predict convective and interfacial instabilities during unidirectional solidification of binary alloys in the vertical direction. Linear hydrodynamic stability theory incorporating solidification boundary conditions has been formulated and has established some criteria for the onset of nonplanar solidification and nonuniform incorporation of solute in the resulting solid, including effects of magnetic fields. This research will delineate the conditions under which gravity-driven convection will occur during constant velocity unidirectional solidification. Numerical algorithms are being developed to solve the non-linear fluid flow, heat flow, and diffusion equations in two spatial dimensions.

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, and the behavior of the doped material is compared to that of ultra-high purity material in which any convective flow must be attributed to thermal gradients or solidification shrinkage.

## Publications

Boettinger, W. J., Coriell, S. R., et al., "Solutal Convection Induced Macrosegregation and the Dendrite to Composite Transition in Off-Eutectic Alloys," Met. Trans. 12A, 321-327 (February 1981).

Schaeffer, R. J. and Coriell, S. R., "Convective and Interfacial Instabilities During Solidification of Succinonitrile Containing Ethanol," in Materials Research Society Symposia Proceedings, Materials Processing in the Reduced Gravity Environment of Space, Volume 9 (G. E. Rindone, ed.), North-Holland, 1982, pp. 479-490.



Ampoule configuration which allows vacuum tight feedthroughs for interface demarcation as well as a grooved tube to allow for placement of 6 thermocouples around the outside without changing the overall o.d. of the ampoule.

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

Crouch, R. K., Fripp, A. L., et al., "Experiment Requirements and Implementation Plan (ERIP) for Semiconductor Growth in Low-g Environment, Experiment No. MPS 77F087," NASA TM-81943, February 1981.

Clark, I. O., Fripp, A. L., et al., "Solutal Diffusion Coefficient for Liquid PbTe-Sn-Te," to be submitted to J. Electrochem. Soc.

Crouch, R. K., Debnam, W. J., and Ryan, R., "Vacuum Tight Quartz Ampoule for Bridgman Growth of Crystals with Interface Demarcation," J. Cryst. Growth 56, 215-216 (1982).

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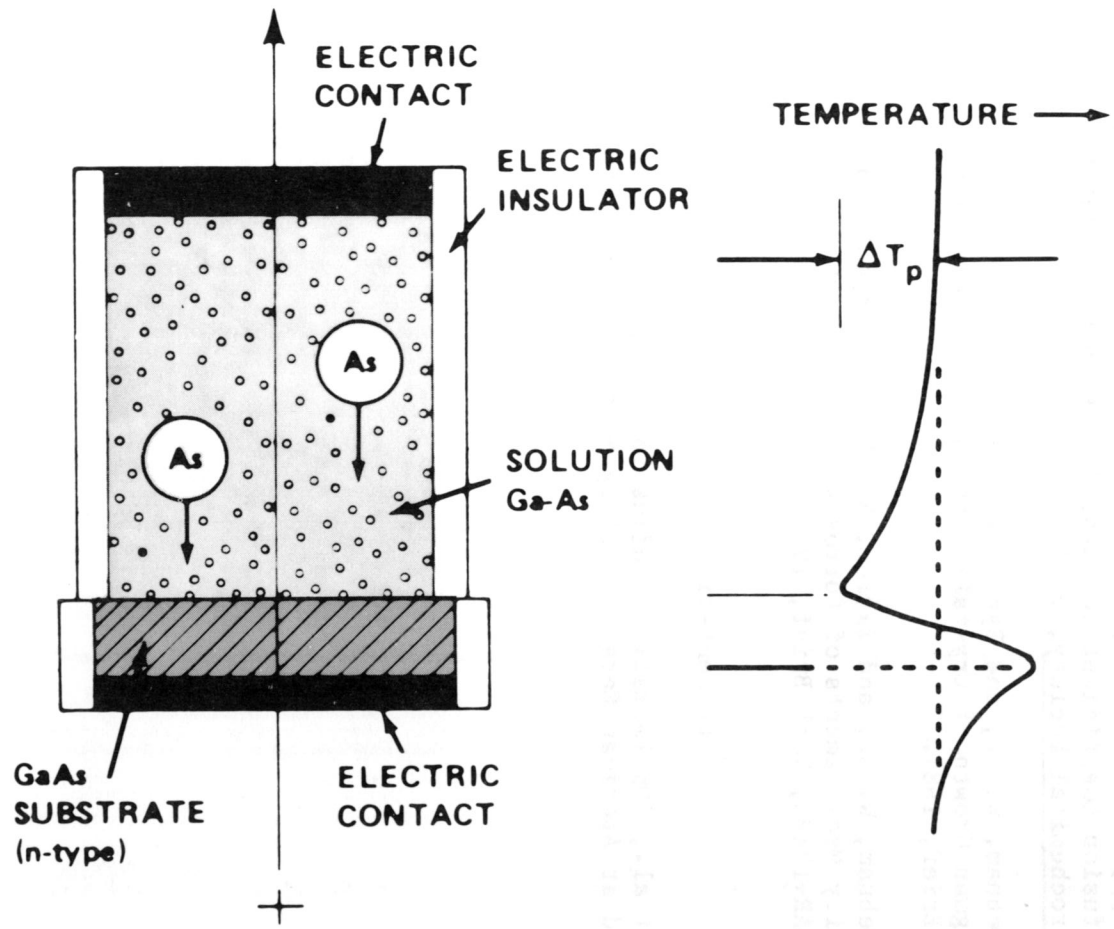
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Crouch, R. K., Debnam, W. J., and Ryan, R., "Vacuum Tight Quartz Ampoule for Bridgman Growth of Crystals with Interface Demarcation," LAR-12899, Tech Brief, 1981.

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Schematic representation of electroepitaxial growth.

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.

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- Okamoto, A., Lagowski, J., and Gatos, H. C., "Enhancement of Interface Stability in Liquid Phase Electroepitaxy," to be published in Journal of Applied Physics, 1982.
- Lagowski, J., Gatos, H. C., Parsey, J. M., Wada, K., Kaminska, M., and Walukiewicz, W., "Origin of the 0.82 eV Electron Trap in GaAs in its Annihilation by Shallow Donors," accepted by Applied Physics Letters.

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Microgravity Silicon Zoning Investigation

Westech Systems, Inc.  
Dr. E. L. Kern, Consultant  
G. L. Gill  
Professor Oscar Stafsudd, UCLA  
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 grow silicon crystals with dopant or alloy content in ranges that are impossible to grow at 1-g. These crystals are needed for new infrared detection and imaging devices. Precursory Materials Experiment Assembly (MEA) flights will show the feasibility of growing deep level and heavily doped silicon crystals and silicon-germanium alloy crystals at small diameters (5-7 mm). The slow and relatively unfluctuating growth, with a tailored heat flux and lack of gravity, should allow for growth with much higher dopant and germanium concentrations. Ground-based research, sufficient to specify the experimental conditions necessary in space and to specify the flight equipment requirements, will be done in the first nine month period. The ground-based research will be completed and the flight hardware designed and built in a second year period. This small diameter zoning in space will further characterize the Marangoni effect under float zone crystal growth conditions and can be correlated to predictions from modeling work now being done by other investigations.



Solution Growth of Crystals in Zero-Gravity

Alabama A&M University

Dr. R. B. Lal

Dr. R. L. Kroes, MSFC

NAS8-32945 Total Cost: \$492K

June 28, 1978 - June 28, 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.

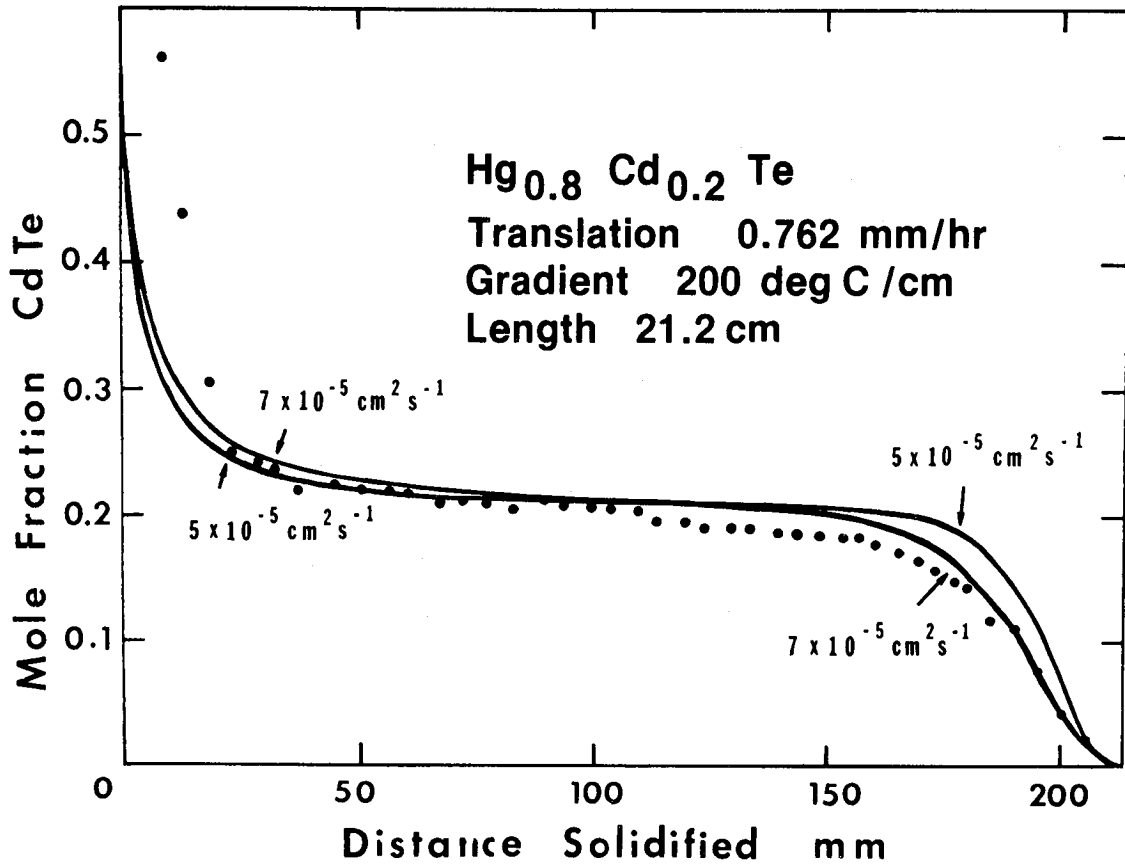
Publications

Lal, R. B., Kroes, R. L., and Wilcox, W. R., "Growth Triglycine Sulfate (TGS) Crystals by Solution Technique," in Materials Research Society Symposia Proceedings, Materials Processing in the Reduced Gravity Environment of Space, Volume 9 (G. E. Rindone, ed.), North Holland, 1982, pp. 399-408.

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Presentations

Wilcox, W. R., Lal, R. B., and Kroes, R. L., "Crystallization in Space," presented at Second World Congress of Chemical Engineering, Montreal, Canada, October 4-9, 1981.



Comparison of experiment and model results for a directionally solidified crystal. The initial liquid composition was  $z = .20$ . Calculated curves are shown for  $D = 5 \times 10^{-5} \text{ cm}^2/\text{s}$  and  $D = 7 \times 10^{-5} \text{ cm}^2/\text{s}$ .



Growth of Solid Solution Crystals

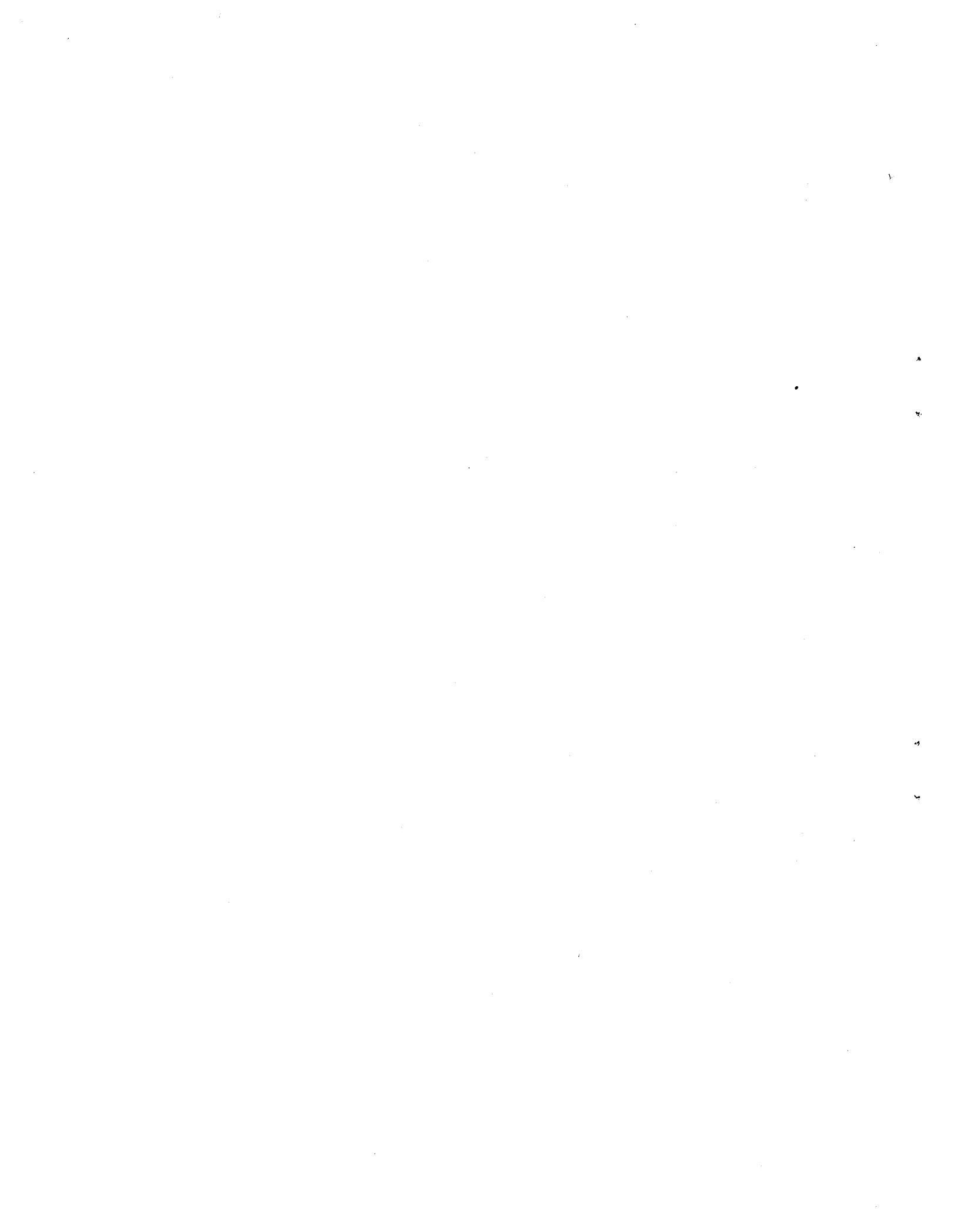
Marshall Space Flight Center  
Dr. S. L. Lehoczky  
Dr. F. R. Szofran  
Dr. L. R. Holland, UAH  
Dr. J. C. Clayton, Semtec  
Dr. D. C. Gillies, Semtec  
In-House Total Cost: \$540K  
October 1977 - October 1982

The major objective of this program is to determine the conditions under which single crystals of solid solutions can be grown from the melt in a Bridgman configuration with a high degree of chemical homogeneity. The central aim is to assess the role of gravity in the growth process and to explore the possible advantages for growth in the absence of gravity. The alloy system being investigated is the solid solution semiconductor  $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$  with x-values appropriate for infrared detector applications in the 8 to 14  $\mu\text{m}$  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.



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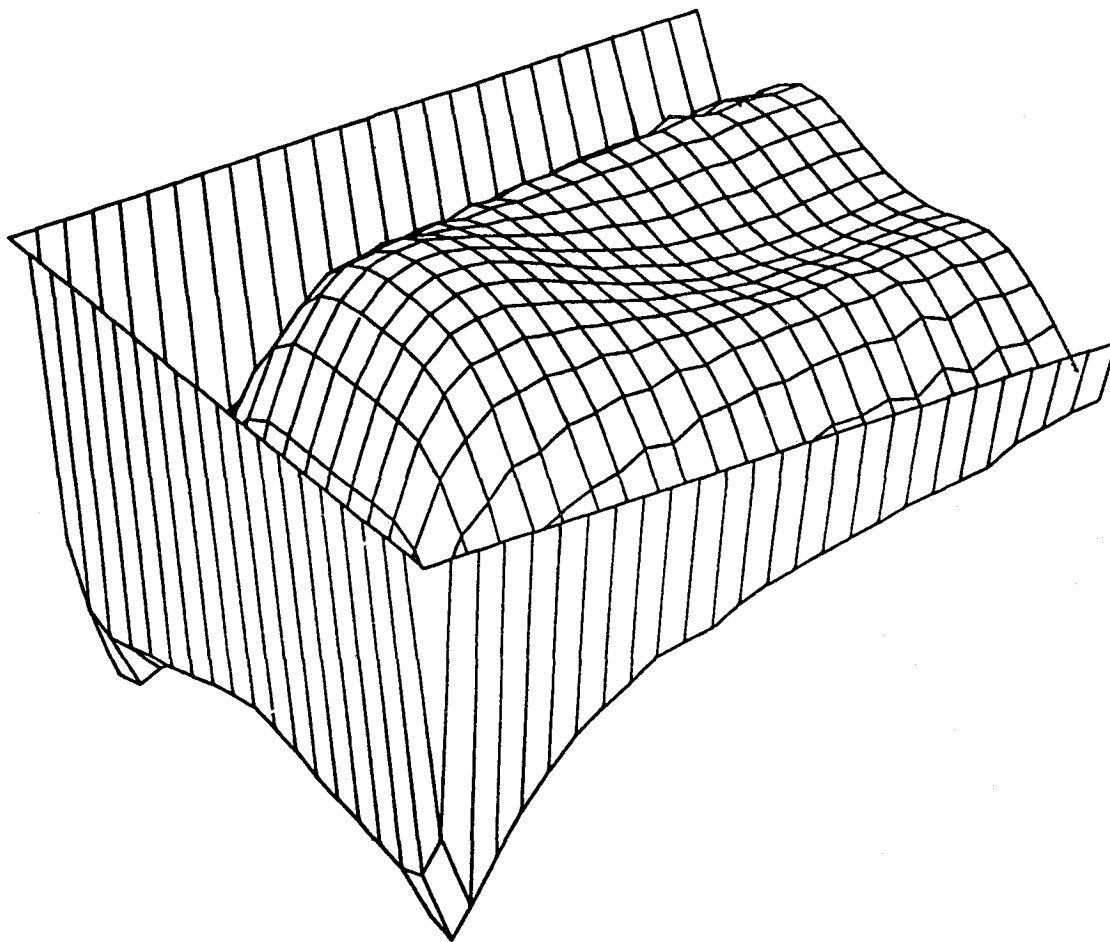
Clayton, J. C., Davidson, M. C., Gillies, D. C., and Lehoczky, S. L., "One Dimensional Analysis of Segregation in Directionally Solidified HgCdTe", J. Crystal Growth (in press).

Clayton, J. C., "Transient and Diffusion Analysis of HgCdTe", NASA CR-162049, 1982.

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

Holland, L. R., "Apparatus and Method for Heating a Material in a Transparent Ampoule", Patent pending (Application Serial Number: 280151).

Gillies, D. C., Clayton, J. C., "Indirect Determination of the Interface in Solid Solution Crystals", J. Crystal Growth (submitted).



Distribution of vertical velocity at midheight of crystal end wall  
in horizontal vapor growth ampoule (laser doppler anemometry study).

Fluid Dynamics of Crystallization from Vapors

University of Utah - Salt Lake City  
 Dr. F. Rosenberger  
 NSG-1534 Total Cost: \$271K  
 June 1978 - September 1982\*

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 destabilizing 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. Modeling of vapor transport across a horizontal cavity has shown that at lower transport rates earlier, simplifying treatments (Klosse-Ullersma, KU), because of fortuitous cancellation of errors, give reasonable results for two-dimensional systems. However, laser Doppler anemometry studies of the convective velocity fields in inclined and horizontal ampoules revealed three-dimensional features of the flow, that had generally not been accounted for in modeling. Titrometric and vapor pressure studies have shown that deviations in stoichiometry of mercuric iodide ( $\text{HgI}_{2+x}$ ) 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.

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Markham, B. L., Greenwall, D. W., et al., "Numerical Modeling of Diffusive Vapor Transport in Vertical Ampoules," J. Crystal Growth 51, 413 (1981).

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\*contract to be extended



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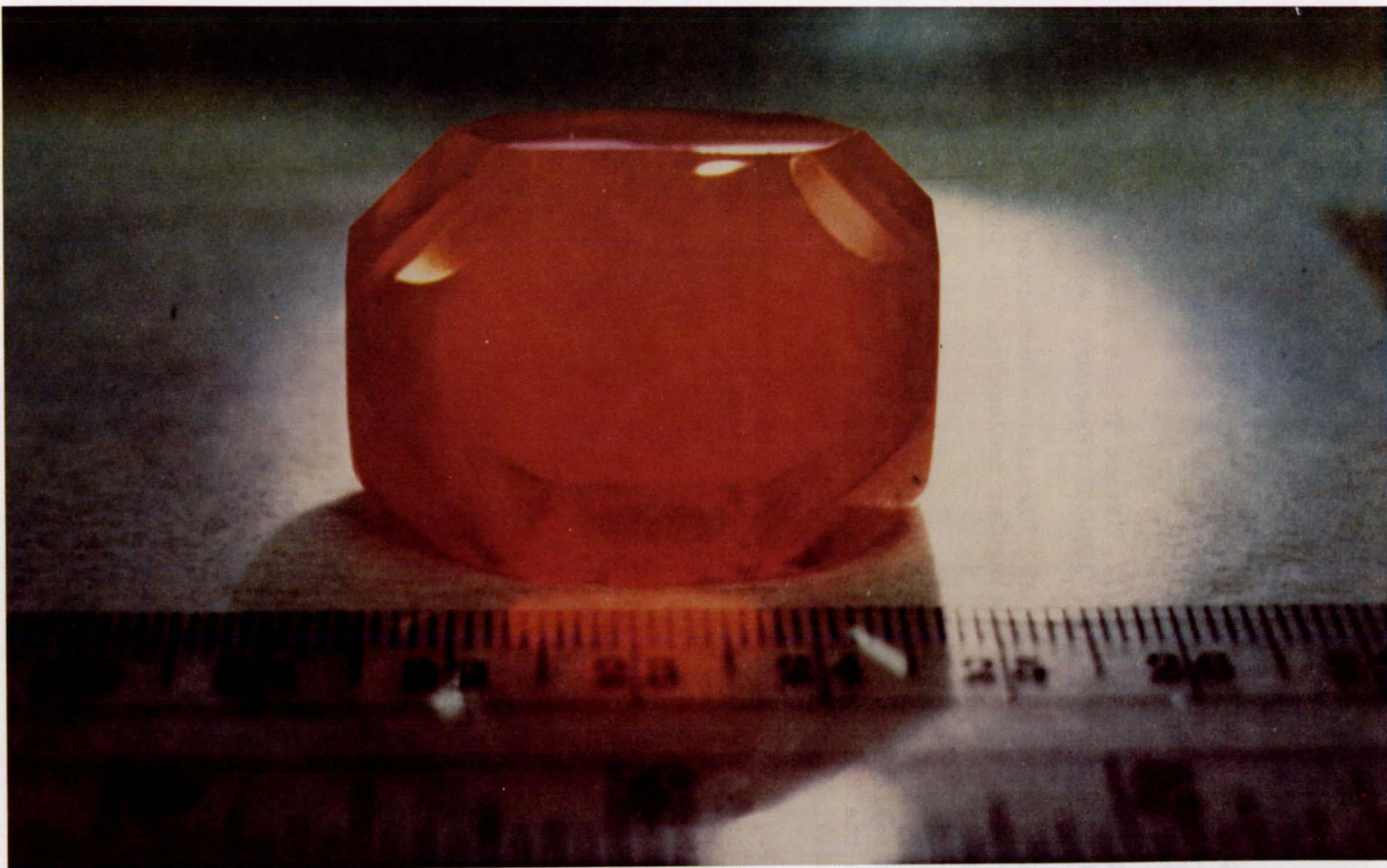
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Rosenberger, F., "Mass Transfer in Closed Ampoule Vapor Crystal Growth," in Materials Research Society Symposia Proceedings, Materials Processing in the Reduced Gravity Environment of Space, Volume 9 (G. E. Rindone, ed.), North-Holland, 1982, pp. 199-210.

Rosenberger, F., "Convection in Vapor Crystal Growth Ampoules," in Convective Transport and Instability Phenomena (J. Zierep and H. Oertel, eds), Braun Verlag, in press.



Terrestrial grown mercuric iodide crystal.

Schnepple  
011



HgI<sub>2</sub> 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 measurements have been performed (uniaxial compression tests) which show that the crystals exhibit slip parallel to the c-planes at stresses as low as 1/2 psi. Preliminary calculations using a simple linearized model indicate the oscillating instabilities in the convection part of the vapor transport system are unlikely, even at 1-g, provided that the utmost care is taken in the preparation of the crystal growth source material.

## Publications

Iwanczyk, J. S., Dabrowski, A. J., Huth, G. C., Del Duca, A., and Schnepple, W. J., "A Study of Low-Noise Preamplifier Systems for Use with Room Temperature Mercuric Iodide (HgI<sub>2</sub>) X-Ray Detectors," IEEE Trans. Nuc. Sci., NS-28, 579 (1981).

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Dishon, G., Schieber, M. M., Ben-Dor, L., and Halitz, H., "On the Stoichiometry of  $\text{HgI}_2$ ," to be published in Mat. Res. Bull.

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Ricker, G. R., Vallergera, J. V., Schneppe, W. J., and Ortale, C., "A Bismuth Germanate-Shielded Mercuric Iodide X-ray Detector for Space Applications," presented at IEEE 1981 Nuclear Science Symposium, San Francisco, CA, October 1981.

Levi, A., Roth, M., and Schieber, M. M., "The Development of Mercuric Iodide Gamma Radiation Detectors for Application in Nuclear Medicine," presented at IEEE 1981 Nuclear Science Symposium, San Francisco, CA, October 1981.

Ortendahl, D., Kaufman, K., Hosier, K., Padgett, L., and Ortale, C., "Operating Characteristics of Small Position-Sensitive Mercuric Iodide Detectors," presented at IEEE 1981 Nuclear Science Symposium, San Francisco, CA, October 1981.

Direct Observation of Interface Stability

Stanford University  
Professor W. A. Tiller  
Professor R. S. Fiegelson  
Dr. Dennis Elwell  
NAS8-33110 Total Cost: \$265K  
December 1, 1978 - January 31, 1982

This task will use a direct observation method to facilitate the careful test of interface stability criteria in a convective environment. This study will form the basis for future experiments under nonconvective conditions. The objective is a careful test of theory with experiment on a model system with all the significant material parameters being measured for this system. Such a careful test has not been performed heretofore.

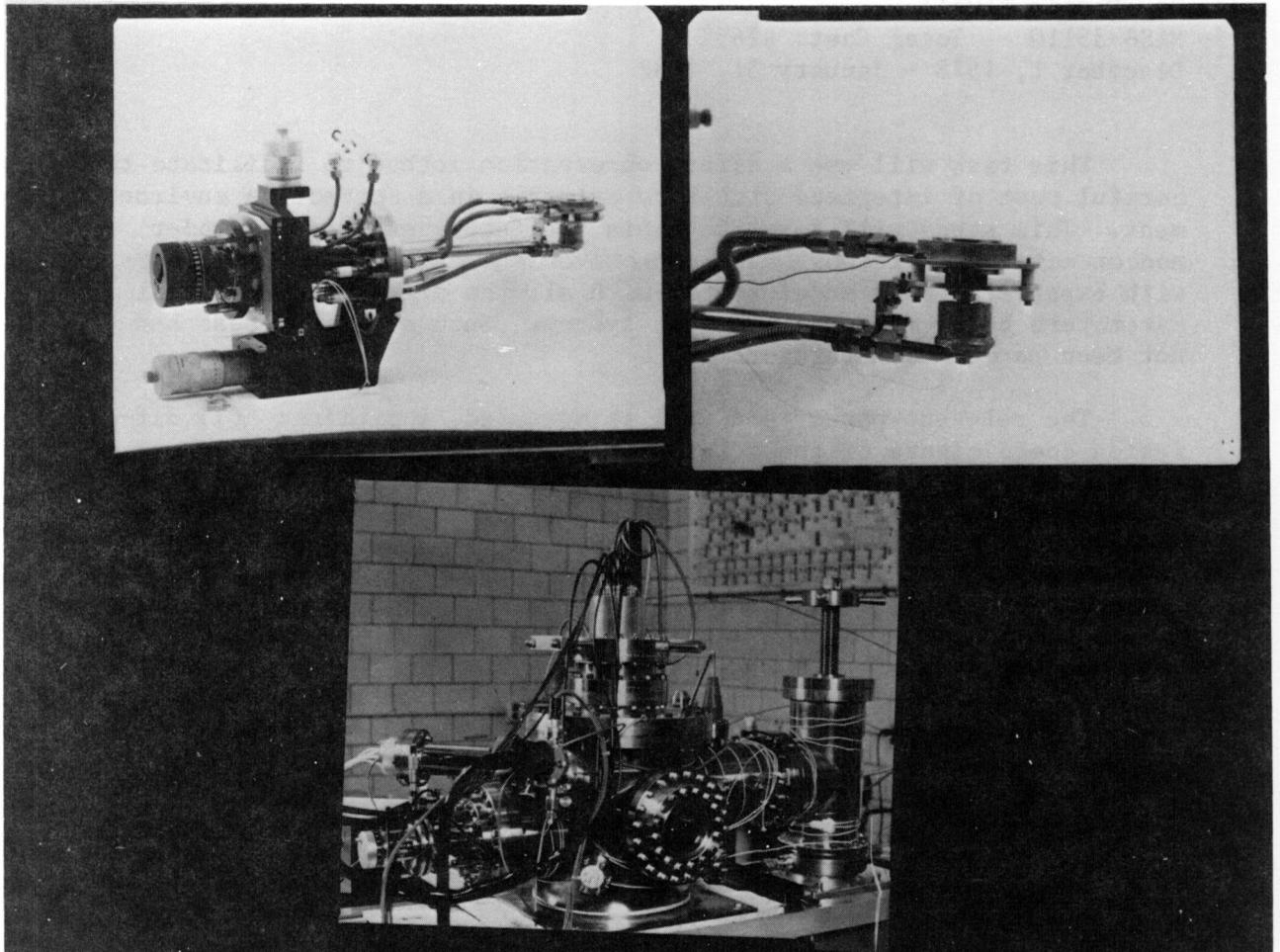
The relevant parameters will be measured, including: (1) diffusion coefficients of the solutes in the liquid, (2) phase diagram and effective distribution coefficients for the solute, (3) liquidus slopes for the chosen solutes, (4) thermal conductivities and diffusivities for both liquid and solid, and (5) the solid-liquid interfacial energy.

Publications

Ahn, K. S. and Tiller, W. A., "Interface Field Effects in the Czochralski Growth of CsCdC<sub>3</sub> Crystals," to be submitted to Journal of Crystal Growth.

Ahn, K. S. and Tiller, W. A., "Interface Field Effects on Constitutional Supercooling," to be submitted to Journal of Crystal Growth.

Tiller, W. A. and Ahn, K. S., "The Effect of Interface Fields on the Stability of a Planar Interface," to be submitted to Journal of Crystal Growth.



Series of 3 photographs of the equipment developed for this NASA project. The photo at the upper right is a close-up view of the disk heater and the upper left photo shows how it is attached to the manipulator which holds it in the UHV system. The UHV system is shown in the lower photo. It includes an Auger analyzer, an ion sputter gun and a residual gas analyzer. The disk heater assembly attaches at the left side port in place of the manipulator or shown in the lower photo.

Float Zone Experiments in Space

Ames Laboratory, Iowa State University  
Dr. J. D. Verhoeven  
H-34328B     \$82K/year  
October 1981 - October 1982

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 oscillatory temperatures. For objective No. 2, solute profiles will be evaluated in the initial transient zones and the quenched zones.

Publications

Verhoeven, J. D., Noack, M. A., and Bevelo, A. J., "Suppression of Marangoni Convection with Oxide Films," in Materials Research Society Symposia Proceedings, Materials Processing in the Reduced Gravity Environment of Space, Volume 9 (G. E. Rindone, ed.), North Holland, 1982, pp. 513-522.



Defect Chemistry and Characterization of (Hg, Cd) Te

Honeywell

Dr. H. R. Vydyanath

NAS8-33245 Total Cost: \$246K

December 18, 1978 - March 17, 1982

( $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$ ) is a variable bandgap alloy semiconductor extensively used for infrared applications. Detector performance close to theoretical limits imposed by infrared imaging systems requires extreme control of compositional uniformity, carrier concentration, and carrier lifetime. The program deals with the study of the nature and concentration of the lattice defects incorporated into ( $\text{Hg}_{1-x}\text{Cd}_x$ )Te alloys as a function of the physiochemical conditions of preparation; namely, the temperature, the partial pressures of the constituent elements, and/or the concentration or the activity of the dopant being incorporated.

Undoped, donor-doped, and acceptor-doped ( $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$ ) samples are annealed at various temperatures in suitable Hg atmospheres. The samples are quenched to room temperature from the high temperatures. Hall effect and resistivity measurements are carried out at 77°K to determine the carrier concentrations and mobilities. The variation of the carrier concentrations as a function of the partial pressure of Hg and/or the dopant concentration is used to arrive at defect models for the doped and the undoped crystals.

At the end of the 24 month period of the program, significant accomplishments have been made toward understanding the nature of lattice defects and the mode of incorporation of different dopants. For the first time in literature, the defect structures of undoped  $\text{Hg}_{0.6}\text{Cd}_{0.4}\text{Te}$  (s), copper doped, indium doped, and iodine doped, phosphorus doped  $\text{Hg}_{0.8}\text{Cd}_{0.2}\text{Te}$  (s) have all been established. The native acceptor defects have been found to be doubly ionized in both  $\text{Hg}_{0.6}\text{Cd}_{0.4}\text{Te}$  (s) and  $\text{Hg}_{0.8}\text{Cd}_{0.2}\text{Te}$  (s). Native donor defects are found to be negligible in concentration in these alloys and the origin of p-type to n-type conversion has been shown to be due to residual foreign donors and not due to native donor defects.

Of the dopants studies, copper and indium have been found to occupy only Hg lattice sites acting with single acceptor and donor electrical activities respectively whereas iodine is found to act as a single donor occupying only Te sites. A large concentration of indium is found to be incorporated in  $\text{In}_2\text{Te}_3$  with only a small fraction acting as donors. Crystals doped with iodine are found to be saturated with the metal iodide with a large fraction of iodine being paired with the native acceptor defects. Results on crystals doped with phosphorus behaves amphoterically acting as a donor on Hg lattice sites and as an acceptor interstitially and on Te lattice sites. Thermodynamic constants have been established for the incorporation of the native

defects as well as the different dopants. These constants satisfactorily explain all the experimental results.

#### Publications

Vydyanath, H. R., "Lattice Defects in  $Hg_{1-x}Cd_xTe$  Alloys, I-Defect Structure of Undoped and Copper Doped  $Hg_{0.8}Cd_{0.2}Te$ ," J. Electrochem. Soc. 128, 2609 (1981).

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Vydyanath, H. R. and Kroger, S. A., "Doping Behavior of Iodine in Mercury  $Hg_{0.8}Cd_{0.2}Te$ ," J. Elect. Mat. 11, 111 (1982).

Vydyanath, H. R. and Abbott, R. C., "Mode of Incorporation of Phosphorus in  $Hg_{0.8}Cd_{0.2}Te$ ," submitted to Journal of Applied Physics, November 1981.



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 properties of these materials, and the existence of microgravity results for some of these systems investigated in previous Skylab and ASTP experiments.

## Publications

Von Schnering, H. G. and Wiedemeier, H., "The High Temperature Structure of  $\beta$ -SnS and  $\beta$ -SnSe and the B16-to-B33 Type  $\lambda$  Transition Path," Z. Kristallog. 156, 143 (1981).

Wiedemeier, H., Pultz, G., Gaur, U., and Wunderlich, B., "Heat Capacity Measurements of SnSe and SnSe<sub>2</sub>," Thermochim. Acta 43, 297 (1981).



Vapor Growth of Alloy-Type Semiconductor Crystals

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  $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$  and  $\text{CuInS}_2$  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  $\text{Hg}_{.8}\text{Cd}_{.2}\text{Te}$  crystals obtained under vertical stabilizing conditions are improved relative to crystals grown under horizontal conditions. The crystal quality of  $\text{CuInS}_2$  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

Wiedemeier, H., Chandra, D., and Klaessig, F. C., "Diffusive and Convective Vapor Transport in the  $\text{GeSe-GeI}_4$  System," J. Cryst. Growth 51, 345 (1981).

Wiedemeier, H., Gaur, U., Pultz, G., and Wunderlich, B., "Analysis of the Heat Capacities of Group IV Chalcogenides Using Debye Temperatures," J. Thermal Analysis 21, 309 (1981).

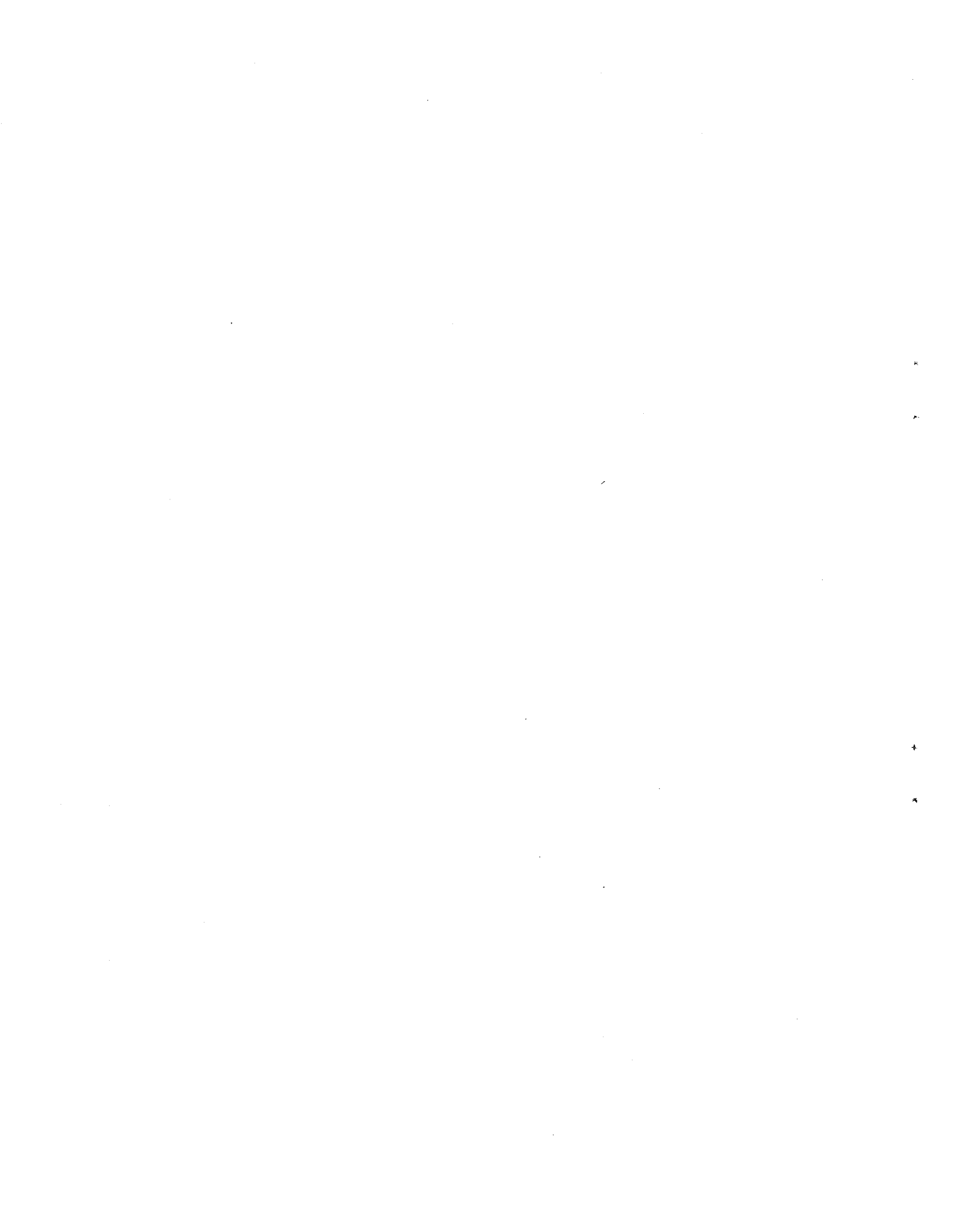
Chandra, D. and Wiedemeier, H., "Chemical Vapor Transport and Thermal Behavior of the  $\text{GeSe-GeI}_4$  System for Different Inclinations with Respect to the Gravity Vector, Comparison with Theoretical and Microgravity Data," J. Cryst. Growth 57, 159-176 (1982).



Wiedemeier, H., Chandra, D., Yang, P. H., and Koniowka, W. J., "Crystal Growth and Chemical Transport Properties of  $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$  and  $\text{CuInS}_2$ ," J. Electrochem. Soc. 81, 157 (1981).

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Wiedemeier, H. and Chandra, D., "Chemical Vapor Transport and Crystal Growth of the  $\text{Hg}_{0.8}\text{Cd}_{0.2}\text{Te}$  System, Crystal Morphology and Homogeneity," Z. Anorg. Allg. Chem., in press.



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 15 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 m/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

Bouret, E. D., Favier, J. J., and Witt, A. F., "Segregation During Directional Melting and Its Implications on Seeded Crystal Growth: A Theoretical Analysis," NASA CR-169072, 1982.

Jasinski, T. J., Rohsenow, W. M., and Witt, A. F., "Heat Transfer Analysis of the Bridgman-Stockbarger Configuration for Crystal Growth. Part I: Analytical Treatment of the Axial Temperature Distribution," accepted for publication in the Journal of Crystal Growth.

Wang, C. A., Carruthers, J. R., and Witt, A. F., "End-Effects on Crystal Growth and Segregation Behavior in Vertical Bridgman Configuration," in preparation for the Journal of Crystal Growth.

Jasinski, T. J., Rohsenow, W. M., and Witt, A. F., "Heat Transfer Analysis of the Bridgman-Stockbarger Configuration for Crystal Growth. Part II: Analytical Treatment of the Radial Temperature Distribution," in preparation for the Journal of Crystal Growth.



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.

Publications

Zoutendyk, J. A. and Akutagawa, W. A., "Gravity-Driven Convection Studies in Compound Semiconductor Crystal Growth by Physical Vapor Transport," in Materials Research Society Symposia Proceedings, Materials Processing in the Reduced Gravity Environment of Space, Volume 9 (G. E. Rindone, ed.), North Holland, 1982, pp. 449-458.

Zoutendyk, J. A. and Akutagawa, W. A., "Diffusive-Convective Physical Vapor Transport of PbTe from a Te-rich Solid Source," J. Cryst. Growth 56, 245-253 (1982).



## 2. METALS, ALLOYS, AND COMPOSITES

TASK NUMBER (TN)	PRINCIPAL INVESTIGATOR (PI)	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 steel wherein small carbide particles are included in the 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 ( $\text{SmCo}_5$ ) 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 techniques 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  $\text{Nb}_3\text{Ge}$ , 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.



Interference pattern created by horizontal cellular motion resulting from side heating a stable vertical density gradient established by stratified sucrose solutions.

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, 1979 - October 1, 1982

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<sup>3</sup> through a 100  $\mu$ m depth and can resolve particles of the order of 2 to 3  $\mu$ m in diameter throughout the entire cell volume. Particle size distribution changes with respect to time and temperature are determined from sequential holograms. Initial experiments using diethylene glycol/ethyl salicylate (DEG/ES) have demonstrated the usefulness of the technique. The thermal system for cell temperature control is presently being refined to give control to at least  $\pm 0.001^\circ\text{K}$  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 phenomenon are observable by holographic photography. Solid-liquid interfacial free energy differences are, in principle accessible by film pressure (via ellipsometry) measurements. Viewing holographic studies and interfacial free energy measurements in light of segregation profiles of model solidified ingots should yield valuable verification of operational limits. Other methods of study, including contact angle measurements and heats of immersion, are under consideration.

A temperature gradient stage with high thermal stability is being assembled to study solidification phenomena in situ using model organic systems. Laser interferometry will be performed with this stage in order to measure concentration gradients in the liquid ahead of an advancing solid/liquid interface. The model organic systems that will be studied 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.

## Publications

Nishioka, G. M., Lacy, L. L., and Facemire, B. R., "The Gibbs Surface Excess in Binary Miscibility Gap Systems," J. Colloid Interface Sci. 80, 197-207 (March 1981).

Kaukler, W. F., "A Quantitative Study of Factors Influencing Lamellar Eutectic Morphology During Solidification," NASA TM-82451, November 1981.

Frazier, D. O., "Solidification Phenomena," presented at TMS/AIME Fall Meeting, Louisville, KY, October 1981.

Witherow, W. K., "Optical Studies of a Model Miscibility Gap System," presented at TMS-AIME Fall Meeting, Louisville, KY, October 1981.



Liquid Phase Miscibility Gap Materials

S. H. Gelles Associates

Dr. S. H. Gelles

Dr. A. J. Markworth - Battelle Columbus Laboratories

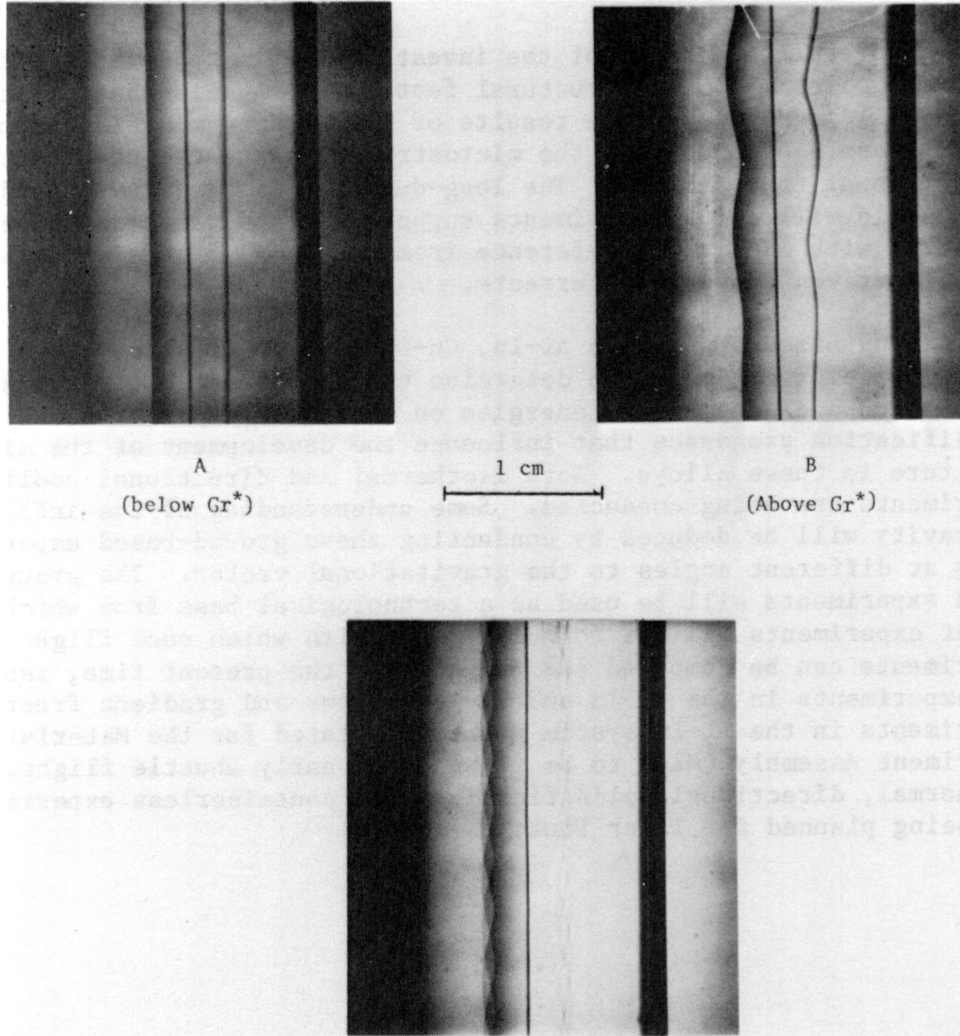
NAS8-32952 Total Cost: \$480K

April 1978 - April 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. Some understanding of the influence of gravity will be deduced by conducting these ground-based experiments at different angles to the gravitational vector. The ground-based experiments will be used as a technological base from which the flight experiments will be formulated and with which such flight experiments can be compared and judged. At the present time, isothermal experiments in the Al-In and Te-Tl systems and gradient freeze experiments in the Al-In system are contemplated for the Materials Experiment Assembly (MEA) to be flown on an early Shuttle flight. Isothermal, directional solidification, and containerless experiments are being planned for later Shuttle flights.

GRAVITY-INDUCED SOLIDIFICATION WAVES



A  
(below  $Gr^*$ )

1 cm

B  
(Above  $Gr^*$ )

C  
(Time Lapse)

Configuration of the solid-liquid interface in a cylindrical convection cell. For small temperature differences between the wire and the solid-liquid interface a cylindrical, stationary interface forms (A). Above a critical temperature difference—described most conveniently by a critical Grashof number,  $Gr^*=200$ ,—the interface becomes helical in shape, and steady rotates in a wave-like manner about the heating wire (B). The time-lapse photograph (3, 20-minute intervals) show the wave progression along the solid-liquid interface (C).

Dendritic Solidification at Small Supercoolings

Rensselaer Polytechnic Institute  
Dr. M. E. Glicksman  
NAS8-32425 Total Cost: \$313K  
March 1, 1977 - June 30, 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

Glicksman, M. E., "Fundamentals of Dendritic Microstructures," published in Proceedings of the Second International Symposium on Aluminum Transformation Technology and Its Applications, Buenos Aires, Argentina, August 1981, American Society of Metals.

Glicksman, M. E. and Huang, S. C., "Fundamentals of Dendritic Solidification, Part I - Steady State Tip Growth," Acta Met. 29, 701-715 (1981).

Glicksman, M. E. and Huang, S. C., "Fundamentals of Dendritic Solidification, Part II - Development of Sidebranch Structure," Acta Met. 29, 717-734 (1981).



Glicksman, M. E. and Huang, S. C., "Boundary Layer Analysis for the Convection/Diffusion Transition in Dendritic Growth," Adv. Space Res. 1, 25-35 (1981).

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Schaefer, R. J. and Glicksman, M. E., "Quantitative Kinetic and Morphological Studies Using Model Systems," in Modeling of Casting and Welding Processes (H. D. Brody and D. Apelian, eds.), The Metallurgical Society of AIME, New York, 1981, pp. 375-384.

Glicksman, M. E., Singh, N. M., and Chopra, M., "Influence of Diffusion and Convective Transport on Dendritic Growth in Dilute Alloys," in Materials Research Society Symposia Proceedings, Materials Processing in the Reduced Gravity Environment of Space, Volume 9 (G. E. Rindone, ed.), North-Holland, 1982, pp. 461-478.

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

#### Presentations

Glicksman, M. E., "Gravitational Influence on Eutectic Solidification," presented at TMS-AIME Fall Meeting, Louisville, KY, October 1981.

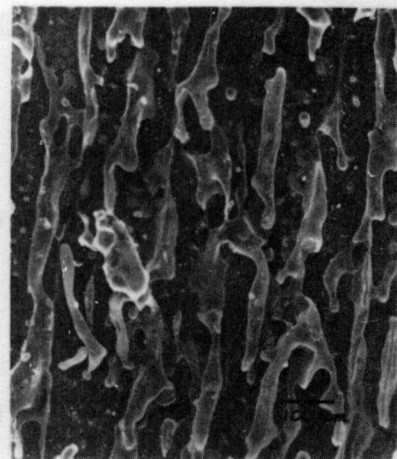


Fig. 1  
Cu-36 Wt.% Pb  $V = 5\mu\text{m s}^{-1}$   $G = 15\text{K mm}^{-1}$   
Deep etch, 50%  $\text{HNO}_3$ , SEM



Fig. 2  
Succinonitrile ~20 Wt.% Ethanol  
 $V = 0.9\mu\text{m s}^{-1}$   $G = 5\text{K mm}^{-1}$   
Transmitted light.

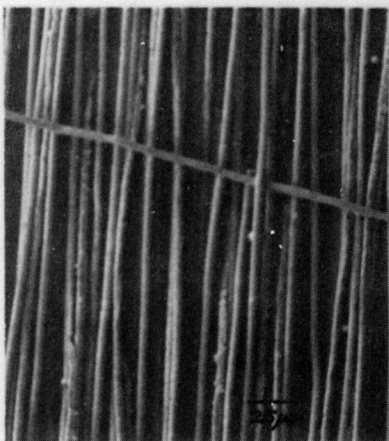


Fig. 3  
Al-17.5 Wt.% In  $V = 2.5\mu\text{m s}^{-1}$   
 $G = 19\text{K mm}^{-1}$ . Deep etch, dilute  
NaOH, SEM

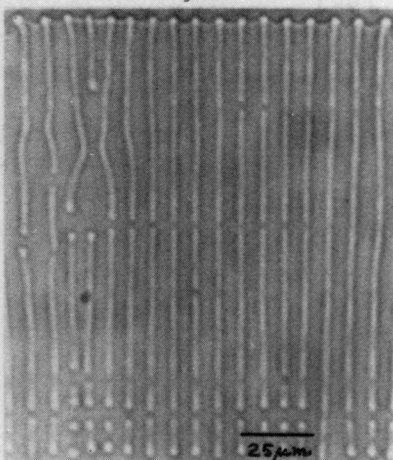


Fig. 4  
Succinonitrile ~7.5 Wt.% Glycerol  
 $V = 0.4\mu\text{m s}^{-1}$   $G = 5.5\text{K mm}^{-1}$   
Transmitted light.

Directionally solidified monotectic alloys. The two pictures on the left, Figs. 1 & 3, are of metallic alloys in which the matrix phase has been partially removed. These illustrate two types of solidification behavior in which, Fig. 1, the minor phase does not wet the matrix but is incorporated as irregular interconnected globules, and Fig. 3, the minor phase grows by steady state diffusion at a coupled growth to give a fine, well-aligned fibrous structure. The two pictures on the right, Figs. 2 & 4, are of transparent organic analogues, taken with a optical microscope in transmission on a temperature gradient stage. These analogues illustrate the same behavior as the metals and at the top of each picture are the advancing growth fronts of the reactions:  
Liquid I  $\rightarrow$  Solid + Liquid II.  
Solidification directions bottom  $\rightarrow$  top

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, and a transparent analogue  $(\text{CH}_2\text{CN})_2\text{-H}_2\text{O}$  as well as the ternary systems Al-In-Sn, Cu-Pb-Al and Cd-Ga-Al. The transparent analogue system is being examined in a temperature gradient stage on an optical microscope in order to study the detailed form of the duplex, solid + liquid growth front. The particular objective here will be to look at the incorporation or rejection of liquid droplets at the growth front and to modify the behavior with ternary additions.

Publications

Grugel, R. and Hellawell, A., "Alloy Solidification in Systems Containing a Liquid Miscibility Gap," Met. Trans. A. 12A, 669-681 (April 1981).

Grugel, R. N., Lograsso, T. A., and Hellawell, A., "Directional Solidification of Alloys in Systems Containing a Liquid Miscibility Gap," in Materials Research Society Symposia Proceedings, Materials Processing in the Reduced Gravity Environment of Space, Volume 9 (G. E. Rindone, ed.), North Holland, 1982, pp. 553-562.

Grugel, R. N. and Hellawell, A., "The Occurrence of Aligned Microstructures in Directionally Solidified Aluminum-Bismuth Alloys," Met. Trans. 13A, 493-495 (March 1982).





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 the samples are being chemically analyzed. To obtain additional low-gravity data, samples will be solidified during KC-135 flights.

Publications

Johnston, M. H. and Parr, R. A., "Low-Gravity Solidification Structures," published in Materials Research Society Symposia Proceedings, Materials Processing in the Reduced Gravity Environment of Space, Volume 9 (G. E. Rindone, ed.), North-Holland, 1982, pp. 651-656.

Johnston, M. H. and Parr, R. A., "The Influence of Acceleration Forces on Dendritic Growth and Grain Structure," Met. Trans. B 13B, 85-90 (March 1982).



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.

Publications

Nair, M., Fu, T. W., et al., "Response of Bi/MnBi Eutectic to Freezing Rate Changes," presented at Fifth International Conference on Vapor Growth and Epitaxy, San Diego, CA, July 1981.

Larson, D. J. and Pirich, R. G., "Characterization of Gravitationally Induced Convection on Solidification of the Bismuth-Manganese Eutectic," presented at Fifth International Conference on Vapor Growth and Epitaxy, San Diego, CA, July 1981.

Pirich, R. G., Larson, D. J., and Busch, G., "Studies of Plane-Front Solidification and Magnetic Properties of Bi/MnBi," AIAA Journal 19, 589 (1981).

Pirich, R. G. and Larson, D. J., "Influence of Gravity Driven Convection on the Directional Solidification of Bi/MnBi Eutectic Composites," in Materials Research Society Symposia Proceedings, Materials Processing in the Reduced Gravity Environment of Space, Volume 9 (G. E. Rindone, ed.), North Holland, 1982, pp. 523-531.



Interfacial Destabilization in Metal Alloys

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.

## Publications

Favier, J. J. Berthier, J., Arragon, Ph., Malmejac, Y., Khryapov, V., and Barmin, I. V., "Solid/Liquid Interface Stability in Normal and Microgravity Conditions," XXXII IAF Congress, Rome Italy, September 6-12, 1981.

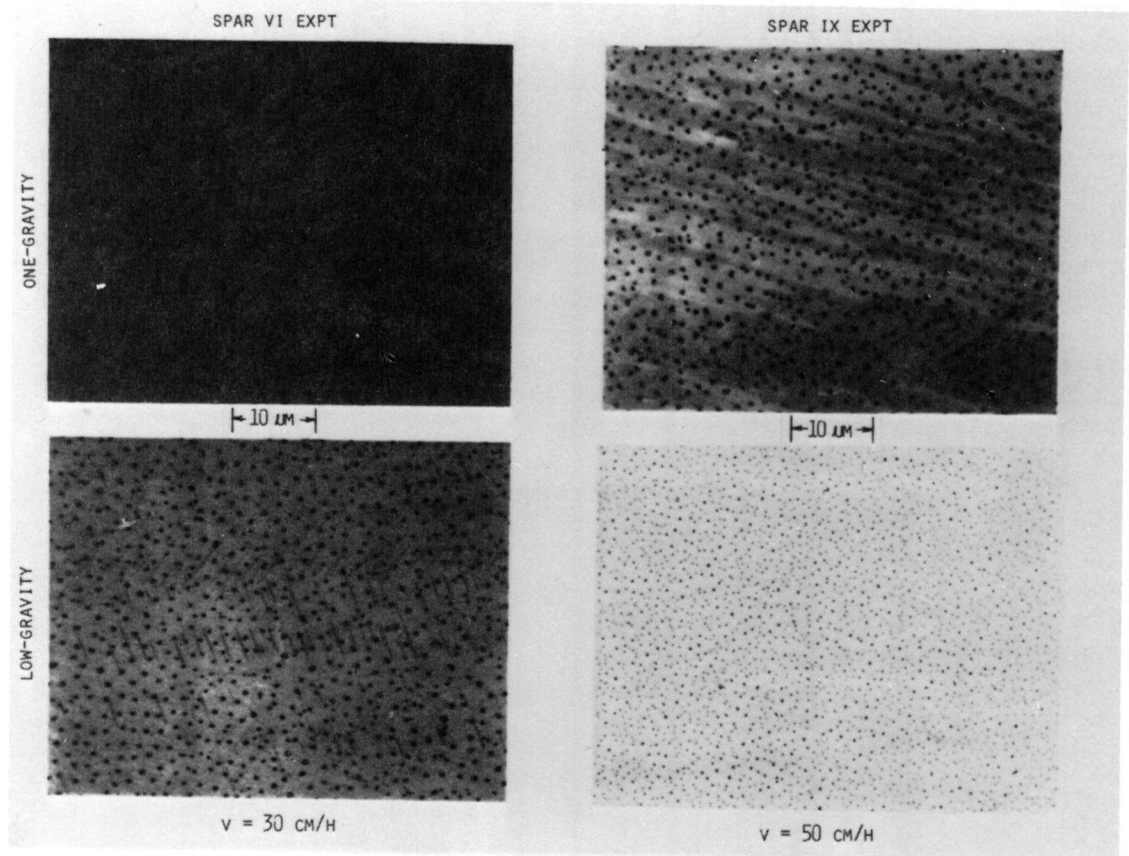
Malmejac, Y., Bewersdorff, A., Da Riva, I., and Napolitano, L. G., "Challenges and Prospectives of Microgravity Research in Space," ESA BR-05, October 1981.



Malmejac, Y. and Huth, U., "Review of the European Microgravity Activities," in Materials Research Society Symposia Proceedings, Materials Processing in the Reduced Gravity Environment of Space, Volume 9 (G. E. Rindone, ed.), North-Holland, 1982, pp. 9-21.

Favier, J. J., "Striations of Kinetic Origin Induced During Non-steady State Solidification," presented at Fifth International Conference on Vapor Growth and Epitaxy, Coronado, CA, July 1981.

COMPARISON OF SPAR FLIGHT ( $10^{-4} g_E$ ) VS GROUND-BASE ( $1 - g_E$ ) EUTECTIC  
Bi/MnBi MORPHOLOGIES TRANSVERSE TO SOLIDIFICATION DIRECTION





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.

## Publications

Pirich, R. G., Larson, D. J., et al., "Studies of Plane Front Solidification and Magnetic Properties of Bi/MnBi," AIAA Journal 19, 589 (1981).

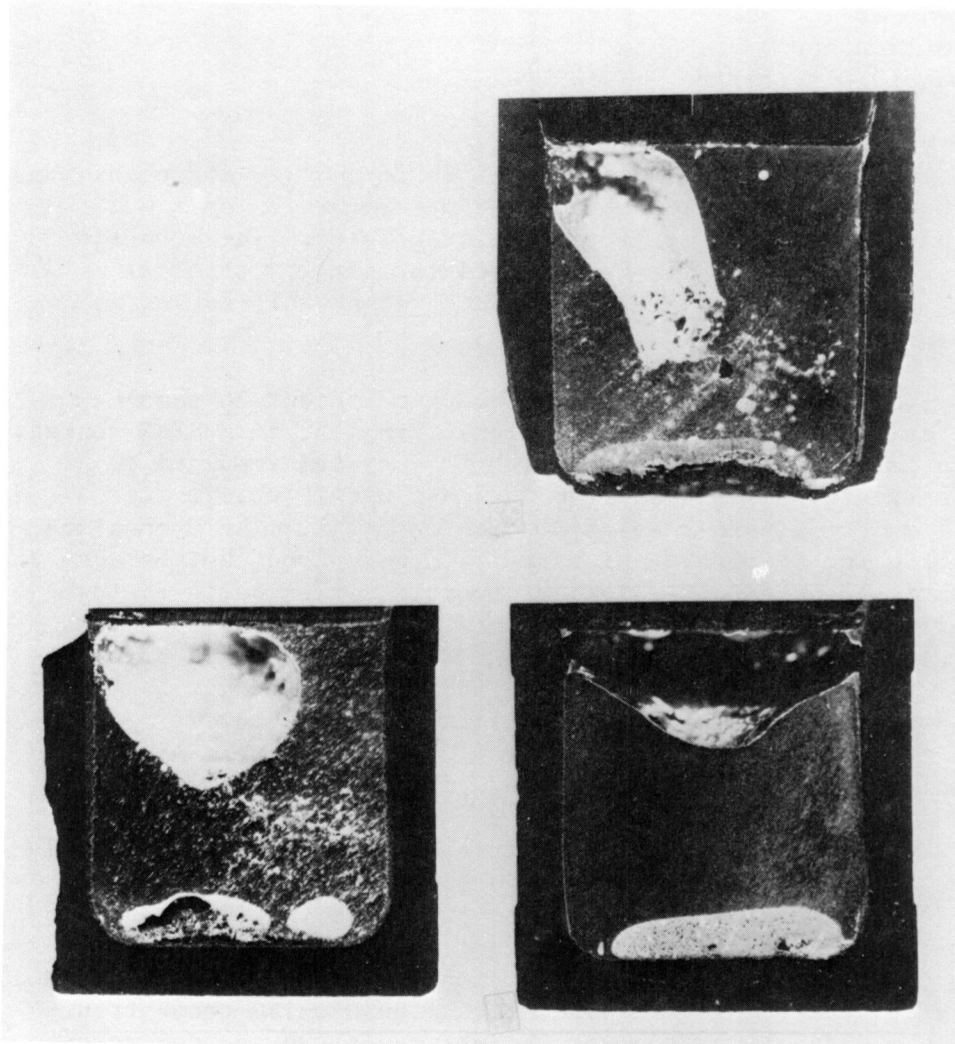
Pirich, R. G., "Gravitationally Induced Convection During Directional Solidification of Off-Eutectic Mn-Bi Alloys," in Materials Research Society Symposia Proceedings, Materials Processing in the Reduced Gravity Environment of Space, Volume 9, (G. E. Rindone, ed.), North-Holland, 1982, pp. 593-602.

Larson, D. J. and Pirich, R. G., "Influence of Gravity Driven Convection on the Directional Solidification of Bi/MnBi Eutectic Composites," in Materials Research Society Symposia Proceedings, Materials Processing in the Reduced Gravity Environment of Space, Volume 9 (G. E. Rindone ed.), North-Holland, 1982, pp. 523-531.



Pirich, R. G., "The Effects of Convection During Directional Solidification of Eutectic Bi/MnBi," submitted to Journal of Crystal Growth, 1981.

Pirich, R. G. and Larson, D. J., "Characterization of Gravitationally Induced Convection on Solidification of the Bismuth-Manganese Eutectic," presented at Fifth International Conference on Vapor Growth and Epitaxy, San Diego, CA, July 1981.



Longitudinal cutting of the hypermonotectic sample together with the x-ray views evidence the complete separation, coalescence and settling of the indium-rich immiscible phase for the hypermonotectic composition alloys of .10 at. of indium, except for the bottom left sample where two globules exist. The indium phase globules remain separated from the walls of the crucible by a film of aluminum. The shrinkage hole in the upper crucible is located at the bottom of the crucible.

Directional Solidification of Monotectic and Hypermonotectic  
Aluminum-Indium Alloys under  $\mu$ -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 wets 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).

Publications

Potard, C., "Structures of Immiscible Al-In Alloys Solidified Under Microgravity Conditions," XXXII IAF Congress, Rome, Italy, September 6-12, 1981.

Potard, C., "Analysis and Control of Directional Solidification by Differential Dilatometry; Application to InSb," J. Cryst. Growth 54, 558-571 (1981).

Potard, C. and Boissier, J., "Final Post Flight Report on SPAR IX Payload R-17 Experiment, Directional Solidification of Immiscible Aluminum-Indium Alloys," November 1981.

Potard, C., "Resultats Preliminaires de "experience de Solidification d'Alliages Immiscibles Aluminum-Indium en Fusee-sonde," Programme CEA-NASA-SPAR, Volume 21, January 1981.

Fredriksson, H., Potard, C., and Walter, H. U., "Materials Science Research with Sounding Rockets, Baseline Considerations for an European Programme," European Space Agency, MAT (81) 1, Paris, March 1981.

Vinet, B., "Etude de l'influence d'un etat de pesanteur sur l'equilibre thermodynamique d'un alliage liquide et sur sa solidification: Application aux alliages Al-Ge eutectiques et Al-In monotectiques," Ph.D. Thesis, Grenoble, December 22, 1981.

Potard, C., "Solidification of Hypermonotectic Al-In Alloys Under Microgravity Conditions," in Materials Research Society Symposia Proceedings, Materials Processing in the Reduced Gravity Environment of Space, Volume 9 (G. E. Rindone, ed.), North Holland, 1982, pp. 543-552.

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 host 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," Journal of Applied Physics, (in press).





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.

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. Finally a twin-forming material will be used to see why twin structures are so different in space-processed ingots.

Numerical finite element models will be developed for the different processes expected to occur, and then will be compared with the experimental results, both these obtained by us on the analogs, and also those obtained by others on technologically important materials.

Study of Eutectic Formation

Clarkson College

Dr. W. R. Wilcox

NAS8-34887 Total Cost: \$65K

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.

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.

### 3. FLUIDS, TRANSPORT, AND CHEMICAL PROCESSES

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



Transient Convective Heat Transfer in Zero Gravity

National Bureau of Standards - Boulder, Colorado  
Dr. V. Arp  
Dr. R. Noble  
H-27954B \$27K

The basic objective of this program is to separate the gravitational contribution from other contributions to dynamic heat and mass transfer measurements, thus allowing a more accurate comparison with theory, leading to improved engineering correlations.

Many advanced technologies involve heat and mass transfer processes as crucial steps in either normal or off-design modes. Current NBS studies are addressing those areas where knowledge of the basic heat and mass transfer processes is inadequate or incomplete for efficient system design. In order to establish the accuracy of the derived transfer components, careful modeling is required to separate, for example, the gravitational contributions from the overall measured values. This includes: (1) separation of "natural" (gravitationally-driven) convection from thermally-driven compressible gas motion in heat transfer, and (2) separation of gravitational from other mass diffusion mechanisms with both neutral and ionic fluids, with and without external electric fields. In both heat and mass transfer, the measurements will extend into the non-linear range, with high wall-to-fluid temperatures, ionic concentrations, etc., where the gravitational contribution is especially difficult or impossible to estimate on a purely theoretical basis.

A modest level with transient heat transfer measurements using a thin film thermometric technique developed by Dr. P. J. Giarratano has begun. Ideally, they would be bolstered by optical measurements which are well-established in NASA's zero-gravity program, but new to our work. However, in the initial tests, only the thermometric measurements would be made. Two types of mass transfer experiments are envisioned for subsequent years. In the first, an ionic solution is isolated from a pure component initially. The barrier is removed and the resulting diffusion is monitored. In a second type of experiment, an ionic solution would be exposed to an external applied field and the resulting diffusion monitored.





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.

An inclusive list of gravitational hypothesis on gravitation effects will be generated. The hypothesis will be based on theoretical and experimental experience of the research team. Consideration will be given to resolution, throughput, boundary layers, convection, and sedimentation. In addition, tests using the previously developed Recycling Isoelectric Focusing (RIEF) apparatus will be made to verify aspects of each hypothesis using standard samples. A study will be conducted to define a meaningful space experiment in isoelectric focusing. Definition of the rationale, general approach, general configuration, and primary hardware requirements will be generated. Another study will refine the current mathematical model for steady state focusing of trace quantity proteins. Conductivity sensors will be designed, constructed, and incorporated into the current hardware.

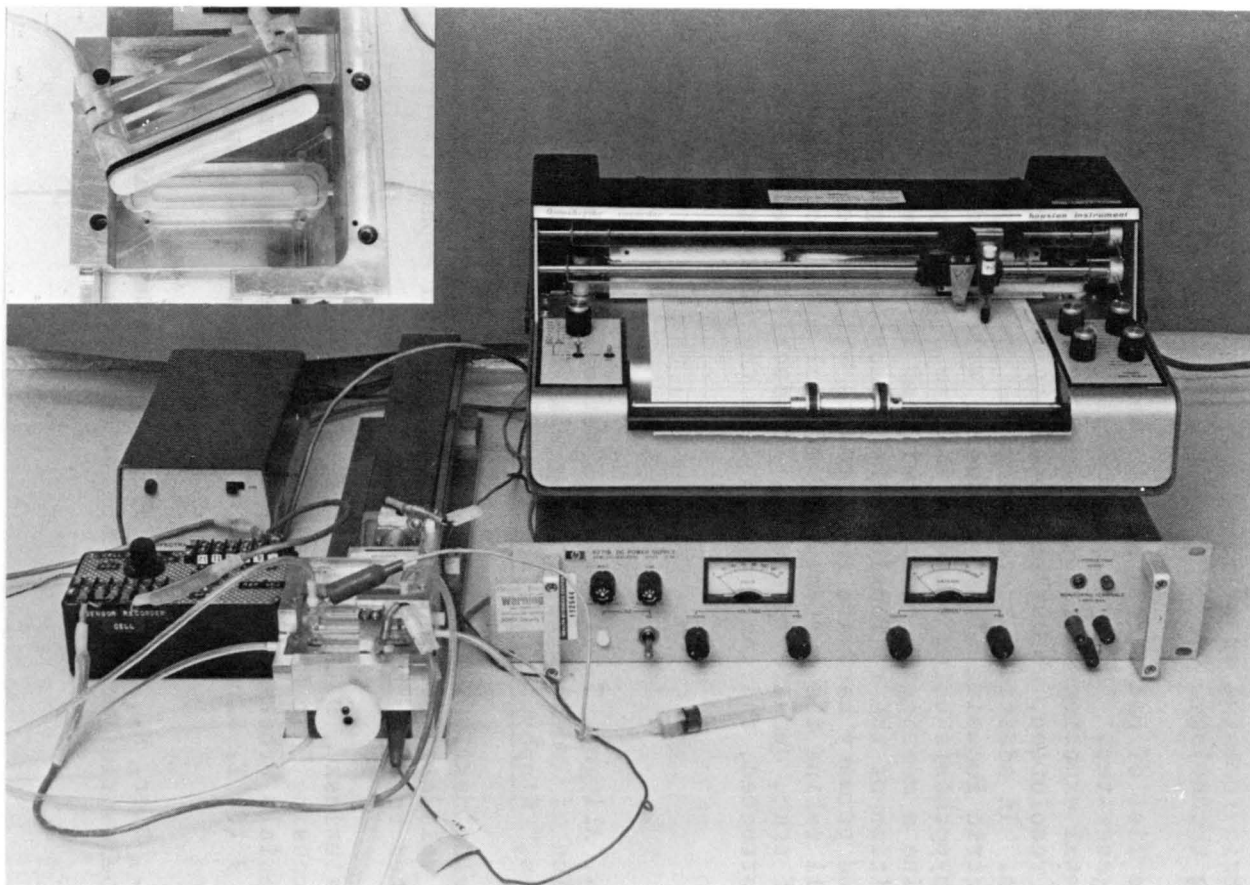
Publications

Palusinski, O. A., Allyger, T. T., et al., "Mathematical Modeling and Computer Simulation of Isoelectric Focusing with Electrochemically Defined Ampholytes," Biophys. Chem. 13, 193-202 (1981).

Mosher, R. A., Palusinski, O. A., and Bier, M., "Computer Simulation and Experimental Validation of Isoelectric Focusing in Ampholine-Free Systems," submitted to J. Chromatography, 1981.

Mosher, R. A., Palusinski, O. A., and Bier, M., "Theoretical Studies in Isoelectric Focusing," in Materials Research Society Symposia Proceedings, Materials Processing in the Reduced Gravity Environment of Space, Volume 9 (G. E. Rindone, ed.), North-Holland, 1982, pp. 255-260.

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.

Countercurrent Distribution of Biological Cells

University of Oregon Health Sciences Center

Dr. D. E. Brooks

NAS8-33575 Total Cost: \$226K

November 16, 1979 - November 15, 1982

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.

The other area in which work has been done is the further development of field-induced phase separation. 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 ( $\sim 0.5 \text{ v cm}^{-1}$ ) enhance the rate at which the phases separate, even in the presence of unit gravity.

Publications

Brooks, D. E. and Bamberger, S., "Studies on Aqueous Two Phase Polymer Systems Useful for Partitioning of Biological Materials," in Materials Research Society Symposia Proceedings, Materials Processing in the Reduced Gravity Environment of Space, Volume 9 (G. E. Rindone, ed.), North Holland, 1982, pp. 233-240.

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.

Blood Flow in Small Vessels

University of Rochester  
Dr. G. R. Cokelet  
Dr. H. Meiselman  
Dr. H. Goldsmith

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 increased 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 individuals 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 flight test conditions and test potential flight experiment components. 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 bifurcation 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 aggregatable 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.



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

Northwestern University  
Dr. S. H. Davis  
Dr. G. M. Homsy, Stanford University  
NAS8-33881 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  $\mu$ -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," to appear in Journal of Fluid Mechanics.

Smith, M. K. and Davis, S. H., "Instability of Sheared Liquid Layers," to appear in Journal of Fluid Mechanics, 1982.

Sen, A. K., Smith, M. K., Davis, S. H., and Homsy, G. M., "Steady Thermocapillary Flows and Their Stability," published in Materials Research Society Symposia Proceedings Materials Processing in the Reduced Gravity Environment of Space, Volume 9 (G. E. Rindone, ed.), North-Holland, 1982, pp. 173-176.

Presentations

Smith, M. K. and Davis, S. H., "Long-Wave Instabilities of Shear Flows with Free Surfaces," 34th Annual Meeting, Division of Fluid Dynamics, American Physical Society, Naval Postgraduate School, 1981.

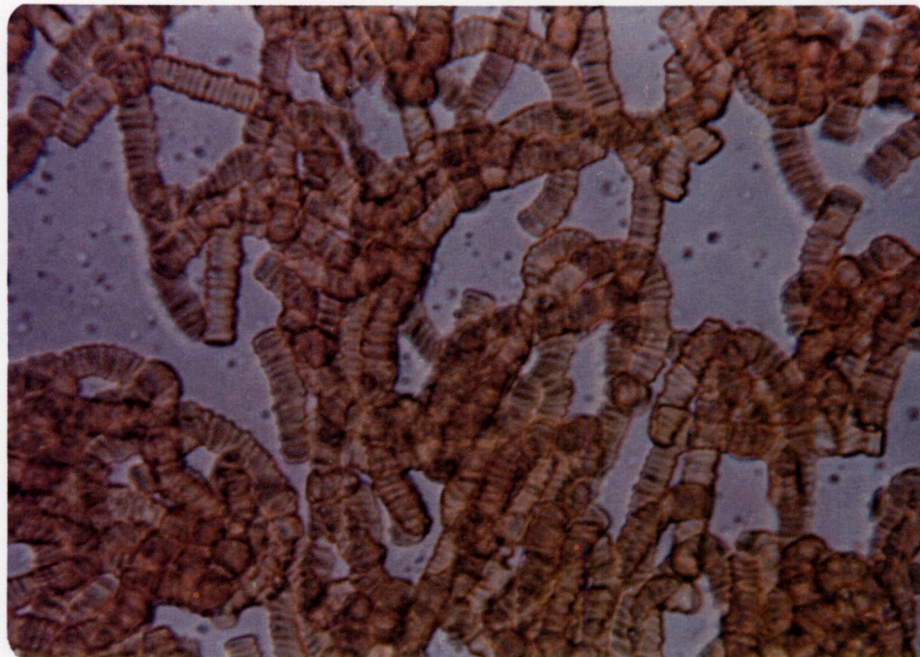
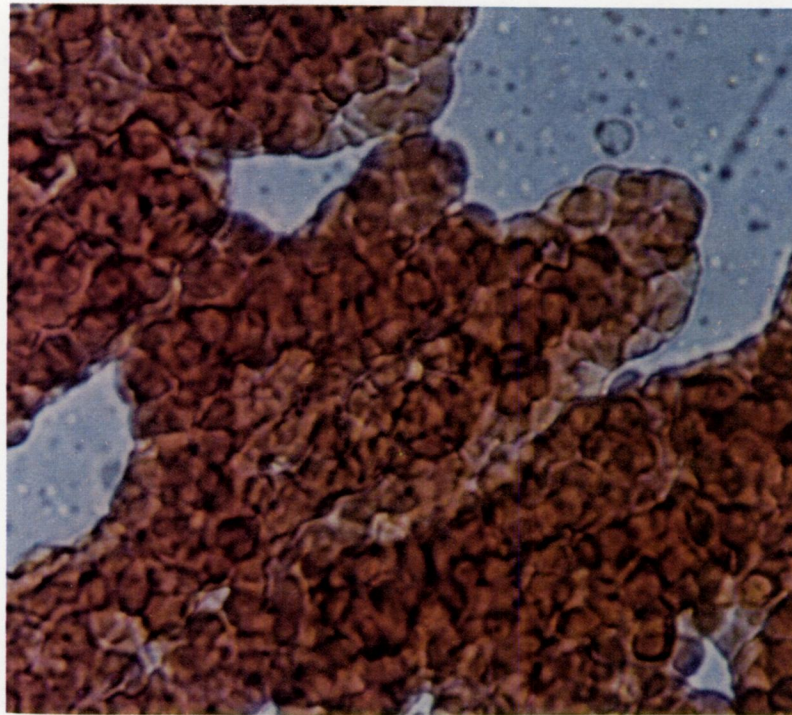




Smith, M. K. and Davis, S. H., "Instability of a Thermocapillary Shear Flow," EUROMECH No. 138, University of Karlsruhe, 1981.

Sen, A. K., Floryan, J. M., Steen, P. H., Davis, S. H., and Homsy, G. M., "Thermocapillary Flow in a Trough or Float Zone," EUROMECH No. 138, University of Karlsruhe, 1981.

Dintenfass  
006



One normal cell and one infarcted cell. Infarction shows clump of cells; the other shows long "rouleaux" (cells one on top of the other forming long strings of cells).

Aggregation of Red Cells

University of Sydney  
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.

## Publications

Dintenfass, L., "The Clinical Impact of the Newer Research in Blood Rheology," Angiology 32, 217-229 (1981).

Dintenfass, L., "Blood Viscosity in Outer Space - a Project for the NASA Space Shuttle," in Blood Viscosity in Heart Disease and Cancer (L. Dintenfass and G.V.F. Seaman, eds.), Pergamon Press, 1981.

Dintenfass, L., "Haemorheology of Capillary Perfusion," in Capillary Perfusion in Health and Disease (R. M. Hardaway, ed.), Chapter, IV, Futura Publishers, New York, 1981.

Dintenfass, L., "Clinical Applications of Haemorheology," in Rheology of Blood, Blood Vessels, and Associated Tissues (D. R. Gross and N.H.C. Hwang, eds.), Sijthoff & Noordhoff, Holland, 1981.

Dintenfass, L., "Haemorheology and the Microcirculation: Role of the Inversion Phenomenon," in Progress in Microcirculation Research (D. Garlick, ed.), Committee on Postgraduate Medical Education, University of New South Wales, Sydney, 1981.

Dintenfass, L., Jedrzejczyk, H., and Willard, A., "Application of Stereological Methods to Evaluation of Aggregation of Red Cells in 12.5  $\mu$  m Slits: a Photographic and Statistical Study," Biorheology 18, 387-404 (1981).

Dintenfass, L. and Willard, A., "Analysis of Results and Evaluation of Size of Aggregates Kinetics and Morphology of Aggregation of Red Blood Cells: Preparations for NASA Experiment. Part I.," Austr. Phys. Engr. Sci. Med. 4, 140-152 (1981).

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.

Publications

Dressler, R. F., "Transient Thermal Convection During Orbital Spaceflight," J. Cryst. Growth 54, 523-533 (September 1981).

Dressler, R. F., Spradley, L. W., and Robertson, J., "Effect of Rayleigh Accelerations Applied to an Initially Moving Fluid," in Materials Research Society Symposia Proceedings, Materials Processing in the Reduced Gravity Environment of Space, Volume 9 (G. E. Rindone, ed.), North Holland, 1982, pp. 203-216.

Dressler, R. F., "Approximate Analysis of Thermal Convection in a Crystal-Growth Cell for Spacelab 3," NASA TP-2016, June 1982.



Surface Tensions and Their Variations with Temperature and Impurities

National Bureau of Standards  
S. C. Hardy  
H-27954B \$200K/year  
April 1977 - continuing task

In this program traditional sessile drop surface tension measurements are being used in conjunction with Auger spectroscopy and other modern surface analytic techniques to study the thermodynamics and chemistry of liquid metal interfaces. The materials selected for study are generally, but not exclusively, those of interest in other areas of the space research program. In recent work, for instance, the surface tension of liquid gallium was measured as a function of temperature from the melting point to about 750°C. The temperature dependence of the surface tension was found to be approximately quadratic. This data is being used elsewhere in the design of experiments which will use gallium to study Marangoni flow.

Currently we are working on the application of Auger spectroscopy to liquid metal surfaces. The experiments are being conducted in a conventional Auger spectrometer with a vertical cylindrical mirror analyzer and a horizontal sample manipulator. The samples are in the form of sessile drops which permits the surface tension to be measured simultaneously with the Auger spectrum. Initial work with gallium drops has been promising because we found that the surface of the drop can be cleaned by sputtering with argon ions. Fluid flows are generated in the sputtering which draw solid impurities such as oxides into the ion beam where they are sputtered away. The mechanism generating this flow is not yet identified. At present we are studying the segregation of tin at the surface of gallium-tin alloys.

## Publications

Fine, J. and Hardy, S. C., "Ion Bombardment Cleaning of Liquid Gallium Surfaces," J. Vac. Sci. Techn. 18, 1310-1311 (1981).

Hardy, S. C. and Fine, J., "Studies of Liquid Metal Surfaces Using Auger Spectroscopy," in Materials Research Society Symposia Proceedings, Materials Processing in the Reduced Gravity Environment of Space, Volume 9 (G. E. Rindone, ed.), North-Holland, 1982, pp. 503-512.





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

University of Alabama in Huntsville  
Dr. J. Milton Harris  
NAS8-33978 \$47K/year  
September 1, 1980 - August 31, 1982

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).

Three new types of water-soluble polymers have been synthesized. These are: (1) polyethylene glycols with attached crown ethers; (2) polyethylene glycols with attached cyclodextrins; and (3) dextans with attached long-chain hydrocarbons. The crown ethers and cyclodextrins are of interest because of their ability to selectively form complexes with, respectively, metal cations and hydrophobic anions. These nitrogen crowns upon protonation should also bind hydrophobic anions. Consequently, these materials present the possibility of specifically binding groups on the cell surface. The polymers with long-chain hydrocarbons attached are of interest because of the probable attraction of the hydrocarbon for lipophilic areas on the cell surface. Testing of the properties of these new materials has begun. An interesting spin-off has been the observation of catalytic activity for the crown polymers.

Publications

Harris, J. M., Hundley, N. H., Shannon, T. G., and Struck, E. C., "Substituted Polyethylene Glycols as Soluble, Recoverable Phase-Transfer Catalysts," accepted for publication Journal of Organic Chemistry, 1982.



Purification and Cultivation of Human Pituitary Growth Hormone-Secreting Cells

Pennsylvania State University  
Dr. W. C. Hymer  
NAS9-15566 \$65K/year  
June 1981 - June 1982

Human growth hormone (hGH) is in demand for treatment of pituitary disease, osteoporosis, stress ulcers, etc. However, the demand is far greater than available supply. The problem could be solved by the culture of purified hGH cells that produce hormone. This program addresses the problem of (a) separation of the pituitary growth hormone cell, (b) its maintenance in vitro, and (c) assessment of the role that gravity plays in establishing limits of these current laboratory technologies.

In an effort to fulfill the above objectives a human pituitary column perfusion method has been developed to sustain hGH release from pituitary tissue over extended periods (1-3 days). On the basis of experimental results from 144 human pituitary glands removed 1-18 hours, postmortem, it has been found that prostaglandin  $E_1(10^{-9}M)$  or epinephrine ( $10^{-9}$ ) stimulates release of a "GRF" from rat hypothalmi which is, in turn, capable of sustaining hGH release for at least 24 hours. Tissue samples stained immunocytochemically for hGH cells reveal large numbers of well-preserved cells in this experimental protocol. These results support the notion that the human postmortem pituitary gland contains functional growth hormone cells.

Results from numerous experiments demonstrate that we can prepare  $\sim 15 \times 10^3$  cells/mg postmortem human pituitary tissues. These cell preparations are  $\sim 80\%$  viable, and by electron microscopy contain membrane and granule systems characteristic of intact tissue. Concerted efforts have been made to separate GH cells from both rat and human pituitaries by chemistry gradient electrophoresis. Results indicate that somatotrophs (GH cells) apparently have low electrophoretic mobilities and possibilities for their eventual purification by this technique appear encouraging. Finally, methodology has been developed for the implantation of human pituitary cells in rats. With this methodology it should be possible to assess function of hGH cells in vitro, as well as eventually being able to isolate hGH from the animal.

Publications

Hymer, W. C., Wilbur, D. L., and Page, R., "Pituitary Hollow Fibers Units In Vivo and In Vitro," Neuroendocrinology 32, 339-349 (1981).

Hymer, W. C., Harkness, J., Bartke, A., et al., "Pituitary Hollow Fiber Units and the Dwarf Mouse," Neuroendocrinology 32, 350-354 (1981).

Kelsey, R., Hymer, W. C., and Page, R., "Pituitary Cell Transplants to the Cerebral Ventricles Suppress Host Pituitary Cell Function," Neuroendocrinology 33, 312-316 (1981).

Hymer, W. C. and Todd, P., "Purification of Rat Pituitary Somatotrophs by Density Gradient Electrophoresis," submitted to Cell Biophysics, 1981.

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

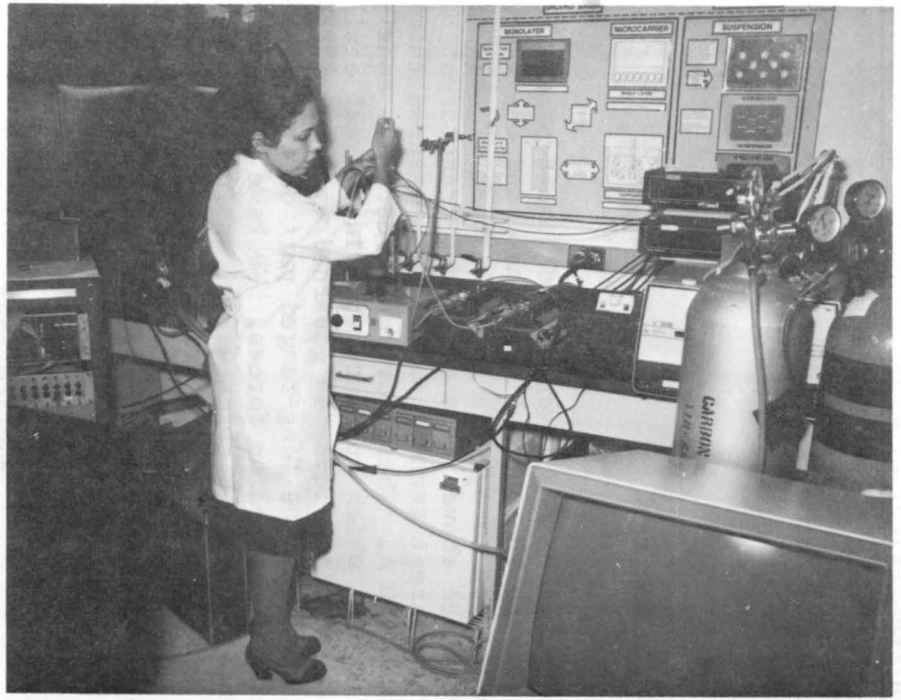
H-27954B

April 1981 - continuing task

In certain solutions the lower of two liquid phases forms a layer which intrudes between the upper liquid phase and the vapor. We have used optical techniques to measure the thickness of this layer at the liquid vapor interface above three different transparent binary solutions and one transparent ternary solution. Measured thicknesses have ranged from 60 Å - 400 Å. These particular data are consistent with theoretical model which predicts that in thermal equilibrium the thickness of the intruding layer is governed by a competition between gravitational forces and the long-ranged part of the intermolecular force; the so-called dispersion forces. We have also discovered a phase transition at which the thickness of an intruding layer increases from less than 20 Å to approximately 400 Å as a two phase liquid sample is heated less than 0.05°C. A theory has been developed for phase equilibria among grain boundary structures and for transitions between various grain boundary phases.

## Publications

Kwon, O'D., Beaglehole, D., Webb, W. W., Widom, B., Schmidt, J. W., Cahn, J. W., Moldover, M. R., and Stephenson, B., "Thickness of the Liquid-Vapor Wetting Layer," Phys. Rev. Lett. 48, 185 (1982).



The photograph on the left is the Electrophoresis unit which flew on STS-3 as a joint MSFC/HSC Verification Test. The photograph on the right is the Bioreactor-Suspension Cell Culture System.

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

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.

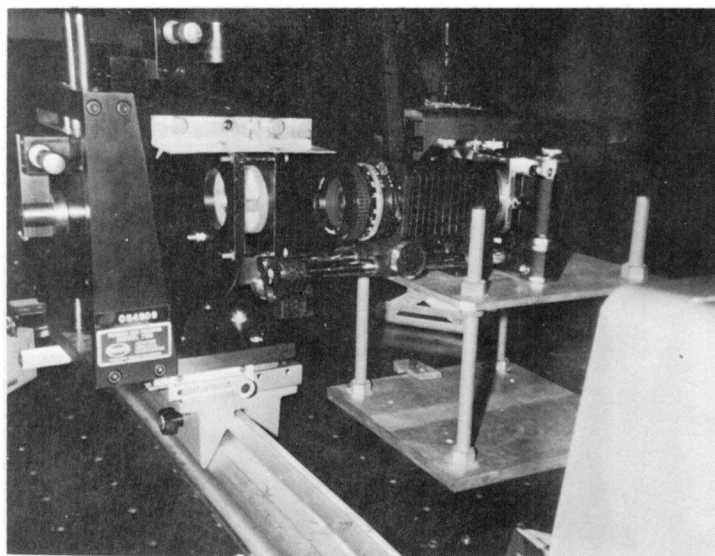
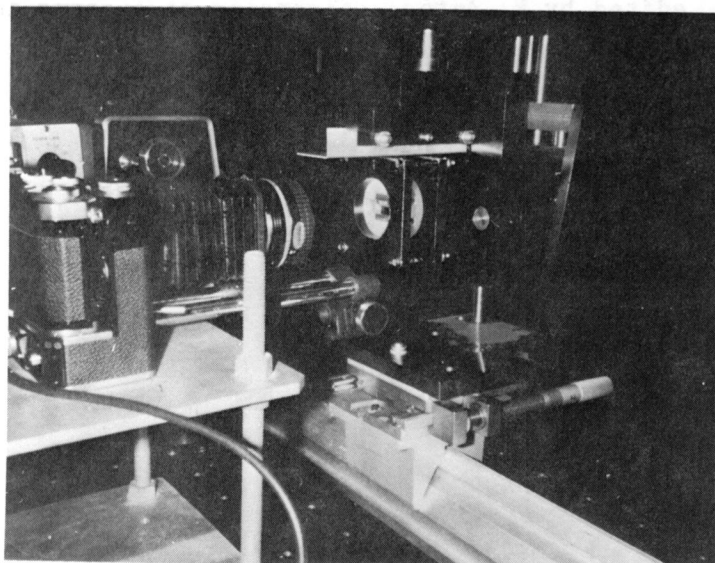




## Publications

Lewis, M. L., Morrison, D. R., Mieszkuc, B. J., and Fessler, D. L., "Problems in Bioassay of Products from Cultured Cells: Plasminogen Activator," published in Proceedings of Second International Cell Culture Congress, edited by R. Acton, University of Alabama in Birmingham, 1981.

Riley  
013



Closeup views of the set-up to study movement of neutral particles in an electrolytic cell system.

Mass Transfer in Electrolytic Systems Under Low Gravity Conditions

University of Alabama in Huntsville

Dr. C. Riley

Dr. H. D. Coble

Dr. R. B. Owens, MSFC

Gordon Fisher, INCO

NAS8-33542 \$33K

September 1979 - June 1982

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.

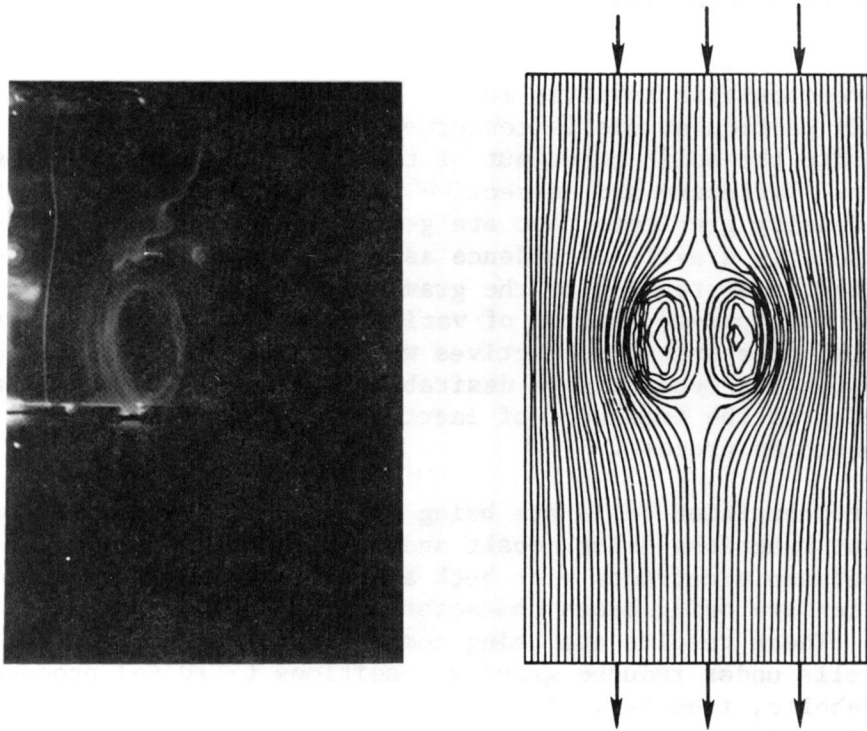
Electrodeposition cells are being utilized to study simple metal-in/metal-out reactions using cobalt and copper. The density flow patterns between electrodes with both a vertical and horizontal configuration are being bench characterized using interferometry detection. These results are being compared 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.

## Publications

Owen, R. B., "Optical Measurements and Tests Performed in a Low Gravity Environment," Opt. Eng. 204, 634 (1981).

Owen, R. B., "Interferometry and Holography in a Low Gravity Environment," Applied Optics 21, 1349-1355 (April 1982).

Riley, C. and Coble, H. D., "Electrodeposition in Microgravity," NAS8-33812, Final Report, June 1982.



Recirculating Flows in Continuous Flow Electrophoresis Chambers.

Small thermal inhomogeneities ( $<0.1\text{C/cm}$ ) can cause recirculations to appear. The photo on the left shows a flow pattern photographed by P. H. Rhodes of MSFC. The pattern depicted on the right was computed using a mathematical model developed at Princeton

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 ( $>100\text{v/cm}$ ). Mathematical models will be developed and tested to establish the roles of the various interactions.

## Publications

Saville, D. A. and Lynch, E. D., "Heat Transfer in the Thermal Entrance Region of an Internally Heated Flow," Chem. Eng. Comm. 9, 201-211 (1981).

Saville, D. A. and Deiber, J. A., "Flow Structure in Continuous Flow Electrophoresis Chambers," in Materials Research Society Symposia Proceedings, Materials Processing in the Reduced Gravity Environment of Space, Volume 9 (G. E. Rindone, ed.), North-Holland, 1982, pp. 217-224.

Saville, D. A., "The Sedimentation Potential in Dilute Suspensions," Advances in Colloid Science and Interface Science, in press, 1981.

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, submitted June 1981.

Saville, D. A., "Electrohydrodynamics and other Hydrodynamic Phenomena in Electrophoresis," Proc. Ninth U.S. National Congress of Applied Mechanics, to appear, 1982.

Electrophoresis Technology

Marshall Space Flight Center  
Dr. R. S. Snyder  
In-House \$75K/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. Optimum operating parameters for resolution and throughput have been established and the two devices can be compared, and (7) these optimized conditions are being used for the separation of biological cells and macromolecules with reproducibility.

Publications

Snyder, R. S. and McGuire, J. K., "Characterization of Continuous Flow Electrophoresis for Improving Resolution and Throughput," submitted to Electrophoresis.

McGuire, J. K. and Snyder, R. S., "Operational Parameters for Continuous Flow Electrophoresis of Cells," in Electrophoresis '81, Walter De Gruyter & Co., Berlin, 1981, pp. 947-960.

Omenyi, S. N., Snyder, R. S., et al., "Reduction of Droplet Formation and Sedimentation of Fixed Erythrocytes in Stationary and Flowing Systems," in Electrophoresis '81, Walter De Gruyter & Co., Berlin, 1981, pp. 961-970.

Rhodes, P. H. and Snyder, R. S., "Numerical Analysis of Continuous Flow Electrophoresis," in Electrophoresis '81, Walter De Gruyter & Co., Berlin, 1981, pp. 899-917.

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Rhodes, P. H., Miller, T. Y., and Snyder, R. S., "Design Consideration of a Thermally Stabilized Continuous Flow Electrophoresis Chamber," in Electrophoresis '81, Walter De Gruyter & Co., Berlin, 1981, pp. 971-981.

Snyder, R. S., "Review of the NASA Electrophoresis Program," in Electrophoresis '81, Walter De Gruyter & Co., Berlin, 1981, pp. 883-897.

Rhodes, P. H. and Snyder, R. S., "The Effect of Small Temperature Gradients on Flow in a Continuous Flow Electrophoresis Chamber," in Materials Research Society Symposia Proceedings, Materials Processing in the Reduced Gravity Environment of Space, Volume 9 (G. E. Rindone, ed.), North-Holland, 1982, pp. 225-232.

Dunning, J. D., Herren, B. J., Tipps, R. W., and Snyder, R. S., "Fractionation of Mineral Species by Electrophoresis," submitted to Journal of Geophysical Research, 1982.



Fluid Dynamics Numerical Analysis

Lockheed Missiles & Space Company  
Dr. L. W. Spradley  
Dr. J. Robertson  
NASW-3281 Total Cost: \$200K  
August 1979 - August 1982

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 is 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. Currently underway is a calculational effort to determine the effect of container shape on the magnitude of microgravity convection.

Publications

Dressler, R. F., Robertson, S. J., and Spradley, L. W., "Effect of Rayleigh Accelerations Applied to an Initially Moving Fluid," in Materials Research Society Symposia Proceedings, Materials Processing in the Reduced Gravity Environment of Space, Volume 9 (G. E. Rindone, ed.), North Holland, 1982, pp. 203-216.



Theoretical Studies of the Surface Tension of Liquid Metals

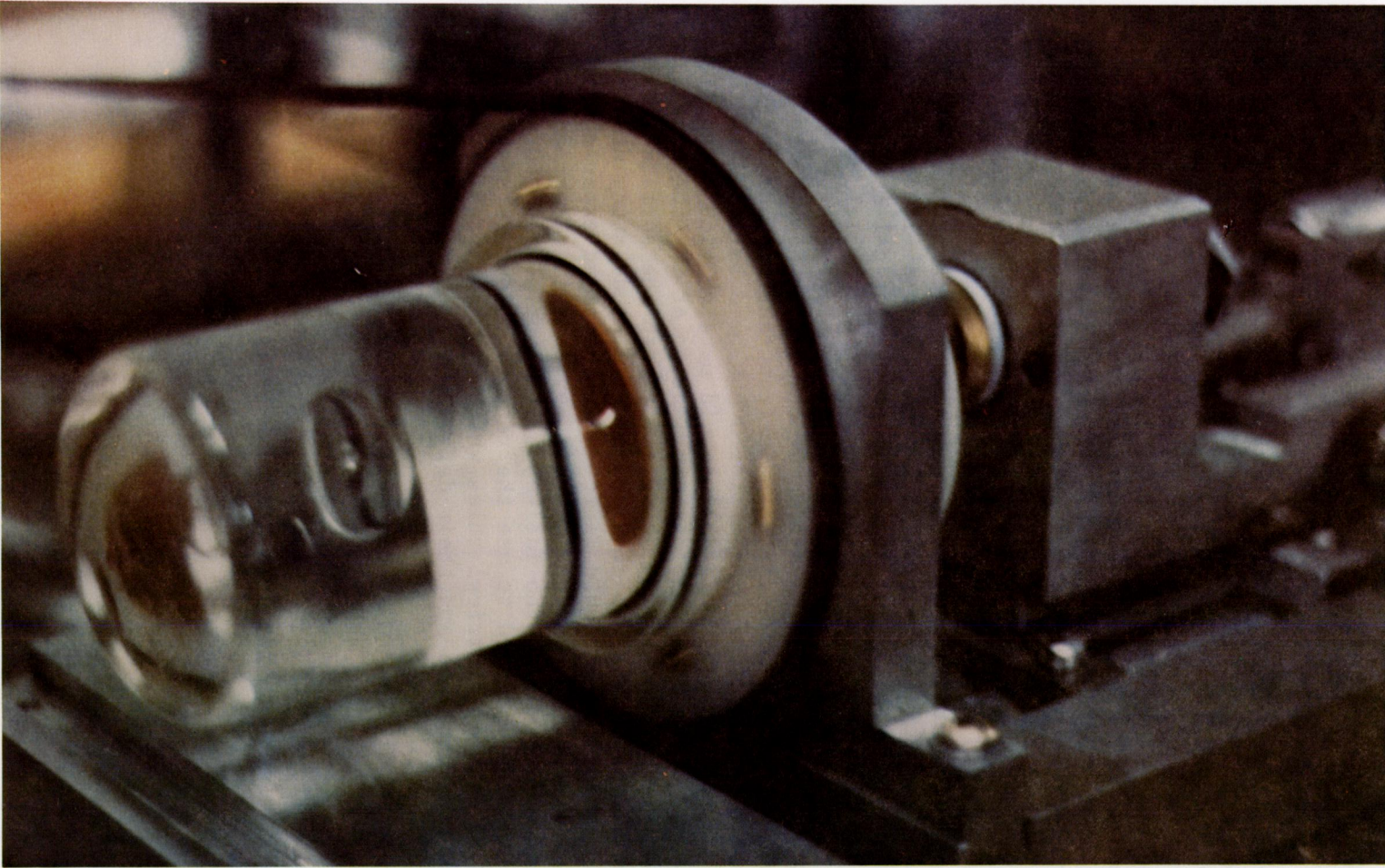
The Ohio State University  
Professor D. G. Stroud  
Dr. D. M. Wood  
Total Cost: \$70K  
February 1982 - February 1984

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  $\text{Li}_x\text{Na}_{1-x}$ ; 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.



Apparatus used for studying bubble migration in an unconstrained rotating drop. The drop is located near the rotation axis of a host fluid which fills the cylinder, and the bubble may be observed near the center of the drop.

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 1982

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

- Subramanian, R. S and Weinberg, M. C., "Asymptotic Expansions in Gas Bubble Dissolution," AICHE J. 27, 739-748 (1981).
- Shankar, N., Cole, R., and Subramanian, R. S., "Thermocapillary Migration of a Bubble in a Drop in a Space Laboratory," Int. J. Multiphase Flow 7, 581-594 (1981).
- Meyyappan, M., Wilcox, W. R., and Subramanian, R. S., "Thermocapillary Migration of a Bubble Normal to a Plane Surface," J. Colloid & Interface Sci. 83, 199-208 (1981).
- Subramanian, R. S., "The Slow Migration of a Gas Bubble in a Thermal Gradient," AICHE J. 27, 646-654 (1981).
- Weinberg, M. C. and Subramanian, R. S., "The Dissolution of a Stationary Bubble Enhanced by Chemical Reaction," Chem. Eng. Sci. 36, 1955-1965 (1981).
- Annamalai, P., Shankar, N., Cole, R., and Subramanian, R. S., "Bubble Migration Inside a Liquid Drop in a Space Laboratory," Appl. Sci. Res. 38, 179-186 (1982).



Meyyappan, M., Wilcox, W. R., and Subramanian, R. S., "Interaction Effects in Thermocapillary Bubble Migration," in Proceedings of Second Colloquium on Drops and Bubbles, JPL Publication 82-7, March 1982, pp. 15-25.

Shankar, N., Annamalai, P., Cole, R., and Subramanian, R. S., "The Motion of Bubbles Inside Drops in Containerless Processing," in Proceedings of Second Colloquium on Drops and Bubbles, JPL Publication 82-7, March 1982, pp. 26-30.

McNeil, T., Cole, R., and Subramanian, R. S., "Surface-Tension Driven Flow in Glassmelts and Model Fluids," in Materials Research Society Symposia Proceedings, Materials Processing in the Reduced Gravity Environment of Space, Volume 9 (G. E. Rindone ed.), North-Holland, 1982, pp. 289-300.

Annamalai, P., Subramanian, R. S., and Cole, R., "Bubble Motion in a Rotating Liquid Body," in Materials Research Society Symposia Proceedings, Materials Processing in the Reduced Gravity Environment of Space, Volume 9 (G. E. Rindone ed.), North-Holland, 1982, pp. 187-190.

Meyyappan, M., Subramanian, R. S., Wilcox, W. R., and Smith, H. D., "Bubble Behavior in Molten Glass in a Temperature Gradient," in Materials Research Society Symposia Proceedings, Materials Processing in the Reduced Gravity Environment of Space, Volume 9 (G. E. Rindone, ed.), North-Holland, 1982, pp. 311-314.

Shankar, N., Cole, R., and Subramanian, R. S., "Thermocapillary Motion of Bubbles Inside Drops," in Materials Research Society Symposia Proceedings, Materials Processing in the Reduced Gravity Environment of Space, Volume 9 (G. E. Rindone, ed.), North-Holland, 1982, pp. 249-254.

Smith, H. D., Mattox, D. M., Wilcox, W. R., Subramanian, R. S., and Meyyappan, M., "Experimental Observation of the Thermocapillary Driven Motion of Bubbles in a Molten Glass under Zero Gravity Conditions," in Materials Research Society Symposia Proceedings, Materials Processing in the Reduced Gravity Environment of Space, Volume 9 (G. E. Rindone, ed.), North-Holland, 1982, pp. 279-288.

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Shankar, N. and Subramanian, R. S., "The Slow Axisymmetric Thermocapillary Migration of an Eccentrically Placed Bubble Inside a Drop in a Space Laboratory," submitted to Journal of Fluid Mechanics, 1981.

Annamalai, P., Subramanian, R. S., and Cole, R., "Bubble Migration in a Rotating Liquid-Filled Sphere," submitted to Physics of Fluids, 1981.

Jayaraj, K., Cole, R., and Subramanian, R. S., "Combined Thermocapillary and Buoyant Flow in a Drop in a Space Laboratory," J. Colloid & Interface Sci., in press, 1982.

#### Presentations

Subramanian, R. S., "The Thermocapillary Migration of a Bubble in a Large Body of Liquid," presented at European Mechanics Colloquium 138, Karlsruhe, West Germany, March 1981.

Shankar, N., Meyyappan, M., et al., "Some Interaction Effects in Thermocapillary Bubble Migration," presented at European Mechanics Colloquium 138, Karlsruhe, West Germany, March 1981.

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McNeil, T. J., Cole, R., et al., "Thermocapillary Convection in a Cylindrical Zone," presented at International Chemical Engineering Congress, Montreal, Canada, October 1981.

Shankar, N., Cole, R., and Subramanian, R. S., "Thermocapillary Migration of a Bubble Eccentrically Located Inside a Drop," presented at AIChE 74th Annual Meeting, New Orleans, Louisiana, November 1981.





Kidney Cell Electrophoresis

Pennsylvania State University  
Dr. Paul Todd  
NAS9-15584 \$40K/year  
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.

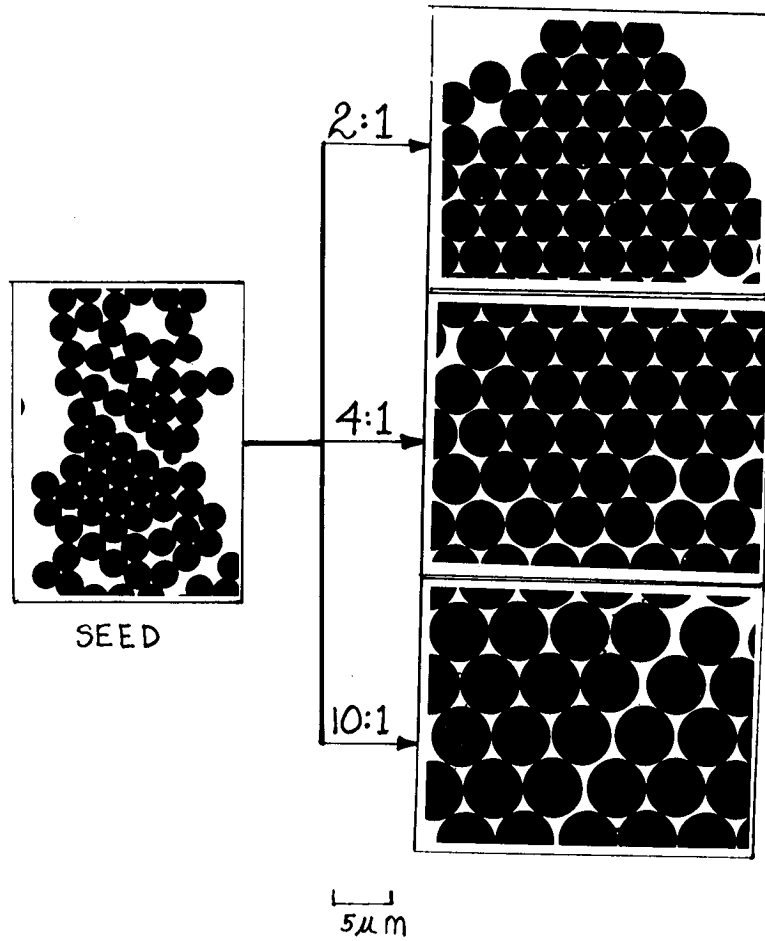
This study effort will perform the ground-based research necessary to establish all of the optimum experimental conditions required to accomplish the best possible electrophoretic separation of human kidney cell fractions, which produce urokinase, granulocyte stimulating factor, or erythropoietin. This 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, and (5) acquisition of the ground control data to be compared with results using cells returned from the electrophoretic separations carried out in microgravity.

Cells from cultures obtained from 26 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 also must be used as a medium for freezing viable cells, has indicated that the low ionic strength required for effective electrophoresis in microgravity experimented compromises the viability of the cells. A buffer designated "D-1", which contains DMSO and EDTA, is phosphate buffered and is made isotonic with sucrose, has been chosen for use in microgravity experiments. Optimum culturing conditions for urokinase production by cells in monolayer have been established. In close collaboration with Johnson Space Center a human kidney cell explant designated "HEK-8514" has been chosen for microgravity experiments. In close collaboration with Michael Reese Research Foundation in Chicago, procedures have been established for urokinase assay of cultures derived from cells separated in in microgravity experiments, which will take place on Shuttle OFT-3 mission.



## Publications

Todd, P. C., Hymer, W. C., Plank, L. D., Marks, G. M., Kunze, M. E., Giranda, V., and Mehrishi, J. N., "Separation of Functioning Mammalian Cells by Density-Gradient Electrophoresis," in Electrophoresis '81, Walter De Gruyter & Co., Berlin, 1981, pp. 871-882.



Electron Micrographs of "seed" latex  $2.5 \mu\text{m}$  used on STS-3 Columbia to prepare larger-size latex-particles:

- $3.5 \mu\text{m}$  (labeled 2:1)
- $4.0 \mu\text{m}$  (labeled 4:1)
- $5.0 \mu\text{m}$  (labeled 10:1)

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 2.5  $\mu\text{m}$ -size seed latex and produced monodisperse latexes of 3.4, 4.1, and 5.0  $\mu\text{m}$  size. The fourth experiment was a control which used a 0.19  $\mu\text{m}$ -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.

Publications

Tseng, C. M., El-Aasser, M. S., and Vanderhoff, J. W., "Modeling the Equilibrium Swelling of Latex Particles with Monomers," Org. Coatings Plastics Chem. 45, 317-322 (1981).



#### 4. ULTRAHIGH VACUUM AND CONTAINERLESS TECHNOLOGIES

TASK NUMBER (TN)	PRINCIPAL INVESTIGATOR (PI)	SHORT TITLE
CP-001	Bonnell	Measurement of High Temperature
CP-002	Cezairliyan	Dynamic Thermophysical Measurements
CP-003	Das	Ultimate Intrinsic Coercivity of $\text{SmCo}_5$
CP-004	Day	Containerless Processing of Glass
CP-005	Downs	Gel Precursors as Glass and Ceramic
CP-006	Dunn	The Upgrading of Glass Microballoons
CP-007	Elleman	Electrostatic Control and Manipulation
CP-008	Ethridge	Homogeneous Crystallization of Glass
CP-009	Frost	Rework of SPAR EML for MEA
CP-010	Margrave	Measurement of the Properties of Tungsten
CP-011	Mukherjee	Ultrapure Glass Optical Waveguide
CP-012	Nordine	Containerless High Temperature Property
CP-013	Robinson	Undercooling in Metastable Peritectics
CP-014	Schmid	Free Cooling at High Temperatures
CP-015	Szekely	Convection in Grain Refining
CP-016	Turnbull	Crystal Nucleation in Glass-Forming Alloy
CP-017	Wang	Fusion Target Technology
CP-018	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 commercial processes can be realized through the elimination of detrimental gravitational effects.



Measurement of High Temperature Thermophysical Properties of Tungsten Liquid and Solid

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

The primary aim of this task has been to evaluate experimental procedures used in the interaction between General Electric Advanced Application Laboratory (GE) and the Rice University to measure the high temperature enthalpy increments of liquid and solid tungsten. GE has demonstrated a unique apparatus capable of levitating molten tungsten and Rice has wide experience in high temperature enthalpy increment measurements, being particularly successful in the use of drop-type isoperibol calorimeters with levitation heating.

The results of this research are of great theoretical and engineering interest. From the scientific viewpoint, the unique location of tungsten at the upper extreme 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 and periodic table correlations are currently used for extrapolation. A definitive value for the heat of fusion of this element will provide a valuable test of such models, perhaps indicating a significant deviation in such predictions based on lower melting refractory metals. Questions concerning the liquid state heat capacity function, i.e., whether it is constant as would be suggested from studies of lower melting refractory metals of the discontinuity from  $C_p(s)$  to  $C_p(l)$  are even reasonable, are crucial in testing theoretical models and may provide clues to establishing new parameters in existing or new models. The engineering applications begin with the fact that, without knowledge of the heat of fusion and liquid enthalpy function, melting, forming, and casting equipment must of necessity be over-designed to allow a satisfactory safety/performance margin. These engineering effects pervade all high temperature uses of tungsten-containing materials.



Dynamic Thermophysical Measurements in Space

National Bureau of Standards

Dr. A. Cezairliyan

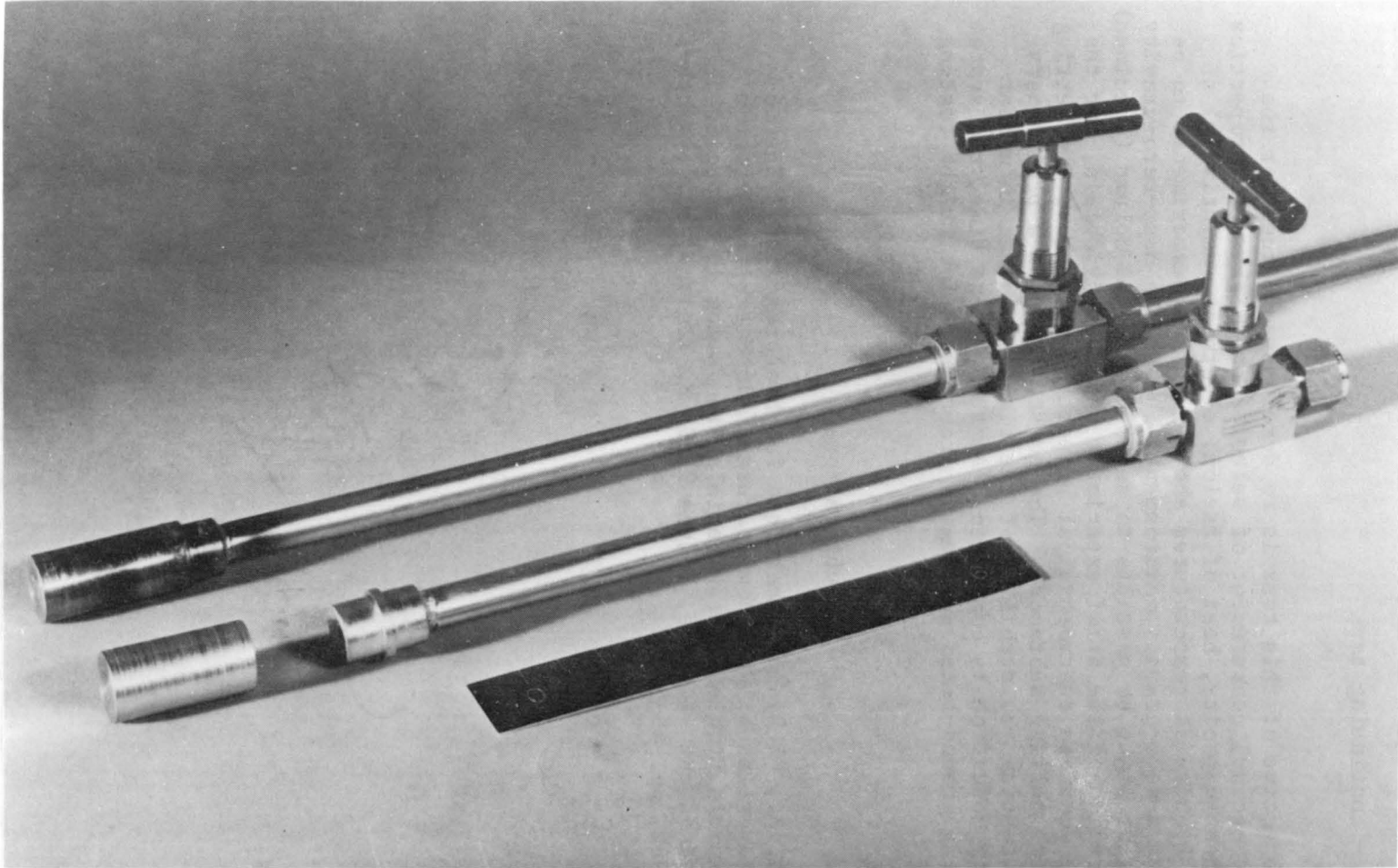
H-27954B

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, electrical 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 substances. 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 1983.

Publications

Cezairliyan, A., "A Dynamic Technique for Measurements of Thermophysical Properties at High Temperatures," in Materials Research Society Symposia Proceedings, Materials Processing in the Reduced Gravity Environment of Space, Volume 9 (G. E. Rindone, ed.), North-Holland, 1982, pp. 49-57.



Welded hot-isostatic-pressing cannister assembly with a  $\text{SmCo}_5$  compact sealed inside. This was performed inside the comminution chamber.

Ultimate Intrinsic Coercivity SmCo<sub>5</sub> Magnet

Charles Stark Draper Laboratory  
Dr. Dilip K. Das  
Dr. R. T. Frost, General Electric  
NAS8-33607 Total Cost: \$396K  
September 24, 1979 - July 15, 1982

The objective of the proposed research is to produce Sm-Co magnets of reasonably high maximum energy product with intrinsic coercivity. These magnets will have to be composed of extremely fine, defect-free crystallites. A very large amount of oxygen enters the magnet during the fabrication process, which involves (1) cold compaction of magnetically aligned powder, (2) comminution of the alloy to fine particle size powder, and (3) thermal densification of the cold compact. The normal practice is to perform the first two steps in air, and the thermal densification is produced by sintering in a purified noble gas atmosphere.

In preparation for the design of a high quality comminution and encapsulation chamber, some preliminary comminution experiments in a laminar flow nitrogen glove box have been performed. The oxygen pick-up in the above powder has been reduced by 60 percent over the powder prepared in air. Based on this encouraging result, using a high quality chamber in a pure quiescent noble gas should lower the oxygen content to a few decades of ppm instead of the 0.6 weight percent in the state-of-the-art magnets.

The comminution chamber described in last year's summary has been built by an outside vendor and has just been installed at the Draper Laboratory. The chamber is capable of attaining  $10^{-6}$  torr pressure, and can be back filled with purified Argon gas with an oxygen level of 0.1 ppm by weight. Experiments to achieve powder comminution, compaction and encapsulation of compacts in the oxygen-free atmosphere inside the chambers will commence soon.

Further experiments in R.F. levitation melting of SmCo<sub>5</sub> alloy have not resulted in significant lowering of oxygen content. We have, however, produced some SmCo<sub>5</sub> alloys with very low oxygen and other contaminant contents, using the expertise and facilities at the Ames Laboratory of Iowa State University.

Publications

Das, D. K., Kumar, K., et al., "Ultimate Intrinsic Coercivity Samarium-Cobalt Magnet," Draper Laboratory, Monthly Progress Report, February 1981.





Containerless Processing of Glass Forming Melts in Space

University of Missouri-Rolla  
Dr. D. E. Day  
NAS8-34758 \$80K  
February 1982 - January 1983

The principal objectives are to (a) measure the effectiveness of containerless melting in extending the compositional limits for glass formation, (b) develop the processing procedures needed to produce multicomponent precursor specimens which will yield chemically homogeneous melts in microgravity, and (c) assess the suitability of the flight hardware for levitating melts in microgravity (Space Shuttle).

The first major task is the preparation of precursor specimens that will be used for containerless glass melting experiments in the MEA facility (acoustic levitator) of the Space Shuttle. Specimens containing known chemical and optical inhomogeneities are being prepared for the purpose of determining the degree of homogeneity achieved in microgravity where homogenization by gravity driven convection is greatly reduced. A comparative property analysis of earth-processed and flight-processed samples will provide the data needed to identify the processing technology (degree of initial homogenization) required for preparing precursor samples that will yield homogeneous, multicomponent melts when processed within the time/temperature capability of the flight hardware.

A second group of specimens will be used to assess the effectiveness of containerless processing in suppressing heterogeneous nucleation/crystallization which should, thereby, extend the compositional range of glass formation. Because of their potentially unique optical properties, the critical cooling rate for glass formation in binary calcia-gallia melts will be determined. Specimens whose measured critical cooling rate are greater or less than the cooling rate in the flight hardware will then be remelted and quenched in microgravity for the purpose of determining whether enhanced glass formation is obtained due to the suppression of heterogeneous nucleation/crystallization. The properties of space-processed glasses will also be evaluated for comparison with equivalent earth-processed glasses to identify any property enhancement due to space processing.



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

KMS Fusion, Inc.  
Dr. R. L. Downs  
W. J. Miller

The overall objective of the proposed work is to determine experimental procedures to produce gels 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 final choice of compositions to be investigated have not been chosen, but potential candidates are the  $\text{GeO}_2\text{-SiO}_2$  system, binary mixtures of the alkali and alkaline earth oxides with the transition metal oxides and/or mixtures of the transition metal oxides.



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 \$43.8K/year  
August 28, 1978 - August 31, 1982

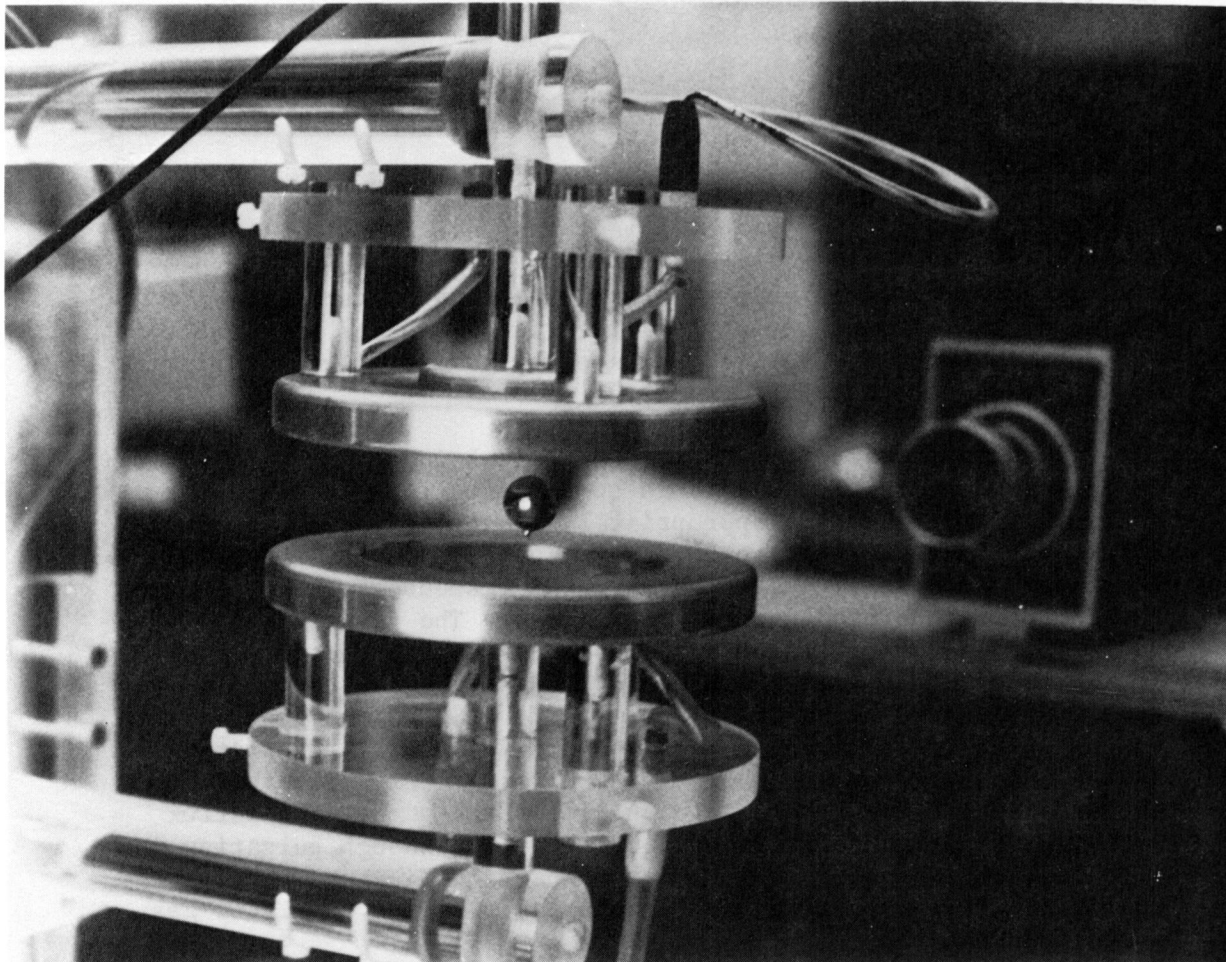
The objective of this program is to study extensively the processes and mechanisms involved in producing glass microballoons of acceptable quality for laser fusion by gas jet levitation and manipulation in the molten condition.

The levitation microfurnace (LMF) is being adopted to variable and microgravity through computerized optical feedback control of multiple CHSs appropriately located on orthogonal tri-axial 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 correspondingly sinusously 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 vector 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.

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 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 a symmetrically packed and confined bundle uniformly sized wires.

Publications

Ethridge, E. C. and Dunn, S. A., "Air Jet Levitation Furnace System for Observing Glass Microspheres During Heating and Melting," in Materials Research Society Symposia Proceedings, Materials Processing in the Reduced Gravity Environment of Space, Volume 9 (G. E. Rindone, ed.), North Holland, 1982, pp. 121-130.



JPL Electrostatic Levitator. The photograph shows the levitator positioning and holding a hollow glass shell that is approximately 1 cm in diameter and weighs about 0.1 gm. The potential difference across the electrodes is about 7 Kilovolts. In the background one can see a CCD camera which is used in a digital feedback system that controls the voltage on the electrodes. The position of the sample between the electrodes can be changed as desired by altering the computer program i.e., typing in a command to position the sample at a new position. This levitator can position low density materials in a low-g environment or high density materials in a reduced gravity.

Electrostatic Control and Manipulation of Materials for  
Containerless Processing

Jet Propulsion Laboratory  
Dr. D. D. Elleman  
Dr. W. K. Rhim  
In-Center \$1050K(to date)  
October 1978 - continuing task

The objective of this task is to develop electric field positioning/manipulation techniques and technology for the containerless processing of materials in bulk and dispersed forms. This method obviates limitations of other methods. Two principal demonstrations to prove technique and technology will be completed no later than CY 83. An important part of the task is to ensure that facilities to be developed satisfy requirements of identified user applications. Another part of the task is to pursue study of the electrohydrodynamics of liquid drops (that must be done as part of the development) as a fundamental investigation.

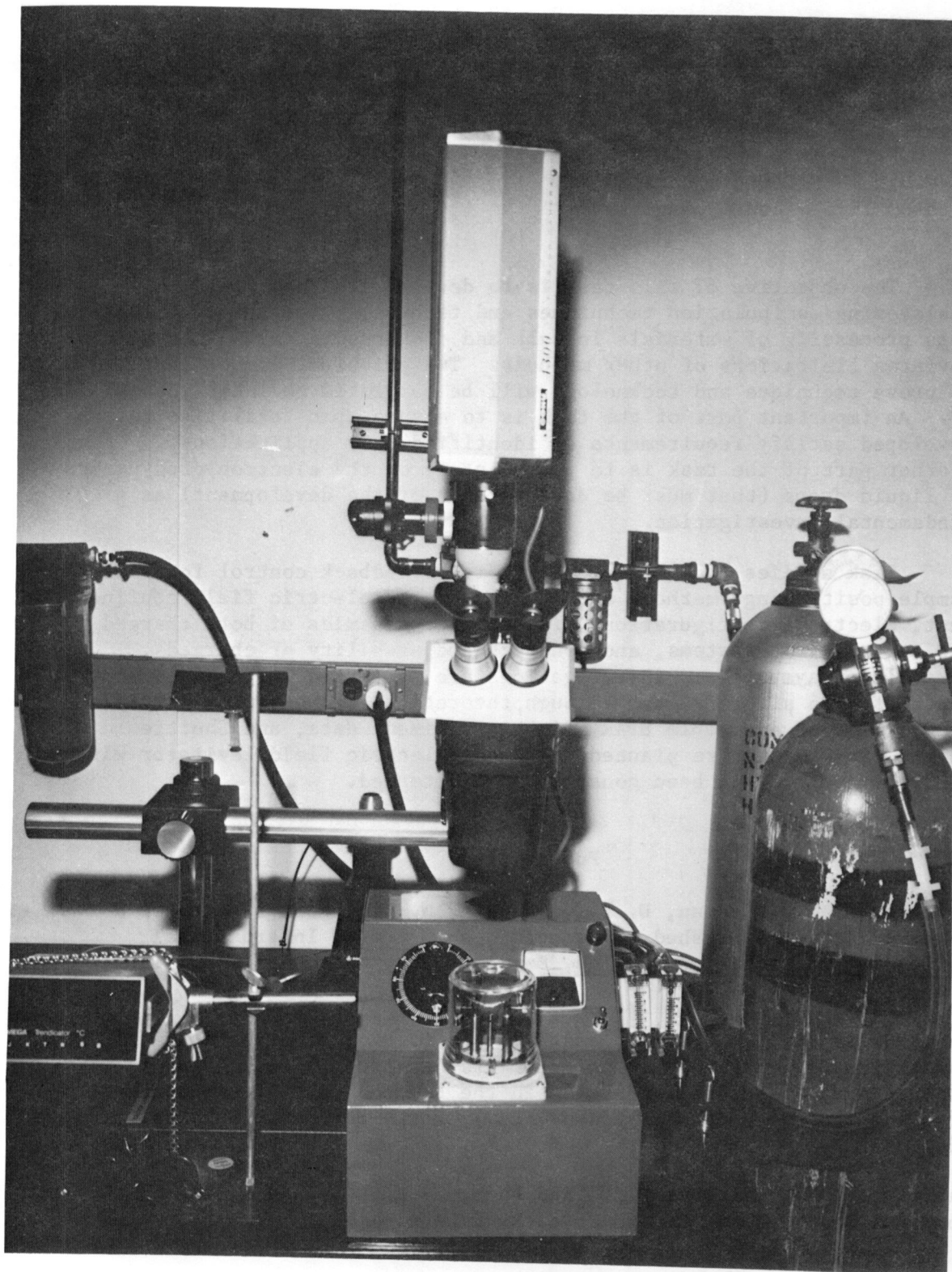
Task studies include: electric field feedback control for bulk sample positioning, methods and limitations of electric field confinement, electrode configuration, electro-hydrodynamics of both charged and neutral drop systems, and dynamics and stability of charged particle arrays. These investigations are performed in the laboratory, in the KC-135 aircraft, and through theoretical and numerical study. Use is made of available SPAR flight experiment data, and Shuttle flight experiments are planned. A one-g electric field levitator with feedback control has been constructed and tested.

Publications

Saffren, M. M., Elleman, D. D., and Rhim, W. K., "Normal Modes of a Compound Drop," published in Proceedings of Second International Colloquium on Drops and Bubbles, Monterey, CA, November 19-21, 1981, pp. 7-14.

Rhim, W. K., Saffren, M. M., and Elleman, D. D., "Electrostatic Levitation Devices at JPL," in Materials Research Society Symposia Proceedings, Materials Processing in the Reduced Gravity Environment of Space, Volume 9 (G. E. Rindone, ed.), North-Holland, 1982, pp. 115-120.

Saffren, M. M., Elleman, D. D. and Rhim, W. K., "Dynamics of a Compound Drop," submitted to Phys. Rev. Lett.



Strip heater furnace used in crystallization studies of glasses.



Homogeneous Crystallization Studies of Borderline Glass Forming Systems

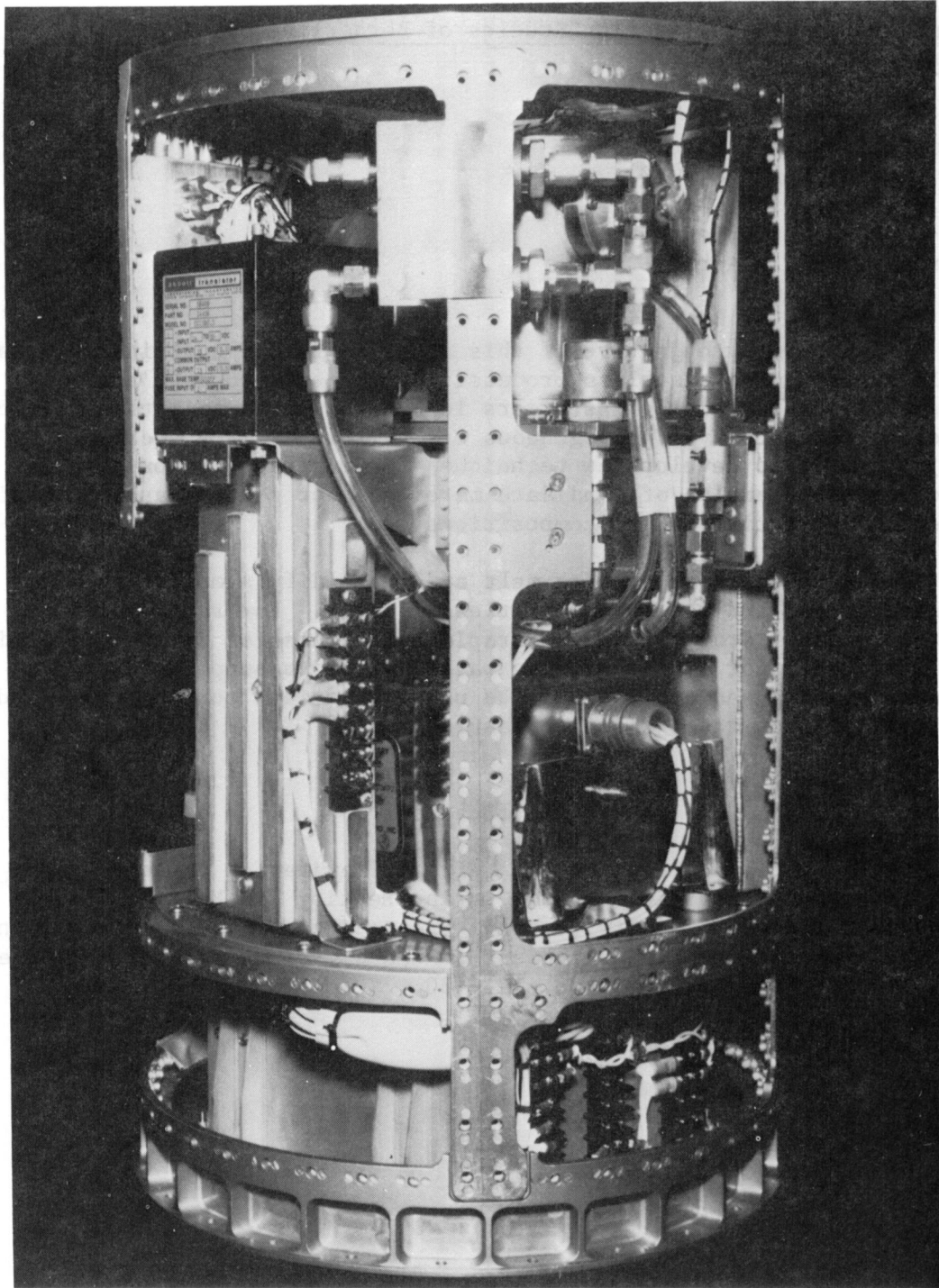
Marshall Space Flight Center  
Dr. E. C. Ethridge  
Dr. P. Curreri  
In-House \$60K/year  
April 1, 1981 - April 1, 1984

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. Critical cooling rates and crystallization rates will be measured.

Publications

Ethridge, E. C. and Dunn, S. L., "Air Jet Levitation Furnace System for Observing Glass Microspheres During Heating and Melting," in Materials Research Society Symposia Proceedings, Materials Processing in the Reduced Gravity Environment of Space, Volume 9 (G. E. Rindone, ed.), North-Holland, 1982, pp. 121-130.



Electromagnetic Containerless Processing Payload. This fully automated electromagnetic levitator/induction melter can be sequentially operated at near zero power in a microgravity environment. Experiments in which metals were solidified under quiescent weightless, containerless conditions have been carried out in sounding rockets. Improved versions of this apparatus will become available in the space shuttle program to permit greater variety of experiments in which simultaneous weightlessness and a free melt surface are required.

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

General Electric Company  
Dr. R. T. Frost  
NAS8-34231     \$126k/year  
October 1981 - October 1982

The general goals of this project are to study the upgrade requirements and approaches needed for incorporation of an EML in the MEA carrier, to design and develop an engineering version of a multi-sample specimen exchanger, to develop and test improvements in high temperature drop calorimetry techniques including new techniques for low gravity work, and to carry out support tasks for the Electromagnetic Containerless Processing Task Team.

Publications

Frost, R. T. and Stockhoff, E. H., Minutes of the Containerless Processing Task Team Meeting, General Electric Space Center, October 27-28, 1980, issued March 1981.

Frost, R. T. and Chang, C. W., "Theory and Applications of Electromagnetic Levitation," in Materials Research Society Symposia Proceedings, Materials Processing in the Reduced Gravity Environment of Space, Volume 9 (G. E. Rindone, ed.), North Holland 1982, pp. 71-80.



Measurement of the Properties of Tungsten at High Temperatures

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

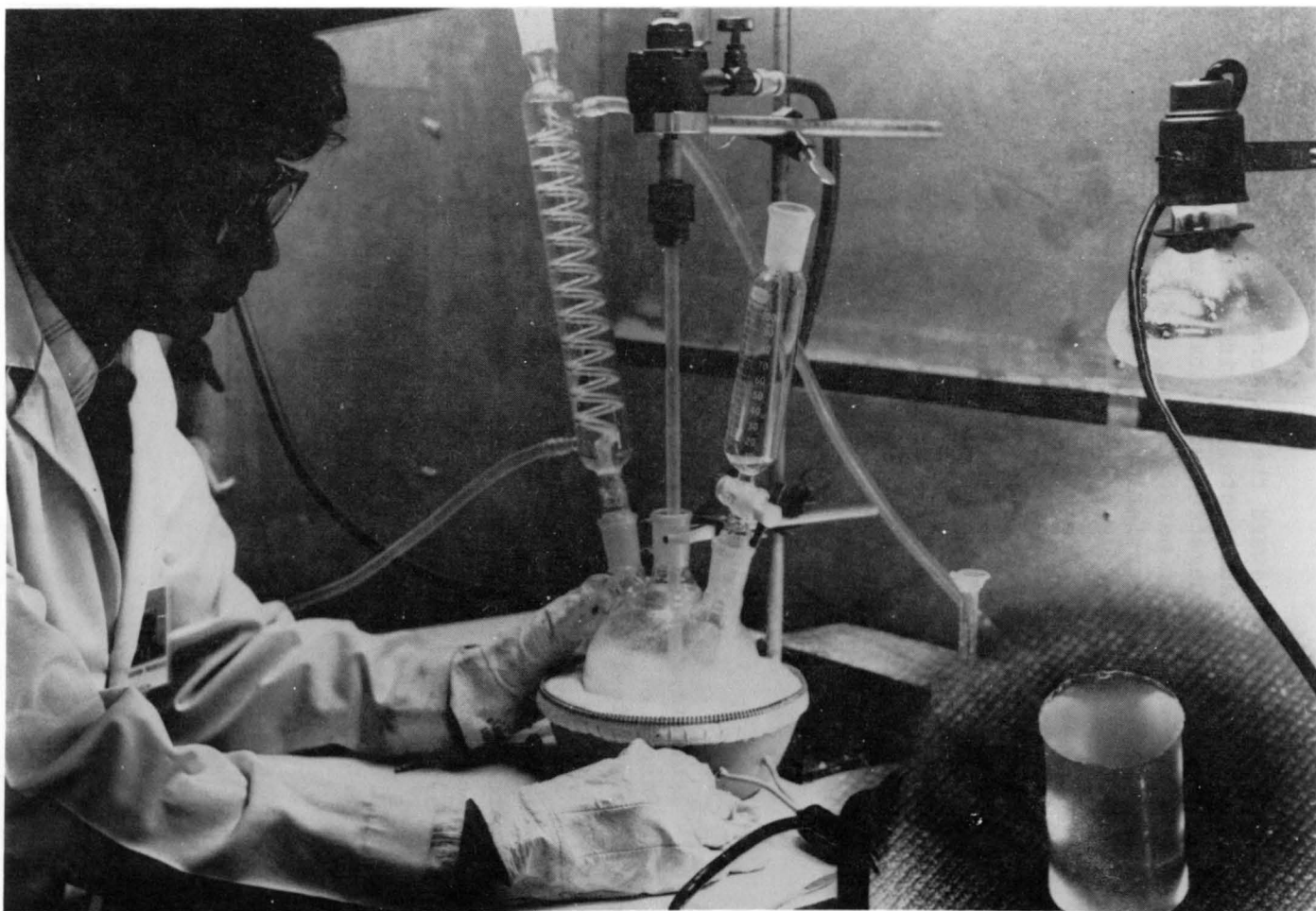
This research is directed toward the measurement of the thermo-physical properties of tungsten and tantalum using containerless techniques. The properties of tungsten and tantalum are of interest because they lie at the extreme of metal melting points and are key data in any extrapolation or interpolation 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.

Publications

Margrave, J. L., "Estimating Thermodynamic Properties of Solids and Liquids over Wide Ranges of Temperatures," published in Proceedings of Conference on Thermophysical Properties," NBS, Washington, D.C., June 1981.

Margrave, J. L., "Heat Capacities of Liquid Metals above 1500 K," in Materials Research Society Symposia Proceedings, Materials Processing in the Reduced Gravity Environment of Space, Volume 9 (G. E. Rindone, ed.), North-Holland, 1982, pp. 39-42.



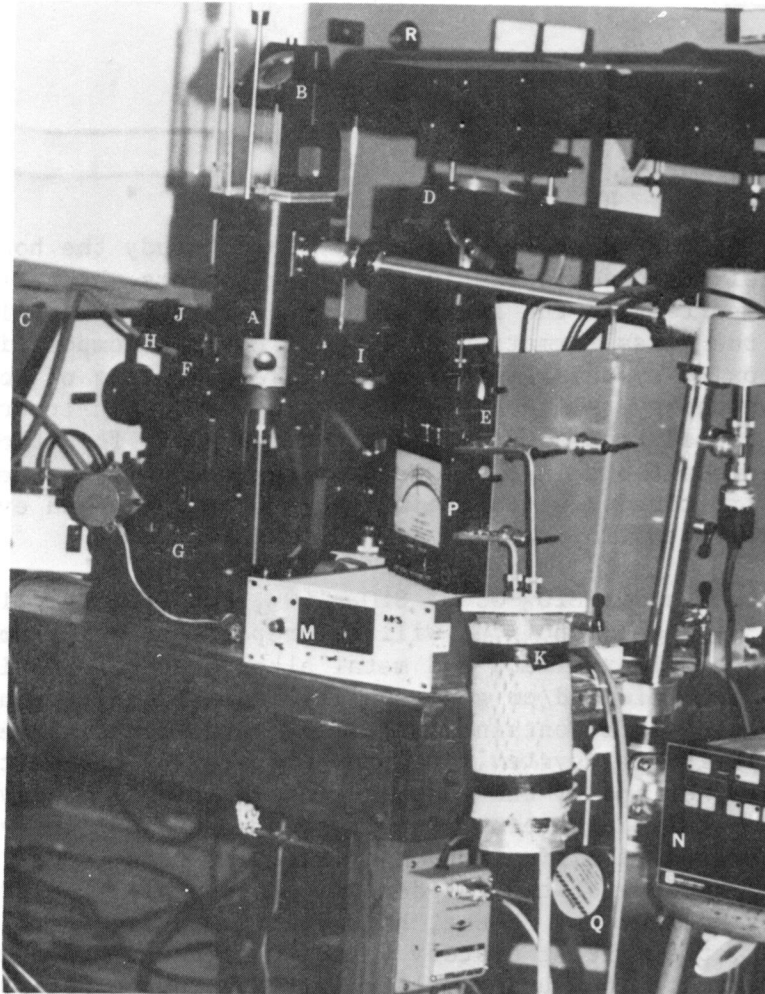
Low temperature synthesis of homogeneous gels and gel-monoliths and a piece of glass-like porous gel-monolith preform after thermal treatment at 500°C.

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

Battelle Columbus Laboratories  
Dr. S. P. Mukherjee  
NAS8-34894 Total Cost: \$200K  
June 1982 - June 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  $\text{SiO}_2\text{-GeO}_2$ ,  $\text{SiO}_2\text{-TiO}_2$ , and  $\text{GeO}_2\text{-PbO/Bi}_2\text{O}_3$  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.



(a) Chamber with levitated, CW CO<sub>2</sub> laser heated alumina sphere, (b) directed infrared laser beam for heating, (c) ultraviolet dye laser to produce atomic fluorescence, (d) optical positioning for directing UV laser beam through the levitation apparatus, (e) fluorescence focusing mirror, (f) fluorescence detector with slits, (g) detector positioning devices with stepper motor drives, (h) optical fiber to monochromator, photodetector and electronics, (i) ambient temperature chamber, (j) ambient detector with optical fiber to second monochromator, photodetector and electronics, (k) isothermal flow chamber for adding mercury vapor to argon gas flow, (l) stepper motor controller, (m) manometer readout for chamber and critical flowmeters, (n) power controller for CW CO<sub>2</sub> laser, (p) automatic optical pyrometer, (q) throttle valve to vacuum pump, and (r) induction heater for liquid metal levitation.



Containerless High Temperature Property Measurements by Atomic Fluorescence

Yale University

Dr. P. C. Nordine

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.

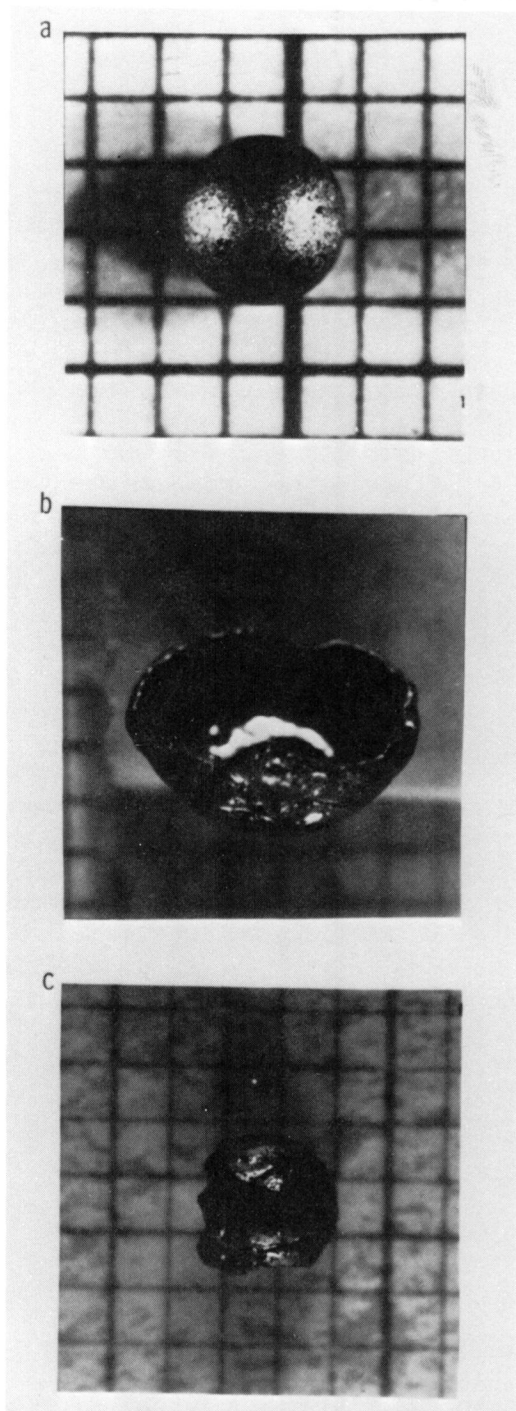
Containerless experiments employ aerodynamic and/or electromagnetic levitation with CWCO<sub>2</sub> laser and/or induction heating of the specimens. Atomic Hg fluorescence at 253.7nm, has been studied to 2327K, the melting point of sapphire (Al<sub>2</sub>O<sub>3</sub>). Experiments with Hg and K fluorescence (in Ar) are in progress, with specimens of known spectral emittance (and temperature) to establish thermal diffusion and inertial separation effects on the relation between concentration and temperature. Absolute temperature measurement on liquid metal and/or non-opaque specimens via ideal gas fluorescence thermometry is one goal of the ambient species fluorescence studies. Vaporization of elemental species from metals, metal borides, and metal oxides will also be investigated.

## Publications

Nordine, P. C. and Atkins, R. M., "Aerodynamic Levitation of Laser Heated Solids in Gas Jets," Rev. Sci. Instrum. 53 (September 1982).



Nordine, P. C., Schiffman, R. A., and Sethi, D. S., "Fluorescence Study of High Temperature Aerodynamic Levitation," in Materials Research Society Symposia Proceedings, Materials Processing in the Reduced Gravity Environment of Space, Volume 9 (G. E. Rindone, ed.), North Holland, 1982, pp. 43-48.



Photomicrograph of several drop tube samples of Nb-22 at .% Ge  
(a)  $\Delta T \leq 100K$  solidified in tube, (b)  $\Delta T = 300K$ , solidified in  
high viscosity oil, (c)  $\Delta T = 300K$ , solidified in low viscosity  
oil.

Undercooling Studies in Metastable Peritectic Compounds

Marshall Space Flight Center  
 M. B. Robinson  
 In-House \$80K/year  
 March 1, 1979 - March 1, 1982

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 temperature 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.

## Publications

Steinberg, J., Lord, A. E., Lacy, L. L., et al., "Production of Bulk Amorphous  $Pd_{77.5}Si_{16.5}Cu_6$  in Continuous Low-Gravity Environment," Appl. Phys. Lett. **38**, 135-137 (February 1981).

Robinson, M. B., "Undercooling Measurement in a Low Gravity Containerless Environment," MS Thesis, University of Alabama in Huntsville, 1981.

Lacy, L. L., Robinson, M. B., and Nisen, N. B., "Containerless High-Temperature Calorimeter," Patent #4,248,083, issued Spring, 1981.

Lacy, L. L., Rathz, T. J., and Robinson, M. B., "Containerless Undercooling and Solidification of Bulk Metastable Nb<sub>3</sub>Ge Alloys," J. Appl. Phys. 53, 682-689 (January 1982).

Lacy, L. L., Rathz, T. J., and Robinson, M. B., "Containerless Undercooling and Solidification in Drop Tubes," J. Cryst. Growth 51, 47-61 (January 1981).

Lacy, L. L., Robinson, M. B., Rathz, T. J., Evans, N. D., and Bayuzick, R., "Solidification Studies of NbGe Alloys at Large Degrees of Supercooling," in Materials Research Society Symposia Proceedings, Materials Processing in the Reduced Gravity Environment of Space, Volume 9 (G. E. Rindone, ed.), North Holland, 1982, pp. 87-94.

Lacy, L. L., Robinson, M. B., and Rathz, T. J., "Containerless Undercooling and Solidification of Bulk Metastable Nb<sub>3</sub>Ge Alloys," presented at TMS/AIME Fall Meeting, Louisville, KY, October 1981.

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.





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

El-Kaddah, N. and Szekely, J., "The Calculation of Transport Phenomena in Levitation Melted Drops," in Materials Research Society Symposia Proceedings, Materials Processing in the Reduced Gravity Environment of Space, Volume 9 (G. E. Rindone, ed.), North-Holland, 1982, pp. 191-198.

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.

#### Presentations

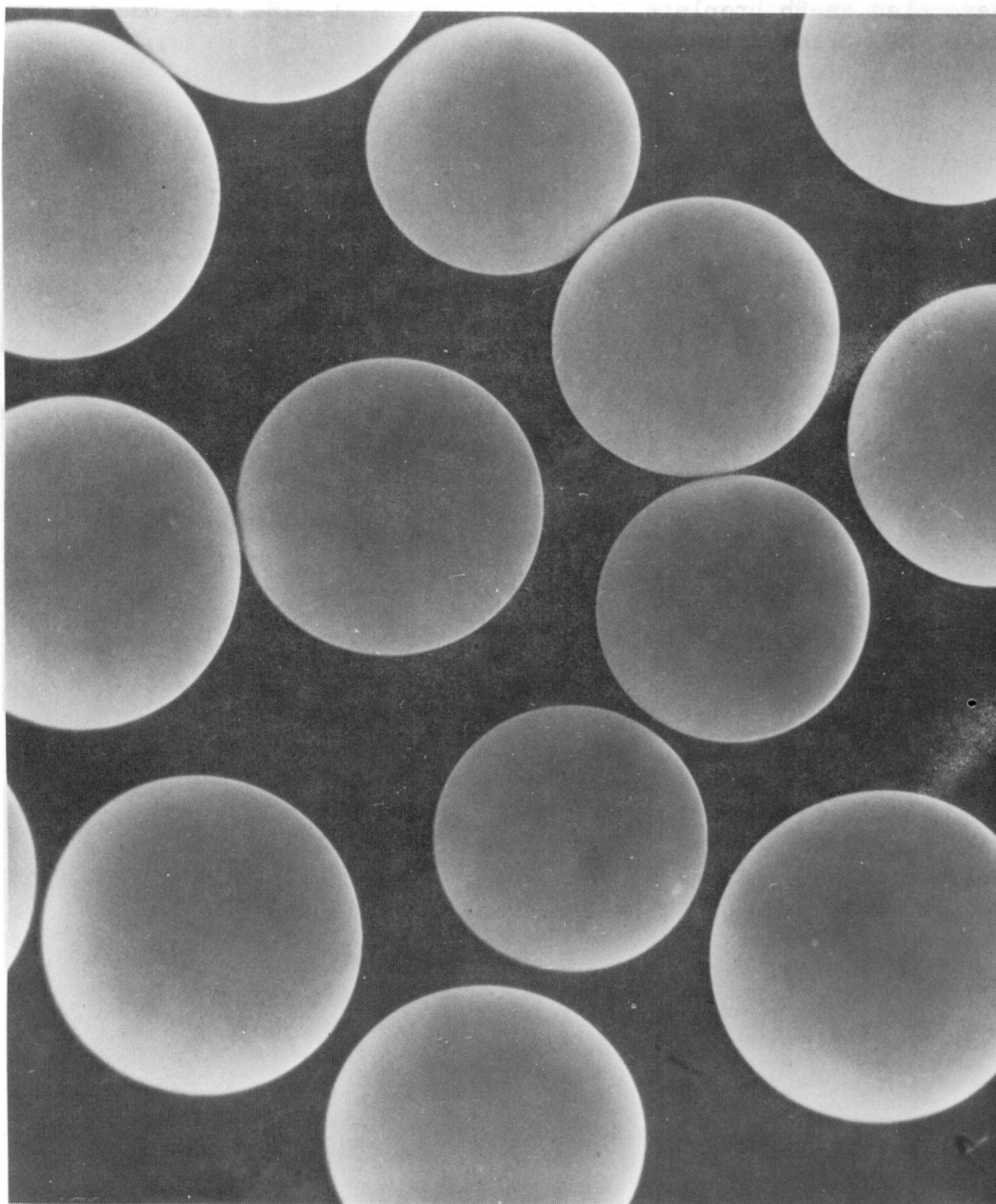
Shiohara, Y., Chu, M. G., MacIsaac, D. G., and Flemings, M. C., "Structure of Undercooled Liquid Droplets," presented at 1982 Spring Meeting, The Metallurgical Society of AIME, Crystallization of Non-Equilibrium Structure IV, Microstructural Development, Dalls, Texas, February 1982.

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MacIsaac, D. G., Shiohara, Y., Chu, M. G., and Flemings, M. C.,  
"Solidification of Highly Undercooled Iron and Nickel Base Alloy  
Droplets," to be presented at AIME Fall Meeting, St, Louis, MO,  
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St. Louis, MO, October 1982.

Turnbull  
016



$\text{Ni}_{40}\text{Pd}_{40}\text{P}_{20}$  glassy spheres formed in drop tube.

Crystal Nucleation in Glass-Forming Alloy and Pure Metal Melts Under Containerless and Vibrationless Conditions

Harvard University  
Professor David Turnbull  
NAS8-32691 Total Cost: \$160K (approx.)  
June 1978 - December 1982

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,  $\Delta T_0$ , 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,  $\Delta T_0$  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 ( $\sim 1^\circ$  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.

#### Publications

Turnbull, D., "On Anomalous Prefactors from Analysis of Nucleation Rates," Progress in Materials, Chalmers Anniversary Volume, (J. W. Christian, P. Haasen, and T. B. Massalski, eds.), p. 269, Pergamon Press, 1981.

Dreham, A. J. and Turnbull, D., "Crystal Nucleation in Pd-Si Alloys," in Materials Research Society Symposia Proceedings, Materials Processing in the Reduced Gravity Environment of Space, Volume 9 (G. E. Rindone, ed.), North-Holland, 1982, pp. 81-86.

Drehman, A. J. and Turnbull, D., "Solidification Behavior of Undercooled Pd<sub>83</sub>Si<sub>17</sub> and Pd<sub>82</sub>Si<sub>18</sub> Liquid Droplet," Scripta Met. 15, 543 (1981).

Thompson, C. V. and Spaepen, F., "The Effect of Solute on the Homogeneous Crystal Nucleation Frequency in Metallic Melts," in Materials Research Society Symposia Proceedings, Materials Processing in the Reduced Gravity Environment of Space, Volume 9 (G. E. Rindone, ed.), North Holland, 1982, pp. 603-610.

Thompson, C. V., Greer, A. L., and Drehman, A. J., "Crystal Nucleation in Easy Glass-Forming Alloys," to be published in Proceedings of Fourth International Conference on Rapidly Quenched Metals, Sendai, Japan, August 1981.

Thompson, C. V., "Crystal Nucleation in Easy Glass-Forming Metallic Alloys," Ph.D. Thesis, Harvard University, 1982.

Fusion Target Technology

Jet Propulsion Laboratory  
 Dr. T. G. Wang  
 In-Center \$300K/year  
 October 1979 - continuing task

The objectives of this task are to (1) study the physical processes that are associated with the fabrication of inertial confinement fusion (ICF) targets in a weightless environment, (2) determine jointly with Department of Energy (DOE) centers the need for extended zero gravity in the future production of ICF targets, and (3) provide technological information to DOE centers that is pertinent to their current target fabrication research.

The approach to be taken is to (1) study the fluid dynamic processes that pertain to pellet fabrication processes such as bubble centering, coating uniformity, and various instabilities; (2) study the effects of various temperature levels and temperature gradients on pellet fabrication; (3) construct Earth-based high temperature and high temperature gradient drop towers; (4) initiate development of a process for the studies, numerical analyses, and computer calculations on various pellet fabrication processes.

## Publications

- Young, A. T. Lee, M. C., Feng, I., Elleman, D. D., and Wang, T., "Polymer Coating of Glass Microballoons Levitated in a Focused Acoustic Field," in Materials Research Society Symposia Proceedings, Materials Processing in the Reduced Gravity Environment of Space, Volume 9 (G. E. Rindone, ed.), North Holland, 1982, pp. 67-70.
- Lee, M. C., Kendall, J. M., Elleman, D. D., Rhim, W. K., Helizon, R. S., Youngberg, C. L., Feng, I., and Wang, T. G., "Application of Microgravity and Containerless Environments to the Investigation of Fusion Target Fabrication Technology," in Materials Research Society Symposia Proceedings, Materials Processing in the Reduced Gravity Environment of Space, Volume 9 (G. E. Rindone, ed.), North Holland, 1982, pp. 95-104.
- Lee, M. C., Kendall, J. M., Wang, T. G., and Johnson, W. L., "Investigation of Metallic Glass Hollow Spheres for Fusion Target Application, in Materials Research Society Symposia Proceedings, Materials Processing in the Reduced Gravity Environment of Space, Volume 9 (G. E. Rindone, ed.), North Holland, 1982, pp. 105-113.
- Lee, M. C., Kendall, J., and Johnson, W., "Spheres of the Metallic Glass  $Au_{55}Pb_{22.5}Sb_{22.5}$  Alloys and its Surface Characteristics," Appl. Phys. Lett. 40, 382-384 (March 1982).

Kendall, J., "Hydrodynamic Performance of an Annular Liquid Jet; Production of Spherical Shells," in Proceedings of Second International Colloquium on Drops and Bubbles, Monterey, CA, November 19-21, 1981, pp. 79-87, JPL Publication 82-7.

Lee, M. C., Feng, I., Elleman, D. D., Wang, T. G., and Young, A. T., "Generation of a Strong Core Centering Force Submillimeter Compound Droplet System," in Proceedings of Second International Colloquium on Drops and Bubbles, Monterey, CA, November 19-21, 1981, pp. 107-111, JPL Publication 82-7.

Lee, M. C., Feng, I., Elleman, D. D., Wang, T. G., and Young, A. T., "Coating of Glass Microballoon Using an Acoustic Technique," J. Vac. Sci. Tech. 20, 1123-1124 (April 1982).

Kendall, J. M., Lee, M. C., and Wang, T. G., "Metal Shell Technology Based Upon Hollow Jet Instability," J. Vac. Sci. Tech. 20, 1091 (April 1982).



Advanced Containerless Processing Technology

Jet Propulsion Laboratory  
Dr. T. G. Wang  
In-Center \$350K/year  
October 1978 - continuing task

The primary 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 design information on a flight version of this chamber for materials science studies in a contactless and zero gravitation environment, and (3) provide potential MPS investigators with a set of ground-based facilities with which to perform precursor experiments.

The approaches to be taken in FY 82 are experimental and theoretical studies of (1) acoustic positioning and manipulation capabilities of a rectangular chamber as a function of temperature and pressure, (2) various acoustical geometries which may have special application in materials science studies, (3) loss mechanisms associated with high-intensity and high-temperature acoustic waves, (4) an aeroacoustic positioning system which will allow us to levitate heavy samples in the laboratory, (5) a liquid-liquid positioning system which will allow us to study the dynamics of liquid melts, and (6) positioning and manipulation capabilities of a KC-135 acoustic module.

Publications

Barmatz, M., "Overview of Containerless Processing Technologies," in Materials Research Society Symposia Proceedings, Materials Processing in the Reduced Gravity Environment of Space, Volume 9, (G. E. Rindone, ed.), North Holland, 1982, pp. 25-38.

Barmatz, M. and Gaspar, M., "Scattering Effects of a Sphere in a Cylindrical Resonant Cavity," JASA Sup.1, 70 (1981).

Trinh, E., Wang, T. G., and Robey, J., "A Non-uniformly Heated Resonant Chamber for Levitation Studies in Air," JASA Sup. 1, 70 (1981).

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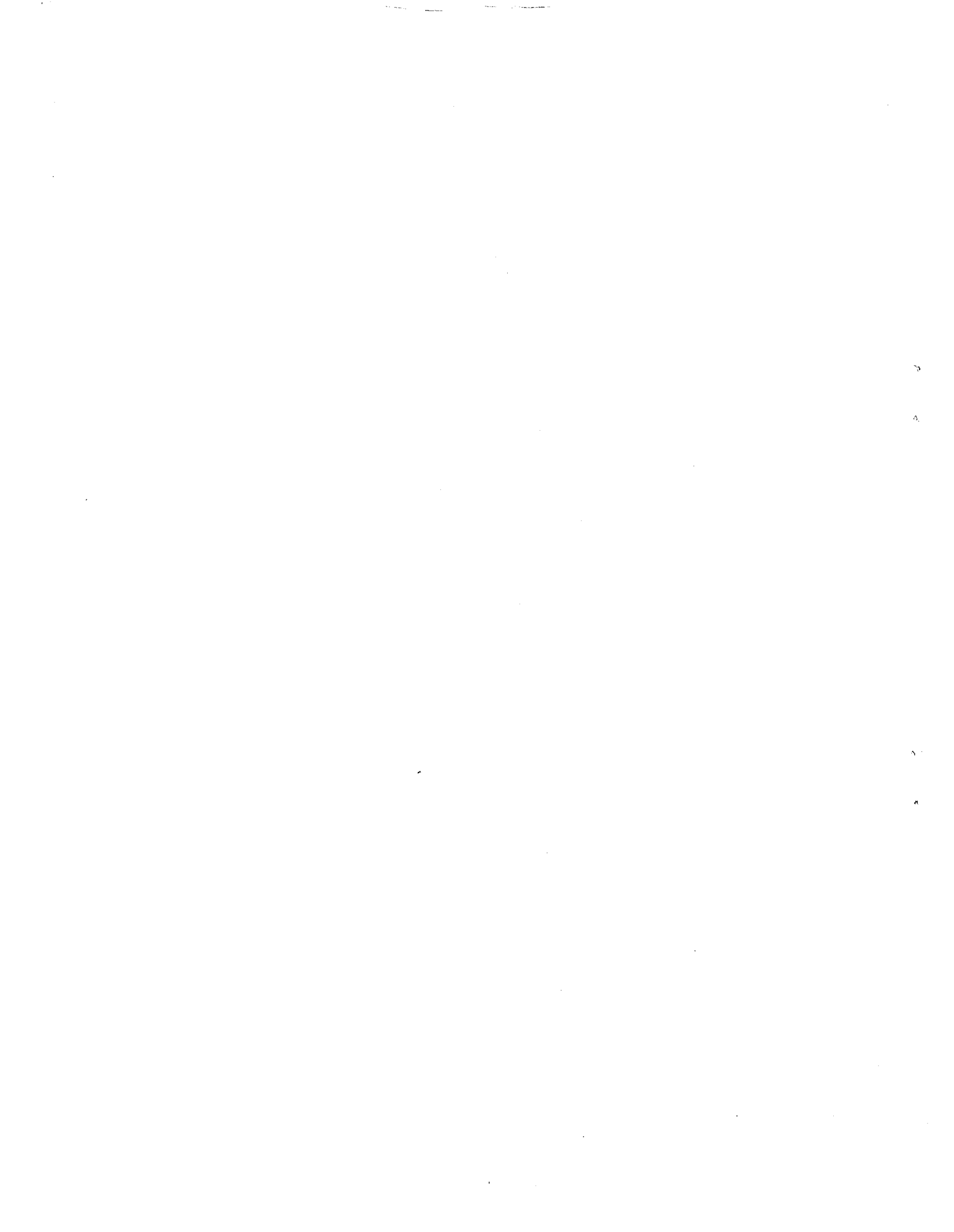
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**APPENDIX A**  
**MPS ORGANIZATIONS**



MPS ORGANIZATIONS

Alabama A&M University  
Huntsville, AL

Battelle Columbus Laboratories  
Columbus, OH

Bjorksten Research Laboratories  
Madison, WI

Charles Stark Draper Laboratory  
Cambridge, MA

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.

Honeywell Research Laboratories  
Lexington, MA

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

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

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

Semtec, Inc.  
Huntsville, AL

Stanford University  
Stanford, CA

University of Alabama, Huntsville (UAH)

University of Arizona  
Tucson, AZ

University of Missouri-Rolla  
Rolla, MO

University of Oregon  
Health Sciences Center  
Portland, OR

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

Yale University  
New Haven, CT





**APPENDIX B**  
**INDEX OF PRINCIPAL INVESTIGATORS**

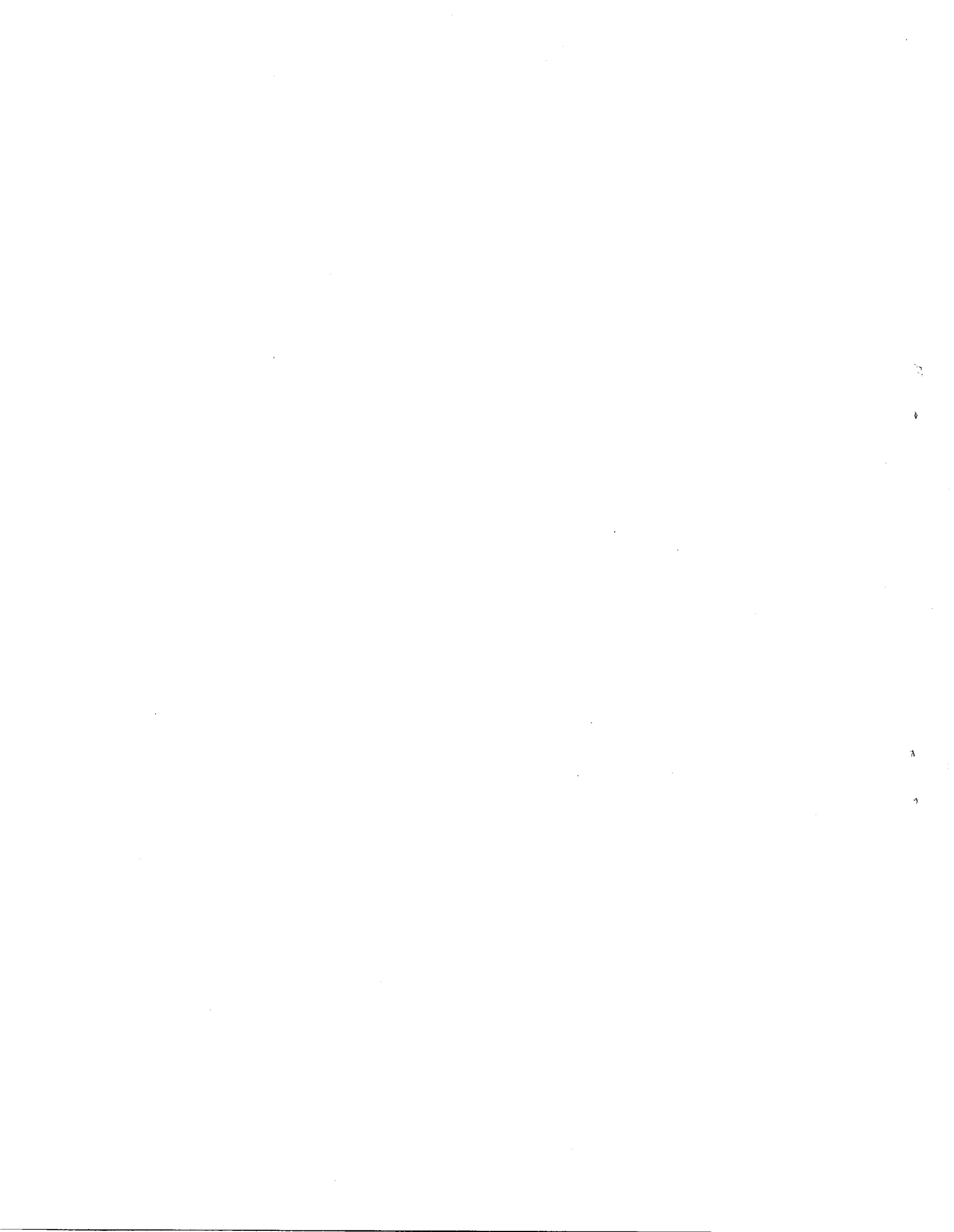


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APPROVAL

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.



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A. J. Dessler  
Director, Space Science Laboratory





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