

# **NASA Technical Memorandum 4191**

## **Microgravity Science and Applications Program Tasks**

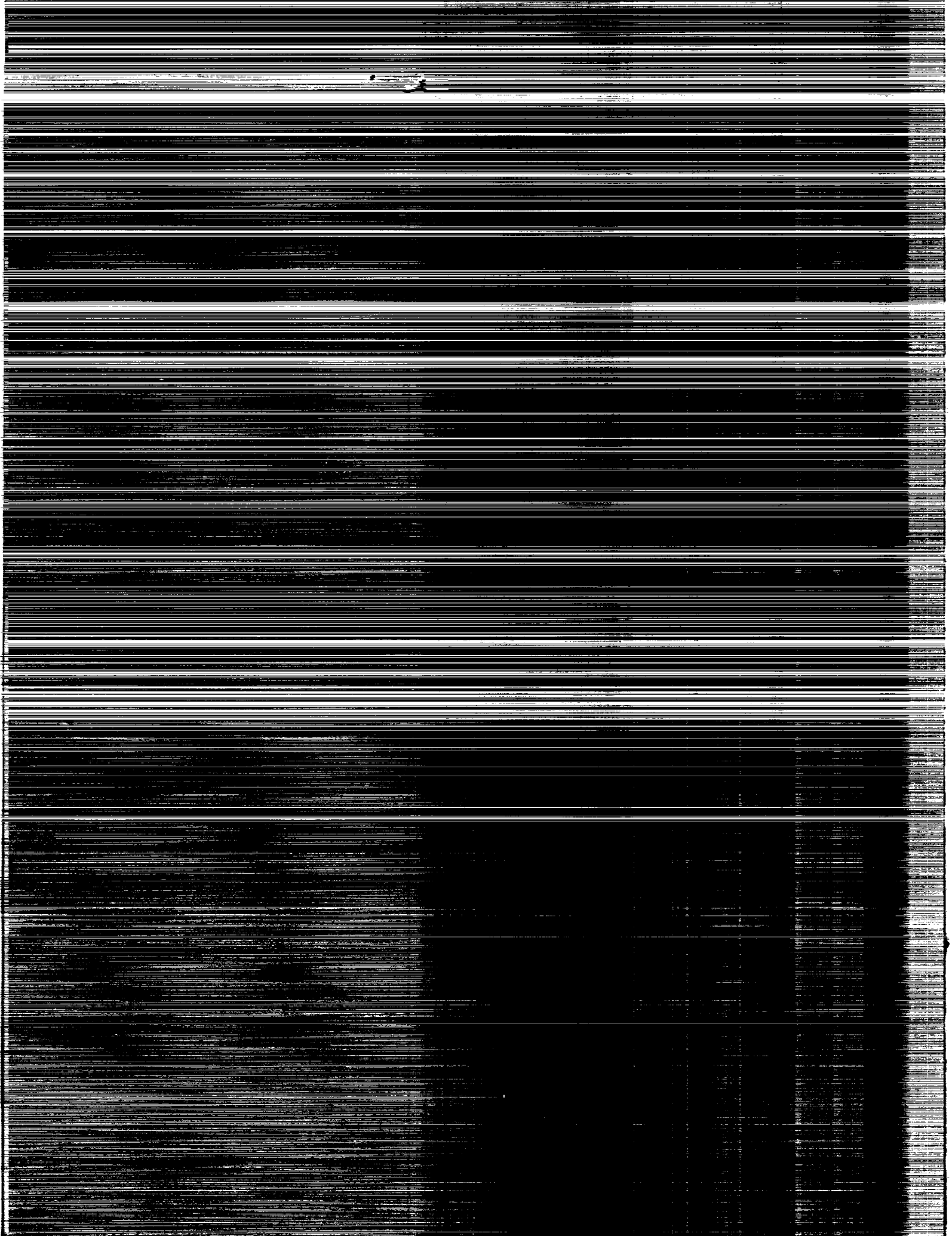
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NASA Technical Memorandum 4191

# Microgravity Science and Applications Program Tasks

*1989 Revision*

*NASA Office of Space Science and Applications  
Washington, D.C.*



National Aeronautics and  
Space Administration  
Office of Management  
Scientific and Technical  
Information Division

**1990**

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## **I. INTRODUCTION**





## NASA TECHNICAL MEMORANDUM

### I. INTRODUCTION

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

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

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

Emphasis will be placed on the expansion of currently funded activities for ground-based and space flight investigations to maximize the outputs from these opportunities. Initiatives requiring new hardware will be encouraged at a low level until funds can be made available. The expansion of current efforts is occurring as a result of focusing support for current space flight investigations on forming facility experiment teams to provide advice and identify future involvement. Emphasis has been placed on experiments involving the Materials Experiment Assembly and Mid-deck experiments on the Space Shuttle.

The Microgravity Science and Applications Program Task Document covers the period of January 1989 - December 1989. The document includes research projects now being funded by the Office of Space Sciences and Applications, Microgravity Science and Applications Division, NASA Headquarters.

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



## II. TASKS

1. The first part of the document discusses the importance of maintaining accurate records of all transactions and activities. It emphasizes the need for transparency and accountability in financial reporting.

2. The second part of the document outlines the various methods used to collect and analyze data. It includes a detailed description of the sampling process and the statistical techniques employed to interpret the results.

3. The third part of the document presents the findings of the study. It shows that there is a significant correlation between the variables being studied, which supports the hypothesis that was tested.

4. The fourth part of the document discusses the implications of the findings for future research and practice. It suggests that the results of this study could be used to inform policy decisions and to guide the development of new programs and initiatives.

5. The fifth part of the document provides a conclusion and a summary of the key points. It reiterates the importance of the study and the need for further research in this area.

6. The sixth part of the document includes a list of references to the sources used in the study. It also includes a list of appendices that provide additional information and data.

7. The seventh part of the document is a list of figures and tables that are included in the study. It provides a brief description of each figure and table and explains how it is used to present the data.

8. The eighth part of the document is a list of footnotes that provide additional information and references. It also includes a list of abbreviations and a list of symbols that are used in the study.

9. The ninth part of the document is a list of acknowledgments that thank the individuals and organizations that provided support and assistance during the study.

10. The tenth part of the document is a list of appendices that provide additional information and data. It includes a list of tables and figures that are not included in the main body of the document.

**A. GROUND BASED EXPERIMENTS**

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## 1. ELECTRONIC MATERIALS



Process Modelling for Materials Preparation Experiments

University of Alabama in Huntsville  
Center for Microgravity and Materials Research  
Dr. J. Iwan D. Alexander  
Dr. Thomas A. Nyce  
Dr. Jalil Ouazzani  
Professor Franz Rosenberger  
NAG8-790 (NASA Contact: S.L. Lehoczy, MSFC)  
August 1989 - July 1992

*Objectives:* The objectives of this program focus on the exploitation and improvement of available numerical techniques in order to formulate and solve useful models of transport processes in (microgravity) materials preparation experiments. Recognizing the fact that the potential of the most sophisticated modelling techniques cannot be fully realized if the physical properties, boundary and operating conditions are not fully characterized, we aim to develop a comprehensive research program which coordinates a study of these essential ingredients with the development of numerical models for two specific crystal growth systems: (1) growth of mercury cadmium telluride (MCT) by the Bridgman-Stockbarger technique and (2) growth of triglycine sulfate (TGS) from solution.

*Research Task Description:* This program entails the following tasks:

- Transport property studies under representative crystal growth conditions (including undercooling and supersaturation):

Kinematic viscosity and solute diffusivities of molten mercury cadmium telluride as a function of temperature and composition using an oscillating crucible method and electrical conductivity method, respectively.

Solute diffusivity in triglycine sulphate-water solutions using an interferometric technique.

- Numerical modelling studies (using experimentally determined boundary conditions):

The development of an algorithm which employs spectral techniques to solve a moving boundary problem. Subsequent incorporation of this technique into a pseudo-spectral collocation method that we have previously applied to convective-diffusive transport in melts. This will be followed by the development of numerical methods for the specific moving boundary problems associated with the growth of MCT by the Bridgman-Stockbarger technique and the growth of TGS from solution.

## Electronic Materials

NASA Lewis Research Center  
Thomas K. Glasgow  
In-House

The objective of this project is to develop a synergistic approach to crystal growth of electronic materials combining both theoretical and experimental techniques. Activities in the arena of electronic materials fall into four categories: growth of opto-electronic salts, study of the interaction of radiation and convection during physical vapor transport, modelling of crystal growth processes, e.g. GaAs Bridgman growth, and study of residual acceleration effects.

Salts having favorable infrared transmission characteristics have been under joint study by researchers from Westinghouse and Lewis. Both physical vapor transport and melt growth by Bridgman techniques have been used, the first for  $\text{Hg}_2\text{Cl}_2$ , the second for  $\text{PbCl}_2$  and  $\text{PbBr}_2$ . All three of these materials have been identified by Westinghouse for their potential use in advanced opto-electronic devices. Preliminary experiments have delineated thermal gradient and growth rate regimes for quality crystal growth. Based on work performed at Lewis and at Westinghouse a flight proposal was prepared by Westinghouse in response to the Announcement of Opportunity. Dr. Walter Duval will serve as the project scientist for this accepted proposal.

Because the salts are transparent themselves and because they can be grown in quartz(transparent) crucibles radiative heat transfer must be considered to understand their crystal growth. When modelling growth of opaque materials, schemes very similar to traditional finite element modelling of stress fields may be used. Here each element interacts only with its adjacent neighbors. But when transparent or semitransparent materials are involved, an element may exchange energy with a distant element, greatly complicating the mathematics. Radiative heat transfer is often ignored in modelling, even in vapor transport where evaporating and condensing surfaces are within direct view. While this may be reasonable for low temperature work, such as the vapor transport of organic molecules, it is not appropriate for the high temperatures at which semiconductors and salts are grown. (Note that ignoring radiative transport is not appropriate for organic materials transport when temperature gradients and temperature differences are high as in the 3M PVTOS experiments.) At Lewis we have begun to include radiative transfer in treatment of physical vapor transport. First results indicate that interface shape is influenced by radiation; the influence is slight for earthbound growth, but becomes more important in space as convection decreases. Dr Mohamed Kassemi has led this work; he has prepared a paper describing early results for the Reno AIAA meeting.

Modelling has been extended at Lewis to the GaAs growth experiment planned by GTE. Their approach is to employ a GAS canister to hold their own fairly simple power down furnaces. In a GTE furnace, about three inches of a four inch sample are to be melted then regrown to study solute redistribution and crystal growth in microgravity. The sample and its dual walled(quartz and BN) crucible have been modelled and growth has been simulated for various acceleration environments, including one-g, Shuttle, sounding rocket, and aircraft flight. For samples of the diameter and viscosity of the GTE experiment, the results indicate that aircraft offer insufficient time at low acceleration to allow damping of initial convection. Rocket flight would allow damping of initial convection and growth of a small amount of undisturbed material. Shuttle flight would allow growth dominated by diffusion for the planned growth rate. The growth interface is predicted to be gently curved. A broadly applicable, simple formula for estimating the time required for an initial annular convection cell to damp to a desired fraction of its original value was obtained. The equation, in dimensional units, is

$$t \sim 0.0291n(1-FR)*r^2/v$$

where  $FR$  is the fraction remaining of the original convection velocity,  $r$  is the ampoule radius, and  $v$  is the

melt kinematic viscosity. The relation was obtained considering several independent numerical studies of directional solidification. Because there remains significant interest in this flight crystal growth experiment by the Air Force, Dr. Arnon Chait and Thomas Glasgow presented results of recent studies to Wright Patterson AFB.

Starting with the work on DMOS, 3M's flight experiment for diffusive growth of organic crystals, Lewis researchers have been developing mathematical tools for investigating convective mixing in microgravity environments. Both steady and time varying accelerations have been examined. It has been found that completely reversed accelerations, ie g-jitter, can cause complete mixing at appropriate interrelations of frequency, amplitude, viscosity, density, and density difference. Steady acceleration is much less effective at mixing, though it can cause bulk fluid flow. Developed for understanding the DMOS experiments, these tools are now available to other researchers through Lewis. In the past year the modelling associated with the question of varying accelerations has been graphically displayed on video by Dr Walter Duval; copies may be obtained from him.

In the past year the efforts of the Lewis group have been augmented by Dr. Christophe Mennetrier, a post doctoral from France; he has investigated the role of crucible inclination during the physical vapor transport of a heavy molecule in a lighter diluent gas. His results, again presented on video, show that even slight inclinations lead to nonaxisymmetric flows and that the nominally stable orientation of hot end elevated is not necessarily stable.

#### Publications

Jacqmin, D., and Duval, W. M. B., "Instabilities Caused by Oscillation, Accelerations Normal to a Viscous Fluid-Fluid Interface," J. Fluid Mech. 196, 495-511 (1988).

Chait, A., and Korpela, S. A., "The Secondary Flow and its Stability for Natural Convection in a Tall Vertical Enclosure," J. Fluid Mech. 200 (1989).

Duval, W.M.B. and Jacqmin, D. A., "Mixing of Two Fluids Under Steady Microgravity Acceleration," *7th International PCH Conference*, MIT, Cambridge, MA, June 25-29, 1989 .

Kassemi, M. and Duval, W.M.B., "Effects of Gas and Surface Radiation on Crystal Growth from the Vapor Phase," *7th International PCH Conference*, MIT, Cambridge, MA, June 25-29, 1989 .

Kassemi, M., and Duval, W.M.B., "Interaction of Surface Radiation with Convection in Crystal Growth by Physical Vapor Transport," *27th Aerospace Sciences Meeting*, January 9-12, 1989, Reno, NV.

Mennetrier, C. D., and Duval, W.M.B., "Coupled Effects of Conduction in the Crystal and Thermo-Solutal Convection in a Rectangular Inclined Enclosure," *28th ALAA Aerospace Sciences Meeting*, Reno, NV, January 8-11, 1989 .

Gokoglu, S. A., Kuczmarski, M., Tsui, P., and Chait, A., "Convection and Chemistry Effects in CVD: A 3D Analysis for Silicon Deposition," J. de Phys., 1989.

Young, G. W. and Chait, A., "Steady-State Thermal-Solutal Diffusion in a Float Zone," J. Cryst. Growth 96, 1989.

Gokoglu, S. A., Arnold, W. A., Tsui, P., and Chait, A., "A 2D Analysis for Convection Effects in a CVD Reactor Configured for Deposition on Single Fibers," in *Proceedings of the 1990 ASME Winter Annual Meeting* (accepted).

### Crystal Growth by Two Modified Floating-Zone Processes

University of Wisconsin

Professor Sindo Kou

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

March 1, 1988 - February 28, 1991

**Objectives:** Floating-zone crystal growth under microgravity, though essentially free from natural convection, can still suffer from undesirable thermocapillary (Marangoni) convection. The objectives of this study are: (1) to help reduce melt-zone convection in floating-zone crystal growth under microgravity, and (2) to help control crystal cross-sectional geometry.

**Research Task Description:** To effectively reduce this convection while at the same time help produce single crystals of a uniform diameter and smooth surface, two modified floating-zone processes are being studied. The first of the two processes uses a ring heater in contact with the melt surface and the second a sheet heater immersed in the melt, both (heaters) with careful temperature control during crystal growth. The approach is as follows: (1) Flow visualization using a transparent melt in which Marangoni (rather than natural) convection dominates so as to allow melt-zone convection under microgravity to be simulated in ground-base experiments; (2) Crystal growth and examination of cross-sectional variation along the crystal axis; and (3) Computer simulation using general body-fitting curvilinear coordinates, allowing consideration for shapes of unknown melt/crystal interfaces.

**Progress to Date:** (1) Flow visualization using  $\text{NaNO}_3$  melt suggests significantly less convection in both modified processes; (2) Computer simulation confirms flow-visualization experiments; (3) Growth of  $\text{NaNO}_3$  crystals shows good cross-sectional control in modified process 2 (immersed heater); and (4) Computer simulations for floating-zone crystal growth under microgravity describes not only Marangoni convection but also the unknown melt/solid interfaces.

### Publications

Kim, Y. J., and Kou, S., "Thermocapillary Convection in Zone Melting Crystal Growth-An Open-Boat Physical Simulation", J. Cryst. Growth, 1989 (in press).

Lan, C. W., Kim, Y. J., and Kou, S., "A Half-zone Study of Marangoni Convection in Floating-Zone Crystal Growth Under Microgravity", submitted for publication.

Lan, C. W., and Kou, S., "Thermocapillary Flow and Melt/Solid Interfaces in Floating-Zone Crystal Growth Under Microgravity", submitted for publication.

Lan, C. W., and Kou, S., "Thermocapillary Flow and Natural Convection in a Melt Column with an Unknown Melt/Solid Interface", submitted for publication.

Theoretical Studies in Support of the 3-M Vapor Transport (PVTOS) Experiments

Yale University  
Dr. Daniel E. Rosner  
Dr. David E. Keyes  
NAG3-898

This 'pilot' program consists of a preliminary theoretical study of the coupled mass, momentum-, and heat-transfer conditions expected within small ampoules used (3M Corp.) to grow oriented organic solid (OS-) films, by physical vapor transport (PVT) in microgravity environments. Previous theoretical studies made familiar restrictive assumptions (e.g., smallness of  $\Delta T/T$ , equality of molecular diffusivities) not valid under PVTOS conditions. But, undoubtedly even more important to the future of the PVT/CVT field, the phenomena of sidewall gas 'creep', Soret transport of the organic vapor, and large vapor phase supersaturations associated with the extraordinarily large prevailing temperature gradients were not previously considered, and are concluded here to be potentially extremely significant. Rational estimates are being made of molecular transport properties relevant to the highly non-spherical molecule: copper-phthalocyanine monomer in a gas mixture containing  $H_2(g)$  and  $Xe(g)$ . Efficient numerical methods are outlined/illustrated for making steady axisymmetric gas flow calculations within such ampoules, allowing for realistic  $\Delta T/T_w$ -values, and ultimately even corrections to Navier-Stokes-Fourier 'closure' for the governing 'continuum' differential equations; eg. including "thermal stress convection". High priority follow-on studies have been defined based on these new results, and a 3-year program of research in these promising undeveloped areas of vapor transport theory has been proposed to NASA Headquarters (April 1989).

Publications

Rosner, D. E., "Side-wall Gas 'Creep' and 'Thermal Stress Convection' in Microgravity Experiments on Film Growth by Vapor Transport," Phys. Fluids A, 1989.

Rosner, D. E. and Keyes, D. E., "Theoretical Studies in Support of the 3M-Vapor Transport (PVTOS-) Experiments," NASA CR 185122, July 1989.

Garcia-Ybarra, P. and Rosner, D. E., "Thermophoretic Properties of Non-spherical Particles and Large Molecules", AIChE J. **35**, 139-147 (1989)



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

Westinghouse Science & Technology Center  
Dr. N. B. Singh  
NAS3-25274

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

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

#### Publications

- Singh, N. B., "Evaluation of Transport Conditions During PVT Growth," Phys. Chem. Hydro. **11**, 41 (1989).
- Singh, N. B., "Crystal Characterization by Birefringence Interferometry," J. Cryst. Growth **96**, 114 (1989).
- Singh, N. B., "On the quality of mercurous chloride crystals," Mat. Lett. **7**, 397 (1989).
- Singh, N. B., "Effect of Temperature Gradient on the Optical Quality of Mercurous Chloride Crystals," J. Cryst. Growth **96**, 969 (1989).
- Singh, N. B., "Design and Performance of Optical Activity Based Bragg Cells," Adv. Opt. Inf. Process **119**, 936 (1989).
- Singh, N. B., "Evaluation of Transport Conditions During Vapor Growth of Mercurous Chloride in 1-g Conditions," J. Thermo-Phys. & Heat. Trans., 1989 (in press).

#### Presentations

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*Solution Crystal Growth of Organic and Polymetric Materials for Nonlinear Optics Applications*

NASA Marshall Space Flight Center  
Dr. Marcus Vlasse  
Dr. Donald O. Frazier  
Dr. H. J. Caulfield, University of Alabama, Huntsville  
Dr. S. C. Narang, Stanford Research Institute

*Objectives:* The major objective of the research is a thorough ground-based investigation of the basic aspects of solution growth of several representative diacetylenes and other organic compounds with nonlinear optical properties. Among the aspects to be studied are the influence of solvents, thermal gradients, concentration gradients, and, foremost, the influence of convective flows on the growth process and perfection of the crystal. Further problems to be studied are the evaluation of the influence of interface effects on defect incorporation into the crystals. Size and shape and growth habit of the crystal may play important roles in the growth process.

The results of the study will be used to determine the advantages of growing such crystals in space under a reduced gravity environment. Furthermore, these findings will be useful in solving Current problems in the crystal growth of diacetylenes and other organic or biorganic substances with nonlinear optical properties.

*Research Task Description:* In this ground-based crystal growth experiment the plan of attack is the control of convection in a predictable manner as to be able to measure its effects and extrapolate to microgravity environment. The following basic steps will form the bulk of the research.

A small scale growth experiment will be carried out using the evaporatin method to produce crystals for further study and seeding. The bulk of the work will be performed using the controlled cooling method to produce the necessary supersaturation and induce nucleation and growth. Both seeded and unseeded runs will be introduced into this growth faces. Reduction of convection will also be tried by the use of more viscous medial such as gels, where the process is quasidiffusion controlled.

Detailed characterization of the crystals will be performed to determine the densities of chemical and physical impurities. Such characterizations will be performed mainly by spectroscopic means as well as microscopy and x-ray diffraction and topography. These data will be correlated to the various growth parameters and methods in order to determine the influence of such parameters on the quality and size of crystals. Some simple modeling of the growth process will be attempted to acquire a better understanding of the mechanisms involved.

*Progress to Date:* The apparatus and method have been set up for the preparation of a urethane substituted diacetylene ( $R-C \equiv C-C \equiv C-R$ ), TCDU, not available in commerce.

The conditions (solvents, temperature, saturation, etc.) have been investigated for the growth of unseeded and seeded crystals of TCDU by the evaporation method. Single crystals of TCDU, in the form of plates of good optical quality have been grown from ethyl acetate-ethanol mixtures at room temperatures.

Work has been initiated to study the crystal growth of L-arginine phosphate (LAP), a very promising material for NLO applications. The dependence of its solubility on temperature and pH in aqueous solutions has been determined. Saturation and speed of evaporation have been studied. Seeded growth experiments in various configurations have been performed. The results of this work so far have allowed us to grow single crystals of LAP of extremely good optical quality for further experimentation.

The apparatus for the application of controlled and programmable cooling has been set up and the

cooling regimes have been determined. Temperature stability of  $\pm 0.1^{\circ}\text{C}$  is expected. Growth trials using this method have started. This growth method, apparatus and growth parameters will lead eventually to a possible flight experiment.

#### Publications

Vlasse, M., *Proceedings of the Polymer Investigations in Space Workshop*, Huntsville, AL, March 17, 1988.

## Heat Flow Control and Segregation in Directional Solidification

Massachusetts Institute of Technology  
Professor August F. Witt  
NSG -7645  
October 1, 1988 - September 30, 1989

*Objectives:* The objectives of the proposed research are: (1) establishment of the limits of melt stabilization in Bridgman growth configuration by axial and transverse magnetic fields; (2) development of a modular hot zone configuration that provides for quantifiable and controllable heat flow about the growth interface and for adjustable axial thermal gradients in the solid, liquid and vapor phase regions in Bridgman growth configuration; and (3) development of a data base for the optimization of space growth experiments directed at the exploration of the potential of micro-g conditions for electronic materials processing.

*Research Task Description:* In conjunction with efforts to develop a Bridgman type growth system that meets fundamental requirements for space experimentation it was considered essential to be able to operate with a heat transfer model which yields parametric information without the requirement for extensive computational work. The primary merits of previous studies was the identification of two effects, the "crucible effect" and the "interface effect". These efforts, while significant, suffered from two drawbacks:

The oversimplified basic assumptions of initial and boundary conditions which lead to a qualitative understanding and non-applicable results.

The desperate attempt to study "effects" one after the other, neglecting a priori some of the parameters, while all parameters are important a posteriori and all effects are coupled.

With this in mind an analytical approach was to be developed that allows the simultaneous consideration of all effects, neglecting a posteriori only those parameters which are found to be numerically insignificant.

*Progress to Date:* The parameters taken into account are: the thermal conductivities of the melt, the crystal and the crucible, the liberation of latent heat at the growth interface, the charge and crucible diameters, the heat transfer coefficients between the crucible and the furnace, the gradient zone length, the charge length, and the charge lowering rate.

The model developed provides analytical expressions for: (1) the axial temperature gradient of the crucible in the gradient zone, (2) an infinite length criterion, (3) axial temperature gradients of the charge at the interface, and (4) the interface location within the gradient zone. It permits analysis of experimental boundary conditions for transient growth behavior. In a second model, under development, it is found that the quantification of the heat transfer between the charge and the crucible in the gradient zone yields information pertaining to the axial temperature gradients of the charge at the interface. Theoretical expressions are directly compared to the existing experimental data base.

### Publications

C. Lin, D. J. Carlson, and A. F. Witt, "Growth Related Residual Strain in LEC GaAs," J. Cryst. Growth 92, 53-60 (1988).

C.-H. Hirsch and A. F. Witt, "Analytical Approach to the Determination of Axial Temperature Gradients in Bridgman System," J. Cryst. Growth (submitted).

C.-H. Hirsch and A. F. Witt, "Analytical Approach to the Determination of Growth Interface Morphologies in Vertical Bridgman Geometry," J. Cryst. Growth (submitted).





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

1. The first part of the document discusses the importance of maintaining accurate records of all transactions and activities. It emphasizes that proper record-keeping is essential for transparency and accountability, particularly in financial matters. The text suggests that organizations should implement robust systems to track and document every aspect of their operations, from procurement to sales.

2. The second part of the document addresses the challenges of data management in a rapidly changing environment. It highlights the need for flexible and scalable solutions that can adapt to new technologies and data sources. The author argues that organizations must invest in training and development to ensure their workforce is equipped to handle complex data sets and analyze them effectively.

3. The third part of the document focuses on the role of leadership in driving organizational success. It stresses that leaders must provide clear vision and direction, while also fostering a culture of innovation and collaboration. The text suggests that effective leaders are those who can inspire their teams to achieve their full potential and overcome any obstacles that may arise.

4. The fourth part of the document discusses the importance of risk management in ensuring the long-term sustainability of an organization. It outlines various strategies for identifying, assessing, and mitigating risks, from financial fluctuations to operational disruptions. The author emphasizes that proactive risk management is key to avoiding costly setbacks and ensuring the organization remains resilient in the face of uncertainty.

5. The fifth part of the document explores the impact of external factors on organizational performance. It discusses how changes in the market, regulatory environment, and global events can influence an organization's ability to succeed. The text suggests that organizations must stay informed about these external factors and be prepared to adjust their strategies accordingly to maintain their competitive edge.

6. The sixth part of the document concludes by summarizing the key points discussed throughout the document. It reiterates the importance of accurate record-keeping, effective data management, strong leadership, proactive risk management, and awareness of external factors. The author encourages organizations to embrace these principles as a means to achieve long-term success and growth.

Immiscible Phase Incorporation During Directional Solidification of Hypermonotectics

University of Alabama in Birmingham  
Dr. J. Barry Andrews  
Dr. R. T. DeHoff, University of Florida  
NAG8-768 (NASA Contact: P.A. Curreri, MSFC)  
April 1, 1989 - March 31, 1991

*Objectives:* The objectives of this work are to determine the manners in which the immiscible phase is incorporated into samples during directional solidification of hypermonotectic alloys and to develop a model describing the process. Immiscible alloys show great promise for use in applications ranging from new types of bearing materials, to the production of in-situ composite superconductor materials, to the processing of nuclear fuel materials. For these future engineering applications, a high volume fraction of the immiscible phase is desired in the structure. Currently, the volume fraction is limited to the value provided by the monotectic composition. This research should provide a better understanding of the ways in which the immiscible phase is incorporated into the structure and lead to the ability to produce aligned composite structures in hypermonotectic alloys which contain a high volume fraction of the immiscible phase than alloys of monotectic composition.

*Research Task Description:* Direct observation of the behavior of immiscible samples at the solidification front will be utilized to help model the incorporation of the hypermonotectic phase during solidification. This will be accomplished through the use of hypermonotectic transparent metallic analog samples and solidification of these samples on a temperature gradient stage microscope. The study will encompass both transparent monotectic systems that produce an irregular microstructure (low dome-height systems) and monotectic systems that produce aligned composite-like microstructures (high dome-height systems). In addition to binary systems, transparent ternaries will be utilized in which the miscibility gap height can be varied in order to bring about a transition in morphology from an irregular to an aligned composite-like structure. Results will be compared with those obtained from recent research on metallic hypermonotectic Cu-Pb-Al alloys of various dome heights that were processed aboard NASA's KC-135 zero-g aircraft. These metallic samples have yielded results that do not appear to be predicted by theory.

Solidification of transparent analog hypermonotectics will be carried out in thin glass cells that will be oriented horizontally in order to minimize separation of the immiscible phases due to gravity. The cells will be coated in order to prevent preferential wetting of the cell walls by the hypermonotectic liquid; an effect that could obscure events at the interface. The program will test the effect of growth rate, thermal gradient, the miscibility gap height on the ability to incorporate the hypermonotectic liquid into the structure at the growth front.

*Progress to Date:* A fully automated directional solidification apparatus has been designed and is under construction. The apparatus will permit directional solidification of immiscible transparent analog samples and viewing of the solidification front. This miniturized temperature gradient stage apparatus will be positioned on an optical microscope to permit video recording and still photography of the solidification process. Computer control of hot zone temperature, cold zone temperature and sample position will be utilized to vary solidification rate and thermal gradient over the range of interests. Tests will be carried out over growth rate and thermal gradient ranges that are known to result in microstructural changes from aligned rod-like composite structures, to arrays of spheres, to randomly dispersed structures. The information gathered will be used in an attempt to model the solidification process in immiscible systems.

## Publications

Andrews, J. B., Sandlin, A. C., and Curreri, P. A., "The Influence of Gravity Level and Interfacial Energies on Dispersion Forming Tendencies in Hypermonotectic Cu-Pb-Al Alloys," *Met. Trans.* **19A**, 2645-2650 (1988).

Sandlin, A. C., Andrews, J. B., and Curreri, P. A., "Influence of Gravity Level and Interfacial Energies on Directionally Solidified Hypermonotectic Alloys," *Met. Trans.* **19A**, 2665-2669 (1988).

Andrews, J. B., Briggs, C. J., and Robinson, M. B., "Containerless Low Gravity Processing of Immiscible Gold-Rhodium Alloys," in *Proceedings of VIII European Symposium on Materials and Fluid Sciences in Microgravity*, ESA, 1989 (in press).

Sandlin, A. C., Andrews, J. B., and Curreri, P. A., "Directional Solidification of Immiscible Cu-Pb-Al Alloys Processed under Alternating High-g/Low-g Conditions," in *Proceedings of VII European Symposium on Materials and Fluid Sciences in Microgravity*, ESA, 1989 (in press).

## Containerless Processing of Refractory Metals and Alloys

Vanderbilt University  
Dr. Robert J. Bayuzick  
Dr. William H. Hofmeister  
Dr. Michael B. Robinson, MSFC  
NAG8-765 (NASA Contact: M. B. Robinson, MSFC)  
March 1, 1989-February 28, 1992

*Objectives:* Research is being conducted on the containerless processing of refractory metals and alloys. There are three primary objectives of the work. One is to understand the kinetics of solid phase nucleation from refractory containerless melts. The second is to determine the growth velocity of the solid phase as a function of undercooling of refractory melts. The third is to relate the microstructure to degree of undercooling and solidification velocity in refractory melts.

*Research Task Description:* The first of the research objectives involves both maximum undercooling measurements and nucleation frequent determination. Maximum undercooling simply requires conducting a set of experiments on a number of samples under the best conditions possible to obtain the highest possible undercooling. The effort on nucleation frequent requires repeated experiments on given samples to evaluate the distribution of nucleation temperatures as a function of cooling rate. The second objective involves monitoring the rate of recalescence in the sample and extracting the solidification velocity from that data. The solidification velocity is then related to the degree of undercooling. The third objective is accomplished by postprocessing analysis on specimens for types, sizes, amounts, distribution and morphology of resulting phases. This includes detailing the distribution of elements and therefore characterizing segregation. A number of techniques for microstructure analysis are used including x-ray diffractational optical microscopy, scanning electron microscopy with energy dispersive analysis, microprobe and analytical transmission electron microscopy

A considerable number of experiments have already been completed in the Marshall Space Flight Center drop tube. Nb-Ge, Nb-Pt, Nb-Si alloys as well as refractory metals have all been investigated. Additional experiments are being performed in the drop tube, especially to improve the statistics. In addition, in-situ experiments using levitation melting are being run "on the bench" in the laboratory. These experiments employ solidification with undercooling in the coil and combinations of deep undercooling with rapid heat removal by splat quenching between pistons.

*Progress to Date:* Niobium-silicon alloys from 16 to 26 at.% Si were containerlessly processed using an electromagnetic levitation technique and subsequently splat quenched between two copper plates. Different degrees of superheating and undercooling were obtained in the levitation coil prior to releasing the samples for splat quenching.

In the eutectic range, from approximately 16 to 20 at.% Si, differing eutectic morphologies were found to prevail as a function of the release temperature of the drops and also as a function of the distance away from the splat surface. However, deep bulk undercooling prior to splat quenching always resulted in very fine microstructures throughout the complete specimen. Amorphous phase was found in those splats released from the levitation coil with superheat in the eutectic range. A metastable eutectic composed of Nb + Nb<sub>5</sub>Si<sub>3</sub> was observed when samples of hypereutectic composition showed evidence of Nb<sub>5</sub>Si<sub>3</sub> primary solidification. In these samples, the equilibrium tetragonal Nb<sub>3</sub>Si phase did not form part of the microstructure. When the sample composition was close to the Nb<sub>3</sub>Si stoichiometry, the only component of the microstructure was indeed tetragonal Nb<sub>3</sub>Si, particularly so if the drops were released with very deep undercoolings. The metastable A1-5 phase has never been found in the microstructures.

Solidification rates from recalescence data were estimated yielding approximately 15 cm/s for eutectics when undercooled 360 K. This is an agreement with the fastest growth rates reported in the literature for eutectic growth. On the other hand, the solidification rate estimated for Nb<sub>5</sub>Si<sub>3</sub> dendrites growing in a drop undercooled 268 K is of the order of 0.5 m/s. This is slower than theoretical predictions based on solute diffusion limited growth and indicates growth difficulties due to attachment kinetics.

Definitive results on maximum undercooling and nucleation frequency in the Nb-Si alloys employing the MSFC drop tube and/or electromagnetic levitation have not yet been obtained. However, undercoolings of Nb-Si alloys by electromagnetic levitation (solidification in the coil) tend to be similar to those obtained in the MSFC drop tube.

#### Publications

Bayuzick, R.J., Hofmeister, W.H., and Robinson, M.B., "Some Aspects of Containerless Processing," *International Forum for the Scientific Utilization of Space Station*, ISAS, Sagamihara Japan (October 1989).

Hofmeister, W.H., Robinson, M.B., and Bayuzick, R.J., "Non-Contact Temperature Measurement of a Falling Drop," *Int. J. Thermophys.* **10**, 279 (1989).

Robinson, M.B., Bayuzick, R.J., and Hoffmeister, W. H., "Undercooling Studies in Nb-Pt and Nb-Si Alloys Using the 105-Meter Drop Tube," *Adv. in Space Res.* (in press).

Bertero, G.B., Hofmeister, W.H., Bayuzick, R.J., and Robinson, M.B., "Splat Quenching of Undercooled Nb-Si Alloys With Nominal Composition of 25 Atomic Percent Silicon," in *Proceedings of Symposium on Materials Processing in Space*, ASM International, in press.

### Dynamic Thermophysical Measurements in Microgravity

National Institute of Standards and Technology

Dr. Ared Cezairliyan

W-16,247 (NASA Contact: R. Crouch, HQ)

January 1, 1989 - September 30, 1989

*Objectives:* The objective of this research is to develop accurate dynamic techniques which, in a microgravity environment, will enable the performance of thermophysical measurements on high-melting-point solids and liquids at temperatures above 1500 K. The initial goal is the development of techniques for electrically conducting materials which, when completed, will enable, for the first time, the extension of accurate thermophysical measurements to temperatures above the limit (melting point) of the highly successful ground-based millisecond-resolution pulseheating experiments. Thermophysical properties of interest include heat of fusion, heat capacity, electrical resistivity, thermal conductivity, surface tension, hemispherical total and normal spectral emittances. The longer-range goal is the development of techniques for electrically nonconducting materials (ceramics) which, when completed, will enable the accurate measurement of selected thermophysical properties of these technologically-important materials at high temperatures. The research is aimed at the ultimate development of a general-purpose facility for high temperature materials research on the Space Station.

*Research Task Description:* The technique for electrically conducting materials is based on resistively heating the specimen up to its melting point and above in about one second by passing a large current pulse through it, and simultaneously measuring the experimental quantities with millisecond resolution. The initial phase of this research requires the establishment of criteria for stability of the specimen when heated rapidly to temperatures above its melting point in a microgravity environment. For this purpose, a test package, which permits rapid heating of specimens in various geometrical forms (solid rod, tubular, triaxial, etc.), has been designed and constructed, and tested during microgravity simulations with NASA's KC-135 aircraft. The test package, which contains a singlewavelength high-speed pyrometer and a high-speed framing camera as the major diagnostic instruments, has been flown eight times. A theoretical study of the stability of a current-carrying liquid is being carried out in conjunction with the experimental work to optimize the specimen geometry and operating conditions of the measurement system. The second phase of this work involves applying the technique to definitive measurements of selected thermophysical properties of one or more refractory metals (niobium, tungsten, etc.) at and above their melting points. In order to accomplish this, additional capabilities must be added to the measurement system to enable rapid and accurate measurements of the specimen temperature, temperature gradients in the specimen, and electrical power imparted to the specimen. The temperature measurements will be performed by two new high-speed pyrometers, a multiwavelength pyrometer (already developed) and a linear spatial scanning pyrometer which is under development. The development of instrumentation for measuring imparted power is also underway.

The technique for electrically non-conducting materials is based on heating the specimen to an initial high temperature state by means of an external radiation source (laser, solar, etc.) and then, during the free cooling of the specimen, measuring the pertinent experimental quantities (specimen temperature and power radiated from the specimen) with millisecond resolution. This technique will utilize multiwavelength and 2-D spatial scanning pyrometers for temperature measurements and a pyroelectric detector for measurements of radiated power. Preliminary tests have demonstrated the feasibility of using pyroelectric detectors for the measurement of radiated power under transient conditions. The positioning of the specimen may be achieved by acoustic or electrostatic methods. In order to determine selected thermophysical properties from the experimental quantities, one needs an accurate description of the heat transfer in the specimen as a function of time, subject to realistic initial and boundary conditions. This will require the development of analytical models for the transient heat transfer in both opaque (conducting and non-conducting) and semi-transparent (non-conducting) specimens.

*Progress to Date:* Experimental and analytical work have continued on the stability of specimens in various geometries when rapidly heated to their molten state under microgravity conditions. A promising geometry for tubular specimens is the triaxial configuration in which the heating current is returned along the tube axis. An analytical model of this arrangement, developed earlier, has shown that surface tension of the liquid specimen can be determined if static equilibrium between the magnetic and surface tension forces acting on the specimen tube can be achieved. This was confirmed by subsequent microgravity experiments on copper specimens with NASA's KC-135 aircraft which established a new technique for measuring surface tension. Preliminary microgravity experiments have now been performed on tantalum specimens in order to improve the accuracy of the technique and to provide baseline data for future definitive measurements of the surface tension of high-melting-point refractory metals for which there is a paucity of data.

A study of the stability of composite specimen geometries, in which the liquid specimen is supported by an interior higher-melting-point metal, has been started. Preliminary experiments on composite specimens, performed on board the KC-135 aircraft, have yielded significant temperature excursions above the melting point.

The design of a new automated multi-purpose measurement system has begun. The system is modular in design to optimize the use of measuring instruments common to experiments on electrically conducting and non conducting specimens while, at the same time, accommodate the different heating methods, namely, volume (resistive) heating of conducting specimens or surface (laser) heating of non-conducting specimens. The measurement system will include: a computer-based data acquisition and control module, a high-speed camera module, and two experiment chamber modules, one for conducting specimens, the other for non-conducting specimens.

Testing of the high-speed linear spatial scanning pyrometer was successfully completed. This pyrometer, the only one of its kind, can measure spectral radiance temperature at about 1000 points along a straight line (25 mm long) on the specimen, with a complete cycle of measurements in about 1 ms. This pyrometer will be used to measure temperature gradients in a rapidly heating or cooling specimen providing data for diagnostic purposes and for determination of thermal conductivity, which will be a novel approach suitable for measurements at very high temperatures. The design to automate the pyrometer has started. This involves the addition of a computer-controlled incremental translation device needed for proper aiming and calibration.

As the first step toward developing a novel high-speed 2-dimensional spatial scanning pyrometer, a commercially available 2-D detector array (128x128 elements) was tested. The preliminary results demonstrated the suitability of such a detector for accurate pyrometric work. Such a pyrometer is needed in measuring and mapping the temperature of a suspended or levitated spherical specimen during rapid cooling.

Heat of fusion of titanium and a titanium alloy (90Ti-6Al-4V) was measured with the ground-based microsecond-resolution pulse-heating facility to generate data for comparison in future experiments in a microgravity environment with the millisecond-resolution system.

#### Publications

Cezairliyan, A. and Müller, A.P., "A Dynamic Technique for Thermophysical Measurements at High Temperatures in a Microgravity Environment," Int. J. Thermophysics, in press.

Müller, A.P. and Cezairliyan, A., "A Dynamic Technique for Measuring Surface Tension at High Temperatures in a Microgravity Environment," Int. J. Thermophysics, in press.



McClure, J.L. and Cezairliyan, A., "Measurement of the Heat of Fusion of Molybdenum by a Microsecond-Resolution Transient Technique," Int. J. Thermophysics, in press.

Chang, R.F. and Cezairliyan, A., "The Use of a Pyroelectric Detector for Rapid Measurements of Total Radiant Power," Int. J. Thermophysics, in press.

MacDonald, R.A., "The Stability of a Current Carrying Hollow Liquid Metal Cylinder," J. Appl. Phys., submitted.

McClure, J.L. and Cezairliyan, A., "Measurement of the Heat of Fusion of Titanium and a Titanium Alloy (90Ti-6Al-4V) by a Microsecond Resolution Transient Technique," Int. J. Thermophysics, submitted.

### Gravitational Effects on Liquid Phase Sintering

Rensselaer Polytechnic Institute  
Randall M. German  
Sita Mani  
Sung-Chul Yang  
NAG3-978 (NASA Contact: G. Santoro, LeRC)  
February 15, 1989 to February 14, 1990

*Objectives:* The focus of this research is on identification of the gravitation effects during liquid phase sintering. The primary concerns are with the causes, rates, and mechanisms of slumping, solid-liquid separation, and differential microstructural coarsening due to gravity. The dimensional changes, microstructure, mechanical properties, gradients are being measured and modeled versus position, composition, heating rate, sintering temperature, and gravitational orientation. We are establishing the theoretical basis for predicting the limiting compositions that can be sintered under normal gravity. The theoretical work has established the minimum energy configurations and the possible connectivity for compact distortion during sintering. Possible secondary factors have been identified and are under study to improve the models for grain growth in connected microstructures. A change in coarsening mechanism at long sintering times has been identified.

*Research Task Description:* The role of gravity in liquid phase sintering is based on experiments using tungsten heavy alloys (W-Ni-Fe compositions) that are processed at temperatures near 1500°C. The large density difference between the liquid and solid phases leads to considerable solid-liquid separation and compact slumping. These effects are being quantified using distortion measurements, dilatometry, and quantitative metallography. By quantification of the gravitation effects, a better understanding of the magnitude and source of such changes is possible. Furthermore, kinetic models can be assessed versus the experimental determinations.

### Publications

R. M. German, S. Farooq and C. M. Kipphut, "Kinetics of Liquid Phase Sintering," Mat. Sci. & Engr. 105, 215-224 (1988).

C. M. Kipphut, A. Bose, T. Kishi and R. M. German, "The Gravitational Effects on Liquid Phase Sintering," Advances in Powder Metallurgy, Volume 1, 1989 (in press).

## Metals and Alloys

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Thomas K. Glasgow  
In-House

There are three general categories of experiments in this area, those aimed at understanding the influence of undercooling on nucleation and on macro and microsegregation in bulk samples, those directed at fundamental understanding of dendritic and cellular growth in directional solidification, and those in support of a proposed Space Shuttle experiment on macrosegregation in metallic alloys.

Experiments on bulk undercooling have led to an increased appreciation of the role of surface tension in heterogeneous nucleation. In the Pb-Sn system one primary phase, Sn is an effective nucleant for Pb, but Pb is not an effective nucleant for Sn. Based on traditional considerations of lattice matching the nucleation behavior would be expected to be reciprocal. Instead, because of differences in interfacial energies, nonreciprocal nucleation is observed. Contrary to expectation, macrosegregation was found to increase in this system with increasing undercooling. The macrosegregation was strongly influenced by melt off and settling of lead dendrites in the less dense alloy liquid.

Experiments designed to test our ability to differentiate among theories describing cellular or dendritic growth have been performed using PbAu and PbSn alloys. Directional solidification was conducted with the hot end above the cold for thermal stability. The PbAu alloys also offered solutal stability. In the slow growth regime where predictions of the theories differ most, convection still interfered with microstructural development. (Only if the solute rich liquid were equal in density to the original liquid for all compositions and temperatures could success be expected for ground based studies.) In keeping with the findings of the ground based work, a flight experiment was proposed for performance in the MASA. Additional studies performed this year indicate that prior work reported in the literature suffers from various effects of convection frequently not acknowledged by the investigators.

Other experiments designed to improve understanding of solidification of alloys used superalloys cast on the KC-135 aircraft. With commercial alloys chosen by Pratt and Whitney, it has been shown that the solute rejection behavior of multicomponent alloys can, to a first order, be described as a sum of binary alloy results, ie the binary alloy phase diagrams predict the behavior of at least some multi-component alloys. The partition coefficients appeared to be constant over a wide range of composition. And the temperature for the final portion of solidification was approximately constant, simplifying analysis of the experimental work. The superalloys showed macrosegregation which could be explained by the directional solidification model of Bower, Brody, and Flemmings. Dr Tewari and co-authors Curreri and Lee of Marshall have four papers in preparation describing this work. A major finding is that for aligned single crystal alloys the primary dendrite spacing does not change as the KC-135 executes variable acceleration maneuvers.

## Publications

de Groh, H. C., and Laxmanan, V., "Macrosegregation in Undercooled Pb-Sn Eutectic Alloys," in *Solidification Processing of Eutectic Alloys* (D.M. Stefanescu, G.J. Abbaschian and R.J. Bayuzick, eds.), TMS, 1988, p. 229.

de Groh, H. C., "Macrosegregation and Nucleation in Undercooled Pb-Sn Alloys," Master Thesis, Case Western Reserve University, 1988, NASA TM 102023.

de Groh, H. C., and Probst, H. B., "Effects of Crucible Wetting During Solidification of Immiscible Pb-Zn Alloys," J. Spacecraft and Rockets **26**, 476-479 (1989); also published as a NASA TM 101372, 1988.

de Groh, H. C., "Metallurgical Programs," NASA Tech Brief, in press.

Tewari, S. N., Nesariker, V. V., and Lee, D., "Side Branch Morphology and Coarsening in Directionally Solidified Pb-8.4 at% Au," Met. Trans. A **20A**, 1889-1893 (1989).

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## The Development and Prevention of Channel Segregation During Alloy Solidification

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Dr. Angus Hellawell  
NAG3-560 (NASA Contact: H. deGroh, LeRC)  
July 15, 1986 - July 14, 1989

**Objectives:** The objective of the research is to identify the conditions under which channel segregation occurs during the solidification of alloys over a freezing range, and to make comparisons between materials having different physical properties, summarized by the Lewis and Prandtl numbers.

**Research Task Description:** During solidification of materials which have a significant freezing range, segregation channels can develop in the mushy zone of the partially solidified dendrite array. These channels extend approximately vertically as pencils of solute rich material and in alloy castings they can be regarded as serious defects. The cause of such channel formation is a density inversion between interdendritic and bulk liquid which is brought about when the solute(s) is less dense than the solvent component, e.g., tin in lead, carbon in iron, water in ammonium chloride, ethanol in succinonitrile. The incidence of channels depends upon the growth conditions, temperature gradient and growth rate, and upon the alloy composition which influences the permeability of the mushy zone. The onset of convection which leads to channel formation originates at or just ahead of the dendritic growth front and depends upon the thermal, solutal diffusivity ratio (Lewis number) and the ratio of the kinematic viscosity to thermal diffusivity (Prandtl number).

**Progress to Date:** A fully automated gradient furnace has been assembled with facility for digital recording of information from multiple thermocouples: this is being used to vary the interdendritic spacing which is thought to influence the onset of channel convection. In addition to the data which has been assembled for metallic (lead base) systems and the aqueous ammonium chloride system, observations of channel formation are being made in organic materials (succinonitrile-ethanol) and ionic salts (sodium fluoride - sodium chloride), these having different Lewis and Prandtl numbers.

The composition of the liquid in lead base channels is being measured by EMPA and in transparent solute plumes by direct sampling. The resulting data is providing estimates of the buoyancy forces which drive the convection and these are being related to the observed flow rates in transparent systems, allowing extrapolation to those operating in metallic systems.

### Publications

Sarazin, J. R. and Hellawell, A., "Channel Flow in Partly Solidified Alloys System," in *Proceedings of Symposium on Advances in Phase Transitions*, Pergamon Press, 1988, pp. 101-115.

Sarazin, J. R. and Hellawell, A., "Channel Formation in Pb-Sn, Pb-Sn and Pb-Sn-Sb and Comparison with the System  $\text{NH}_4\text{Cl-H}_2\text{O}$ ," *Met. Trans.* **19A**, 1861-1871 (1988).

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Sarazin, J. R. and Hellawell, A., "Fluid Flow in Partly Solidified Systems," *TMS Spring Meeting, Las Vegas, 1989.*

### Containerless Processing of Undercooled Melts

University of Wisconsin-Madison  
Professor John H. Perepezko  
Grant NAG8-771 (M. B. Robinson MSFC)  
March 1, 1989 - September 21, 1989

*Objectives:* The main objective of the research project is to evaluate the undercooling and resultant solidification microstructures in containerless drop tube processing of alloys as an experience base for microgravity experiments. The degree of liquid undercooling attainable in a laboratory scale (3m) drop tube can be altered through the variation of processing parameters such as melt superheat, droplet size and gas environment. In a given sample, the competitive nucleation and growth kinetics between equilibrium and metastable phases controls microstructural development. This solidification behavior is evaluated through metallography, thermal analysis and x-ray diffraction examination in conjunction with a heat flow model of the processing conditions.

*Research Task Description:* In drop tube processing, a quantitative analysis of the thermal history is required for sample evaluation. Direct thermal measurement is difficult, but thermal histories can be determined through alternate techniques. In previous work it has been established that measurements of the falling distance for solidification can be used to judge undercooling through a heat flow analysis. During the current program the development of a more direct in-situ temperature monitoring system is being pursued to allow for a complete analysis of thermal history.

*Progress To Date:* In the present work which has started recently, unique microstructures and microstructural transitions will be identified in selected Fe, Ni and Mn alloys during microgravity containerless processing. Based upon an understanding of microstructure development kinetics and processing a sample scale up is intended to be compatible with space station facilities. It is also planned to evaluate the influence of positioning fields and other disturbances on sample undercooling and solidification behavior.

### Publications

J.H. Perepezko and W.P. Allen, "Processing of Undercooled Melts", in *Proceedings of the Third International Colloquium on Drops and Bubbles*, ed. T. Wang (in press).

### Presentations

J.H. Perepezko, "Metastable Phase Formation in Undercooled Melts" 1989 Gordon Conference on Gravitational Effects in Materials and Processes.

### Role of Gravity on Macrosegregation in Alloys

University of Arizona  
Professor D. R. Poirier  
Professor C.F. Chen  
Professor J. C. Heinrich  
NAG3-723 (NASA Contact: A. Chait, LeRC)  
April 1986 - April 1989

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

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

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

Linear stability analyses and nonlinear calculations for a fully convecting system were done for a liquid layer above a stationary porous medium with constant porosity for both isotropic and anisotropic media. The model was also extended to treat alloy solidification more realistically by considering an all liquid zone above a mushy zone moving with a prescribed solidification velocity and with a variable volume fraction of liquid in the mushy zone that is consistent with solidification thermodynamics. Linear stability analyses were done in order to define marginal stability curves for the onset of convection in Pb-20 wt pct Sn alloys, in terms of thermal gradient versus the horizontal wave number at various gravitational constants. Calculations of the nonlinear convection were also done. These calculations verified the linear stability calculations and showed that, when there is convection, flow in the upper part of the mushy zone is driven by the convection in the all-liquid region.

Another accomplishment was the development of a code to treat steadystate directional solidification of a binary alloy in the form of a circular cylinder. The code can be used to examine the sensitivity of the interdendritic convection and macrosegregation to a slight curvature of the nominally horizontal isotherms in the mushy zone of a DS casting.



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- Yeum, K. and Poirier, D. R., "Modelling Directional Solidification of a Dendritic Alloy," *Cast Metals* **1**, 161-170 (1988).
- Chen, F. and Chen, C. F., "Convection in a Superposed Fluid and Porous Layer," *J. Fluid Mech.*, 1988 (submitted).
- Heinrich, J., Felicelli, S., Nandapurkar, P., and Poirier, D. R., "Thermosolutal Convection During Dendritic Solidification," *27th Aerospace Sciences Meeting*, January, 1989, Reno, Nevada, Paper no. AIAA 89-0626.
- Chen, F. and Chen, C. F., "Experimental Investigation of Convective Stability in a Superposed Fluid and Porous Layer when Heated from Below," *J. Fluid Mech.* **207**, 311-321 (1989).
- Heinrich, J. C. and Felicelli, S., "Finite Element Simulation of Thermosolutal Convection in Vertical Solidification of a Binary Alloy," *Proceedings of the 7th International Conference on Numerical Methods in Flow Problems* (T.J. Chung and G.R. Kerr, eds.), University of Alabama in Huntsville Press, 1989, pp. 596-603.
- Heinrich, J. C. and Felicelli, S., "Thermosolutal Convection during Dendritic Solidification of a Binary Alloy," *Finite Elements in Flow Problems* (T.J. Chung, ed.), Hemisphere Publishing Co., 1989 (in press).
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- Chen, C. F., "Double-Diffusive Convection and Its Effect Under Reduced Gravity," *Progress in Low-Gravity Fluid Dynamics and Transport Phenomena*, AIAA, Washington, D.C., 1990.
- Yeum, K. S., Speiser, R., and Poirier, D. R., "Estimation of the Surface Tensions of Binary Liquid Alloys," *Met. Trans. B*, 1989 (in press).
- Ganesan, S. and Poirier, D. R., "Solute Redistribution in Dendritic Solidification with Diffusion in the Solid," *J. Cryst. Growth*, 1989 (in press).
- Yeum, K. S., Laxmanan, V., and Poirier, D. R., "Efficient Estimation of Diffusion During Dendritic Solidification," *Met. Trans. A*, 1989 (in press).
- Ganesan, S. and Poirier, D. R., "Conservation of Mass and Momentum for the Flow of Interdendritic Liquid during Solidification," *Met. Trans. A* (in press).
- Poirier, D. R., "Segregation and Convection in Dendritic Alloys," *Progress in Low-Gravity Fluid Dynamics* (J. N. Koster and R. L. Sani, eds.), AIAA, 1990, to be published.

## **Presentations**

**Chen, C. F., "Convection at Reduced Gravity," *28th AIAA Aerospace Science Meeting*, Reno, Nevada, January 8-11, 1990.**

Thermosolutal Convection and Macrosegregation in Dendritic Alloys

University of Arizona  
Professor D. R. Poirier  
Professor J. C. Heinrich  
University of Illinois at Urbana-Champaign  
Professor A. J. Pearlstein  
NAG 3-1060 (NASA Contact: T. K. Glasgow, LeRC)  
July 18, 1989 - July 17, 1991

**Objectives:** The major objective of the program is to develop a computer model for studying thermosolutal convection and associated macrosegregation phenomena in dendritically solidifying alloys. As a possible means of controlling macrosegregation, the effect of rotation during solidification will be studied by means of linear stability analyses.

**Research Task Description:** Comparison between the calculated macrosegregation and experimental results of other NASA-sponsored investigators will be made. A major feature of the code will be to simulate the nonlinear convection that leads to the formation of a macrosegregation defect, called freckles. The calculations will include the effects of varying solidification rate, thermal gradient, and composition in Pb-Sn and Pb-Sb alloys. Time permitting, comparisons between calculated and experimental results in solidified  $\text{NH}_4\text{Cl-H}_2\text{O}$  solutions will also be made.

The effects of reducing the magnitude of  $g$  and changing its direction on thermosolutal convection and the attendant macrosegregation, will be studied by using our finite element code for the fully nonlinear convection problem. The goal of these calculations will be to assist experimentalists in planning work in the low- $g$  environment. The effect of rotation on convection and macrosegregation will be examined by means of linear stability analyses.

**Progress to Date:** For solidification of Pb-Sn alloys at low growth velocities, we have shown that the Coriolis acceleration associated with uniform rigid rotation leads to substantial increases (one order of magnitude) in the allowable bulk concentration of Sn below which plane-front solidification is stable with respect to thermosolutal convection. The stabilization is due to the Taylor-Proudman mechanism which suppresses long wavelength instabilities. Not surprisingly, the Coriolis acceleration does not affect the short wavelength instability associated with dendrite formation at higher growth velocities.

The finite element code (used for grant NAG3-723) is being modified so that it can be used to simulate unsteady dendritic solidification in a vertical mold. An important feature of the code will allow for the adjustment of the volume fraction of the interdendritic liquid, as convection occurs in the mushy zone.

Presentations

Oztek, A. and Pearlstein, A.J., "Coriolis Suppression of Convection in Unidirectional Solidification of a Binary Alloy," *APS/DFD Annual Meeting*, NASA, Ames Research Center, November 19-21, 1989.

Microgravity Solidification Processing of Monotectic Alloy Metal Matrix Composites

Massachusetts Institute of Technology  
Professor Kenneth C. Russell  
NAG8-084 (NASA Contact: P.A. Curreri, MSFC)  
December 12, 1986 - continuing

*Objectives:* The objectives of this research are to: (1) utilize the combination of the microgravity environment and the unique wetting characteristics of monotectic alloys to produce microstructures not obtainable elsewhere; (2) provide understanding of the basic principles governing the solidification of monotectic alloys; (3) conduct a theoretical and experimental study of monotectic alloy solidification in the constrained spaces between ceramic fibers and particles; and (4) understand the role of the gravity vector in the solidification of monotectic alloys.

*Research Task Description:* The proposed program was to proceed along both experimental and theoretical fronts. The program is designed as a coordinated, cooperative effort between MIT, NASA, Applied Research Labs, and United Technologies Research Center.

Experiments are to be conducted on several monotectic alloys, beginning with the Aluminum-Indium system. Solidification is to be both with and without reinforcing ceramic particulates or fibers. Initial experiments are to be conducted in normal gravity in a Bridgman-type furnace built specifically for this project. The furnace is designed so that the key solidification variables, thermal gradient and crystal growth rate, can be varied independently.

After obtaining baseline data at MIT, solidification experiments will be conducted under sub-orbital microgravity conditions. A mathematical modelling study is to be conducted in parallel with the experiments. Diffusion and fluid flow equations will be solved to predict the solidification morphology as a function of processing parameters. Theoretical results will be of great value in planning and interpreting experimental work.

*Progress to Date:* A unidirectional solidification apparatus, capable of attaining high temperature gradients and low growth rates, has been constructed. The entire apparatus can be rotated to study the effects of the direction of the growth vector relative to the gravity vector on the solidification behavior. Gradients as high as 400K/cm have been obtained.

Samples of monotectic composition have been directionally solidified at various temperature gradients and growth rates. This was done to check results of our experiments against those found in the literature as well as provide conditions for our modelling work that were not reported in the literature (i.e. extremely slow growth rates). The samples were found to obey the reported  $\lambda^2 R = \text{constant}$  relationship.

Composite samples with monotectic and hypermonotectic matrices, reinforced with SiC or Al<sub>2</sub>O<sub>3</sub> particulates have been formed. These samples were made by pressure infiltration of the molten alloys into a powder compact which had been packed to a uniform density. Al-In Al<sub>2</sub>O<sub>3</sub> fiber composites have also been produced. These samples were formed by pressure infiltration of the alloy into an aligned, fibrous compact that is contained within a closed bottom alumina tube of the appropriate diameter for the directional solidification. Directional solidification of the monotectic composition fibrous composites is currently being done.

Following the Jackson-Hunt model for eutectic growth, we have been able to predict, from material parameters, the constant in the  $\lambda^2 R = \text{Constant}$  relation. A value of  $C = 9.8 \times 10^{-17} \text{ m}^3/\text{sec}$  is predicted, which is in good agreement with the experimentally reported value of  $C = 4.5 \times 10^{-16} \text{ m}^3/\text{sec}$ . The radius of the  $L_2$  fiber tubes in the regular monotectic structure was also compared to the critical particle radius for entrapment in an Uhlmann, Chalmers and Jackson particle pushing analysis.

Preliminary modelling of the stability of the regular monotectic structure within the interfiber spaces, as a function of the fiber composition, fiber spacing, temperature gradient and growth rate have been done. Further modelling will be done after the completion of the experimental studies.

#### Publications

Frier, N., Shiohara, Y., and Russell, K.C., "Solidification Processing of Monotectic Alloy Metal Matrix Composites", in *Proceedings of 1988 Materials Research Society Symposium on Materials Processing*, 1989 (in press).

#### Presentations

Frier, N., Shiohara, Y., and Russell, K.C., "Solidification Processing of a Monotectic Alloy Matrix Composite", *Fall TMS-AIME Meeting*, Chicago, September 1988.

Russell, K.C., Frier, N., and Shiohara, Y., "Overview of Monotectic Solidification and Related Microgravity Efforts," *Symposium on Materials Processing in Space, ASM World Conference*, Chicago, September 1988.

Russell, K. C., Frier, N. L., Mortensen, A., and Curreri, P. A., Directional Solidification of Fiber Reinforced Composites, *Fall TMS-AIME Meeting*, Indianapolis, October, 1989.

### Containerless High Temperature Property Measurements

Interionics Incorporated  
Dr. Robert Schiffman  
NAS8-37472 (NASA Contact: M. Robinson, MSFC)  
September 1988 - September 1989

*Objectives:* The objective of this research is to examine advanced containerless processing and materials research at high temperatures on earth and in space. In this way, the production of very pure and high quality forms of important ceramic, superconducting, semiconducting, very hard, very strong and other useful kinds of materials may be achieved.

This research program is directed to the measurement of properties such as emissivity, temperature, vapor pressure, formation enthalpies, melting temperatures, etc., at high temperatures for technologically and scientifically interesting materials and to the development of advanced new measurement techniques.

*Research Task Description:* New techniques adaptable to in-space processing have been developed in earth-based research and the limits of earth-based containerless experiments are advanced and defined and new techniques for microgravity environments developed so that good choices for in-space R&D can be made. Noncontact measurement and containerless levitation methods are used including gas jet and electromagnetic levitation. The latter were used in combination with CW-CO<sub>2</sub> laser or EM heating and laser induced fluorescence or mass spectrometric measurements of vapor and ambient gas concentrations. Non-contact temperature measurement is achieved by optical pyrometry. A new absolute method for liquid specimen emittance measurement has been defined and tested and development will continue.

*Progress to Date:* Emissivity, temperature and vapor pressure measurements were obtained on solid boron and the enthalpy of boron vaporization was derived from the vapor pressure vs temperature results. Gas jet levitation was used in combination with laser heating to achieve specimen temperatures up to the melting point. Laser reflectance polarimetry was used to measure the spectral emittance of boron from 1020 to 1960R. Emitted light polarimetry was used to measure boron emissivities at 0.63 and 0.65  $\mu$  m from 1820K to the melting point. Laser-induced fluorescence of atomic boron was used to measure equilibrium vapor concentrations at temperatures from 1950K to the melting point. Results include a melting temperature for pure boron equal to 2335  $\pm$  9K and a vaporization enthalpy of boron equal to 562.5  $\pm$  3.7 kJ/mol at 298K.

Electromagnetic (EM) levitation of liquid silicon was achieved in combination with CO<sub>2</sub> laser heating. This allowed the solid, which is a semiconductor, to be heated and melted to form the "metallic" liquid, for which EM levitation was possible. Spectral emittance and optical property measurements were obtained on solid and liquid silicon at a wavelength of 0.63  $\mu$  m and temperatures from 1160-1390K and 1620-1910K, respectively. These results include measurements on supercooled liquid silicon at temperatures below the 1688K melting point.

Optical properties were also measured for EM-levitated liquid aluminum at 0.63  $\mu$  m and temperatures from 1200 to 1860K. Emissivity values were measured at 1800K for wavelengths from 0.40 to 0.65  $\mu$  m.

The laser reflection polarimetric technique was applied to measure optical properties and spectral emissivities of refractory transition metals and carbides. These included measurements at 632.8 and 1064 nm. on C (graphite), W, Ta, WC and TaC from 1200 - 2700R. Polarimetric measurements for studies on thin films have been carried out on Si-C and Si-C-W films used for x-ray optics.

Based on the emitted light polarimetry results, the next generation device to measure spectral emissivity from the degree of polarization of emitted light was designed and is under construction. This device will allow absolute temperature measurements on liquids of unknown or variable emissivity during the second and third year of this program. Work has been initiated on Ti, Ta, W, C, and metal-carbon systems, as well as metal-silicon alloys.

*Future Plans:* Construction of an advanced apparatus for determining emissivity from the degree of polarization of emitted light. Measure emissivity vs temperature for EM levitated liquids, including titanium, Ti-Si alloys and other metals such as Ta, W, Zr, U, Ni, and Mo. Establish performance of the emissivity measurement apparatus by melting temperatures on Ni, Mo, Si, Ti, etc.

Carry out LIF intensity vs temperature measurements on Ti-Si alloys as well as pure titanium and silicon. In this work, the component activities for the alloys will be calculated from the LIF intensity ratios for the alloy and pure components at temperatures where the liquid is in equilibrium with solid Ti-Si phases. The thermodynamic properties of the solid phases will then be calculated from the activity measurements over the liquid.

Investigate the optical properties and spectral emissivities of supercooled metals and alloys, such as Nb, Ni and Zr metals and related alloys. This work will be carried out in collaboration with The Center for Space Processing at Vanderbilt and MSFC.

Analyze results to determine limitations and opportunities for earth-based research on alloy systems and pure liquid metals.

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P. C. Nordine and R. A. Schiffman, "Direct Emissivity Measurements on liquids and Corrections to Multi-Color Pyrometers," NASA Conf. Publ. 2503, 1988, pp 173-81.

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S. Krishnan, R. A. Schiffman, P. C. Nordine, and J.K.R. Weber, "Temperature Measurement in Containerless Experiments," *Symposium on Space Commercialization: Roles of Developing Countries*, Nashville, TN, March 5-10, 1989.

#### Presentations

S. Krishnan, "Optical Properties of Liquid Metals," presented at LaRC, Hampton, VA, April 5, 1989.

P. C. Nordine, "Containerless Laser-Induced Fluorescence Study of Boron Vaporization," presented at the *IUPAC Sixth International Conference on High Temperatures-Chemistry of Inorganic Materials*, Gaithersburg, MD April 3-7, 1989.

Nordine, R. A. Schiffman, J. K. R. Weber, and S. Krishnan, "The Vapor Pressure and Spectral Emissivity of Boron," presented at the *IUPAC Sixth International Conference on High Temperatures-Chemistry of Inorganic Materials*, Gaithersburg, MD April 3-7, 1989.

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R. A. Schiffman, P. C. Nordine, S. Krishnan, and J.K.R. Weber, "Containerless High Temperature Measurements," *MSFC Ground-Based Review*, Huntsville, August 29-30, 1989.



Theoretical Studies of the Surface Properties of Liquid Metals and Semiconductors

Professor David G. Stroud  
Dr. Zhiqiang Wang  
Ohio State University  
NAG3-999 (NASA Contact: R.A. Wilkinson, LeRC)  
March 1989 - March 1990  
September 21, 1989

*Objectives:* The principal objective of this research is to calculate the surface tension and other thermophysical properties of a number of liquid semiconductors and liquid metals as functions of temperature and impurity composition. Knowledge of these quantities will permit a better understanding of the forces underlying convection in a low-gravity environment of space, as well as of many other material parameters necessary to model the fluid dynamics of crystal growth in a low-gravity environment.

*Research Task Description:* Our initial goal has been to calculate the surface tension of liquid CdTe as function of temperature and concentration, using empirical two and three-body potentials fitted to the bulk cohesive energy lattice constant and alloy heat of formation. The method involves direct Monte Carlo evaluation of the work required to create the surface. Calculations are being carried out on the CRAY Y-MP 8/64 of the Ohio Supercomputer Center. In order to take account of the metal-insulator transition in CdTe as a function of stoichiometry, we will add a concentration dependence to the potentials.

Additional planned work includes the following: (i) calculation of bulk properties of liquid compound semiconductors, including viscosity and thermal conductivity, possibly by molecular dynamics techniques; (ii) extension of the calculation to other compound semiconductors, such as  $Ga_xAs_{1-x}$ , and to pseudobinary semiconductors, such as  $Hg_xCd_{1-x}Te$ . Later planned work includes calculations of the liquid-solid surface tensions of elemental and compound semiconductors and metals, and theoretical studies of the properties of high-temperature liquid metals, especially thermal emissivity, which is an essential parameter in interpreting contactless temperature measurements.

*Progress to Date:* We have successfully calculated the liquid vapor surface tension and surface entropy of stoichiometric  $Cd_xTe_{1-x}$  in quite good agreement with experiment, following earlier calculations for liquid Si. Calculations on nonstoichiometric  $Cd_xTe_{1-x}$  using the potentials developed for the stoichiometric case, predict a reversal of slope in the variation of the surface tension with composition near  $x = 0.5$ . Initial work on a density-functional theory to calculate the liquid-solid interfacial tension gives good results for bcc metals and promising results for tetrahedrally bonded semiconductors.

Publications

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X. C. Zeng and D. Stroud, "Ginzburg-Landau Theory for the Solid-Liquid Interfacial Tension of BCC Elements. II. Application to the Classical One-Component Plasma, the Wigner Crystal, and  $^4\text{He}$ ," Phys. Rev. A **39**, 4761 (1989).

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

Cleveland State University  
Dr. S.N. Tewari  
Dr.A. Chopra  
NCC-395 (NASA Contact: T.K. Glasgow, LeRC)  
September 1987- September 1989

**Objectives:** The objective of this research was to study the development of cellular/dendritic microstructures during directional solidification of binary metallic model alloys in a positive thermal gradient.

**Research Task Description:** Experimental facilities, such as vacuum/controlled atmosphere casting apparatus to prepare cylindrical specimens with uniform composition, directional solidification furnace for controlled rate directional solidification and rapid quenching of metallic alloys and transparent directional solidification furnace for visual observation of the liquid-solid interface were designed and fabricated as part of this research in the Microgravity Materials Science Laboratory at NASA-Lewis Research Center.

Directionally solidified Pb-8 at pct Au and Pb- 10 wt pct Sn have been examined. Cell/dendrite tip morphology and solutal profile in the melt at the tip have been measured in Pb-Au alloy. Distortion of the macro liquid-solid interface caused by convection in the melt results in a large scatter in the tip radii and tip compositions. Dendrite growth models are therefore not able to predict the tip radii and tip compositions individually. However, the relationship among the destabilizing solutal gradient, stabilizing thermal gradient and stabilizing capillarity at the tip assumed by the "marginal stability criterion" is supported by the experimental data. Previously reported observation in Al-Cu alloy, where with decreasing growth speed a minimum in primary arm spacings was observed in the cellular growth regime, is not confirmed by Pb-10 wt pct Sn alloy.

### Publications

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S.N. Tewari, V.V. Nesariker, and D. Lee, "Side Branch Morphology and Coarsening in Directionally Solidified Pb-8.4 at% Au", Met. Trans. **20A**, 1989 (in press).

## Containerless Studies of Nucleation and Undercooling

Jet Propulsion Laboratory  
Dr. Eugene H. Trinh  
January 1989-January 1990

**Objectives:** The long term research objectives are experimental and theoretical studies to determine the achievable limits of undercooling using acoustic and other means of sample levitation and manipulation, to study the characteristics of heterogeneous nucleation using levitated materials in 1 G and in microgravity, to measure the physical properties of significantly undercooled melts, and to determine the characteristics of solidification under conditions allowed by both containerless processing and low gravity.

**Research Task Description:** Ground-based experiments and investigations aboard the NASA KC-135 aircraft are carried out to levitate, melt, undercool, and solidify 0.5 to 3 mm specimens of low-melting pure metals and alloys (Ga, In, Sn, Al, Al-Cu alloys), as well as inorganic compounds (O-Terphenyl, Succinonitrile), and low melting glasses. Non-perturbing measurement techniques for the surface tension, viscosity, density, sound velocity, and optical refraction index are to be used and refined to probe the physical state of undercooled levitated melts. New methods for the measurement of the specific heat and thermal diffusivity are currently being investigated. The quantitative evaluation of the effects of external physical stimuli on the nucleation onset is also being carried out to rigorously document the advantages of experimentation in microgravity. Current investigations revolve around controlling the processing environment in ultrasonic levitators, increasing the undercooling and cooling rate of samples in 1 G through atomization, and refining the non-contact measurement techniques for the thermophysical properties of levitated undercooled melts.

**Progress to Date:** An experimental determination of the level of undercooling achievable in 1 G using ultrasonic levitators has revealed the capability for undercooling between 10 to 20% of the melting point temperatures for a variety of low-melting metals and inorganic compounds. The measurement of the surface tension, viscosity, and density of undercooled levitated samples has been carried out. A quantitative characterization of the processing environment of ultrasonic levitators in 1 G and in the KC-135 has been obtained, and preliminary results of the effects of external stimuli on the onset of nucleation have been obtained.

## Publications

Trinh, E. H. "Levitation Studies of Physical Properties and of the Nucleation of Undercooled Liquids," in *Proceedings of the VIIth European Symposium on Materials and Fluid Sciences in Microgravity*, ESA, 1989 (in press).

Trinh, E. H. and Perepezko, J. H. "Fluid Dynamics and Solidification of Drops and Shells," in *Progress in Low Gravity Fluid dynamics and Transport Phenomena* (J. Koster, ed.), 1989 (in press).

Ohsaka, K. and Trinh, E. H. "Melting and Solidification of Acoustically Levitated Drops," *J. Cryst. Growth*, 1989 (in press).

Seshan, P. K. and Trinh, E. H. "Contamination Concerns in the Modular Containerless Processing Facility," *27th AIAA Aerospace Meeting*, AIAA paper 89-0403, 1989.

Trinh, E. H., Gaspar, M., and Leung, E. "Optical Measurement of Sound Pressure", New Technology Report NP0-17565, 1989.

### Influence of Convection on Microstructure

Clarkson University  
Dr. William R. Wilcox  
NAG8-753 (NASA Contact: F. Szofran, MFSC)  
August 1988 - August 1991

*Objective:* To gain an understanding of the influence of microgravity on the microstructure of eutectics especially MnBi-Bi.

*Research Task Descriptions:* David Larson and Ron Pirich of Grumman have shown that directional solidification of the MnBi-Bi eutectic in space or with a magnetic field applied results in a fiber spacing  $1/2$  of that obtained by solidification on earth. We had shown previously that the microstructure is unaffected by temperature gradient, that the microstructure responds more quickly to a change in freezing rate than the freezing rate changes in response to a change in ampoule translation rate, that insertion of convection into the Hunt and Jackson model of lamellar and fibrous eutectics predicts that normal buoyancy-driven convection on earth is insufficient to account for the two-fold change in fiber spacing observed, that the fibers tend to grow very straight with very little branching, that the fibers project out into the melt an average of one diameter during solidification, and that temperature oscillations are common in the melt.

During our current grant period we plan to do the following:

1. Perform theoretical work for the cooperative solidification of MnBi-Bi two phase mixtures, as follows:
  - a. Influence of the Soret effect on the average composition vs distance of off-eutectic mixtures in the absence of convection.
  - b. Influence of convection on the microstructure of off-eutectic mixtures using a linear velocity gradient in the melt.
  - c. Influence of volumetric change of solidification on microstructure.
  - d. Influence of convection on microstructure when the fibers project out in front of the matrix
2. Tabulate the physical properties of all eutectics solidified in space to see if point the way to an explanation of the different effects of microgravity on microstructure; volume fraction eutectic, eutectic temperature, densities of the pure constituents, density changes upon solidification, etc.
3. Measure the Soret coefficient and the diffusion coefficient for MnBi-Bi melts near the eutectic composition.

*Progress to Date:* After a one year hiatus in funding, this project was re-instated in the fall of 1988 with two new M.S. students. One student dropped out at the end of the semester. A post-doc paid by the Brazilian government began in February of 1989. We hope to attract a new graduate student in the fall of 1989.

We designed, constructed and debugged an apparatus for measuring the Soret coefficient and the diffusion coefficient in Mn-Bi mixtures. Techniques were developed for preparing suitable small diameter ampoules containing eutectic mixtures of Mn and Bi. We are developing methods for analyzing composition versus distance.

The equations for Soret effect combined with diffusion have been written down and solved numerically on the computer. This will enable us to interpret our experimental data, derive the ratio of

Soret coefficient to diffusion coefficient from the data from Dr. Larson's last flight experiment, and predict the result of experiments with a temperature gradient in the absence of convection.

Results for all flight experiments on eutectic solidification are being tabulated, along with the physicochemical properties of the systems.

We are preparing to perform the theoretical work described in the work statement above.

#### Publications

M. F. Larrousse and W. R. Wilcox, "Interfacial Mass Transfer to a Cylinder Endwall during Spin-up/Spin-down," Chem. Eng. Sci. (in press).

### Modelling Directional Solidification

Clarkson University  
Dr. William R. Wilcox  
NAG8-541 (NASA Contact: F. Szofran, MSFC)  
September 1985 to July 1989  
Renewal expected October 1989

*Objective:* To develop an improved understanding of some phenomena of importance to directional solidification, to enable us to explain and predict differences in behavior between solidification on earth and solidification in space.

*Research Task Description:* The following tasks were performed under this grant.

1. Development of a computer simulation for radial segregation with arbitrary interface shape in the absence of convection.
2. Determination of cross-sectional and axial impurity segregation as a function of furnace temperature profile, convection in the melt, interface shape and freezing rate using a transparent organic compound.
3. Development of a method for measuring the heat transfer coefficient between the furnace wall and a conductive material contained in an insulating ampoule.
4. Developed theory for diffusional decay of striations.
5. Investigated the influence of current pulses, spin-up/spin-down, interface shape and vibrations on the compositional homogeneity and microstructure of InSb-GaSb alloy semiconductors.

*Progress to Date:* Our experiments on organic compounds showed that in contrast to computer models the convection in a vertical Bridgman-Stockbarger ampoule is usually not axisymmetric and may vary with time. If the temperature in the furnace increases with height, the convection may be greatly suppressed. On the other hand, if the temperature decreases with height, the convection may be vigorous. Theoretical models have built into their initial equations steady state, axisymmetric flow, and a constant heater temperature. Experiments were performed to determine the influence of convection, freezing rate, interface shape and furnace temperature profile on impurity doping homogeneity of directionally solidified organic compounds. It was found, for example, that even when the convection is too small to be observed by particle movement in the melt, the convection may be sufficient to cause large cross-sectional variations in impurity doping when the freezing rate is low.

A computer model was developed for influence of interface shape on the radial variation in doping. It was found that the influence of interface shape depends strongly on the freezing rate.

Computer and analytical models were developed for the diffusional decay of compositional or doping striations during solidification. Striations can disappear altogether at low freezing rate, closely spaced striations, and high diffusion coefficient in the solid. Thus the absence of striations cannot be taken as conclusive evidence of an freezing rate or convection during crystal growth. This may explain why, for example, striations were not reported in HgTe-CdTe alloys solidified with spin-up/spin-down accelerated crucible rotation technique).

Apparatus and procedures were developed to determine the influence of spin-up/spin-down, freezing rate fluctuations, interface shape et cetera on the homogeneity and crystallographic perfection of directionally solidified InSb-GaSb alloys. (Mullard laboratories in Southampton, England have

shown both increased homogeneity and much larger grain size caused by spin-up/spin-down during solidification of HgTe-CdTe alloys. The mechanism responsible for increased grain size is unknown.) Experiments are underway.

In the last two decades many models have been developed for heat transfer in the Bridgman-Stockbarger technique. Many of these models utilize the heat transfer coefficient between the material and the furnace wall. However the value of this heat transfer coefficient is unknown, and has been estimated only occasionally from heat transfer theory. We have developed a technique for measurement of the heat transfer coefficient. The method is valid when the furnace temperature is reasonably isothermal over several ampoule diameters and the material has a much higher thermal conductivity than the ampoule. The rate of change of temperature in the material is measured following a sudden change in thermal environment, as caused, for example, by moving the charge to a different part of the furnace with a different temperature, or by the sudden cessation of passing an environment should not be such as the cause a phase change, i.e. the material should remain all solid or all melt. The time constant of the exponential temperature change in the material is directly related to the heat transfer coefficient.

#### Publications

G. T. Neugebauer and W. R. Wilcox, "Convection in the Vertical Bridgman-Stockbarger Technique," *J. Crystal Growth* 92, 143-154 (1988).

R. T. Gray, M. J. Larrousse and W. R. Wilcox, "Diffusional Decay of Striations," *J. Crystal Growth* 92, 530-542 (1988).



### **3. FLUIDS, INTERFACES, AND TRANSPORT**



### Thermo-Diffuso Capillary Phenomena

NASA Lewis Research Center  
Dr. R. Balasubramaniam  
Dr. L. H. Dill  
In-House

*Objectives:* The main objective of this program is to understand the motions of bubbles and droplets due to thermocapillary effects (Marangoni flow) in a reduced gravity environment.

*Research Task Description:* Analytical and numerical studies are being performed to predict the terminal and transient velocities of immiscible bubbles and droplets in a host fluid possessing a uniform temperature gradient. The terminal velocity of a bubble has been calculated including the effects of inertia and convection, i.e., for non-negligible values of the Reynolds and Marangoni numbers. The steady thermocapillary migration of a gas bubble in the presence of an insoluble surfactant is also being analyzed.

Experiments are also being conducted using an immiscible density matched liquid-liquid system to obtain migration data for droplets and also visualize the thermocapillary flow that is induced within the droplet.

*Progress to Date:* The research performed in this area has resulted in several publications/presentations that are cited below. Analytic studies have been completed to predict (i) the terminal velocity and small deformations of a droplet for small Ma and arbitrary Re, (ii) the migration velocity of a large gas slug in a heated tube, and (iii) the transient migration velocity and the time taken to reach steady state for bubbles and droplets when Re and Ma have negligibly small values. Numerical studies have been completed predicting steady velocities for a spherical bubble up to Re and Ma of a thousand. The finite difference code that was developed has been modified to account for surfactant effects. Preliminary results suggest that surfactants can substantially reduce thermocapillary migration rates. Migration data and flow visualization using particle tracking in a laser light sheet have also been obtained using a vegetable oil-silicone oil density matched system.

#### Publications

Hasan, M.M., and Balasubramaniam, R., "Thermocapillary Migration of a Large Gas Slug in a Tube," J. Thermophys. & Heat Transf. **3**, 87-89 (1989).

Balasubramaniam, R., and Lavery, J. E., "Numerical Simulation of Thermocapillary Bubble Migration Under Microgravity for Large Reynolds and Marangoni Numbers," Num. Heat Transf., in press.

Dill, L. H. and Balasubramaniam, R., "Unsteady Thermocapillary Migration of Bubbles," in *Proceedings of Third International Colloquium on Drops and Bubbles*, Monterey, CA, September 1988.

#### Presentations

Dill, L. H., and Balasubramaniam, R., "Unsteady Thermocapillary Migration of Droplets," *AIChE Annual Meeting*, Washington, D.C., Nov.-Dec. 1988.

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Rashidnia, N., and Balsubramaniam, R., "Thermocapillary Flow in Droplets in a Vertical Temperature Gradient Using a Density-Matched Liquid-Liquid System," *7th International PCH Conference*, MIT, Cambridge, MA, June 1989.

Dill, L. H., "Thermocapillary Migration of a Gas Bubble: Simulation of Surfactant Effects at Finite Reynolds and Marangoni Numbers," *7th International PCH Conference*, MIT, Cambridge, MA, June 1989.

## Experimental and Theoretical Studies of Wetting and Multilayer Adsorption

National Institute of Standards and Technology

Dr. J. W. Cahn

Dr. M. R. Moldover

Dr. J.W. Schmidt

W-16, 170

*Objectives:* The objective of this work is to understand the important role that interfaces between phases of matter play in low gravity conditions. These results have important consequences for materials processing techniques including containerless processing in space.

*Research Task. Description:* The research involves two specific areas. These are (a) Measurements on interfaces between phases of matter with an attempt to uncover the universal character of the underlying structure of these interfaces and (b) measurements of much needed properties of specific materials such as alternative refrigerants. Research techniques include high precision ellipsometry, refractometry, and low thermal gradient thermostating important for materials processing.

*Progress to Date:* The following results apply to both ground based and low-gravity environments,

### (1) Structure of Fluid Interfaces

Ellipticity measurements from liquid-liquid interfaces in polymer + solvent mixtures were interpreted via a combined capillary-gravity wave and mean-field model. Previous measurements of 5 simple binary mixtures were scaled according to this model to a universal constant  $\chi (= \bar{\rho} / \Delta n \xi)$  where  $\bar{\rho}$  is the ellipticity,  $\Delta n$  is the refractive index difference between the two liquid phases and  $\xi$  is the correlation length. The measurements have been extended to include another class of fluid mixtures, namely, high molecular weight (MW=96,400-355,000) polystyrene + methylcyclohexane systems. Results indicate that  $\chi$  for these systems is the same as  $\chi$  for the simple low molecular weight binary mixtures. These results make clear the universal behavior of broad classes of materials under a variety of conditions and should apply in low g environments as well.

### (2) Adsorption at the Vapor-Liquid Interface

The ellipticity of the vapor-liquid interface in the mixture MCH + pFMCH was measured in the vicinity of the consolute point. An excellent fit to the data has been obtained using a model with one free parameter that incorporates a physically realistic dielectric constant profile of this interface. These results show the correspondence between simple low molecular weight and high molecular weight mixtures.

### (3) High Pressure, Ellipsometry

A miniature ellipsometer has been designed and is presently being fabricated. The ellipsometer will be placed in a high pressure cell containing SF<sub>6</sub>. This arrangement will remove the effects of cell window strain from measurements of ellipticity taken in a pressurized environment. When development is completed the ellipsometer will be used to further test the capillary-gravity wave theory for liquid interfaces in which density rather than composition is the order parameter. Previous earth based results have been difficult to interpret because of pressure-induced window strain.

### (4) Surface Tension in New Refrigerants

New refrigerants that are potentially environmentally acceptable are being investigated. These replacement refrigerants will be used as the nation phases out presently used refrigerants. We, have developed the capability to accurately measure capillary rise in the refrigerants via differential technique

from ambient temperature to their critical points. Five refrigerants R123a, R134, R141b, R14b and R152a were measured. In situ refractive indices were also measured over the same temperature range. The refractive indices were combined with the density measurements of Morrison (NIST) at lower temperatures and used to obtain densities and surface tensions up to the critical points for these refrigerants.

In addition to the above measurements, light scattering techniques are being developed to measure interfacial tension in refrigerant + oil mixtures. Good agreement was obtained between our light scattering measurements and published values for 4 pure fluids including ethanol R123, SF<sub>6</sub> and pFMCH.

#### Publications

H. B. Chae, J. W. Schmidt, and M. R. Moldover, "Surface Tension of Refrigerants R123 and R134a," J. Chem. Eng. Data, in press.

M. O. McLinden, J. S. Ballagher, L. A. Weber, G. Morrison, D. Ward, A.R.H. Goodwin, M. R. Moldover, J. W. Schmidt, H. B. Chae, T. Bruno, J. F. Ely, and M. L. Huber, "Measurement and R134a and R123," ASHRAE Trans., in press.

J. W. Schmidt, "Adsorption at the Vapor-Liquid Interface Near the Consolute Point," Phys. Rev. A, in press.

D. G. Miles and J. W. Schmidt, "Structure of the Polymer Solvent Interface," J. Chem. Phys., in press.

H. B. Chae, M. R. Moldover, and J. W. Schmidt, "Five Alternative Refrigerants Capillary Rise, Critical Points, Refractive Indices, Coexisting Densities, and Surface Tensions", (in preparation).

### Interfacial Phenomena

NASA Lewis Research Center  
Dr. A. T. Chai  
Mr. J. B. McQuillen  
Mr. M. Weislogel  
In-House

#### *Contact Angle Measurements - A. Chai*

The objective of this research is to experimentally determine the feasibility of utilizing the constant curvature of liquid-vapor interface encountered at low Bond number (i.e., low gravity) conditions to provide accurate measurements of contact angles. Special sample container and rig for use in the 5.5 sec. Zero Gravity Facility have been designed and fabricated. Initial testing was only partially successful. A revised design of the original sample container has been planned. Concurrently different methods and approaches are being evaluated.

#### *Two Phase Flow Through Fittings - J. McQuillen*

A precursor study is being performed on two phase flow in reduced gravity to examine the effects of changes in conduit geometry (tubing diameter and axial direction) on two phase flow behavior such as flow patterns, mixing and pressure drop. Preliminary tests were conducted on the Learjet using a 0.95cm to a 0.62cm ID contraction and a 0.95cm to a 2.54cm ID expansion. Results are being analyzed and further testing and measurements are planned.

#### *Capillary Phenomena - M. Weislogel*

This research encompasses several tasks. (1) Surface reorientation and settling with step reduction in gravity, where drop tower data on the transients (settling time and frequency) of a vessel/fluid pair are correlated with the characteristic parameters of the system. (2) Low gravity surface tension measurement, where a conceptual technique is being explored employing a differential capillary tube to accurately measure the surface tension of a liquid/gas pair in a low-g environment. The device is under fabrication and is targeted to fly aboard the Learjet in early fall. (3) Visualization of flow near the contact line (3 phase line). This qualitative study will provide guidance for analytical investigations. (4) Surface uniqueness experiment. By selection of an irregular vessel geometry/liquid pair, a degree of insight into the applicability of the existing theory of static surface configurations (in low-g) may be obtained. One such vessel has been machined and is currently under testing at the Zero-g facility.

### Publications

Chai, A. T., "Accurate Contact Angle Measurements," *ALAA 27th Aerospace Sciences Meeting*, January 1989, Reno, Nevada.

Weislogel, M., "Surface Reorientation and Settling in a Right Circular Cylinder with Step Reduction in Gravity," in preparation.

## Convective and Morphological Stability During Directional Solidification

National Institute of Standards and Technology

Dr. S. R. Coriell

Dr. G. B. McFadden

Dr. J. R. Manning

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

*Research Task Description:* The main focus of this task is the interaction of fluid flow in the melt with the crystal-melt interface, and the solute segregation which is controlled by interface morphology and fluid flow. In the absence of fluid flow, the conditions for morphological instability during directional solidification of an alloy at constant velocity are well established. However, for processing conditions for which instability occurs, the resulting non-planar interface morphologies (cellular or dendritic) are an active area of current research. Although progress has been made in calculating cellular morphologies, the question of wavelength selection is unresolved, and definitive experiments (without convection) are needed to provide guidance and a bench mark for theoretical developments. Numerical algorithms to calculate cellular morphologies in the absence of convection are being developed. Linear stability analyses of the effect of various types of fluid flow on the onset of morphological instability are carried out in order to delineate the role of convection. Fluid flow in the melt is calculated by a time-dependent, two-dimensional finite difference algorithm as a function of the gravitational acceleration. This allows prediction of the differences between experimental results obtained on earth and in space. The effect of time-dependent gravitational accelerations (g-jitter) is studied both by linear stability analyses and numerical solution of the nonlinear fluid flow equations. Since the MEPHISTO furnace uses electrical pulsing and Seebeck measurements, research on the effect of electrical currents on the solidification process and solute redistribution is also underway.

*Progress to Date:* The onset of morphological instability during the directional solidification of a single-phase binary alloy at constant velocity vertically upwards is treated by a linear stability analysis. For the case in which a heavier solute is rejected at the solidifying interface, the effect of natural convection on the critical concentration for the onset of instability is calculated. For tin containing lead we find a small destabilization of the system at low growth velocities, and a large increase in the wavelength of the instability at the onset. Calculations show that the destabilization is enhanced as the variation of density with solute concentration is reduced, and in the limit of neutrally-dense solute, there is a long wavelength instability for which the critical solute concentration is several orders of magnitude lower than that predicted by the Mullins and Sekerka analysis in the absence of convection. The destabilization is very sensitive to the ratio of crystal and melt thermal conductivities.

In collaboration with L. N. Brush, we have developed a numerical method to study the free boundary problem for the motion of a planar crystal-melt interface during the directional solidification of a binary alloy in the presence of a time-dependent electric current. The model includes the Thomson effect, the Peltier effect, Joule heating, and electromigration of solute in the coupled set of equations governing heat flow in the crystal and melt, and solute diffusion in the melt. For a variety of time-dependent currents, the temperature fields and interface velocity are calculated as functions of time for the pure materials, indium antimonide and bismuth, and for the binary alloys, germanium-gallium and tin-bismuth. For the alloys, we also calculate the solid composition as a function of position, and make quantitative predictions of the effect of an electrical pulse on the solute distribution in the solidified material.



In collaboration with M. E. Glicksman and M. E. Selleck, a linear stability analysis has been carried out for the axisymmetric Taylor-Couette instability of the flow between infinite coaxial rotating cylinders for the case that one of the bounding cylinders is a crystal-melt interface. Results have been obtained for various rotation ratios of the outer and inner cylinders in the narrow gap approximation. The Taylor-vortex mode is destabilized for large Prandtl numbers, and the critical Taylor number varies inversely with the Prandtl number. In the limiting case of small Prandtl number the results tend to classical values for a rigid-walled system.

Calculations on the effect of time-dependent gravitational accelerations on solutal convection during directional solidification, and on the accompanying solute segregation, have continued. In collaboration with B. T. Murray, a linear stability analysis for the onset of solutal convection is being carried out for the case of a time-dependent gravitational acceleration aligned with the growth direction. For conditions of instability, the nonlinear states are calculated by finite differences.

#### Publications

Coriell, S. R., McFadden, G. B., and Sekerka, R. F., "Convective and Interfacial Instabilities during Solidification, *Physicochemical Hydrodynamics* (M. G. Velarde, ed.), Plenum, 1988, pp. 559-569.

Coriell, S. R., McFadden, G. B., Wheeler, A. A., and Hurle, D. T. J., "The Effect of an Electric Field on the Morphological Stability of the Crystal-Melt Interface of a Binary Alloy II. Joule Heating and Thermoelectric Effects, *J. Cryst. Growth* 94, 334-346 (1989).

Coriell, S. R. and McFadden, G. B., "Buoyancy Effects on Morphological Instability during Directional Solidification," *J. Cryst. Growth* 94, 513-521 (1989).

McFadden, G. B., Coriell, S. R., Glicksman, M. E., and Selleck, M. E., "Instability of a Taylor-Couette Flow Interacting with a Crystal-Melt Interface," *PCH Physicochem. Hydrodyn.* 11, 387 (1989).

Modeling of Coalescence, Agglomeration and Phase Segregation in Microgravity Processing of Bimetallic Composite Materials

University of Colorado at Boulder

Dr. Robert H. Davis

NAG3-993 (NASA Contact: R. Balasubramanian, LeRC)

February 2, 1989-February 1, 1990

*Objectives:* The primary objective of this research is to develop models to predict drop-size distribution evolutions due to droplet collisions and coalescence during processing within the miscibility gap of bimetallic liquid-phase-miscibility-gap materials. The individual and collective action of gravitational and nongravitational mechanisms on the relative motion and coalescence of drops are considered.

*Research Task Description:* When bimetallic liquid-phase-miscibility-gap materials, which are thought to have a variety of desirable properties, are cooled through the miscibility gap, droplets rich in one of the metals form in the liquid matrix rich in the other metal. Droplet coalescence and phase segregation then occur due to buoyancy and to thermocapillary and other nongravitational mechanisms. In order to gain a predictive understanding of these phenomena, population dynamics models are used to follow drop-size distribution evolutions in time as the droplets grow due to collisions and coalescence. Continuous drop size distributions are discretized into a large number of categories. Drops of a given mass are destroyed by coalescing with other drops and are formed by the coalescence of smaller drops. The population dynamics model tracks the formation and destruction of drops in each size category.

The relative motion of drops which gives rise to their collision and coalescence is considered to occur by gravity sedimentation, Marangoni migration, Brownian motion, and bulk flow. These collision mechanisms are considered either individually or collectively in the population dynamics models. In general, different mechanisms dominate for different processing conditions, materials properties, and drop-size ranges.

The collision kernels appearing in the population dynamics models require expressions for the collision rate between drops of two different sizes. Classical expressions attributed to Smoluchowski are improved to include attractive, repulsive, and hydrodynamic interactions between drops. In particular, trajectory calculations are used to predict collision efficiencies, which represent the ratio of the collision rate with these interactions to the Smoluchowski collision rate without these interactions, as functions of the size ratio, viscosity ratio, and other relevant dimensionless parameters.

*Progress to Date:* A computer program has been completed for solving the population dynamics model to follow droplet size evolutions with time in homogeneous dispersions due to collisions arising from gravity sedimentation and Marangoni migration acting collectively or individually. It is shown that droplet growth rates may be reduced significantly by reducing the driving force (gravity level or temperature gradient), the droplet volume fraction, the average initial droplet size, or the degree of spread in the initial size distribution. Coalescence may also be greatly reduced by appropriate antiparallel alignment of the gravity vector and the temperature gradient. A bimodal distribution will have much more rapid coalescence due to gravity sedimentation or Marangoni migration than will a unimodal distribution. Moreover, a unimodal initial distribution will evolve into a bimodal distribution and then into a shifted and broadened unimodal distribution.

Collision efficiencies for Brownian motion have been computed for drops having a range of viscosity and radius ratios. Owing to the absence of complete fluid dynamic information on asymmetric interaction of two drops in another fluid, only approximate collision efficiencies for gravity sedimentation have been computed. In both cases, it is found that spherical liquid drops have nonzero collision efficiencies in the absence of attractive forces. This is in contrast to rigid particles which require an attractive force (such as the London-van der Waals induced-dipole force) in order to

squeeze the fluid out from between them and come into contact. Further work on the interaction of drops is in progress, including a study of their deformation when in near contact.

#### Publications

Davis, R. H., Schonberg, J. A., and Rallison, J. M., "The Lubrication Force Between Two Viscous Drops," Phys. Fluids A 1, 77-81 (1989).

Bamocky, G. and Davis, R. H., "The Lubrication Force Between Spherical Drops, Bubbles and Rigid Particles in a Viscous Fluid," Int. J. Multiphase Flow, 627-638 (1989).

Rogers, J. R. and Davis, R. H., "Modeling of Coalescence in Microgravity Processing of Zn-Bi Immiscible Alloys," Met. Trans. A, (in press).

## Theory of Solidification

NASA Lewis Research Center

Prof. Stephen H. Davis

NAG3-747

October 14, 1986 - October 13, 1989

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

*Research Task Definition:* The research entails the study of coupled systems using analytical methods, with the aim of identifying new mechanisms of behavior and new physical effects.

*Progress to date:* In the past year we have concentrated on two aspects. On the one hand we have reexamined the weakly nonlinear morphological problem near the minimum  $c$  and were able to describe simultaneously the high and low speed transitions predicted by Mullins and Sekerka. We find conditions for various types of secondary or tertiary transitions and the existence of isolated solutions. Further, we have identified experimentally-accessible conditions under which shallow cells appear and might serve as a quantitative test of the Mullins and Sekerka model and its weakly nonlinear extensions.

On the other hand we have found a new class of long-wave models for directional solidification and have examined how morphological instabilities couple with convective instabilities. These models provide "cheap" systems for the study of certain strongly-nonlinear behaviors of the interface.

## Publications

Brattkus, K. and Davis, S. H., "Cellular growth near absolute stability," Phys. Rev. B **38**, 11452 (1988).

Merchant, G. J. and Davis, S. H., "Morphological stagnation-point flow and steady streaming," J. Fluid Mech. **198**, 543 (1988).

Young, G. W. and Davis, S. H., "Morphological instabilities in directional solidification of a binary alloy: end effects," SIAM J. Appl. Math. **49**, 152 (1988).

Brattkus, K. and Davis, S. H., "Directional solidification with heat losses," J. Cryst. Growth **91**, 538 (1988).

Canright, D. R. and Davis, S. H., "Similarity solutions for phase-change problems," Met. Trans. **20A**, 225 (1989).

Merchant, G. J. and Davis, S. H., "Shallow cells in directional solidification," Phys. Rev. Lett. **63**, 573 (1988).

Merchant, G. J. and Davis, S. H., "Flow-induced morphological instabilities due to temporally-modulated stagnation-point flow," J. Cryst. Growth, 1988 (in press).

Riley, D. S. and Davis, S. H., "Eckhaus instabilities in generalized Landau-Ginzburg equations," Phys. Fluids A, 1988 (in press).

Riley, D. S. and Davis, S. H., "Hydrodynamic stability of a melt during the solidification of a binary alloy with small segregation coefficient," Physica D, 1989 (in press).

Davis, S. H., "Hydrodynamic interactions in directional solidification," J. Fluid Mech., 1989 (in press).

Riley, D. S. and Davis, S. H., "Long-wave morphological instabilities in the directional solidification of a dilute binary mixture," SIAM J. Appl. Math., 1989 (in press).

Riley, D. S. and Davis, S. H., "Long-wave interactions in morphological and convective instabilities," (pending publication).

Merchant, G. J. and Davis, S. H., "Directional solidification near minimum  $c$  : two-dimensional isolas and multiple solutions," (pending publication).

## Numerical Simulation of Fluid Transport Phenomena

Lewis Research Center  
Dr. J. C. Duh  
In-House

*Objectives:* The main objective of this program is to develop a custom code to enhance the scientific understanding of the fluid transport phenomena of interest to the microgravity experiments program. With the code developed, it could also help in assessing the design of microgravity experiments and evaluating the real users requirements for microgravity experiments from an informed position.

*Research Task Description:* The main goal of this research is to develop a numerical code which is "general" and "flexible" enough to cover the fluid transport phenomena of interest to the microgravity science community, but "specific" enough that some of the long troublesome points in these calculations can be resolved. The research thus involves two areas: (1) numerical techniques development, and (2) direct numerical simulation and/or numerical experiments on fluid transport problems. On the numerical techniques development part, an adaptive grid system has been developed to automatically provide better resolution in the high temperature and/or velocity gradients area. A velocity map method has been devised to determine the onset of imbedded secondary cells in the enclosure convection problem. Future activities will include the development of unstructured grid system and to further explore higher order accurate numerical schemes. On the numerical simulation and/or experiments, several subjects have been investigated including Marangoni convection inside an evaporating liquid droplet, thermal natural convection inside an inclined enclosure, and the Rayleigh-Benard stability. With a better understanding of the subjects mentioned above, future effort will be expanded into study on the Marangoni stability, the effect of g-jitter on the transport phenomena, and the implementation of deformable free-surface modelling.

*Progress to Date:* Results from the numerical simulation of natural convection inside tilted enclosures have been compared favorably with the existing experimental data. In the horizontal-heating cases, the velocity map method has been used to determine the critical Rayleigh number for onset of imbedded secondary cells, the results predicting a much lower value than can be determined experimentally. In the Rayleigh-Benard stability cases, a second transition has been observed from the numerical experiments, which transfers the transport mode from stable unicellular convection to periodic multicellular oscillation. These efforts have been documented in the publications cited below:

### Publications

Duh, J. C., and Yang, W.-J., "Numerical Analysis of Natural Convection in Liquid Droplets by Phase Change," *J. Num. Heat Transf.* 16, 129-154 (1989).

Tan, C.-H., Duh, J. C., "Pressure Gradient Method for Solving Incompressible Navier-Stokes Equations with Curvilinear Coordinate System," in *Proceedings of the 7th International Conference on Mathematical and Computer Modelling*, Chicago, August 1989 (in press).

Tan, C.-H., and Duh, J. C., "Comparison Between Pressure Gradient Method and MAC Method on High-Reynolds Flows," in *Proceedings of the 5th International Symposium on Numerical Methods in Engineering*, Lausanne, Switzerland, September 1989.

Arpaci, V. S., Selamet, A., Chai, A. T., and Duh, J. C., "Multiscale Dimensional Foundations of Surface Tension and/or Buoyancy Driven Flows," in *Proceedings of the 7th European Symposium on Materials and Fluid Sciences in Microgravity*, ESA, 1989 (in press).

Chai, A. T., Duh, J. C., and Arpaci, V. S., "A Proposed Study on Liquid Droplet Evaporation under Microgravity," in *Proceedings of the 7th European Symposium on Materials and Fluid Sciences in Microgravity*, ESA, 1989 (in press).

#### Presentations

Duh, J. C., Wang, L. W., Rashidnia, N., and Tan, C.-H., "Natural Convection Inside an Inclined Low-Aspect-Ratio Enclosure," *7th International Conference on PhysicoChemical Hydrodynamics*, MIT, June 1989.

## Two Phase Gas Liquid Flow under Reduced Gravity Conditions

University of Houston  
Professor A. E. Dukler  
NAG 3-510 (NASA Contact: J. McQuillen, LeRC)  
December 1888 - December 1991

*Objectives:* The objective of this research is to carry out experiments designed to reveal the mechanisms controlling gas-liquid flow at the microgravity and to develop general models which can predict the hydrodynamics and heat transfer under these conditions. This includes predicting flow patterns which exist and their transitions, pressure drop, stability of the flow, the size, velocity and frequency of the long gas bubbles and liquid slugs during slug flow and interfacial structure during annular flow. The work will also include studies of droplet formation and transfer which is controlled by the hydrodynamic behavior.

*Research Task Description:* Experiments are carried out on the LeRC Learjet. Sixty trajectories have been executed at  $0 \pm 0.02$  g and 16 at 0.17 g (moon gravity). The test loop consists of 1.27 cm diameter tube about one meter long. Air and water are metered to the tube and films of the flow are taken through a transparent section using a 400 frame/s camera. In addition, measurements are made of time varying pressure drop, and void fraction. The films and data are analyzed to give information of the flow pattern, characteristics dimensions of the flow, velocity and sizes of slugs and bubbles, wave frequency, void fraction, etc. Future experiments will involve different fluids, test section sizes and entry configurations. Trajectories provide reduced g for 12-20 seconds as indicated by accelerometers



Transient Heat Transfer Studies in Low-gravity

National Institute of Standards & Technology, Boulder Labs  
Dr. Patricia J. Giarratano  
W-16,170 (NASA Contact: R.K. Crouch, HQ)  
October 1988 - October 1989

*Objectives:* This project has investigated thermally induced motion in highly compressible fluids which have been subjected to a transient heat pulse from a thermometer/heater test section. The tasks designated for the past year are intended to finalize the applicability of the optical measurement technique (holographic interferometry with diffuse light source) to measurement of the temperature profile in the thin thermal boundary layer.

*Research Task Description:* The tasks are to (1) complete holography measurements in the laboratory to optimize the exposure times and magnifications of the test section, and thereby provide bench marks for the holograms to be obtained in low-g during KC135 flights, (2) fly the equipment on the KC135 and obtain data, and (3) write a final report summarizing the work.

*Progress To Date:* We have completed the ground tests, item (1) above, and are currently awaiting the opportunity to fly on the KC135 to complete the measurements. Our flight has been delayed due to mechanical problems with the aircraft; therefore, the completion of item (2) and (3) are dependent on the availability of the KC135 equipment.

Publications

Giarratano, P. J., Kumakawa A., Arp, V. D., and Owen, R. B. "Transient Heat Transfer Studies in LowGravity Using Holographic Interferometry," J. Thermophys. & Heat Transf. 4, 53-58 (1990).

## Acoustic Forcing of a Liquid Drop

West Virginia University  
Dr. M. J. Lyell

*Objectives:* The objective of this project is to determine the flow field in an acoustically forced drop. This is to be done for the cases in which the acoustic field is modulated and unmodulated, respectively. This goal involves the development of a formulation in which the tangential radiation stress contributes to drop forcing.

*Research Task Description:* Advanced technology which will enable the manipulation of a liquid sample in a microgravity environment is currently under development. One such system is the three-axis acoustic levitation chamber which utilizes acoustic radiation pressure forces to not only position but also to manipulate the droplet. For example, the drop can be made to oscillate via forcing at its resonant frequencies. This research investigates the fluid dynamics involved in the acoustic forcing of a liquid drop. In particular, the tangential components of the acoustic radiation pressure vector responsible for the tangential forcing are to be taken into account. The appropriate governing equations are formulated, and the nature of the coupling between the first order sound field and the second order hydrodynamic field is elucidated. Ultimately, the specific form of the acoustic field and the radiation pressure vector are to be displayed. The hydrodynamic field will then be obtained. The response amplitude of the forced oscillating drop will be obtained. This will be compared to the approximate result determined through restricting the forcing to be that due to the radial component of the radiation pressure vector only. In the case of the non-modulated acoustic forcing, the flow field interior and exterior to the drop will be determined. Finally, an extension to the case of forcing of specific compound liquid drops will be considered.

*Progress to Date:* The form of the viscous acoustic field in the case of a traveling wave has been determined. The standing wave formulation is underway. This is needed for the computation of the radiation pressure vector (with viscous effects). Attention for the problem of forcing by a modulated acoustic wave is focused on the situation in which the acoustic field couples to the hydrodynamic field only at the drop/host interface. That is, viscous effects are taken into account in the acoustic field, but no effect of streaming is considered. In the case of forcing by an unmodulated acoustic wave, the acoustic field is coupled to the hydrodynamic governing equations. This coupling appears as forcing terms. The viscosity is taken to be large. This calculation is underway. The streaming velocity is to be determined. Some results have been obtained for forcing of a compound drop in the case in which core and shell fluids are restricted to have the same density and compressibility.

### Presentations

M. J. Lyell, "On Aspects of Forced and Free Compound Fluid Drop Behavior," *ALAA 27th Aerospace Sciences Meeting*, Reno, NV, 1989, AIAA Paper 89-0066.

M. J. Lyell, "The Effect of Tangential Radiation Stress on the Acoustic Forcing of Liquid Drops," *ALAA 28th Aerospace Sciences Meeting*, Reno, NV, January, 1990.

## Fundamental Study of Nucleate Pool Boiling Under Microgravity

University of Michigan  
Professor Herman Merte, Jr.  
NAG3-663 (NASA Contact: F. Chairamonte, LeRC)  
October 1985 - February 1990

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

The initial research focuses on the net forces acting on the growing/collapsing vapor bubbles when buoyancy has been drastically reducing, and consist of (a) internal vapor bubble pressure, (b) bulk liquid momentum, (c) molecular momentum of evaporation/condensation, (d) liquid viscosity, (e) liquid-solid-vapor surface tension, (f) Marangoni convection, (g) residual buoyancy. It is desired to determine how these forces are related to the temperature distribution in the liquid, which itself is influenced by buoyancy.

Additionally, attempts will be made to describe the heterogeneous inception of boiling, using known liquid temperature distributions in the vicinity of the heater surface. This is possible in circumstances where nucleation takes place before distortion of the temperature distribution due to buoyancy occurs.

*Research Task Description:* A relatively large (19 mm x 38 mm) flat heat transfer surface is part of one wall of a closed vessel, maintained at a constant pressure and initially uniform temperature. The independent variables are subcooling and heat flux, with a step increase from zero to a prescribed power input. Measurements of surface temperature are made simultaneously with motion photography during the transient heating process including the onset of boiling, until a terminal condition is reached appropriate to the particular circumstances present. Two heating surfaces are being developed: a semitransparent layer of gold vacuum deposited on a quartz substrate, which acts simultaneously as a well-defined electrical heater and resistance thermometer, and which permits viewing simultaneously from the side and beneath the boiling surface, and a copper surface indirectly heated electrically, gold coated so as to present the same surface energy conditions to the boiling fluid. Testing will be conducted in the laboratory at  $a/g = \pm 1$ , and in a drop tower for short term microgravity. Plans are being developed for subsequent orbital flight in the shuttle to provide the longer time periods necessary. R-113 is selected as the initial fluid to be used since it is in a class of "space fluids" such as cryogenics, ammonia and fluorocarbons, and being nonpolar is compatible with the use of a transparent gold-film heater carrying an electrical current.

*Progress to Date:* Work is underway to describe the heterogeneous vapor nucleation taking place at the heater surface using known and well-defined liquid temperature distributions in the vicinity of the heater surface. This is possible in circumstances where nucleation takes place before distortion of the temperature distribution due to buoyancy occurs. Testing in a microgravity environment removes buoyancy for this purpose, as does heating in an inverted position at  $a/g = -1$  to a limited extent.

Experiments with the test package for operation at  $a/g = \pm 1$  are underway. Fabrication of the test package for operation in the 5 second drop tower at NASA-Lewis has been completed, tested at  $a/g = +1$ , and delivered to NASA-Lewis. A Science Requirements Document for an orbital experiment has been completed, along with the Conceptual Design Review and the Requirements Definition Review.

## Publications

Merte, H., "Combined Roles of Buoyancy and Orientation in Nucleate Pool Boiling," Heat Transfer 2, 179-186 (1988).

Merte, H. and Judd, R. L., "Variation of Superheat with Subcooling in Nucleate Pool Boiling," J. Heat Transf., submitted.

## Presentations

Merte, H., "Buoyancy Effects in Nucleate Pool Boiling" at the University of Colorado at Boulder, Colorado, Center for Low-Gravity Fluid Mechanics and Transport Phenomena, October 10, 1988. Also at Idaho National Engineering Laboratory, Idaho Falls, Idaho, January 26, 1989. Also at Institute A for Thermodynamics, Technical University of Munich, Munich, FRG, February 17, 1989.

"Boiling Nucleation in Microgravity" at Session on Two-Phase Flow and Boiling, *7th International Conference on Physico Chemical Hydrodynamics*, MIT, Cambridge, Massachusetts, June 28, 1989.

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

Arizona State University  
G. Paul Neitzel  
Daniel F. Jankowski  
NAG 3-568 (NASA Contact: A.T. Chai, LeRC)

*Objectives of Research:* The objectives of this research were to calculate energy-stability limits for basic states which are models of the float-zone-crystal-growth process. Energy-stability limits provide sufficient conditions for stability of a basic state. The successful computation of such a limit for an actual float-zone basic state would identify conditions under which oscillatory convection (and hence striations in the final material) would be absent.

*Progress to Date:* Energy-stability limits for axisymmetric disturbances have been computed for the basic state of a model half-zone with a non-deformable free surface. The influence of gravity and material properties has been examined through variation of the dimensionless Grashof and Prandtl numbers, respectively. Earlier problems in solving the symmetric, but indefinite, generalized eigenvalue problem have been overcome through the implementation of an inverse-iteration algorithm. The results have been compared to the experimental results of Preisser, Schwabe & Scharmann. Progress has also been made on the calculation of the basic state of a half-zone with a deformable free surface and on the implementation of energy-stability theory to this case.

#### Publications

Y. Shen, "Energy Stability of Thermocapillary Convection in a Model of Float-Zone Crystal Growth," Ph.D. Dissertation, Arizona State University, May 1989.

Y. Shen, G. P. Neitzel, D. F. Jankowski, and H. D. Mittelman, "Energy Stability of Thermocapillary Convection in a Model of the Float-Zone, Crystal Growth Process," *J. Fluid Mech.*, submitted.

G. P. Neitzel and D. F. Jankowski, "Application of Energy-Stability Theory to Problems in Crystal Growth," *Progress in Low-Gravity Fluid Dynamics and Transport Phenomena* (J.N. Koster and R.L. Sani, eds), in press.

#### Presentations

G.P. Neitzel, *Applied Mathematics Seminar*, May, 1988, Department of Engineering Sciences and Applied Mathematics, Northwestern University, Evanston, IL.

G.P. Neitzel, Y. Shen, D.F. Jankowski, "Energy Stability of Thermocapillary Convection in a Model Half-Zone. Finite-Difference Results," *41st Annual Meeting, Division of Fluid Dynamics, American Physical Society*, November, 1988, Buffalo, NY.

D.F. Jankowski, *Mechanical Engineering Seminar*, April, 1989, Department of Mechanical Engineering, University of New Mexico, Albuquerque, NM.

D.F. Jankowski, G.P. Neitzel, Y. Shen, and H.D. Mittelman, "Energy Stability of Thermocapillary Convection in a Model Half-Zone. New Results," November, 1989, *42nd Annual Meeting, Division of Fluid Dynamics, American Physical Society*, NASA Ames Research Center.

G.P. Neitzel, J. R. Hyer, and D. F. Jankowski, "Thermocapillary Convection in a Model Float-Zone," *28th AIAA Aerospace Sciences Meeting*, January, 1990, Reno, NV.

*In-Space Experiment on Thermoacoustic Convection Heat Transfer Phenomenon Experiment Definition*

University of Tennessee, Knoxville  
Professor M. Parang  
NAS3-25359 (NASA Contact: A. T. Chai, LeRC)  
August 1988 - August 1990

*Objectives:* The objective of this research is to define a potential flight experiment to evaluate the significance, enhance the understanding and to access the phenomenon of the thermoacoustic convection and its effects on fluid storage handling and transport, as well as, material processing in space environment.

*Research Task Description:* The research tasks include: (1) conduct numerical and supporting ground-based experiments to help establish and define the objectives of the in-space experiments and the necessary technical requirements for the experiment hardware; (2) develop conceptual designs for the experiment; and (3) develop preliminary plans, schedules, and costs for implementing the experiment aboard a shuttle mission.

*Progress to Date:* A series of laboratory experiments have been conducted in which air is rapidly heated in a rectangular cavity. One wall of the cavity is subjected to a rapid rise of temperature and the resulting fluid response to the thermal input has been investigated. The transient fluid temperature profile, bulk fluid pressure and velocity are among the flow parameters that are experimentally measured. The instantaneous spatial distribution of the fluid temperature was accomplished by the design of an optical system. The preliminary ground experimental results are being compared at the present time with the numerical models of the cavity flow. In addition, flow visualization experiments are completed and the feasibility of including flow visualization in the space experiment is being studied. Finally, a preliminary design of the in-space experimental apparatus is in progress which will provide rapid heating of a boundary (up to 1500 C in 3 seconds) in a sealed cavity containing air and includes provisions for the measurement of temperature, velocity, and pressure. The in-space experiment design will also have such capabilities as high-speed film recording which will be used as part of the optical temperature measurement system and the flow visualization experiments.

Publications

Parang, M., and Catt, J.A., "Convective Air Motion Generated by Rapid Heating in an Enclosure", 28th AIAA Aerospace Sciences Meeting, Reno, Nevada, January 8-11, 1990.

Breakdown of the Non-Slip Condition in Low-Gravity

Los Alamos National Laboratory

Donald R. Pettit

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

May 1988 - June 1990

*Objectives:* The objective of this research is to investigate whether slip of liquids at a solid wall occurs in low gravity.

*Research Task Description:* The purpose of the experiment is to measure the extent of fluid-wall slip in a concentric cylindrical cell where the fluid fills the annular gap. Couette flow is introduced into the fluid by rotating the inner cylinder at constant RPM. Alteration of the boundary condition is determined from the torque ratio between low-g and normal g.

*Progress to Date:* The concentric cylinder fluid cell was made so either fire polished pyrex glass walls or smooth ground teflon wall could be used. The inner cylinder diameter was 25mm by 115mm long. The gap could be varied from 0.7mm to 2.5 mm. Two Brookfield torque meters were modified for low gravity operation and had a range from 0-670 and 0-7200 dyn-cm with a report 1% accuracy. Rotation rates of 5, 10, 20, 50 and 100 RPM were used with silicone oils with viscosities of 5, 10, 20, 50, 100 and 500 mPa-s. KC-135 flights are currently in progress.



### Fluid Dynamics and Low Gravity Effects of Chemical Vapor Transport

University of Alabama in Huntsville  
Center for Microgravity and Materials Research  
Professor Franz Rosenberger  
Dr. Thomas Nyce  
NAG8- 704 (NASA Contact: S.L. Lehoczky. MSFC)  
February 1988 - February 1990

*Objectives:* Chemical vapor deposition (CVD) processes are used on a large scale for the preparation of electrically active and insulating layers for opto-electronic solid state devices. The performance of these devices often depends sensitively on the uniformity in thickness and composition of the as-grown layers. These, in turn, depend upon the heat and mass transfer conditions during the deposition process, i.e. on the fluid dynamics in the nutrient vapor phase. Currently, the fluid dynamics of CVD processes is not too well understood, though recent research has revealed that buoyancy-driven convection is responsible for various layer non-uniformities, that seriously limit the yield of CVD materials processing.

This research program will delineate the effects of gravity in current CVD practice, give guidance in reactor design for reducing these effects on earth, and explore the potential of CVD experiments under low gravity conditions. Beyond these questions of great technological importance, this research can be expected to clarify some fundamental aspects of combined (superimposed buoyancy-driven and forced) convection.

*Research Task Description:* A complimentary theoretical and experimental research program is being pursued to quantify the effects of gravity ( $1\text{-}10^{-6}g$ ) and total pressure (760-0.1 torr) on compositional and thickness uniformity attainable in horizontal reactor geometries used in silicon CVD and GaAs MOCVD. The work includes extensive numerical modelling (2-D, 3D, compressible, multicomponent steady-state flow, including homogeneous gas phase reactions and some account of interfacial kinetics). In the experimental part of the study, velocity distributions in CVD reactor models are being determined with laser Doppler anemometry, with some emphasis on the characterization of the parameter combinations that lead to (undesirable) non-steady transport.

*Progress to Date:* In order to clarify the limitations of 2-D theoretical simulation and to establish a well-defined foundation for our 3-D modelling, we have performed a systematic numerical study and compared the results with published experimental data for the MOCVD of gallium arsenide. This study shows that 2-D models can produce realistic results for the growth rate distribution only for reactors with large crosssectional width-to-height (aspect) ratios that are operated at subcritical Rayleigh numbers. In addition, Soret diffusion has been identified as a major factor for growth rate uniformity. Furthermore, velocity corrections for finite aspect ratios must be made, and buoyancy effects can be significant even in large aspect ratio reactors at the leading edge of the susceptor. Whereas the temperature dependence of the transport parameters can be ignored for the prediction of growth rates (due to a compensating effect of property averaging on the diffusive and convective fluxes at the susceptor), the composition dependence of the transport properties must be retained in a realistic model.

In the three-dimensional modelling work we have compared solutions obtained from full 3-D and 3-D parabolic formulations. We have found that depending on the aspect ratio and lateral thermal boundary conditions used, buoyancy-driven 3-D flow under conditions judged stable against natural convection rolls when using only criteria based on vertical temperature gradients (i.e. subcritical Rayleigh numbers). In particular, the modelling results illustrate the importance of the proper choice of thermal boundary conditions on the sidewalls of the reactor for lateral growth rate uniformity. The much higher computational costs associated with a full 3-D model, as compared to 3-D parabolic solutions, are justifiable only for small aspect ratio reactors operated at low flow rates.

The experimental studies, conducted on a model reactor with aspect ratio 2, have shown that, depending on the underlying Reynolds and Rayleigh numbers, combined convective flows cover three distinctly different flow regimes: (a) Steady flow throughout the entire channel with two maxima in the crosswise profile of the axial velocity developing symmetrically about the vertical center-plane; (b) Steady flow throughout with asymmetries about the center plane in a very limited regime; and (c) Non-steady flow with asymmetries about the center plane, evolving with increasing distance from the leading edge of the heated susceptor for Rayleigh numbers in excess of 14,000. The finding of these unexpected asymmetries, is extremely important for numerical modelling. All CVD modelling work to-date has assumed (computer timesaving) symmetry of the flow about the vertical center plane.

#### Publications

Ouazzani, J., Chiu, K-C., and Rosenberger, F., "On the 2D Modelling of Horizontal CVD Reactors and its Limitation", J. Cryst. Growth **91**, 497-508 (1988).

Ouazzani, J. and Rosenberger, F., "Three-Dimensional Modelling of Horizontal Chemical Vapor Deposition I: MOCVD at Atmospheric Pressure, J. Cryst. Growth (accepted).

*Disorder-Order Transitions in Colloidal Suspensions: Computer Simulations and Experimental Observations*

Princeton University

William B. Russel

Pablo G. Debenedetti

NAG3-859

January 20, 1988 - February 19, 1990

*Objectives:* The goal of this work is to understand the rate and mechanisms of formation of ordered, crystalline structures in concentrated colloidal suspensions, experimentally and computationally. Experiments will be aimed at developing a technique for the production of macroscopic single crystals by the application of periodic electric fields. From a scientific viewpoint, the dynamics of this phase transition has been largely unexplored. Practically, understanding the disorder-order transition is of importance in areas such as ultrafiltration and ceramics fabrication from submicron particles via colloidal processing routes.

*Research Task Description:* Our most important experimental task is to grow macroscopic single crystals by applying a periodic electric field at the bottom of a container to induce order in the first few layers and provide a pattern for subsequent crystal growth. This will be done by evaporating conducting electrodes on a quartz plate, interdigitating two combs of electrodes, and applying opposite voltages to each. This will produce a periodically modulated electric field gradient whose profile extends a short distance into the sample volume. The resulting force on the colloidal particles will induce order which is periodic in two direct

## Hydrodynamic Instability as the Cause of Morphological Breakdown during Electrodeposition

Massachusetts Institute of Technology

Professor D.R. Sadoway

Professor R.A. Brown

NSG-7645

October 1, 1988 - September 30, 1989

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

**Research Task Description:** The work combines experiments to measure flow characteristics and resulting surface structures with calculations of critical physical parameters. The study is unique in several respects. The causes of morphological breakdown during electrodeposition has never been the subject of a systematic study that seeks to investigate the problem under conditions where the kinetic processes are clearly defined. Specifically, this study is being conducted in a physical model system that will permit the observation of simple electrocrystallization under conditions of strict mass transfer control. Furthermore, this study is unique in its attempt to combine experimental observations with mathematical models of complex fluid flow behavior originally developed for the analogous solidification problem in crystal growth. There are, however, important differences between solidification and electrocrystallization, and these differences serve as the basis for this comparative study.

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

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

**Progress to date:** The experiments involved galvanostatic deposition of silver from a stagnant, non-complexing, aqueous solution of nitrates (0.5 M  $\text{AgNO}_3$ -5 M  $\text{NaNO}_3$  - HNOS). On the basis of additional electrochemical, spectroelectrochemical and microscopic studies a solution of pH 2 was found to provide optimal deposition characteristics and thus was used throughout the principal investigation.

For the purposes of modelling solutal Benard convection the deposition experiments were conducted with horizontal and upward-facing cathodes. Below a critical value of Rayleigh number Benard convection is characterized by stable stratification of fluid, inverted with respect to buoyancy. The electrode configuration employed in this investigation allowed the deposits to form from both convecting and stagnant electrolytes. The onset of hydrodynamic instability in the concentration boundary layer was observed in situ by laser interferometry. The critical wavelength of convective instability was determined from interferograms by two dimensional Fourier image analysis. Deposits were characterized using microscopic (ex-situ) techniques.

The results of this study indicate that there are two distinct regimes of behavior with respect to both convection and deposit morphology. In the first regime, which is characterized by low galvanostatic current, convective instability is the primary influence on deposit morphology. In the second, a high current regime, convection has no influence on deposit morphology.

In the low current regime, the onset of convection precedes the morphological change that also occurs in the deposits. Specifically, this change refers to the emergence of nodular deposits. The mean distances between nodules correlate well with measured critical convective wavelengths. The critical Rayleigh number ( $Ra_c$ ) was computed on the basis of the time required to attain hydrodynamic instability. Both  $Ra_c$  and critical wavelength values compare favorably with theoretical values for the Benard model of convection. This agreement permits the use of the hexagonal convective pattern, typical of Benard convection, to explain the 1:2 ratio between nodular and convective dimensions.

In the high current regime, dendritic deposits formed rapidly, well before the onset of convection. In addition, there were large increases in overvoltage during the formation of these dendrites. Large overvoltage increases indicate depletion of the ionic species in the diffusion boundary layer.

The crossover point between the low and high current behaviors occurs when the convective and diffusive time scales are similar. At current levels at which ion depletion occurs before the onset of convection, dendritic deposits grow. At lower currents such depletion is prevented by the increased mass transport associated with convection. In fact, the hexagonal pattern of convection causes modulation in the amount of material delivered to the cathode and promotes the formation of nodular deposits.

#### Publications

J. K. Koziol, *The Effect of Electrolyte Convection on the Morphology of Silver Electrodeposits*, Ph.D. Thesis, Department of Materials Science and Engineering, MIT, September, 1989.

Statistical Mechanics of Fluids: the Effects of an External Gravitational Field

NASA Lewis Research Center  
Dr. R. E. Salvino, Sverdrup Technology  
Dr. R. A. Wilkinson, NASA LeRC  
In-House

*Objectives:* The objective is to clarify, quantify, and assess the role of gravity in the thermophysical description of matter from a first principles statistical mechanical point of view. The basic formulation of the inclusion of an external field in equilibrium statistical thermodynamics without an appeal to local equilibrium assumptions is a primary starting point. The role of an external field that couples to the center of mass only in a Newtonian microscopic description of a many--particle assembly is also an important building block. The activity will develop both the formal and computational aspects of the statistical mechanics. Questions of a fundamental physics nature will be addressed as well as the computation of specific gravitational effects in the thermophysical description of matter. The systems to be investigated may be categorized into equilibrium systems without walls, equilibrium systems with walls, flow systems without walls, and flow systems with walls.

*Research Task Description:* The activity entails theoretical studies of both an analytical and a computer simulational nature. Both lines of investigation will focus on bulk thermodynamic, hydrodynamic, and transport properties as well as typical surface quantities such as the liquid/vapor interfacial surface tension. The computer simulations will focus exclusively on classical systems but the formal developments will allow investigation of both classical and quantum systems. The analytical studies start with the formulation of the equilibrium statistical thermodynamics of a many--particle system under the influence of an external scalar field. This is accomplished without the use of any local equilibrium assumptions. The microscopic many--body equations of motion or conservation laws are investigated to help indicate where local equilibrium assumptions and constitutive relations may have weak points. Simple model calculations are used as illustrations and to provide a primitive understanding of the results. On the simulation side, a molecular dynamics program which can be used in the description of a wide variety of equilibrium and nonequilibrium phenomena is now operational. The program is very flexible in that any analytical two--body potential can be readily used and it shows a high degree of stability in that there is no detectable energy drift even for runs of over 100,000 time steps. The energy fluctuations for the microcanonical ensemble calculations are consistently of the order of one part in a million. However, the calculation of a number of time correlation functions which are necessary to the calculation of transport properties have not yet been coded. At present, all simulation work is being performed with conventional sequential Fortran on a PC--Transputer system. Eventually the system size and the interest in fluid flow problems will necessitate the implementation of parallel Fortran if the PC--Transputer system is to remain an acceptable hardware option. Consequently, time must be spent on successfully implementing the parallel Fortran.

*Progress to Date:* Two preprints have been prepared which summarize the results found to date. The results may be classified into formal results and computational results. The basic formal results are threefold: a system in an external gravitational field does not have the extensivity property it would have without the external field, the thermodynamic description develops an anisotropy due to the presence of the external field, and the assumptions used to augment local equilibrium arguments are suspect, that is, they appear to be inconsistent with the microscopic equations of motion or conservation laws. Explicit computational results in closed form for the effects on some thermophysical properties of a single phase system without any external walls has been obtained. This was accomplished through the analysis of the Newtonian equations of motion that are used as the basis of the molecular dynamics simulation program. The interpretation of the results, however, is not as straightforward as one would like. A basic understanding and a logically consistent interpretation of the results needs to be done. A possible interpretation of the results is that the coupling to the center of mass is similar to a collective effect in that the external constant field influences all particles in the same way thus compounding the single particle effect, which by itself is quite small, in a simple additive manner. For example, the velocity autocorrelation

function shows a large contribution from the external field as does the pressure tensor autocorrelation function. An attempt to connect the time integrals of these time correlation functions to transport properties leads to very large gravitational terms in these properties. This has led to a re-examination of the constitutive relations which introduces the transport properties into the thermophysical description. Modifications to the constitutive relations, or reinterpretation of the transport coefficients, may be required to bring the results more in line with the present status of the data. Once the results are judged logical, we will return to the molecular dynamics simulations. After we assess to what extent the simulations can duplicate the analytical results, the simulation of systems that will have no analytical solution available will be implemented, in particular, fluid systems flowing in the presence of an external wall. The parallel Fortran will need to be operational by then.

#### Publications

R. E. Salvino, "The Surface Tension of  $^4\text{He}$ : Low--Temperature Behavior," J. Low Temp. Phys. **76**, 121 (1989).

R. E. Salvino, "The Equilibrium Statistical Thermodynamics of a Many--Particle System Coupled to an External Scalar Field," preprint (1989).

R. E. Salvino, "The Newtonian Mechanics of a Many--Particle Assembly Coupled to an External Body Potential," preprint (1989).

## The Roles of Fluid Motion and Other Transport Phenomena in the Morphology of Materials

Princeton University  
Dr. D A. Saville  
NAG 3-447 (NASA Contact: R. Snyder, MSFC)  
October 1988-September 1990

*Objectives:* The objective is to find out how certain transport phenomena influence the morphology of crystalline materials. Two problems are under study: one deals with the effects of convection on the crystallization of pure materials, the other with the crystallization of proteins from solution. In the first study we are interested in how convection alters relations between undercooling and the speed and stability of growth. In the second, we seek to find out why protein crystals grow as slowly as they do and how crystal morphology depends on the growth rate and crystal size.

### *Research Task Description:*

#### Crystal Growth in the Presence of Convection

A computation scheme has been developed which simulates the evolution of a needle-shaped crystal in an undercooled melt when convection is present. The computer program will be exercised to explore a wider range of undercoolings, flow strengths, and material properties. The algorithm will also be expanded to permit better resolution of the tip region and to allow for crystal anisotropy.

#### Protein Crystal Growth

Experimental apparatus has been constructed to record the growth process with a digital imaging system. Using model proteins we will study the relations between crystal growth rate, size, and quality. Structure determinations will be carried out in collaboration with Professor C. Schutt, a protein crystallographer in Princeton's Chemistry Department.

*Progress to Date:* Theoretical work has focussed on testing the computer algorithm for calculating the evolution of a needle-shaped crystal developed under the previous contract. On the experimental side we have begun accumulating data on the growth rate of lysozyme using the digital imaging system.

### Publications

Saville, D. A. and Beaghton, P. J., "Growth of Needle-Shaped Crystals in the Presence of Convection," Phys. Rev. A **37**, 3423-3430 (1988).

### Presentations

Saville, D. A. and Beaghton, P. J., "Stability of Needle-Shaped Crystal Interfaces," *AIChE Annual Meeting*, Washington D.C., December 1988.



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

Cornell University

Dr. Paul H. Steen

NAG3-801 (NASA Contact: L. Dill, LeRC)

June 1, 1987 - May 31, 1989

**Objectives:** This research aims to determine under what conditions in a low-gravity environment small-to-moderate shear stresses inhibit the capillary instability of a liquid bridge. Hydrodynamic stability theory is used to predict windows in parameter space where stabilization occurs in model systems and these predictions guide ground-based experiments which utilize a Plateau-chamber. This apparatus simulates low-gravity by matching the densities of two immiscible liquids.

**Research Task Description:** The low gravity environment of space allows the containment of liquids by surface tension in a range of configurations not possible in conventional ground-based applications. The distortion of the free liquid interface may be suppressed to advantage in containerless energy exchange, in the transportation of liquids, and in the processing of materials, to name a few. Nevertheless, the surface tension (capillary) instability limits static shapes to be spherical or of small aspect ratio, depending on the constraints of contact. For example, even in zero-gravity, surface tension limits a circular cylinder to be shorter than its circumference; longer cylinders tend to break up into droplets. We study the influence of liquid motion on the capillary instability. A major result is the prediction, by means of a linear stability analysis, that axial motion through an annular cross-section can completely stabilize the capillary instability; cylindrical interfaces longer than their circumference are possible. Furthermore, we have shown that such complete stabilization also exists when the axial motion is generated by thermocapillary-induced stresses. As an application, these results may explain the apparent observation of a very long stable interface (nearly twice the static limit) in a molten indium sample taken from a float-zone experiment in a space laboratory (STS Flight 41D, September 1984).

**Progress to Date:** Our research effort (i) has discovered and mapped out the windows in parameter space where hydrodynamic shear forces can stabilize capillary break-up in long cylindrical interfaces according to linear stability theory, (ii) has designed, built, and tested an apparatus capable of exploring the influence of shear on capillary instability in a parameter range where theory suggests stabilization may occur, and (iii) has developed a simpler analog experimental system, the soap-film bridge, by means of which many of the fundamental influences of motion on stability may be observed and studied. The soap-film bridge has already illustrated details of the rupture phenomenon never before documented. Finally, the stabilization predicted by theory for an isothermally generated shear has led to (iv) an examination of a thermocapillary-generated shear and its influence on an annular film of liquid.

### Publications

Russo, M. J., and P. H. Steen, "Shear-stabilization of the Capillary Breakup of a Cylindrical Interface", Phys. Fluids, 1989 (in press).

Dijkstra, H. A. and P. H. Steen, "Thermocapillary Stabilization of the Capillary Breakup of an Annular Film of Liquid," J. Fluid Mech., 1989 (in press).

Dijkstra, H. A., "The Coupling of Marangoni and Capillary Instabilities in an Annular Thread of Liquid," J. Colloid & Interface Sci., 1989 (in press).

## Presentations

Cryer, S. A. and P. H. Steen, "Capillary Rupture in low Gravity: Soap-film Prototype," *Physico-chemical Hydrodynamics Meeting (PCH-7)*, Boston, Massachusetts, June 25-29, 1989.

Physical Phenomena in Containerless Glass Processing

Clarkson University

Dr. R. Shankar Subramanian

Dr. Robert Cole

NAS8-32944 (NASA Contact: R. Daugherty, MSFC)

December 1977-January 1990

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

*Research Task Description:* At this time, all of the research is ground-based. The research task involves conducting experimental studies of bubble/drop motion in a temperature gradient and in rotating liquid bodies. Supporting theoretical models of ground-based experiments as well as planned flight experiments are also to be developed.

*Progress to Date:* Experiments were conducted on liquid drops migrating in a vertical temperature gradient. The thermocapillary contribution to their velocities was found to be consistent with predictions from theory in scaling linearly with drop radius and applied temperature gradient. Some experiments have been performed on drops migrating normal to a plane surface under the action of an applied temperature gradient.

Some experiments have been performed on the motion of bubbles contained in freely suspended rotating drops. The drops first move to the rotation axis during which period the bubbles are normally found to stay in their relative locations within the drops. However, upon reaching the axis of rotation, the drops reach a rotation speed close to that of the surrounding fluid, and the bubbles are found to migrate inward toward the rotation axis.

#### Publications

H. S. Kim and R. S. Subramanian, "The Thermocapillary Migration of a Droplet With Insoluble Surfactant Part I. Surfactant Cap," J. Colloid & Interface Sci. **127**, 417-28 (1989).

H. S. Kim and R. S. Subramanian, "The Thermocapillary Migration of a Droplet With Insoluble Surfactant Part II. General Case," J. Colloid & Interface Sci. **130**, 112-29 (1989).

R. M. Merritt and R. S. Subramanian, "Migration of a Gas Bubble Normal to a Plane Horizontal Surface in a Vertical Temperature Gradient," J. Colloid & Interface Sci., 1989 (in press).

K. D. Barton and R. S. Subramanian, "The Migration of Liquid Drops in a Vertical Temperature Gradient," J. Colloid Interface Sci., 1989 (in press).

J. S. Ruggles, R. G. Cook, and R. Cole, "Microgravity Bubble Migration in Rotating Flows," J. Spacecraft and Rockets, 1990 (in press).

#### Presentations

K. D. Barton and R. S. Subramanian, "Droplet Motion in a Vertical Temperature Gradient," *AIChE 81st Annual Meeting*, Washington, D.C., December 1988.

P. A. Kondos, R. S. Subramanian, and M. C. Weinberg, "The Influence of an Insoluble Gas on the Dissolution or Growth of a Gas Bubble Inside a Drop in Zero Gravity," *AIAA Aerospace Sciences Meeting*, Reno, Nevada, January 1989.

K. D. Barton and R. S. Subramanian, "Migration of a Liquid Drop in a Vertical Temperature Gradient-Interaction Effects Near a Horizontal Surface," *Physicochemical Hydrodynamics (PCH-7) International Conference*, Cambridge, MA, June 1989.

D. S. Morton and R. S. Subramanian, "Thermocapillary Migration of a Compound Drop," *Physicochemical Hydrodynamics (PCH-7) International Conference*, Cambridge, MA, June 1989.

J. S. Ruggles, D. Brone, R. Cook, and R. Cole, "Bubble and Drop Trajectories in Rotating Liquid Flows," *VIIth European Symposium on Materials and Fluid Sciences in Microgravity*, Oxford, England, September 1989.

*The Study of Electromagnetically-Driven Flow in Aqueous Systems Using Laser Velocimetry*

Massachusetts Institute of Technology

Professor Julian Szekely

David Forrest

NAGW-1762 (NASA Contact: M. Lee, HQ)

*Objectives:* The objective of this research is to develop a numerical model to predict 3-D magnetohydrodynamic (MHD) fluid flow and to test the numerical model against experimental measurements. The experimental design in this proposal is intended to be a prototype for future equipment for Learjet low gravity and Shuttle microgravity experiments. The specific goals of this research are to: (1) develop a numerical model to predict heat flow, velocities, and turbulence in 3-D Cartesian coordinates for MHD flow of an aqueous electrolyte in an enclosed cavity, driven by alternating current passing between two electrodes, (2) build an experimental apparatus to measure MHD flow in this system; the design will be optimized based on initial numerical calculations, (3) measure velocities and turbulence using a laser Doppler velocimeter, (4) compare the computed results with the experimental measurements, (5) determine the suitability of this system for Learjet and Shuttle flights.

*Research Task Description:* The proposed system, with the general design consisting of an applied current between two electrodes of unequal size, is applicable to a variety of materials processing operations with similar geometries: electroslag refining, direct arc electric furnace steelmaking, the Hall-Heroult cell, and electroslag welding. This experimental work will quantify how accurately we can predict transport phenomena in these materials processing operations, and help us to learn how our models can be improved. The use of 3-D Cartesian coordinates will allow us to model complex geometries. The use of a transparent system will allow us to take full advantage of the powerful data acquisition capabilities of laser Doppler velocimetry to better study the turbulence structure.

Future low gravity and microgravity experiments may be necessary if this experiment lacks the ability to discriminate MHD flow. The heat generated when electric current passes through the system causes buoyancy-driven flow, which may mask the MHD flow. This ground-based work will nevertheless provide useful data to show whether the experiment will be appropriate for further measurements in low gravity and microgravity.

*Progress to Date:* Scoping calculations have been performed, the code to calculate the magnetic field from the electric current density field has been written and tested, and the hardware requirements have been established, as well as the choice of an aqueous system--19.53% HCl.

### Ostwald Ripening of Solid-Liquid Mixtures

Northwestern University  
Professor P. W. Voorhees  
Dr. S. C. Hardy, NIST  
H-85025B (NASA Contact: D. Frazier, MSFC)

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

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

*Progress to Date:* We have located a solid-liquid mixture in which the materials parameters required to compare the experimental results to the theoretical predictions are known and developed an experimental protocol necessary to produce a dispersion of solid particles in a liquid. We have examined the coarsening kinetics of solid particles in a liquid in the volume fraction solid range above 0.6 where the development of a solid skeletal structure inhibits particle sedimentation. The experimentally measured coarsening rate constants are found to exceed those calculated from theory by factors ranging from 2 to 5. Possible causes for the disagreement between theory and experiment are the movement of particles within the skeletal structure due to density differences between the solid and liquid phases or convection of the liquid matrix. Only experiments in a micro-gravity environment will eliminate conclusively these possibilities.

The effects of particle motion on the ripening process have been investigated in solidliquid mixtures of lead-tin which have been rotated with the axis of rotation perpendicular to the gravity vector. The experiments show that the effects of convection are strongly dependent on the volume fraction solid employed in the experiments. At volume fractions of solid above approximately 0.7, rotation rates up to 5 revolutions/sec have no effect on the coarsening process. For volume fractions below 0.6, the amplitude of the temporal power law for the average particle radius changes with the rotation rate, but the exponent remains unchanged. Finally, at extremely low volume fractions, the effects of convection become still more dominant and the exponent of the temporal power law for the average particle radius changes from the classical value of  $1/3$  to  $3/8$ . These experiments show that particle motion in a fluid can have a major effect on the ripening process and that the magnitude of the effects of this particle movement is strongly dependent on the stability of the solid skeletal structure.

We are investigating the effects of convection on the ripening process theoretically. In the low Peclet number limit, we have assumed that the particles move in the liquid at a rate which is proportional to  $R^m$  where  $R$  is the radius of the particle and  $m$  is an exponent whose value depends on the particular fluid flow conditions. We find that longtime solutions to the equations defining the ripening process which show an effect of convection are possible only for  $m = -1$ . However, if we assume that the fluid flow rate in the liquid scales with the average particle radius, then solutions to the ripening equations can be found which yield the classical temporal exponents characteristic of a ripening process in the absence of fluid flow. We have determined the amplitudes of the temporal power laws and find, in contrast to the exponents, that they

are a function of the magnitude of the fluid flow in the liquid. These results are qualitatively consistent with the experimental results discussed above of ripening with a moderate volume fraction of solid.

#### Publications

Hardy, S. C. and Voorhees, P. W., "Ostwald Ripening in a System with a High Volume Fraction of Coarsening Phase," Met. Trans. 19A, 2713 (1988).

Voorhees, P. W., "Coarsening in Binary Solid-Liquid Mixtures," Met. Trans. A (in press).

### Drop Coalescence Studies

Vanderbilt University  
Professor Taylor G. Wang  
Dr. A. V. Anilkumar

*Objectives:* The objective of this investigation is to understand the detailed mechanics of the coalescence of liquid drops. The experiments are being conducted in an immiscible acoustic levitator with degassed water as the host medium.

*Research Task Description:* Typically, a quasineutrally buoyant drop of silicone oil mixed with bromobenzene is levitated close to the velocity node of the levitator. A second drop of the same liquid is introduced, and as it slowly seeks the same levitation position, the drops coalesce. Coalescence is delayed until the host film between the drops is completely drained. The final events of film rupture followed by drop coalescence are rapid, and are studied with high-speed video photography (1000 fps).

Following coalescence, the excess surface energy in the coalesced drop is dissipated through shape oscillations. The mode of oscillation depends on diameter ratio of coalescing drops. There is no evidence of induced mixing during the coalescence process.

Currently, efforts are being directed towards studying the dynamics of host film drainage and in particular events leading to film rupture. To achieve this, the host will be seeded with a fluorescent dye and the film will be illuminated with a thin argon ion laser sheet ( $\sim 1\text{mm}$ ) and photographed with high-speed video.

### Presentations

A.V. Anilkumar and T.G. Wang, "Drop Coalescence Experiments," *42nd Annual Meeting of the APS Division of Fluid Dynamics*, November 18-22, 1989, Palo Alto, California.



### Critical Fluid Thermal Equilibration

Dr. R. Allen Wilkinson, NASA Lewis Research Center  
Dr. R. W. Gammon, University of Maryland  
Dr. R. F. Berg, NIST (Gaithersburg)  
Dr. M. R. Moldover, NIST (Gaithersburg)  
Dr. J. Straub, Technical University of Munich (FRG)

*Objectives:* This experiment will examine the thermal relaxation and the fluid density profile as a function of time after a temperature perturbation of sulfur hexafluoride (SF<sub>6</sub>) near its liquid-vapor critical point in the low-gravity environment of the Shuttle. Past low-g critical fluid experiments yielded unexpected results which were perhaps due to unanticipated time scales for relaxation dynamics. Future critical fluid experiments will depend on achieving thermal equilibrium to within a specified tolerance and on knowing how phases develop or disappear. This work is intended to determine the practical time scale needed to execute meaningful critical fluid space experiments and characterize the location and dynamics of density or phase domains within the sample.

*Research Task Description:* The experiment timeline allows observation of large phase domain homogenization without and with stirring right after thermostat installation in the Critical Point Facility (CPF), time evolution of heat and mass after a temperature step applied to one-phase equilibrium sample, phase evolution and configuration upon going two-phase from one-phase equilibrium, effects of stirring on a low-g two-phase configuration, two-phase to one-phase healing dynamics starting from a two-phase low-g configuration, and quantifying the mass and thermal homogenization time constant of a one-phase system under logarithmic temperature steps. Two Critical Fluid Thermal Equilibration (CFTE) test cells will be developed to be flown in the European Space Agency's CPF, which is manifested on IML-1 for December 1990 launch. During the full experiment, accelerometry data, time correlated with the video records, will identify the compressible fluid dynamics associated with Shuttle acceleration events and provide the investigators with intuition about gravity effects in a non-vibration isolated Shuttle environment.

*Progress to Date:* The CFTE was initiated and will ship flight hardware cells within calendar year 1989. Pancake shaped fluid constant volume cells have been designed and tested for structural and thermal performance, 100 atmosphere pressure leak tests, and launch vibration qualification. Considerable optical design discussion has been carried out with ESA and its subcontractors to assure the sapphire window thickness was correct to allow the imaged object plane to be located in the 1 mm thick fluid. A unique magnetic stirrer was developed to allow stirring of the 0.09 cm<sup>3</sup> fluid volume. This will guarantee being able to reach a homogeneous one-phase state within the experiment time-line of 60 hours.

### Publications

"Critical Phenomena in Microgravity Environments", Bull. Am. Phys. Soc. **34**, 718 (1989).



#### 4. BIOTECHNOLOGY



Protein Crystal Growth in Low Gravity

Stanford University, Center for Materials Research

Robert S. Feigelson

Robert C. De Mattei

(January 1989 to present)

Previous grant (NAG 8-489) ended 3/31/88 and present grant

(NAG 8-774) started 4/27/89

*Objectives:* The objective of this research is to study the effect of low gravity on the growth of protein crystals and those parameters which will affect growth and crystal quality. The proper design of the flight hardware and experimental protocols are highly dependent on understanding the factors which influence the nucleation and growth of crystals of biological macromolecules. Thus, the primary objective of this research is centered on investigating those factors and relating them to the body of knowledge which has been built up for "small molecule" crystallization. This data also provides a basis of comparison for the results obtained from low-g experiments.

*Research Task Description:* The main component of this research program is the study of mechanisms involved in protein crystallization and those parameters which influence the growth process and crystalline perfection. Both canavalin and lysozyme are being used as the basic model proteins in these studies. Other biological macromolecules such as isocitrate lyase have been included in this research program when they provide an opportunity to better understand the nature of the crystallization process. The program involves four broad areas:

- (1) The application of both classical and novel chemical and physical techniques to study the fundamentals of protein crystallization. Included in this area are the study of the phase relationships in the systems of interest, primarily the factors controlling solubility, the study of growth kinetics to determine the growth rate controlling mechanism and the relevant activation energy involved in the process. The effects of fluid flow on the growth and perfection of protein crystals will be studied using flow visualization techniques. The use of electrochemical techniques to monitor and/or control crystallization will be studied also. The effects of applied voltages on nucleation and growth are not known nor is the magnitude of the potentials which may develop on the crystal during growth.
- (2) Characterization of protein crystals. Optical microscopy will give a general evaluation of crystal morphology, size and perfection. Phase contrast techniques will give enhanced contrast to the surface features allowing observation down to the  $0.1 \mu$  level. For more detailed surface imaging the application of Scanning Tunneling Microscopy and Atomic Force Microscopy to protein crystals will be investigated. To study the defects occurring in the bulk of the crystals, that applicability of Synchrotron x-ray topography will be studied. The characterization studies will be attempting to associate the defects in protein crystals with the growth conditions to develop insights for growing crystals of greater perfection.
- (3) Control of nucleation and growth. The information developed in the phase relationship studies of section (1) will be used to design experiments to separately control the nucleation and growth processes. The information from section (2) will be used to optimize the growth.
- (4) The design and construction of a prototype of space flight hardware. The design will incorporate the results of section (3) and will be instrumented to gather the types of data that have been acquired in the ground based studies.

*Progress to Date:* The analysis of the flows around growing crystals has been expanded. In a previous report it was noted that flows had been observed around growing crystals of Rochelle salt, lysozyme and canavalin using the schlieren imaging technique. The values for the change of density and index of refraction with change of concentration for each system were also reported. Using these values, the Grashof number (the ratio of buoyant force to viscous drag) and the local change in light intensity for each system have now been calculated under the experimental conditions. The values of the Grashof number are 12.07 for Rochelle salt, 0.36 for lysozyme and 1.06 for canavalin. If the Grashof number is normalized by fixing the size of the crystals at 1mm, then the Grashof numbers become 1.76 for Rochelle salt, 0.11 for lysozyme and 0.14 for canavalin. There is a much lower tendency for the protein solutions to experience convection under the conditions of these experiments even when size considerations are taken into account, but flow does occur in all these systems. The values found for the local change in intensity (an indication of whether images will be seen) are 21.7 for Rochelle salt, 2.93 for lysozyme and 4.39 for canavalin. Based on the image quality of the films used in this study, the value of 2.39 appeared to be near the limit of detectability in our system (400ASA film, f2.8, 1/51 sec exposure).

A study of the growth behavior of isocitrate lyase has recently begun. This is a joint project with Du Pont. When grown by the hanging drop method on earth, the crystal grows in a manner such that the corners grow out rapidly and the crystal quality is poor. Crystals grown in space however are equiaxed. Crystals of this material have been grown in our laboratory by the hanging drop method. The morphology duplicates that of those produced at Du Pont. The growth rates are measured and, consistent with the morphology, the growth rate of the corners is initially high (1.8 microns/min) while that across the face center of the crystal is about 0.7. The growth rate of the corners remains above 0.2 microns/min for at least 1100 minutes while the face velocity drops essentially to 0 after 300 minutes. In order to test whether fluid flow was responsible for this unstable growth, a vapor equilibrium cell was built to allow flow visualization. The cell volume was 20  $\mu$ l (5 times the volume of the 1-g hanging drop, 0.5 the volume of the space experiment). The cross section was 3mm x 1mm giving a much smaller surface area for equilibration than the hanging drops. The crystals grown in this cell were equiaxed with a similar to the space grown crystals. No flow data has been collected yet.

Preliminary results show that the morphology of the isocitrate lyase crystals is not due to the sedimentation effect. Morphological stability seems to be related to the rate of equilibration of the protein solution and the crystal growth rate. Baird's work shows that hanging drops equilibrate faster in 1-g than in space and the reduced surface area of the flow cell guarantees a slower equilibration. This is borne out by the lower nucleation and growth rates observed in the flow cell.

Preliminary controlled nucleation experiments using lysozyme have been conducted. These initial experiments used a small, temperature controlled spot to induce nucleation at a fixed position and to limit the number of nuclei produced. These experiments used lysozyme (20 mg/ml, pH 4.0, 0.1M sodium acetate, and 4 % sodium chloride). This solution will spontaneously nucleate in 4-5 days at room temperature.

By using a cold spot temperature of 9 C, nucleation was accomplished in 5 hours. The number of nucleation was less when compared to the isothermal solutions, but they were not localized to the extent anticipated.

The results of the preliminary localized nucleation experiments has led to the design of the first prototype space flight hardware. This design incorporates a more sophisticated localized temperature gradient control as well as a means of controlling the ambient temperature around the growth cell as an aid to localizing the nucleation as well as a means of controlling subsequent growth. The apparatus also has provisions for in situ microscopy, the inclusion of schlieren optics, and light scattering to detect the onset of nucleation. This apparatus is currently under construction.

### Publications

C. C. Young, R.C. DeMattei, R. S. Feigelson and W. A. Tiller, "Some Implications of Colloid Stability Theory for Protein Crystallization," J. Cryst. Growth **90**, 79 (1988).

R. S. Feigelson, "The Relevance of Small Molecule Crystal Growth Theories and Techniques to the Growth of Biological Macromolecules," J. Cryst. Growth **90**, 1 (1988)

R. C. DeMattei and R. S. Feigelson, "Growth Rate Study of Canavalin Single Crystals," J. Cryst. Growth, 1989 (accepted).

R. C. DeMattei and R. S. Feigelson, "Solubility of Canavalin," *The Third International Conference of the Crystal Growth of Biological Macromolecules*, August 13-19, 1989, Washington, D.C.

R. C. DeMattei and R. S. Feigelson, "Preliminary Comparisons of Buoyancy Driven Flows Around Growing Crystals in Inorganic and Biological Systems-Rochelle Salt, Lysozyme and Canavalin," to be published.

### Cell Partition in Aqueous Phase Systems

University of Alabama in Huntsville  
Dr. J. Milton Harris  
NA58-35362 (NASA Contact: R.S. Snyder, MSFC)  
July 1987 - July 1990

*Objectives:* The goals of this work are to understand and control low-g demixing of two phase systems formed by solution of pairs of polymers (generally dextran and polyethylene glycol) in water and then to use these systems to purify cells by partitioning. The immediate objectives have been to control demixing by controlling wall wetting with covalently-bound polymer coatings and to develop chemistry necessary for performing this function for affinitive partitioning of cells and proteins.

*Progress to Date:* As chemists, we have worked on improving the chemistry used to couple polymers to glass (for control of wall wetting) and to proteins (for affinity purification of cells). This work has focused primarily on synthesis of new aldehydes of polyethylene glycol (or PEG). We have recently completed synthesis and characterization of PEG-benzaldehyde and PEG-propionaldehyde. The benzaldehyde is quite a stable compound and shows good reactivity toward amino groups on glass surfaces. However, it is relatively unreactive toward amino groups on proteins. On the other hand, the propionaldehyde is much more reactive toward amino groups in general. Also this compound is stable toward moisture (unlike any other activated PEGs). Thus we feel the PEG-propionaldehyde may be the ideal activated PEG. Further work with this compound is continuing. In two related works we have developed a new proton NMR technique for characterizing PEGs and we have prepared PEG-picric acid, which appears to have utility for measuring the number of amino groups substituted in a protein (useful in affinity phase partitioning).

It is important when covalently linking polymers (such as dextran and PEG) to glass that good methods for aminating glass be available. We have recently improved glass amination by silination by developing a procedure which utilizes dry aprotic solvents at high temperatures. This procedure is reproducible (unlike other procedures) and it gives a high density of amino groups that are strongly coupled to the surface.

In a related work we have begun study of coupling chemistry poly (methylmethacrylate) (or Plexiglas). This material is very useful for constructing partitioning apparatus, and it would be quite useful if we could effectively couple our polymers to this material. Our initial studies indicate that basic hydrolysis of the material is effective in producing carboxylate groups that can then be converted to amines. Work is proceeding in this area.

### Publications

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J. M. Dust and J. M. Harris, "The picryl ether of polyethylene glycol," J. Polym. Sci. Poly Chem. Edn., 1989 (in press).



J. F. Boyce, B. A. Hovanes, J. M. Harris, J. M. Van Alstine, and D. E. Brooks, "The wetting behavior of aqueous two-phase polymer test systems on dextran coated glass surfaces: Effect of molecular weight," J. Colloid and Interface Sci., 1989 (submitted).

## Separation of Chromosome-Size DNA Molecules

University of Pennsylvania  
Professor Ponzy Lu  
Dr. Young Cho, Drexel University,  
Dr. Lee Silver, Princeton University  
NAG 8-748 (NASA Contact: R. Snyder, MSFC)

*Objectives:* This project addresses the problem of DNA separation by gel electrophoresis. Since we are interested in DNA molecules  $10^6$ - $10^8$  base pairs in length ( $10^9$ - $10^{11}$  daltons), or molecules that are polyanions of 2 nm diameter with lengths of millimeters to many centimeters which distort as they move through the electrophoretic matrix, existing theories and models for electrophoretic transport processes must be modified.

*Research Task Description:* This effort will complement parallel national efforts to map and sequence the entire human genome. There are currently limitations in the human genome project which can be addressed by the technologies to be developed by this research project. Even if the human genome project is completed using existing methods, the technology to be developed here will extend the benefits of those efforts to all biological species, including those used as models of human disease and for agriculture and industry.

This project is an extension of NASA support of electrophoretic separation process development over the past decade. The extrapolation of currently employed methods for DNA separation suggests the use of more dilute electrophoretic media to allow larger porosity and consequently shorter reorientation times of the polymer subjected to pulsed electric fields. The only method to maintain fluid stability in the presence of chemical and physical density heterogeneity is the microgravity environment. Since the separated DNA will be used for genetic analysis, this program will keep space science applications at the cutting edge of biotechnology.

The investigations follow that outlined in the original proposal with emphasis on identifying the microgravity component. Construction of the apparatus to directly observe the orientation of DNA molecules during the electrophoretic process is complete. The electronic and optical components for this instruments are in the laboratory at the Department of Chemistry, and is now being put through calibration experiments.

We have modeled the motion of DNA in Newtonian and non-Newtonian fluids using thin wire as a mechanical model. The results were unexpected and suggest the major experiments for the next 12 months. The findings show that the wire falls end first, axis vertical, in the non-Newtonian fluid and longer wire falls faster. In the Newtonian fluid all wire lengths fall at the same rate, horizontally. This latter observation parallels the observations of Olivera, Baine and Davidson [Biopolymers 2, 245-257 (1964)] where DNA of all length move at identical velocities in free Newtonian fluids in an electric field.

During the second grant year, beginning October, 1989, we plan to electrophorese DNA in a vertical fluid column, stabilized by a density gradient on Earth. In this gradient, un-crosslinked macromolecules will be dissolved which will orient around moving DNA or in the field. The intention is to set up a medium where DNA will move along the helix axis, similar to the wire in a non-Newtonian fluid. The actual orientation of the DNA will be monitored using the apparatus built to measure fluorescence polarization.

It is anticipated that conditions can be found where longer DNA will move faster than shorter DNA. Since this is non-crosslinked system, the need for microgravity to prevent convection becomes obvious. Given the length of naturally occurring DNA, the dimensions of the electrophoresis system would need to be measured in meters rather than centimeters making Earth bound stabilization by density gradient less

useful, if not useless. An absolute requirement for convection free fluid is necessary for resolution in the separation process.

We have also examined the initial step of the separation process for gel electrophoresis of chromosome-sized DNA molecules. This requires the liberation of chromosomes from the cell and subsequent removal of proteins which condense the molecule geometrically. For the DNA molecules of interest to this project, this entire process must be done in the absence of hydrodynamic shear forces which break the long thin DNA molecules. Current methodology uses low temperature melting agarose to immobilize the cell as reagents are diffused to this cell for lysis and chromosome deproteinization procedure. With this method, chromosomes from more complex eukaryotes such as mammals do not liberate DNA which will electrophore into the gel matrix unless nucleases are also used to break the DNA. As an alternative matrix for the initial DNA liberation process, glass bead material described in the original proposal was explored. Experiments are in progress to establish that megabase lengths of DNA are indeed liberated into gels in the presence of glass beads of 170-200 $\mu$  diameters.

#### Publications

Kim, I. and Cho, Y., "The Hydrodynamics of Bent Cylinder in Viscoelastic Fluid," J. Non Newt. Fluid Mech., 1989 (in press.)

Kim, J. and Cho, Y., "The Hydrodynamics of a Thin Cylinder Falling in a Viscoelastic Fluid," J. Fluid Dynam. (submitted).

## Nucleation and Growth Control in Protein Crystallization

University of Alabama in Huntsville  
Center for Microgravity and Materials Research  
Professor Franz Rosenberger  
Dr. Edward Meehan  
Dr. Thomas Nyce  
NAG8- 711 (NASA Contact: R.S. Snyder, MSFC)  
March 1988 - March 1990

*Objectives:* The inability to routinely grow crystals of sufficient size and quality is considered the major bottleneck in the further widespread development of the field of protein crystallography. By comparison with inorganic crystal growth, the fundamental understanding of the dynamics and kinetics of protein crystallization is still rather rudimentary. Correspondingly, the existing protein crystal growth technology provides only for limited control of essential growth parameters such as supersaturation and, consequently, the number and timing of nucleation events, and the growth rate. Most protein growth experiments to-date result in a large number of small crystals. In low gravity growth experiments these uncontrolled growth conditions can become somewhat mitigated. The reduction of buoyancy-driven, solutal convection reduces the probability for concurrent nucleation in the vicinity of initially formed crystallites. This may be one of the reasons for the increase in size and decrease in the number of protein crystals that have been obtained in space.

The objectives of this research program are: (a) contributions to the understanding of the dynamics and kinetics of protein crystallization in general; (b) the determination of the advantages and drawbacks of using temperature as the control parameter in protein growth; and (c) the development of a semi-automated protein crystal growth technology that excludes further nucleation after select initial nucleation or seeding, and provides for the controlled, non-interfering growth of the initial nuclei or seeds.

*Research Task Description:* Towards these objectives we are conducting (a) nucleation and growth experiments with lysozyme and canavalin solutions at low salt concentrations in temperature-programmable, optically accessible growth cells, in which information on nucleation and growth events is obtained via microscopic observation and a simple light scintillation and photodetection arrangement; (b) phase diagram work with lysozyme and canavalin; (c) studies of protein growth and dissolution kinetics and morphology as a function of supersaturation through temperature variation using interferometric microscopy with image storage and processing; and (d) model calculations on the diffusive transport dynamics of the hanging drop crystallization technique.

*Progress to Date:* The feasibility of restricting nucleation and growth to a cold spot in a thermostated growth cell has been demonstrated with lysozyme solutions. Furthermore, we have shown that the initially large number of nuclei, that results at the high supersaturations required to drive nucleation in protein solutions in general, can be drastically reduced by temperature cycling. The microscopic observations associated with these studies revealed that temperature changes must be limited to certain rates if damage to seeds is to be avoided. In addition, we have demonstrated that faceted morphologies of crystallites can be retained under slow enough, thermally controlled dissolution. This condition is important for the use of seeds for later growth with low defect density.

Using a simple laser light scintillation and photodetection setup, we have demonstrated that (a) the formation of crystallites can be detected at an early stage of growth run, and (b) the solubility of protein can be determined through appropriate temperature programming much more rapidly than with traditional batch methods. Both the growth cell and scintillation cell can be miniaturized to become applicable to proteins less abundantly available than lysozyme.

The phase diagram work (solubility vs. temperature, salinity and pH) with lysozyme has revealed that the occurrence of the orthorhombic and tetragonal form is not solely determined by temperature and pH, but rather a combination of these parameters and ionic strength. In a few cases, however, it was found that the same combinations lead to either polymorph. Hence, there is likely some other, unidentified control parameter involved.

In preparation for the design of the in-situ optical growth kinetics studies we have found that surface morphological features, such as growth and etching steps, on protein crystals inside layers of solution can be observed with great image quality employing brightfield and interferometric microscopy.

The transport model calculations for the hanging drop technique have shown that, depending on the diffusivity (i.e. size) of the protein involved, significant protein concentration gradients can develop in the drop under purely diffusive conditions, whereas the drop will remain well mixed with respect to the salt concentrations.

#### Publications

Fehribach, J. D. and Rosenberger, F., "Analysis of Models for Two Solution Crystal Growth Problems", J. Cryst. Growth, 1988 (in press).

Nyce, T. A. and Rosenberger, F., "Growth of Protein Crystals Suspended in a Closed Loop Thermosyphon", J. Cryst. Growth (accepted).

## The Fluid Mechanics of Continuous Flow Electrophoresis

Princeton University

Dr. D A. Saville

NAG 8-759 (NASA Contact: R. Snyder, MSFC)

August 1986-September 1989

*Objectives:* The work is a collaboration between Princeton and MSFC with the overall objective of establishing theoretically and confirming experimentally the ultimate capabilities of continuous flow electrophoresis chambers operating in an environment essentially free of particle sedimentation and buoyancy. The efforts at Princeton are devoted to: (i) improving the extant mathematical models to predict flow and particle trajectories in the apparatus conceived at MSFC and (ii) a study of the effects of particle concentration on sample conductivity and dielectric constant. The dielectric constant and conductivity have been identified as playing crucial roles in the behavior of the sample and, thus, on the resolving power and throughput of continuous flow devices.

*Research Task Description:* Earlier work at Princeton (under contract NAS8-32614) showed that particle concentration has a strong influence on sample conductivity (more specifically, on the "real" or "dc" conductivity). Recent work has shown that the dielectric constant (the "imaginary" part of the complex conductivity) is similarly affected. Concurrently, experimental studies by P.H. Rhodes at MSFC showed that sample conductivity influences the spreading of the sample. A large conductivity mismatch between sample and buffer causes sample to spread rapidly from the front to rear walls of the channel. Rhodes developed an electrohydrodynamic theory of this spreading which shows that in addition to the conductivity, the dielectric constant should also affect spreading behavior. To optimize performance of a continuous flow device, it will be necessary to understand the spreading process and, therefore, how it is influenced by the conductivity and dielectric constant. Accordingly we need to be able to measure the dielectric behavior of the sample as well as the DC conductivity. The Princeton effort consists of 3 tasks:

1. Extension of existing mathematical models of flow and temperature fields in continuous flow chambers.
2. Development of a device to measure the conductivity and the dielectric constant of a suspension.
3. Theoretical work to relate buffer and sample compositions to conductivity and dielectric constant.

*Progress to Date:* Progress has been excellent and we can now measure the properties of a given suspension at frequencies between 500 Hz and 200 KHz. A complete frequency scan takes about 25 minutes. The instrument has been used to measure the dielectric constant and conductivity of latex suspensions similar to those used in Rhodes experiments. The results are linear in the volume fraction of particles for dilute systems ( $< 8$  vol%) as they should be. One surprising result is that the low frequency limit is much larger (over 800 for a 4% by volume suspension) than the extant theory predicts. In addition, the relaxation frequency is much lower than predicted by the theory.

Our results have been reported at several technical meetings and two papers published in the Journal of Colloid and Interface Science. Inasmuch as this is the first time that the theory for the low frequency behavior has been subjected to a rigorous test, our results have attracted considerable attention in the colloid science community.

It follows from these results that the effect of the dielectric constant on the sample's configuration may be even larger than anticipated. Clearly, it is of crucial importance to understand these phenomena if electrophoresis is to be used to fractionate cells or cell fragment populations.

Recent efforts have focussed in two areas: building up our experience base with the dielectric spectrometer and theoretical work on the mobility of particles in concentrated suspensions. On the experimental side we have now studied the properties of two types of synthetic latex particles, amphoteric and anionic particles, in a variety of electrolyte solutions. The device performs well and we continue to measure low frequency dielectric constants much larger than the classical electrokinetic theory allows. Two papers have now appeared on our initial work with the spectrometer.

Earlier work showed that particles undergoing electrophoresis in concentrated suspensions behaved as if they were isolated. The first part of a theoretical study of the phenomena has been completed.

#### Publications

C. F. Zukoski and D. A. Saville, "Electrokinetic Properties of Particles in Concentrated Suspensions: Heterogeneous Suspensions," J. Colloid Interface Sci., in press.

D. F. Myers and D. A. Saville, "Dielectric Spectroscopy of Colloidal Suspensions I. The Dielectric Spectrometer," J. Colloid & Interface Sci. **131**, 448-460 (1989).

D. F. Myers and D. A. Saville, "Dielectric Spectroscopy of Colloidal Suspensions II. Comparisons Between Theory and Experiment," J. Colloid & Interface Sci. **131**, 461-470 (1989).

A. Acrivos, D. J. Jeffrey, and D. A. Saville, "Particle Migration in Concentration Suspensions: Thermophoresis and Electrophoresis," J. Fluid Mech., in press.

#### Presentations

Saville, D. A. and Rosen, L., "Dielectric Spectroscopy of Colloidal Dispersions: A Test of the Classical Theory," *Annual Meeting of the Colloid and Surface Science Division of the American Chemical Society*, Seattle, 1989.

## Electrophoresis Technology

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

*Objectives:* The objectives of this program are to: (1) analyze the fluid flow and particle motions during continuous flow electrophoresis by experimentation and computation; (2) characterize and optimize electrophoretic separators and their operational parameters; (3) develop innovative methods to accomplish electrophoretic separations in space; and (4) analyze the electrophoretic process using apparatus that has been characterized or modified to perform in a predictable manner and according to procedures that have been developed to yield improved separation.

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

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

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

A new electrophoresis-type test chamber has been built and put into operation. This chamber has the feature of two dimensional observation of the sample stream as well as observation of the sample stream cross section. The cross section is observed by means of a cross section illuminator which assures that the sample stream distortions can be accurately recorded.

*Progress to Date:* Through analysis and experiments, it has been found that sample stream distortion is initiated by variations in conductivity and dielectric constant between the sample stream and the buffer medium. While the effect of conductivity variations is clearly understood, the dielectric behavior of the



colloidal suspensions which make up the sample stream is not at all clear. Indeed, a large discrepancy exists between the published experimental dielectric data and classical theory.

A method has been developed to experimentally determine the dielectric constant of colloidal suspensions by observing the distortion of sample streams in the experimental chamber. These studies will have a direct influence on the understanding of sample stream distortion in continuous flow electrophoresis chambers.

#### Publications

Rhodes, P. H., Snyder, R. S., and Roberts, G. V., "Electrohydrodynamic Distortion of Sample Streams in Continuous Flow Electrophoresis," J Colloid & Interface Sci. 129, 78-90 (1989).

Roberts, G. O., Rhodes, P. H., and Snyder, R. S., "Dispersion Effects in Capillary Zone Electrophoresis," J. Chromato. 480, 35-67 (1989).

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*Growth of DNA Crystals in a Microgravity Environment*

University of Pennsylvania  
Dr. Donald Voet  
J. LaLonde  
NAG8-770 (NASA Contact: K.D. Sowell, MSFC)  
July, 1989 - June, 1991

*Objectives:* The objective of the research is to determine the x-ray structure of several large segments of double stranded DNA. To this end it is necessary to crystallize the DNA. Since DNA is difficult to crystallize in a form suitable for x-ray diffraction, we are proposing to study the effects of microgravity on DNA crystallization.

*Research Task Description:* We intend to synthesize and crystallize several non-self complementary segments of B-DNA in the range 12-20 base pairs. The crystals, when obtained, will be used to determine the x-ray structure of the DNA's in order to establish how the DNA conformation varies with its base sequence.

*Progress to Date:* We have synthesized seven different species of non-self-complementary double stranded DNA ranging in size from 12-20 base pairs. We have developed new methods of purifying these substances and have crystallized three of them: (1) a 12-base pair segment which may be suitable for x-ray analysis; (2) a 20-base pair segment which is yet too small for x-ray analysis but which we are hopeful of growing larger, and (3) an 18-base pair segment. The 12-base pair segment will be tested for crystallization under microgravity in STS-32.

## **5. GLASSES AND CERAMICS**



Powder Agglomeration in Microgravity

Ohio State University  
Dr. James D. Cawley  
NAG3-755 (NASA Contact: D. Fox, LeRC)  
November 1986 - November 1989

*Objectives:* The population of agglomerates in a powder suspension strongly affects rheology. The on-going research is designed to extend our understanding of the mechanistic of the agglomeration process through both experimentation and numerical simulation. The concepts of fractal geometry, which have been applied to a wide range of forms and objects including colloidal aggregates, are explored. In the experimental study, alumina aggregates are being analyzed to determine the suitability of a fractal dimension as quantitative measure of agglomerate structure. In earth based experimentation, the aggregation process is constrained to a two-dimensional surface oriented such that it is perpendicular to the force or gravity. Real time observation of the agglomeration process is used to identify the dominant growth mechanism and comparison has been made to existing numerical models. In a separate effort, a numerical model of colloid aggregation has been developed to investigate the screening behavior in terms of biasing growth about the outer periphery of an agglomerate. Experimental and simulated aggregate growth studies have indicated that an inner core screening of growth particles leads to fractal structures.

*Research Task Description:* The experimental Set up consists of a buffer solution in which 0.001 volume percent of  $0.4 \mu\text{m}$  in diameter alpha alumina powder is suspended. The buffer solution of HCl tris(hydroxymethyl) amine, and de-ionized water is adjusted to a pH of 8.2 which is the approximate isoelectric point as determined from microelectrophoresis. Well dispersed drops of suspension are confined to a small hole in an aluminum slide placed on an inverted optical microscope. The particles aggregate on the air-water interface at the bottom of a drop thus restricting growth to two dimensions. The low volume concentration of alumina results in a very low collision probability in the bulk of the drop and virtually all agglomeration occurs at the interface. The numerical approach Consists of ballistic growth model modified to include inner core screening by excluding interior growth on a length scale related to the radius of gyration. The model approximates fluid flow around the agglomerate as two dimensional fluid flow around a cylinder having a radius of scaled from 0.72 to 1.2 of the radius of gyration of the growing agglomerate. Particles are released randomly about an aggregate and constrained to a streamline of constant potential until contact. To examine the effects of particle anisotropy, doublet and triplet particle configurations are used as penetrating growth particles. The effect of particle anisotropy on aggregate restructuring is also investigated in two dimensional sedimentation simulation experiments in which aggregate structures are allowed to settle to an interface under varied levels of gravitational force, but constant interparticle force.

*Progress to Date:* An M.S. research program directed at quantitative analysis of the experimental alumina agglomerates has been completed. The average fractal dimension,  $D$ , of the alumina aggregates analyzed to date was determined to be 1.5. This value was calculated from an equal weighting of the results of analyzing the data using three independent methods: averaged two point density correlation function versus radial distance  $\langle D \rangle = 1.53$  count versus radius of gyration,  $D = 1.51$ ; and averaged count versus radial distance,  $\langle D \rangle = 1.47$ . Direct observation of alumina aggregation indicate that diffusion-limited aggregation (DLA) is a realistic model for the growth process. Despite the constant flux of singlets to the lower surface, cluster-cluster collisions resulting from Brownian Motion are observed to be principally responsible for growth. The fractal dimension resulting from analysis of computer simulations, by others workers, based on cluster-cluster aggregation is in fair agreement with the experimental values.

The results of the modified ballistic numerical simulation have indicated that, although much more ramified clusters result, the scaling behavior is very similar to the simple ballistic aggregation. The fractal dimension of 10,000 particle aggregates generated from modified ballistic growth was equivalent to the Euclidian dimension as is the case with simple ballistic growth. The fractal dimension was insensitive to the

scaling factor used in the calculation of the hydrodynamic radius while the overall geometry was very sensitive such that increasing the effective hydrodynamic radius lead to more pronounced branching. The effect of particle anisotropy on aggregate structure was negligible in the modified ballistic growth and low gravity sedimentation simulations. The influence of particle shape in a high gravity situation was seen in the higher coordination and denser packing of singlets over triplets.

Study of Phase Separation in Glass in Reduced Gravity

NASA Lewis Research Center  
Mark J. Hyatt  
In-Center

*Objectives:* The objectives of this research are to study the fundamental aspects of stable phase separation in glass forming oxide melts, determine the effect of gravity on the final microstructure of the phase separated glass, and observe the effect of microstructure on the physical properties and crystallization behavior of the glass. A research program embodying these objectives was proposed in response to NASA AO No. OSSA-4-88.

*Research Task Description:* The proposed experiment involves obtaining direct evidence of phase separation and the controlling mechanism by observing the change in properties of a containerless glass sample as phase separation occurs. Ground based research in support of the flight experiment includes phase equilibria studies and physical property measurements of the model glass systems chosen for the flight experiment.

*Progress to Date:* The proposal submitted was not selected for support as flight experiment. Ground based work continued with glass formation and phase separation studies, and measurement of critical physical properties in the model glass systems. Physical properties measured for lead and barium borate glasses include viscosity, surface tension, and glass melt density. The phase equilibria study of the ternary bismuth borosilicate system was completed. Crucible contamination and melt volatilization issues in this system were also studied.

Use of Microgravity to Improve the Efficiency and Power Output of Ni-Doped Laser Glasses

University of Missouri-Rolla

Dr. Chandra S. Ray

NAG8-779 ((NASA Contact: E. Ethridge, MSFC)

July 1, 1989 - June 30, 1991

*Objectives:* The efficiency and power output of Nd:glass laser increases initially with increasing concentration of  $\text{Nd}^{3+}$  ions in the host glass and then decreases after a critical concentration is reached. This effect, known as concentration quenching, occurs when the mean separation between two neighboring Nd-ions becomes less than a critical value  $d_c$ , and sets an upper limit to the Nd-ion concentration in a laser glass. At a mean ion separation,  $d < d_c$ , the effect of ion-ion interaction which decreases the ratio for radiative to nonradiative transitions, becomes important and deteriorates the overall laser properties of the glass. The presence of impurities in the glass also causes a similar effect which is called impurity quenching.

Glasses prepared on earth are reported to contain microheterogeneous regions which can cause the mean ion separation in a laser glass to reach the critical value,  $d_c$ , at Nd-ion concentration much less than what is needed to reach  $d_c$  when the ions are homogeneously distributed. Glasses prepared in microgravity are reported to be more chemically homogeneous and to contain fewer impurities than glasses prepared on earth. The major objectives of this investigation are to, therefore:

- 1) increase the concentration of  $\text{Nd}^{3+}$  ions in various glasses prepared in microgravity before concentrations quenching becomes important, thereby, preparing laser glasses with increased output power per unit volume,
- 2) decrease the threshold energy (minimum energy required to start laser action) and further increase the output power by suppressing or eliminating impurity quenching in glasses processed without container,
- 3) compare selected properties such as fluorescence efficiency (ratio of input to output energy), fluorescence lifetime and bandwidth, and crystallization kinetics for the laser glasses prepared in microgravity and on earth.

The anticipated results from this study will provide quantitative data showing the extent to which the power efficiency is increased and the threshold energy is decreased for laser glasses prepared in microgravity. The practical use would be to take advantage of the higher compositional homogeneity and chemical purity for glasses prepared in microgravity to produce laser glasses that would yield more output power per unit volume than the glasses melted on earth.



## Crystallization of Glass

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

*Objectives:* The objectives of this research are: (1) to study bulk (homogeneous) crystal nucleation in lithium diborate and other simple glasses; (2) to assess the viability (and desirability) of performing such nucleation experiments in a containerless facility in space; and (3) to provide accurate methods of computing the volume fraction crystallized in complex systems and/or systems of small particles; (4) to explore the factors which prevent glass formation near the extreme compositional edge of glass-forming systems.

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

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

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## **6. COMBUSTION SCIENCE**



The Combustion of Free or Unsupported Fuel Droplets at Low Gravity

Cornell University  
Professor C. Thomas Avedisian  
NAG3-987 (NASA Contact: J. Haggard, LeRC)  
February 17, 1989- September 1, 1989

*Objectives:* The objective of this work is to study the combustion of small (initial diameters less than about  $500\text{ }\mu\text{m}$ ) free or unsupported fuel droplets in a low gravity environment. The information obtained consists of a high speed photographic record of the droplet burning process from which the evolution of droplet and flame diameters are measured. Additional information concerning extinction, sooting, flame structure, and microexplosion is extracted from the movie record. Single component and multicomponent fuels are being studied. The results will be compared with on-going related experiments using larger droplets to ascertain the influence of initial droplet diameter on the phenomena observed.

*Research Task Description:* The experiments are being carried out in a small scale (7.6m) drop tower. The free-fall period of the instrumentation package in this tower is about 1.25s, which is a long enough time that the complete droplet burning history can be recorded for the small droplets being studied. The experiments are initiated by simultaneously releasing the instrumentation package to which the photographic equipment is attached and test droplet into free-fall. During the period of the fall the droplet burning history is recorded by two on-board cameras: a 16mm cine camera and a video camera.

*Progress to Date:* Improvements in four aspects of the experiment were made: the method of ignition, the droplet generator, recording the burning history, and magnet deactivation. Also, new measurements of the evolution of gravity in the instrumentation package were obtained with an improved data acquisition system.

A new ignition system was constructed and tested which consisted of two pairs of electrodes which simultaneously retract after being activated. The use of multiple sparks is believed to create a more symmetrical ignition, while electrode retraction removes the electrodes from the region that the droplets burn thus eliminating possible disturbances from these electrodes on the droplet burning history. The droplet generator was redesigned to allow for removal of liquid fuel from the combustion chamber during the set-up procedure to minimize fuel vapor accumulation in the vicinity of the generator nozzle. A second (video) camera was mounted on the package. This camera allows observation of the burning process during an experiment and permits more precise positioning of the droplet during set-up. Improved backlighting permitted both the droplet and flame to be observed for heptane droplets. Finally, a means of more precisely defining the magnet delay time was found: separation of the instrumentation package from the magnet coincided with a current surge in the magnet.

The above improvements were first tested using suspended droplets, and then free-droplets were studied. The suspended droplet diameters were initially about 1.1 mm equivalent diameter. Results showed that during the first .6s of burning flame shapes were nearly spherical and then gradually became distorted from this shape because of the increase in gravity within the moving frame of reference due to air drag on the instrumentation package. The smaller droplets studied in the free droplet experiments burned in less than .4s. The free-droplet experiments showed that flame shapes are spherical provided the droplets remain stationary.

## Effects of Buoyancy on Gas-Jet Diffusion Flames

Science Applications International Corporation  
Dr. M. Yousef Bahadori  
Dr. Raymond B. Edelman, Rockwell International  
NAS3-22822 (NASA Contact: Dennis Stocker, LeRC)

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

**Research Task Description:** The program is structured in terms of closely interrelated ground-based experiments and theoretical modeling. This program has evolved as a result of theoretical analyses and limited experimental observations which have delineated the requirements to gain a more fundamental understanding of the effects of buoyancy on gas jet diffusion flames. The experimental portion of the ground-based program is designed to provide both additional time and quantitative measurements based on the findings of the past 2.2-second drop tower experiments. These data will be obtained by the combined use of the Lewis 2.2-Second Drop Tower, 5.18-Second Zero-Gravity Facility, and the KC-135 aircraft. The results will be used as a database for the model development, and will clarify the requirements on time for approach toward steady state. This will establish the need for space experiments.

**Progress to Date:** The preliminary 2.2-second drop tower tests for methane and propane flames under different oxygen concentrations and chamber pressures have shown that the characteristics and behavior of flames change dramatically in microgravity compared to their normal gravity counter-parts. Specifically, the following important findings are obtained: (a) refined photographic technique and igniting the flame in microgravity have shown that flames previously thought to have extinguished were still burning during the 2.2-seconds of microgravity; (b) although extremely useful for preliminary studies, 2.2 seconds is not sufficient for definitive conclusions on flame behavior and characteristics; (c) tip-opening and soot escape have been observed in microgravity for many flames, which are attributed to enhanced radiative loss, extensive soot formation, quenching at the tip, and possible thermophoretic effects; (d) extinction mechanism and extinction limits are apparently different in microgravity compared to normal gravity; (e) flame thickness, size, and color are different in microgravity and normal gravity for various oxygen concentrations and pressure levels, which indicates that kinetics, sooting, and diffusive processes are strongly affected by the removal of buoyancy.

The 2.2-seconds drop package has been improved with the addition of a thermocouple, a second fuel regulator (for chamber pressures greater than one atmosphere), mounting for silicon carbide fibers (to be used for flame visualization and approximate temperature indication), and soot probes (using thermophoretic sampling and subsequent analysis of collected soot). Drop testing will continue in the 2.2-Second Drop Tower.

Various analyses and modeling work have been conducted to support experiment fabrication and testing for the 5.18-second microgravity tests. The drop package assembly has been completed, and is now in the initial testing and check-out phase. Preliminary drop tests conducted to-date in the Zero-Gravity Facility have shown that flame radiation (using a thermopile detector), temperature field (using a 3 x 3 thermocouple rake), and chamber pressure (using a transducer) can be obtained successfully in

microgravity. These measurements combined with the species field (which will be obtained in the next phase of the program using a 3 x 3 rake of sampling probes and evacuated sample bottles) will provide a database for model development and validation. The 5.18-second tests will continue throughout 1990. The KC-135 hardware assembly will follow the successful demonstration of the drop hardware and verification of the need for test times longer than 5 seconds. Work will continue on the implementation of the radiation submodel, Soret effect (for thermophoresis), soot processes, and multicomponent diffusion in the numerical model for gasjet diffusion flames. The various findings of the experimental and theoretical efforts have been presented at different meetings which are listed in the following 1989/90 publications section.

#### Publications

Bahadori, M. Y., Edelman, R. B., Stocker, D. P., and Olson, S. L., "Ignition and Behavior of Laminar Gas-Jet Diffusion Flames in Microgravity," AIAA J., 1989 (in press).

#### Presentations

Bahadori, M. Y. and Stocker, D. P., "Oxygen-Concentration Effects on Microgravity Laminar Methane and Propane Diffusion Flames," *1989 Fall Technical Meeting (Eastern Section) of the Combustion Institute*, Albany, New York, October/November 1989.

Bahadori, M. Y., Stocker, D. P., and Edelman, R. B., "Effects of Pressure on Microgravity Hydrocarbon Diffusion Flames," *AIAA 28th Aerospace Sciences Meeting*, Reno, Nevada, January 1990, Paper AIAA 90-0651.

### Particle Cloud Combustion

University of California, San Diego  
Professor A. L. Berlad  
Dr. K. Seshadri  
Dr. V. Tangirala  
NAG3-925 (NASA Contact: H. Ross, LeRC)

*Objectives:* The principal objectives of this microgravity experimental program are to obtain flame propagation rate and flame extinction limit data for several important premixed, quiescent particle cloud combustion systems under near zero-gravity conditions. The data resulting from these experiments are needed for utilization with flame propagation and extinction theory. These data are also expected to provide new standards for the evaluation of fire hazards in particle suspensions in both Earth-based and space-based applications. Both terrestrial and space-based fire safety criteria require the identification of the critical concentrations of particulate fuels and inerts at the flame extinction conditions. Recent findings encourage the broadening of these objectives to include studies of both uniform and nonuniform particle cloud systems.

*Research Task Description:* The Particle Cloud Combustion Experiment employs long flame tubes within each of which a uniform quiescent cloud of particles is to be suspended in reduced gravity. Particulates under study include the fuels Iycopodium, cellulose and coal, as well as a number of inert particulates. Systems of additional interest include initially uniform clouds of inert particulates in premixed combustible gases. Ground-based supportive studies include the use of the LeRC drop tower and Learjet research facilities as well as the laboratories at the University of California, San Diego. Laboratory experiments include studies of particle cloud mixing, particle-particle interactions, pyrolysis-vaporization kinetics, and the radiative attenuation properties of individual particles and of particle clouds. LeRC drop tower and Learjet facilities are employed to study particle cloud mixing and particle cloud combustion phenomena in reduced gravity.

*Progress to Date:* LeRC drop tower and Learjet facilities have been used successfully to study lowgravity particle mixing and flame propagation phenomena supported by quiescent clouds of lycopodium particulates. These studies also include clouds of brown coal, with and without inert particulates. One mode of quasisteady flame propagation observed involves a well behaved flame front whose transport is radiatively driven. Theoretical relations between these observations and those for our previously observed stabilized Iycopodiumair flames have been developed. Additionally, a strikingly novel mode of flame propagation has been observed and analyzed. These latter flames are called *Chattering Flames* and result from flame-induced acoustic excitation of the Kundt's Tube phenomenon. This leads to spatial segregation of the fuel particles into regularly arranged laminae. Flame-induced acoustic segregation of particulates implies important safety-related consequences for systems of combustible particulates in reduced gravity as well as in normal gravity. Theoretical analyses have been conducted for *Chattering Flames* as well as for the more normal flame propagation mode. Analyses have been enlarged to include those for nonuniform concentrations of particulates as well as for clouds of inert particulates in premixed gaseous fuel plus oxidizer.

### Publications

A. L. Berlad and V. Tangirala, "Autoignition of Fuel-Oxidizer Mixtures in Microgravity," *Acta Astron.* 17, 1179 (1988).



A. L. Berlad and V. Tangirala, "Reduced Gravity Combustion of Particle Clouds," NASA CR-182299 (1988).

A. L. Berlad, H. Ross, L. Facca, and V. Tangirala. "Particle Cloud Flames in Acoustic Fields," Comb. & Flame, 1989 (accepted).

A. L. Berlad, H. Ross, L. Facca, and V. Tangirala. "Radiative Structures of Lycopodium-Air Flames in Low Gravity," AIAA Paper 89-0500, presented at *27th Aerospace Sciences Meeting*, Reno, Nevada (1989); also, J. Propul. & Power, 1989 (accepted).

A. L. Berlad, V. Tangirala, H. Ross and L. Facca. "Particle Segregation Effects On the Combustion Safety of Dust-Containing Systems," in *Proceedings of 12th International Colloquium on Dynamics of Explosions & Reactive Systems*, July, 1989), Paper No. 034.

H. D. Ross, L. T. Facca, A. L. Berlad, and V. Tangirala, "Feasibility of Reduced Gravity Experiments Involving Quiescent, Uniform Particle Cloud Combustion," NASA-TM 101371 (March 1989).

### Scientific Support for Space Shuttle Droplet Burning Experiment

Princeton University  
Professor Frederick L. Dryer  
Professor Forman A. Williams  
University of California, San Diego  
NAS3-24640 (NASA Contact: J.B. Haggard, LeRC)  
November 30, 1988 - November 30, 1989

*Objectives:* The general objective of this program is to ascertain how best to utilize reduced gravity facilities to pursue scientific investigations of droplet combustion. The scientific objective is to provide scientific support for development of a droplet combustion experiment module for conducting experiments in the NASA LeRC microgravity facilities, and possibly aboard a space-based platform such as the space shuttle or in the Space Station. The planned experiments are intended to improve fundamental understanding of droplet combustion phenomena, especially in relationship to time-dependent characteristics such as extinction. The scientific support objective encompasses data acquisition and reduction as well as to providing asymptotic and numerical modeling for interpretation of observations.

*Research Task Description:* The research tasks include theoretical modeling using asymptotics and numerical techniques, with emphasis on including transient and detailed chemical kinetic/transport effects, ground based experimentation on droplet burning in both the 2.2 and 5.5 second NASA LeRC drop towers, support to NASA in providing advice on hardware aspects of a flight experiment and analysis of data to be obtained from all experiments. The modeling addresses questions related to transient burning, to soot formation and destruction, to droplet disruption, and to ignition and extinction phenomena. Ground-based experiments have been focused on droplet ignition and on impulses imparted to droplets by ignition sparks; spark designs for minimum impulse were addressed. In addition, drop tower experiments are addressing burning rate measurements, mechanisms of soot production and droplet disruption, as well as extinction phenomena. Work presently encompasses n-decane, n-heptane, and methanol as fuels in oxidizer/inert mixtures at pressures to about 2 atmospheres.

### Publications

Shaw, B. D., Dryer, F. L., Williams, F. A., and Gat, N., "Interaction Between Gaseous Electrical Discharges and Single Liquid Droplets," Comb. & Flame **74**, 233 (1988).

Shaw, B. D., Dryer, F. L., Williams, F. A., and Haggard, J. B., "Sooting and Disruption in Spherically Symmetrical Combustion of Decane Droplets in Air," Acta Astron. **17**, 1195 (1988).

Shaw, B. D., and Williams, F. A., "Theory of Influence of Low Volatility, Soluble Impurity on Spherically Symmetric Combustion of Fuel Droplets," Int. J. Heat & Mass Transf., 1989 (in press).

Choi, M.Y., Dryer, F.L., and Haggard J.B., Jr., "Some Further Observations on Micro-gravity Droplet Combustion in the NASA Lewis Drop Tower Facilities," in *Proceedings of Third International Colloquium on Drops and Bubbles*, Monterey, CA, September 18-21, 1989. To be published in the Refereed Proceedings.

## Presentations

J. B. Haggard, M. H. Brace, J. L. Kropp and F. L. Dryer, "Droplet Combustion Experiment Drop Tower Tests Using Models of the Space Flight Apparatus," *27th Aerospace Sciences Meeting*, Reno, NV, January 9-12, 1989. Paper No. AIAA-890501 .

Choi, M. Y., Yetter, R. A., and Dryer, F. L., "Application of the Moving Finite Element Method to Chemically Reactive Flow Problems," *Eastern States Sectional Meeting of the Combustion Institute*, Clearwater Beach, FL, December 5-7, 1988. Extended Abstract.

Choi, M. Y., Dryer, F. L. and Haggard, J. B., "The Burning Behavior of Methanol Droplets in Humid Air," *Eastern States Sectional Meeting of the Combustion Institute*, Clearwater Beach, FL, December 5-7, 1988. Extended Abstract.

Choi, M. Y., Dryer, F. L., and Haggard, J. B., "Droplet Combustion in Microgravity," *International Microgravity Combustion Workshop*, NASA Lewis Research Center, Cleveland, OH, January 25-26, 1989. Poster Presentation.

Cho, S. Y., Yetter, R. A., Dryer, F. L., "Development of Comprehensive Fuel Particle Combustion Model," *International Micro-gravity Combustion Workshop*, NASA Lewis Research Center, Cleveland, OH, January 25-26, 1989. Poster Presentation.

Dryer, F. L., "Microgravity Research on Droplet and Particle Combustion: Some Thoughts," *International Microgravity Combustion Workshop*, NASA Lewis Research Center, Cleveland, OH, January 25-26, 1989. Invited Presentation as Panel Member.

Cho, S. Y., Choi, M. Y., Dryer, F. L., Haggard, J. B., and Brace, M., "Some Considerations on the Combustion of Heptane Droplets in Microgravity," *Seventh International Conference on PhysicoChemical Hydrodynamics*, MIT, June 25-29, 1989. Abstract.

## Supercritical Vaporization of Liquid Fuel Droplets in a Microgravity Environment

University of Wisconsin-Madison  
Dr. Patrick V. Farrell  
Dr. Gary Borman  
D. E. Foster  
NAG 3-718 (NASA contact: D. Stocker, NASA LeRC)  
April 15, 1986 - January 1, 1990

*Objectives:* This program represents a joint venture between the University of Wisconsin and General Motors Research Laboratories. The study concentrates on the details of droplet vaporization under conditions typical of Diesel engines and liquid fueled rockets. For these combustion systems, liquid fuel is sprayed into an environment which is above the liquid critical pressure and at or above the liquid critical temperature. The result is very rapid vaporization of the liquid. For most liquid fuel combustion systems, fuel vaporization rate is a major factor in the subsequent combustion event. Several phenomena have been observed at near critical conditions which may have a significant impact on the vaporization rate of the droplets. These effects include droplet deformation and breakup due to the elimination of liquid surface tension, significant changes in transport properties near the critical point, and unsteady vaporization characteristics. In order to study these phenomena without the interference of convective effects on transfer and drop shape, a quiescent environment is required. Since the problem is by definition not isothermal, this requires virtual elimination of natural convection as well as forced convection. In this study, natural convection effects are reduced by operating the experiments in microgravity. The specific objectives of the project are:

1. Develop experimental techniques and perform experiments required to investigate supercritical droplet vaporization in microgravity;
2. Develop a numerical model of the supercritical vaporization problem consistent with the experimental conditions;
3. Validate the model with the experimental results obtained in the experimental program.

*Description of Research:* The program is currently in a 6-month no-cost extension stage after completion of the third year of work. A proposal to continue the work is pending.

The experimental program has progressed to the point where an experimental device has been constructed and tested in the NASA Lewis 2.2 second drop tower. The device is designed to generate a single droplet at low pressure and temperature. An opposed piston compression device driven by a hydraulic accumulator then provides a high pressure atmosphere in a short time with a minimum of residual gas motion. The temperature of the atmosphere is increased due to the compression.

At the end of the compression event, the desired experiment initial conditions are attained and data taking begins. At present, the data consists of high speed movies of the droplet. The device has been dropped at NASA Lewis more than 30 times, with minimal damage. The major problems with the device have been the droplet deployment device and internal heat transfer.

A droplet deployment device similar to the device developed for the Droplet Combustion Experiment has been developed. After considerable testing and optimization, the device is capable of producing 1 mm size heptane droplets with residual velocities of about 5 mm/s before compression, and about 2 mm/s after compression.

The second major problem with the compression device has been the problem of rapid heat transfer from the compressed gas to the surrounding metal parts, principally, the pistons. The result is that the gas

cools rapidly and the pressure drops soon after the compression event. As a result, the constant high temperature and high pressure are attained for only about 100 ms, which is too short for 1 mm droplets to vaporize significantly by an order of magnitude. Recent work on this problem has led to a solution in the form of a pre-heater for the piston surfaces, which are the primary site for the heat transfer. Testing on the rig is continuing, but this scheme seems to have reduced the problem to manageable levels.

As soon as the heat transfer improvements are finished, a test matrix of several liquids and operating conditions will be tested in the drop tower.

The numerical model of the droplet vaporization problem has been developed to the point that it can predict droplet lifetimes under a variety of operating conditions. The model is a one-dimensional transient model for a regressing spherical droplet in an infinite field. The major difficulty in modelling the vaporization process is in accurately modelling the interface transfer processes and the interface equilibrium conditions. Since little data exists for validating the code, detailed verification awaits the results of our own experiments. As an adjunct to the vaporization code, a simple combustion code has been added to the vaporization predictions. Since some high pressure droplet combustion data does exist, this version of the model can be compared with experimental results.

In addition to these two main efforts, a series of smaller support projects have been completed. These support projects include a study of non-contact droplet positioning techniques, in particular optical levitation techniques. The technique was demonstrated on solid particles and small ( $20\text{ }\mu\text{m}$ ) silicon oil droplets in l-g. At present, the droplet generator produces relatively motionless drops, so this device has not yet been implemented in the experimental device.

A second support effort has focussed on developing suitable measurement methods for acquiring detailed temperature and species measurements in the gas field and in the droplet. This work continues, with emphasis on Rayleigh scattering methods and interferometric methods.

#### Publications

E. W. Curtis and P. V. Farrell, "Droplet Vaporization in a Supercritical Microgravity Environment," Acta Astron 13 (1988).

E. Curtis, J. Hartfield, and P. V. Farrell, "Microgravity Vaporization of Liquid Droplets", in *Proceedings of Third International Colloquium on Droplets and Bubbles*, September 1988 (in press).

## *A Fundamental Study of the Effect of Buoyancy on the Stability of Premixed Laminar Flames*

University of California at Berkeley  
Professor A. Carlos Fernandez-Pello  
Corey M. Dunskey  
September 1988 - September 1989

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

*Research Task Description:* The research task consists of the experimental determination of the gravity effect on the stability and cellular structure of a burner-stabilized premixed flame. Low gravity-atmospheric pressure tests are performed at the 2.2 sec NASA LeRC drop tower. The results for the normal and micro-gravity tests are compared to observe, through the cellular structure, the effect of gravity on the flame stability. These experiments are complemented with a similar series of tests performed at varied ambient pressure. Since the ambient pressure affects the buoyancy force, diffusivity and reaction rate, the comparison of the different results are used to determine the effect of gravity on the individual mechanisms of flame stabilization.

*Progress to Date:* The research performed during this reporting period includes two tasks: 1) normal gravity experiments on the effect of ambient pressure on the cellular flame structure, with emphasis on comparison of constant mass flow and constant volume flow results; and 2) ambient pressure microgravity experiments utilizing a new drop package and subsequent data analysis. A summary of the accomplishments to date is presented below.

### *Normal Gravity, Low Pressure Experiments*

These experiments are conducted in a low pressure facility that consists of a water-cooled porous plug burner with its supporting flow metering devices and instrumentation, and a pressure vessel with the supporting equipment for pressure control and measurement. A microcomputer and photographic equipment complement the facility.

In the experiments, the oxygen-propane equivalence ratio,  $\phi$ , is varied to determine the range of conditions at which cellular flames occur. The ambient pressure is then reduced while holding the dilution index,  $\delta$ , fixed, and  $\phi$  is again varied until the cellular regime is observed. This procedure is carried out for both constant mass and volume flow rates. The data are then used to obtain the flame stability maps for each mixture flow rate and dilution index. The contours in this map delineate the boundaries of the region where cells are visually observed, as well as curves of constant cell diameter.

Comparison of the constant mass flow and constant volume flow cases indicates that the effect of pressure by itself on the occurrence of the cellular instability is relatively weak. Rather, the flame temperature and resulting burning velocity emerge as the factors controlling the conditions at which the cells appear. This interpretation is further supported by the movement of the cellular regime as  $\delta$  is changed. At smaller values of  $\delta$ , when the mixture is more highly diluted, the band of cellular instability shifts toward the faster-burning mixtures of lower equivalence ratio. This counters the lowering of the flame temperature and speed due to the increased concentration of the diluent.

Currently, this dependence of the cellular regime on flame speed and temperature is being explored more quantitatively through measurements of the burner coolant enthalpy gain.

### *Ambient Pressure Microgravity Experiments*

During the period November 1988 - February 1989, a series of microgravity tests were performed at the NASA LeRC 2.2-sec drop tower, utilizing an experiment package designed and constructed for these experiments. This package features Tattletale microprocessor data acquisition and control; dual 16 mm cameras for lateral and plan views of the flames; gas mixing in a single onboard bottle; additional plumbing for use of the inert-gas burner shroud; closed-loop burner cooling; and control of the flow rate of fresh mixture delivered to the burner. Supporting equipment includes a gas mixing system utilizing the method of partial pressures.

A total of 55 drop were performed with about 50 of these yielding good data. A range of fourteen equivalence ratios were tested, over three dilution levels and five flow rates. The direction of flame propagation was downward, and the effects of using the burner cooling and nitrogen shroud were recorded by testing with and without these features. Early drops were conducted by igniting the flame in normal gravity conditions, letting it stabilize on the burner for several seconds, and then initiating the period of microgravity. This allowed direct visual comparison between normal and microgravity flame structure. Later drops employed ignition in microgravity. In these tests, gas flow to the burner and ignitor sparking were initiated at the beginning of the drop. Ignition generally occurred within 0.2-0.4 sec. This procedure ensured that no transient natural convective flow from the normal gravity burn was present.

The primary information obtained from the film records was that the cells persist in the absence of gravity. It was also observed that motion of the cells becomes de-coupled from that of the buoyant plume above the burner. In normal gravity, this plume is continuously entraining ambient air, which burns with the excess fuel downstream of the premixed flame in a blue diffusion flame. Baroclinicity adds vorticity to the plume, resulting in the periodic generation of toroidal vortices that travel upward with the rising hot gases. When the system is subjected to microgravity conditions, the buoyant effects are no longer present, and the pulsations cease within 50 msec. The outer diffusion flame lifts upward and outward from the burner, becoming fainter as it seeks fresh oxidizer. The premixed cellular flame then becomes quite stationary, except for the cells near the burner edges, which oscillate randomly at high frequency.

Other parameters were found to have little effect on the flame structure. Burner cooling had no observable effect, though this is not surprising considering the short duration of the burns. No difference in cell characteristics was observed in comparisons of most normal gravity and microgravity flames, nor between microgravity flames generated by normal-gravity and microgravity ignitions. The flat/cellular boundary on the lean side of the cellular regime was unchanged. The cellular/lifted boundary, however, shifts toward higher equivalence ratios, widening the cellular band. Visually, flames that are just rich enough to lift in normal gravity are observed to become cellular under microgravity conditions.

### *Publications*

Dunsky, C.M. and Fernandez-Pello, A.C., "Some Experimental Observations of the Stability of Premixed Cellular Flames Under Microgravity Conditions," *1988 Spring Meeting, Western States Section/The Combustion Institute*, Salt Lake City, UT, March 1988.

Dunsky, C.M. and Fernandez-Pello, A.C., "The effect of ambient Pressure on the Cellular Flame Structure," *1988 Fall Meeting, Western States Section/The Combustion Institute*, Dana Point, CA, October 1988.

Dunsky, C.M. and Fernandez-Pello, A.C., "Gravitational Effects on Premixed Cellular Flames," *7th International Conference on Physico-chemical Hydrodynamics*, Cambridge, MA, June 1989.

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

University of California at Berkeley  
Professor A. Carlos Fernandez-Pello  
Professor Patrick J. Pagni  
September 1988 - September 1989)

*Objectives:* The objective of the overall research program is the design and performance of smolder combustion experiments under microgravity conditions, to help understand the mechanisms controlling smoldering, and in turn the prevention and control of smolder originated fires in normal gravity and in space-based environments. The specific objectives are: to develop ground-based experiments to determine the effect of gravity on the different modes of smoldering; to perform drop-tower tests to obtain data on the smolder transition processes of ignition, flaming and extinction; and to use these data to design a space-based smoldering combustion experiment.

*Research Task Description:* The current research program is primarily experimental. Experiments are being conducted to determine the range of conditions for which smolder is affected by gravity, and how gravity affects the smolder process and its transition to flaming. The approach being followed is to measure the smolder velocity through materials of varied void fraction as a function of the velocity of a mixed (free and forced) oxidizer flow, for both downward and upward propagation. Comparison of the measurements for the two geometrical configurations allows the determination of the conditions where gravity affects smoldering. This information is then used toward the design of the space-based experiment.

The smolder process is too slow to obtain significant microgravity information in droptower, or parabolic flight experiments. However, such experiments can provide information about the microgravity behavior of the transition processes of smolder ignition, flaming and extinction. With this objective in mind, the present research task includes a series of droptower tests aimed to obtain information about the suitability of the different ignition methods to initiate the smolder process in a microgravity environment.

*Progress to Date:* Research carried out during this reporting period includes normal gravity experiments to determine the effect of buoyancy on co-current, downward and upward, smoldering, and drop-tower, microgravity experiments to observe ignition under low gravity conditions. All the experiments have been conducted with polyurethane foams of varied void fractions. This material has been selected for the experiments because it has uniform properties and a rigid structure, which is needed for upward smolder tests.

The normal gravity experiments for co-current smoldering, although not totally completed, have resulted in the design of a new method for smolder ignition, and an initial indication of the range of air velocities at which buoyancy plays a role on smolder combustion of polyurethane foams. The ignition device consists of an electrically heated nichrome wire uniformly wrapped between two 1 cm thick layers of a porous ceramic material. The ceramic material provides rigidity to the ignitor while having the dual role of providing insulation to the smolder reaction and permitting the flow of gaseous species to and out of the smolder reaction zone. The range of input power and temperatures needed to initiate the smolder process have been documented. Measurements of the smolder velocity as a function of the forced air flow velocity for downward and upward smolder propagation show that the smolder velocity fluctuates around a constant value for air velocities less than 0.2 cm/sec, and drops sharply for larger air velocities. These preliminary results indicate that the role of gravity in this smolder configuration is limited to air velocities smaller than 0.2 cm/sec. This work is currently being continued to verify the results obtained to date and to obtain additional information on the counter-current and two-dimensional smoldering process.

The drop-tower experiments have required the modification of a drop-tower package designed and constructed to perform premixed laminar flame experiments (see NAG-3-861 progress report). This modification included the adaptation of the smolder test apparatus, and the incorporation of the necessary



instrumentation to perform smolder experiments. A series of 28 tests were conducted on the 2.2 sec NASA LeRC drop tower in order to observe the effect of microgravity on forced flow smolder ignition. The parameter analyzed was the smolder temperature gradient, because the temperature itself, or the smolder velocity, do not change enough in the 2.2 secs to observe significant differences. The results for the smolder temperature gradient variation with the flow velocity indicate that microgravity favors the initiation of smoldering, and that here also 0.2 cm/sec is the upper range of flow velocities at which buoyancy plays a significant role on the smolder ignition process. This task is also being continued to test the new ignition method and to observe the effect of microgravity on extinction and flaming.

#### Publications

Newhall, J.L., Fernandez-Pello, A.C., and Pagni, P.J., "Experimental Observations of the Effect of Buoyancy on Co-Current Smoldering," *1988 Spring Meeting, Western States Section/The Combustion Institute*, Salt Lake City, Utah, March 1988.

Cantwell, E., "Preliminary Observations of Transitional Smoldering at Micro-Gravity," *1988 Spring Meeting, Western States Section/The Combustion Institute*, Salt Lake City, Utah, March 1988.

### Time-Dependent Computational Studies of Premixed Flames in Microgravity

Naval Research Laboratory  
Dr. K. Kailasanath  
Dr. Gopal Patnaik  
Dr. Elaine Oran  
(NASA Contact: Dr. Howard Ross, LeRC)  
August 1988- August 1989

*Objectives:* The objective of this research is to investigate fundamental problems in the combustion of premixed gases such as the differences in the propagation and extinction of gas-phase flames in zero and normal gravity.

*Research Task Description:* We study the dynamics of premixed flames by performing detailed numerical simulations using a time-dependent, two-dimensional numerical model. This model solves the multispecies coupled partial differential reactive flow equations. The model includes detailed chemical kinetics mechanisms, algorithms for thermal conduction, molecular diffusion, viscosity, convective transport and effects of gravity. We also evaluate various simplified models that will reduce the cost of computations by comparing their results to those from detailed simulations.

*Progress to Date:* We first used the two-dimensional model to verify that the predictions of the model are in agreement with experimental observations of flames in both lean and rich hydrogen-oxygen-nitrogen mixtures. Then we used the model to systematically study mechanisms that can lead to cellular structure and identified the thermo-diffusive instability mechanism to be the dominant mechanism. We have also studied the effects of gravity on flame structure and instability by comparing simulations of zero-gravity flames to upward- and downward-propagating flames. These simulations show that the effects of gravity become greater as the lean flammability limit is approached. Furthermore, these simulations have provided additional insight on the interactions between the processes leading to cellular structure and the buoyancy-induced Rayleigh-Taylor instability mechanism. The simulations also suggest that cellular instability grows more rapidly than Rayleigh-Taylor instability.

We have also studied the role of intermediate species on the cellular structure of flames. For this study, we compared the results of detailed simulations to those obtained from simulations using a single-step kinetics model in which the intermediate species are neglected. The simplified model could predict the tendency for instability but the evolved multi-dimensional structure was incorrect. It was also found that the diffusion of minor intermediate species cannot be neglected and that a level of approximation consistent with the chemistry model used is required. Currently we are studying the cell-split limit observed in microgravity experiments.

### Publications

Patnaik, G., Kailasanath, K., Oran, E.S., and Laskey, K.J., "Detailed Numerical Simulations of Cellular Flames," *Proceedings of the 22nd International Symposium on Combustion*, pp. 1517-1526, The Combustion Institute, Pittsburgh, PA., 1988.

Patnaik, G., Kailasanath, K., and Oran, E.S., "Effect of Gravity on Flame Instabilities in Premixed Gases," AIAA Paper No. 89-0502, Proceedings of the 27th *Aerospace Sciences Meeting*, American Institute of Aeronautics and Astronautics, Washington, D.C., 1989.

#### Presentations

Kailasanath, K., Patnaik, G., and Oran, E.S., "Effect of Gravity on Multi-dimensional Laminar Premixed Flame Structure," Paper No. IAF-88-354, presented at the 39th *Congress of the International Astronautical Federation*, Bangalore, India, October 1988.

Patnaik, G., and Kailasanath, K., "Numerical Simulation of Cellular Instability in Hydrogen Flames," Paper No. 32, presented at the 1988 *Technical Meeting of the Eastern Section of the Combustion Institute*, Clearwater Beach, FL, December 1988.

Kailasanath, K., "Numerical Simulations of Flames and Detonations," invited paper, presented at the 3rd *International Conference on Numerical Combustion*, Antibes, France, May 1989.

Patnaik, G., and Kailasanath, K., "The Role of Intermediate Species in the Cellular Structure of Flames," presented at the 12th *International Colloquium on the Dynamics of Explosions and Reactive Systems*, Ann Arbor, MI, July 1989.

## Radiative Ignition in Microgravity Environment

National Institute of Standards and Technology  
Dr. Takashi Kashiwagi  
Dr. Howard R. Baum  
Dr. Colomba di Blasi

*Objectives:* The objective of this study is to develop a theoretical model capable of predicting the radiative ignition of a cellulosic material( for example, filter-paper) and the subsequent flame spread over the sample in a microgravity environment. This study is to lead to applications such as fire safety for spacecraft by understanding and predicting ignition and subsequent flame spreading behavior. It is hoped that validation experiments of the predicted results will be conducted in a microgravity environment in the next phase of the study.

*Research Task Description:* This study consists of four parts: (1) Thermal and thermal oxidative characterization of the cellulosic material will be measured in a normal gravity environment by using a thermal analysis technique. A global degradation scheme involving two or three step reactions will be used and their kinetic constants will be determined by dynamic derivative thermogravimetry at various heating rates and also at various ambient oxygen concentrations. (2) Kinetic constants of global gas phase oxidation reactions of evolved degradation products from the degrading cellulosic material will be determined in a normal gravity environment calculated assuming OH equilibrium. (3) A theoretical model of ignition of the cellulosic material by an external radiation and subsequent transition to flame spreading in a microgravity environment will be developed and solved numerically using the above characteristics of the degradation and of the global gas phase oxidation reactions. The advantage of a microgravity environment is fully utilized (the dominant vorticity creation mechanism in the bulk of the gas is absent), permitting the gas phase flow pattern to be calculated by solving an irrotational flow pattern determined by the thermal expansion of the field and the convective mass transfer of the evolved degradation products to the gas phase. (4) Radiative ignition experiments at a normal gravity environment will be conducted to examine ignition behavior and also to obtain radiative properties of the cellulosic material. The bottom surface of the sample will be ignited by a vertically upward external radiation. Ignition behavior in this configuration in a normal gravity environment is the closest to that in a microgravity environment.

*Progress to Date:* After the completion of the construction of an ignition chamber, the characterization of radiant flux distribution from the Tungsten lamp to the sample surface was completed. The measured flux distribution is nearly Gaussian at various fluxes up to  $10 \text{ W/cm}^2$ . Ignition experiments with ashless filter paper were conducted in normal gravity in the bottom surface ignition configuration. However, no ignition was achieved with this sample even at  $10 \text{ W/cm}^2$  under 35% oxygen concentration. This is due to high surface reflectance of the paper in the visible wavelength where significant amount of energy is emitted from the lamp. Black color papers were used as a sample and ignition was observed even in air. Relations between external incident radiant flux and ignition delay time were obtained in air and 35% oxygen concentration. Also, surface temperatures of the paper (at the irradiated surface and the back surface) during the ignition period were measured at various radiant fluxes and oxygen concentration. The results show that thermal thin approximation is validated for the paper. Hemispherical surface reflectance of the paper was measured from  $0.3$  to  $2.5 \text{ } \mu\text{m}$  and the results will be used as one of the material characteristics in the theoretical model.

The characterization of thermal and thermal oxidative degradation of the cellulosic material was delayed by the problem of the gradual deterioration of the  $\text{H}_2\text{O}$  analyzer. After a long search a new  $\text{H}_2\text{O}$  analyzer was selected, purchased and installed. After careful calibration of the mass balance of  $\text{CO}$ ,  $\text{CO}_2$ ,  $\text{H}_2\text{O}$ ,  $\text{O}_2$  (depletion from the gas phase) and sample weight loss rate, the determination of the degradation kinetics of the paper is in progress at 5 different heating rates and 4 different gas phase oxygen concentrations.

In the present reporting period a mathematical model capable of predicting the pyrolysis of a thermally thin fuel has been coupled to a generalization of the gas phase preheating analysis described earlier. Calculations have been carried out for the pre-ignition phase of the problem. Results have been obtained for the solid phase temperature and density distributions, as well as gas phase temperature, velocity, and species concentrations.

The gas phase model requires the solution of mass, energy, and species concentration equations, supplemented by an irrotationality condition for the induced velocity fields. No momentum equations need be solved. This situation arises due to the unique combination of a microgravity environment and low Reynolds number associated with this problem. A separate formal asymptotic analysis has been undertaken as a NIST research project in collaboration with Prof. I. Wichman of Michigan State University to justify this approach.

The solid phase model is thermally thin, so the temperature and density across its thickness are uniform. The density varies due to the outgassing of pyrolysis products, whose evolution is described by two distinct finite reaction rate processes. The reaction rates are represented by Arrhenius functions, with parameters determined by the laboratory experimental portion of this project. The evolution equations for the solid phase density and temperature (conservation of mass and energy in the solid fuel) are coupled to the gas phase by interfacial conditions representing the exchange of mass, energy, and pyrolysis products.

#### Publications

H. Baum, T. Kashiwagi, and C. Di Blasi, "Radiative Ignition of Solid Fuels in a Microgravity Environment - The preheating Problem," in *Proceeding of the 1988 Eastern States Combustion Meeting*, December 1988.

#### Presentations

H. Baum, T. Kashiwagi, and C. Di Blasi, "Radiative Pyrolysis of Thin Fuels in a Microgravity Environment," presented at the *1989 Eastern States Combustion Meeting*, October 1989.

*Ignition and Subsequent Flame Spread in Cellulosic Materials for Microgravity Applications*

National Institute of Standards and Technology,  
Center for Fire Research  
Dr. Takashi Kashiwagi  
C-3200-K (NASA Contact: R. Friedman, LeRC)  
January 1988 - December 1990

*Objectives:* The objective of this work is to determine the ignition and subsequent transition to flame spreading of cellulosic materials through radiant heating. This study is to lead to applications such as material flammability acceptance standards for spacecraft.

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

The gas-phase analysis will incorporate governing equations without gravity. Hence, the model will serve as a guide to define a specific experimental system and test procedure for microgravity to demonstrate material flammability and acceptance criteria, in follow-on work.

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

NASA Lewis Research Center  
Sandra L. Olson  
In-House

*Objectives:* The objective of this program is to determine the extinction limits and steady burning characteristics of thermally-thin solid fuels over a wide range of oxidizer concentrations and flow velocities. This work will provide insight into the effects of buoyancy on flame spread mechanisms by decoupling natural convective transport processes from forced convective, conductive, and diffusive transport processes that are important in spreading flames. Understanding the mechanisms for flame spread will provide a fundamental link between normal gravity materials flammability and materials flammability in spacecraft environments where low velocity forced flows exist due to environmental control systems.

The approach used in this study is to perform a series of experiments in low gravity varying fuel thickness, oxidizer concentration, diluent composition, pressure, and opposed flow velocity. Tests will be conducted in a low speed combustion tunnel developed for use in all three NASA Lewis Research Center's low gravity facilities. Low gravity is required for these experiments because in normal gravity buoyancy-induced gas flows around the spreading flame are on the order of or greater than the range of forced flow velocities to be studied (0-30 cm/s). These natural convective flows overwhelm or combine with the forced convective flows so that the effect of the forced flow on the flame spread rate cannot be isolated.

*Research Task Description:* The study will be performed in four phases:

- 1) Preliminary normal and low gravity quiescent environment (zero flow) and normal gravity high velocity forced flow experiments will be performed to define fuel burning characteristics and to estimate fuel extinction limits as a function of oxygen concentration and flow velocity.
- 2) Flow field characterization of the combustion tunnel with cold flow in normal and/or microgravity will be performed, and instrumentation will be developed for the low gravity testing.
- 3) Normal gravity tests and low gravity tests in all three Lewis low gravity facilities will be conducted. The flame development and steady burning characteristics of flame spread over solid fuels will be studied and compared with current modelling work underway at Lewis.
- 4) At an appropriate time in the ground-based test program, a Science Requirements Document will be drafted. If necessary, further ground-based research will be proposed as well.

*Progress to Date:* Quiescent environment flame spread and extinction experimental results were presented at the Twenty-Second Symposium (International) on Combustion in August 1988. A significant finding from that work was an extinction boundary which shows quenching and blowoff extinction branches meeting at a fundamental low oxygen limit for flame spread in opposed flow. Additionally, fuel thickness effects on the flame spread rate, especially near the extinction limit, depart significantly from theory.

Quantitative flow visualization using a hot wire smoke pulse tracking technique in microgravity was completed in 1988 also. Combustion Tunnel experiments in the 2.2 second Drop Tower began in February 1989 and are expected to continue through 1990. A 5.18 second Zero Gravity Facility Combustion Tunnel is in the process of being designed as a "multi-user" facility and is estimated to be completed for experimental use by 1991.

Results to date indicate a strong effect of flow velocity on the flame spread rate in low gravity. From the quench limit in a quiescent environment, the flame spread rate increases sharply with increasing

opposed flow until it reaches a maximum spread rate. For opposed flow velocities above this point, the flame spread rate decreases with increasing opposed flow. This trend agrees with normal gravity high forced velocity data and elevated gravity (high buoyant velocity) data, and demonstrates the full range of flame spread behavior. The results of flame spread modelling efforts to look at the importance of solid phase radiative loss on the flame spread behavior were presented at the AIAA meeting in January, 1989.

An additional series of quiescent flame spread tests was conducted using diluents other than  $N_2$  (Ar, He,  $CO_2$ ) to study the heat and mass transport changes caused by the use of different diluents. Results to date indicate that flame spread rates do not vary significantly in different diluent atmospheres, the flame spread rate is primarily a function of oxygen concentration. The effects of diluents are seen only near the extinction limit. Helium, which enhances burning in normal gravity by increasing heat transfer, appears to cause premature (with respect to  $N_2$ ) extinction due to excessive conductive heat losses. Gas phase radiative heat loss is believed to be the cause of the premature carbon dioxide-diluted flame extinctions. Argon-diluted flames, however, demonstrates an extended range of flammability to lower oxygen concentrations than nitrogen.

Research will continue to map out the flame spread behavior for a variety of oxygen concentrations, opposed flow velocities, and for different diluent gases to look at the effects of heat transfer versus mass transfer in these flames. Other fuels will also be studied for potential use in experiments. It is anticipated that by Fall 1990, the research will have progressed to the point so that a Science Requirements Document for a space experiment can be written.

#### Publications

Olson, S. L., "The Effect of Microgravity on Flame Spread Over a Thin Fuel", NASA TM 100195, also CWRU Master's Thesis, 1987.

Olson, S. L., Ferkul, P. V., and T'ien, J. S., "Near-Limit Flame Spread Over a Thin Solid Fuel in Microgravity", *Twenty-Second Symposium (International) on Combustion*, The Combustion Institute, pp. 1213-1222, 1988.

#### Presentations

Fakheri, A., and Olson, S.L., "The Effects of Radiative Heat Loss on Microgravity Flame Spread", *27th AIAA Aerospace Science Meeting*, January, 1989, submitted to AIAA for publication.

Olson, S.L., Stouffer, S.C., and Grady, T., "Diluent Effects on Quiescent Microgravity Flame Spread Over a Thin Solid Fuel", *Fall Technical Meeting of the Eastern States Section of The Combustion Institute*, 1989.



Mechanisms of Combustion Limits in Premixed Gas Flames at Microgravity

Princeton University  
Professor Paul D. Ronney  
Angel Abbud-Madrid  
Kenneth Whaling  
NAG3-965 (NASA Contact: Dr. Howard Ross, LeRC)  
December 15, 1988 - December 14, 1991

**Objective:** The objective of this work is to study the mechanisms responsible for limit phenomena in premixed gas combustion processes. By performing experiments in a microgravity environment it is possible to eliminate the complicating and sometimes ambiguous influences of buoyant convection on these phenomena.

**Research Task Description:** Three types of limit phenomena have been identified which may be affected by buoyant convection: flammability, ignition, and stability limits. The effects of the interactions of chemical reaction, thermal and mass diffusion, flame front curvature, and radiative transport are being examined. Experimental, analytical, and computational methods are employed.

**Progress to Date:** Current microgravity experiments have verified and extended earlier work demonstrating the effects of the aforementioned processes on flammability limits. It is found that the primary mechanism of extinguishment is by heat loss due to radiation from the burned gases. Dynamic behavior is governed by the flame front curvature and the Lewis number of the mixture. The details of the chemical reaction mechanism are of little qualitative importance.

Several new and unexpected phenomena, not observed in earth gravity experiments, have been found at  $\mu g$  under appropriate conditions. Three of these are "flame balls," "double flames," and an "inverse flammability region." Flame balls are apparently stable, stationary spherical flames which are spawned from cellular flame structures that occur in mixtures with low Lewis number. In double flame behavior, after propagation of the primary flame front from the center of combustion vessel radially outward through the mixture to the walls of the vessel, *a second flame front forms near the center of the vessel and propagates outward to the walls*. The "inverse flammability region" refers to cases in which progressive dilution of a combustible mixture with inert gas renders the mixture non-flammable (as expected) but *further dilution renders the mixture flammable again!* The first two of these phenomena have been interpreted in terms of simple analytical models which include the effects of a single-step, high activation energy chemical reaction, radiative heat loss from the combustion products, and flame front curvature and unsteadiness. In the case of "double flames," the presence of two reactants with unequal Lewis numbers (in which case one reactant may "leak" through the flame front) is also employed. The "inverse flammability region" behavior has yet to receive a simple interpretation.

In future work the KC-135 aircraft will be employed to obtain increased  $\mu g$  test time, grab-sampling techniques will be used at  $\mu g$  to test the hypothesis of reactant leakage in "double flames," and inert, radiant particles will be used further examine the effects of radiative transport. Additionally, the effects of isotropic turbulence on the aforementioned limit processes will be studied.

## Publications

Farmer, J. N., Ronney, P. D.: "A Numerical Study of Unsteady Nonadiabatic Flames," Comb. Sci. & Techn., 1989 (submitted).

Ronney, P. D., "Near-Limit Flame Structures at Low Lewis Number," Comb. & Flame, 1989 (in press).

Buckmaster, J. D., Joulin, G., Ronney, P. D., "Effects of Heat Loss on the Structure and Stability of Flame Balls," Comb. & Flame, 1989, (in press).

Ronney, P. D., Sivashinsky, G. I., "A Theoretical Study of Propagation and Extinction of Nonsteady Spherical Flame Fronts," SIAM J. Appl. Math. **49**, 1029-1046 (1989).

Ronney, P.D., "On the Mechanisms of Flame Propagation Limits and Extinction Processes at Microgravity," in *Proceedings of Twenty Second Symposium (International) on Combustion*, Combustion Institute, 1988, pp. 1615-1623.

Ronney, P.D., "Effect of Chemistry and Transport Properties on Near-Limit Flames at Microgravity," Comb. Sci. & Techn. **59**, 123-141 (1988).

## Presentation

Abbud-Madrid, A., Ronney, P. D., "Effect of Lewis Number and Radiative Loss on the Behavior of Near-Limit Flames at Microgravity," *Spring Technical Meeting, Combustion Institute, Central States Section*, May 1-2, 1989, Dearborn, Michigan.

### The Effects of Gravity on Flame Spread Involving Liquid Fuel Pools

NASA Lewis Research Center  
Dr. Howard Ross  
In-House

*Objectives:* The objective of the study is to increase fundamental understanding of the effects of gravity on flame spread in open liquid pool fires. Specifically, the flame spread dependence on gravity and oxygen concentration will be studied for fuels above and below their flash points.

*Research Task Description:* The series of tasks are as follows: determine if a quiescent, flat liquid-gas interface can be achieved in a time frame compatible with ground-based, reduced gravity facilities; (b) Design, fabricate, and test apparatus to study flame spread behavior in these facilities; (c) Collect and compare flame spread data in normal and reduced gravity; and (d) Analyze the data by comparison with existing models (if available).

*Progress to date:* A 2.2 sec drop tower rig was constructed to determine the time of a liquid/gas interface to stabilize and reach quiescence when the gravity changes suddenly from a normal to a reduced level. It was shown that the viscosity and the contact angle significantly affect the total time, and that shallow, viscous pools can probably be used in the ground-based facilities.

In the past twelve months, fabrication and testing of the 5.1 second Zero G Facility flame spread hardware was also completed. Operational testing was successful. A first series of tests in reduced and normal gravity were conducted to determine the roles of gravity and oxygen concentration. Ignition and steady flame spread behavior for near- and superflash temperature liquid pools were observed for the first time in reduced gravity. The effect of higher oxygen concentration was to increase flame spread rates and flame luminosity (presumably due to soot) in both normal and reduced gravity. At lower oxygen concentration, pulsations or asymmetries were observed in reduced gravity, demonstrating the importance of gas phase buoyancy to the pulsating flame spread mechanism. After spreading, the reduced gravity flame appears headed for extinction.

#### Presentations

Ross, H. D. and Pline, A. "Equilibrium Times for Liquid-Gas Systems Exposed to Step Changes in Gravity" *AIChE Annual Meeting*, November 1988, Washington D.C.

Ross, H. D. and Sotos, R.G. "Normal and Microgravity Experiments on Pulsating Flame Spread over Shallow Liquid Pools" *Eastern States Meeting of the Combustion Institute*, October 1989.

## *The Effects of Gravity on Pre-Ignition Processes Involving Liquid Fuel Pools*

NASA Lewis Research Center  
Dr. Howard Ross (co-Investigator with W. Sirignano)  
In-House

*Objectives:* The objective of this program is to verify experimentally a model of the velocity and temperature fields in varying gravity fields and determine their effect on ignition susceptibility of an enclosed liquid fuel pool. The model is being developed by W. Sirignano and co-workers at the University of California-Irvine.

*Research Task Description:* Model and experiment development are to be done in a stepwise fashion: first, only the gas phase is to be studied; then combined liquid and gas phases without vaporization; and finally the combined phases with vaporization.

*Progress to Date:* In the past year, the single phase experiment were completed in a newly fabricated drop rig. The rig successfully employed a He-Ne laser light sheet and video cameras and recorders for the first time in the drop tower. Drop tower tests revealed a distinct gravitational effect (a sudden hot gas expansion upon entering reduced gravity and a rapid deceleration of flow), similar to that predicted by the computer code. The results were presented at the AIAA meeting in Reno; a more complete publication is being prepared at the time of this writing. It will include the effect of heater size, temperature, and emissivity, in addition to gravitational effects. Tests of two phase systems without significant vaporization were begun as well.

### **Presentations**

Ross, H. D., Schiller, D., Disimile, P., and Sirignano, W., "Behavior in Normal and Reduced Gravity of An Enclosed Gas with Nonuniform Heating From Above", *AIAA 27th Aerospace Sciences Meeting*, Reno, Nevada, January 1989.

Ross, H. D., and Disimile, P., "Flow Visualization of a Gas Enclosed in a Circular Cylinder Heated Nonuniformly from Above" *American Physical Society Meeting*, November 1988, Buffalo, NY.

Ignition and Flame Spread Above Liquid Fuel Pools: Gravity Effects

University of California Irvine  
Professor William A. Sirignano  
F. H. Tsau,  
D. N. Schiller  
NAG3-627 (NASA Contact: B. Bane, LeRC)

*Objectives:* To investigate thermal and fluid dynamic behavior of fluids within an axisymmetric container as related to ignition and flame propagation phenomena with liquid fuel pools heated nonuniformly from above. Effects of gravity and surface tension on ignition delay and flame spread are of particular interest. Additionally, the influence of chemical kinetics and vaporization on the flow is also studied. The three-dimensionality of the flow arises from gravity jittering which can come from a variety of sources, e.g., thrust maneuvering of a space vehicle. The jittering effects on the flow are also examined in this research.

*Research Task Description:* (i) A 3-D Cartesian numerical solver is adopted and simple 2-D cases are tested to debug the program; (ii) the above program is then adapted to a more general solver which is capable of doing 3-D rectangular and cylindrical simulations. A simple case is tested with this program; (iii) the above general numerical solver from step (ii) is used to study the target problem. Effects of gravity jittering, variable-property effects and other important consequences from gravity jittering will be examined; (iv) continue parametric studies with two-dimensional code and compare with experimental results from NASA Lewis Research Center; (v) derive scaling laws from the 2-D model suitable for the problem and, eventually, simplify the 2-D model to study more complicated phenomena; (vi) together with Lewis staff, design experiments for reduced gravity facility.

*Progress to Date:* Steps (i) and (ii) were completed successfully. Step (iii) is in progress. Step (iv) and (vi) proceed simultaneously, with numerical model developed at UCI and 2-D experiments carried out at Lewis Research Center. Good agreements were obtained between the numerical and experimental work. Step (v) is in progress and more experiments are planned under Step (vi).

Presentations

H. D. Ross, D. N. Schiller, H. Kim, and W. A. Sirignano, "Behavior in Normal and Reduced Gravity of an Enclosed Liquid/Gas System with Nonuniform Heating From Above," *ALAA 27th Aerospace Sciences Meeting*, Reno, Nevada, January 1989.

D. N. Schiller, B. Abramzon, and W. A. Sirignano, "Enclosed Liquid/Gas System Heated Nonuniformly From Above: Variable Properties and Vaporization Effects," *26th ASME/AICHE Heat Transfer Conference*, Philadelphia, PA, August, 1989.

1. 2. 3. 4. 5. 6. 7. 8. 9. 10. 11. 12. 13. 14. 15. 16. 17. 18. 19. 20. 21. 22. 23. 24. 25. 26. 27. 28. 29. 30. 31. 32. 33. 34. 35. 36. 37. 38. 39. 40. 41. 42. 43. 44. 45. 46. 47. 48. 49. 50. 51. 52. 53. 54. 55. 56. 57. 58. 59. 60. 61. 62. 63. 64. 65. 66. 67. 68. 69. 70. 71. 72. 73. 74. 75. 76. 77. 78. 79. 80. 81. 82. 83. 84. 85. 86. 87. 88. 89. 90. 91. 92. 93. 94. 95. 96. 97. 98. 99. 100.

## 7. EXPERIMENTAL TECHNOLOGY





Residual Acceleration Data on IML-1: Determination of Experiment Sensitivity and Efficient Data Dissemination

University of Alabama in Huntsville  
Center of Microgravity and Materials Research  
Dr. J. Iwan D. Alexander  
Dr. E. Bergman, Charles Stark Draper Labs  
NAG8-759 (NASA Contact: R.S. Snyder, MSFC)  
March 1989 - February 1992

*Objective:* The objective of this research is to develop a mission-specific residual acceleration data reduction and utilization plan. It will be designed to meet the needs of individual experiments and result in a useful, manageable data base. This will enable the principal investigators to assess the acceleration environment (steady and glitter) to which their experiment was exposed, without having to examine the entire acceleration history recorded during the experiment (400 Mbyte/disk, some 20 disks/mission) .

*Research Task Description:* Our basic approach entails the following tasks:

1. Identification of low-g sensitive experiments and sensitivity ranges (magnitude and frequency) through order of magnitude estimates and results of numerical modelling.
2. Research and development toward a plan for reduction and dissemination of residual acceleration data.
3. Use of a "Shuttle Motion Model" to predict accelerations resulting from specified Shuttle maneuvers.
4. Supplementation of the recorded data with calculated predictions for gravity gradient and other accelerations resulting from the basic orbital motion.
5. Implementation on existing residual acceleration data bases (Spacelab 3, etc.)

*Progress to Date:* We are currently addressing the following questions:

1. Can a meaningful "characteristic" background acceleration be defined in terms of steady, oscillatory and transient components?
2. How can significant disturbances (i.e. of concern to a specific experimenter) be identified?
3. How does the orientation and magnitude of both the background level and obvious deviations compare with tolerance limits for a given experiment?

Our work towards answering these questions has involved the time and frequency domain analysis of acceleration data from Spacelab 3. The major problem encountered to date is related to the need for a condensed description of the acceleration environment. During the course of this work we have been able to identify some specific disturbances (such as thruster firings) and the subsequent response of the Spacelab structure. We hope to use these, and previously analysed data, to catalogue recognizable disturbance response signatures characteristic of the Shuttle environment. This will greatly assist future analysis of residual acceleration data.

### Multimode Acoustic Research

Jet Propulsion Laboratory

Dr. Martin Barmatz

NAS7-918

October 1, 1981 - September 30, 1990

*Objectives:* There is a recognized need for high temperature containerless processing facilities that can efficiently position, heat, and manipulate materials in the reduced gravity environment of space. The primary objectives of this task are to develop theoretical models of new classes of levitation, provide experimental validation of these models, and investigate novel methods of sample levitation, manipulation, and heating.

The ultimate goal of this research is to develop sophisticated high temperature positioning and manipulation capabilities. The program tasks include the development of theories for the acoustic potential, forces and torques in uniform and non-uniform temperature environments. These calculations are used to (1) determine those acoustic modes that produce stable levitation, (2) determine operating conditions to avoid translational and rotational instabilities, and (3) determine the shape and position of levitated liquid drops. The task is also experimentally and theoretically investigating the use of microwaves as a sample heating and positioning technique.

Theoretical analyses carried out under this task have predicted stable acoustic levitation in rectangular, cylindrical, and spherical resonators using one acoustic mode of excitation. This single mode levitation theory was verified up to 1000°C in a uniform temperature environment and up to 1500°C for a laser beam heated sample.

This RTOP has been resubmitted for FY'90 with Professor Klaus Bachmann as co-Investigator. The new task titled "Crystal Growth and Directed Reaction of Liquid Metals in a Containerless Environment" will evaluate (a) containerless directional solidification of seeded melts and (b) kinetics of the directed oxidation reaction of liquid metals (Lanxide process).

### Publications

Jackson, H. W., Barmatz, M. and Shipley, C., "Equilibrium Shape and Location of a Liquid Drop Acoustically Positioned in a Resonant Rectangular Chamber," J. Acoust. Soc. Am. 84, 1845, (1988).

Putterman, S., Rudnick, J. and Barmatz, M., "Acoustic Levitation and the Boltzmann-Ehrenfest Principle," J. Acoust. Soc. Am. 85, 68 (1989).

Collas, P. and Barmatz, M., "Acoustic Levitation in the Presence of Gravity," J. Acoust. Soc. Am. 86, 777, 1989.

Rudnick, J. and Barmatz, M., "Theory of Oscillational Instabilities in Acoustic Resonators," J. Acoust. Soc. Am. (accepted).

Barmatz, M., Gaspar, M. and Trinh, E., "Controlled Sample Orientation and Rotation in an Acoustic Levitator," Patent No. 4,777,823, October 1988.

Barmatz, M. "Acoustic Method and Apparatus for controlled Rotation and Orientation of a Levitated Object," Patent No. 4,800,756, January 1989.

## Electrostatic Containerless Processing Technology

Jet Propulsion Laboratory  
Daniel D. Elleman  
Won-Kyu Rhim

*Objectives:* There are several important characteristics of the electrostatic positioning method when viewed against other positioning methods such as the acoustic and the electromagnetic methods. The developmental effort of electrostatic positioning system has been justified on the following basis; (i) It reduces the surface contamination at high temperature since it operates in high vacuum as well as in gaseous environments. (ii) Both conducting and nonconducting samples can be levitated in contrast to the electromagnetic levitation system which requires sample materials having relatively good conductivity. (iii) Since the electrostatic method employs a feedback control principle, the sample experiences control forces only when it is necessary, therefore, allowing a quiescent environment during experiments. Since the first successful demonstration of electrostatic levitation in 1983, the system has been improved and modified in anticipation of various experiments. Initially, we have developed electrostatic positioners which are operable in ambient condition. Feedback-controlled *tetrahedral levitator* and *electrostatic acoustic hybrid system* have opened up the possibilities for containerless crystal growth from solutions as well as the various charged drop-dynamics experiments in micro-gravity environment.

Efforts directed toward high temperature electrostatic-positioning began in FY'89. Main accomplishments have been: (1) the construction of a vacuum system by which electrostatic sample positioning can be investigated at high temperature, (2) Investigation of sample charge variation when sample was heated up to 1300 K, and (3) Development of magnetic sample rotation system operable in vacuum.

Main thrust during FY'90 will be to continue developing high temperature electrostatic positioning system for the microgravity application. Sample temperature will reach 2000 K in selected sample materials. Some of the primary activities will include: (a) Study of sample charging at high temperature, (b) Electrostatic levitation of high density solids and melts, (c) Development of sample manipulation capabilities operable in vacuum, (d) Study of charged drop dynamics, (e) Development of an adaptive feedback control software, and (f) Experiments on board KC-135, the NASA zero-g simulation plane.

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Advanced Containerless Processing Technology: Physical Acoustic Experiments

Vanderbilt University  
Professor Taylor G. Wang  
Dr. Chun P. Lee  
Mr. James L. Allen

**Objectives:** The primary objective of this task is to develop a detailed and quantitative understanding of the interactions between a sample and the acoustic field in a levitation chamber in order to pursue high temperature containerless processing using advanced acoustic methods. Several physical acoustics activities are being undertaken to address this objective. They are to (1) develop advanced chamber systems that provide improved chamber/waveguide coupling and impedance matching and improved sample stability and rotation control; (2) study sample interaction with the higher harmonics generated in the acoustic field; (3) study shaping of liquid drops by acoustic forces; (4) study the acoustic torque on a levitated sample of varying shape and position; and (5) study the effects of feedback systems on levitated sample stability.

**Research Task Description:** Many of the materials research experiments to be conducted in the microgravity science and applications program require manipulation and control of weightless molten materials in a non-contaminating environment. In these experiments, the melt is positioned and formed within a container without physically contacting the container's wall. Acoustic methods developed to date have demonstrated the capabilities of positioning and manipulating a moderate temperature sample. However, at the high processing temperatures desired for many experiments, it becomes increasingly more difficult to introduce the acoustic energy to the chamber due to an increasing acoustic impedance mismatch between chamber and sound sources (acoustic drivers). A new method is being developed that provides improved coupling between the drivers and chamber. By appropriate configuration of the chamber-waveguide-driver system, the efficiency of a pair of drivers can be effectively doubled as compared to the efficiency of a single driver. This will result in a four-fold increase in the acoustic energy transferred to the chamber for a given driver amplitude. This coupling improvement can be extended even further by adding more driver pairs. As a result, improved coupling should allow stable levitation to be achieved at much higher temperatures than are available using current techniques. Experimental testing is required in order to determine the actual coupling improvement and how much higher in temperature stable acoustic levitation can be extended.

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## **B. FLIGHT EXPERIMENTS**





## **1. ELECTRONIC MATERIALS**



### Compound Semiconductor Growth in Space

NASA Langley Research Center  
Dr. A. L. Fripp  
Mr. W. J. Debnam  
Dr. I. O. Clark  
Dr. R. K. Crouch, NASA Headquarters

*Objectives:* The objectives of this research are to determine the effects of gravity driven convection on the growth parameters and crystal properties of compound semiconductors.

*Research Task Description:* The Microgravity Science effort at the Langley Research Center is centered on the growth of the compound semiconductor alloy, lead tin telluride. Lead tin telluride is a substitutional alloy of lead telluride and tin telluride that is miscible over the entire compositional range. The semiconductor properties of this material are dependent on the ratio of the two components and consequently, the uniformity of an array of devices is dependent on good compositional control.

This material was chosen for microgravity research for a number of reasons. Lead tin telluride is not only a useful semiconductor material which has been used for construction of infrared detectors and tunable diode lasers but it also has a similar phase diagram to other compound semiconductors of interest such as mercury cadmium telluride and mercury zinc telluride. Lead tin telluride is very interesting from a purely scientific point of view in that it is both solutially and thermally unstable, but in a one dimensional analysis with growth axis parallel to the gravity vector, only one instability works, per orientation, at a time. This double convective instability cannot be made stable by balancing thermal and solutal expansion in a high temperature gradient. Lead tin telluride is amenable to study for it is easily compounded; it has a relatively low vapor pressure; it is single phase and there is existing, though limited, literature on its growth and properties.

The desired growth mode is of course one in which convection is zero so compositional steady state can be reached. However fluid dynamic calculations have shown that finite convection exists in the physical configuration used in crystal growth experiments even at  $1 \times 10^{-8}$  Earth gravity if there is a density gradient orthogonal to the gravity vector. However, due to the residual atmosphere the minimum gravity level expected on the Space Shuttle is roughly the order of  $1 \times 10^{-7}$  Earth gravity. Hence experiments are designed such that interface movement ie, growth rate, is greater than the anticipated fluid velocity.

Other work at Langley ranges from fundamental studies involving the direct measurement of both steady and oscillatory fluid flow in a crystal growth melt to efforts in furnace design and calibration. Also of interest is an effort to measure the melt-solid interface shape and position as this is of great importance to understanding the crystal growth process.

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### Vapor Crystal Growth of Mercuric Iodide

EG&G Measurements, Inc.  
Dr. Lodewijk van den Berg  
H-78559B

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

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

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

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

Preparations are under way for the next experiment, to be flown on the first flight of the International Microgravity Laboratory. Present groundbased research and experiment development activities concentrate on improving the control system of the flight equipment and increasing the temperature of the growth process so that larger crystals can be obtained in the limited time available during the flight.

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## Vapor Growth of Alloy-Type Semiconductor Crystals

Rensselaer Polytechnic Institute  
Professor Heribert Wiedemeier  
NAS8-32936 (NASA Contact: D.A. Schaefer, MSFC)  
March 1978 - October 1989

*Objectives:* The present effort is part of a continuing research program directed towards the investigation of basic vapor transport phenomena and of crystal growth properties of electronic materials. The primary objectives of ground-based studies are the development and definition of optimum experimental parameters for flight experiments. The specific experiments to be performed in a microgravity environment include the investigation of vapor transport and crystal growth phenomena of the  $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$ - $\text{HgI}_2$  system. Emphasis for this system is on the mass flux, on the unseeded and seeded growth of bulk crystals, and on the growth of epitaxial layers. The above experiments are performed in closed, fused silica ampoules.

The objectives of the  $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$  experiments are to determine the positive effects of microgravity on vapor phase crystal growth of ternary, alloy-type materials in terms of chemical and structural microhomogeneity, and of electrical properties of crystals grown in space. In order to achieve these goals it is necessary to perform corresponding crystal growth studies on ground which will serve as a comparative basis for the meaningful analysis of microgravity effects. Thus, the objectives of ground-based studies are to determine the effects of gravity-driven convection on mass transport rates and on crystal morphology for the bulk and epitaxial growth of  $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$  crystals. These objectives include the thermodynamic and fluid dynamic analysis of the mass transport processes, and the characterization of grown crystals in terms of defects and of electrical properties. The ultimate goal is a quantitative correlation between crystal morphology, electrical properties, and growth parameters for this system.

*Research Task Description:* Earlier studies demonstrated the effects of gravity-driven convection on mass flux and morphology of  $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$  bulk crystals. The continued experimental tasks are directed towards the investigation of the effects of temperature profile and of the geometry of the condensation region of the ampoule on mass flux and crystal morphology. In addition, the effects of aspect ratio of the ampoule on the mass transport and crystal growth properties will be investigated. The ground-based tasks include the investigation of the above effects for different orientations of the density gradient with respect to the gravity vector, and for different transport agent ( $\text{HgI}_2$ ) pressures and temperature profiles. The crystal characterization techniques include various microscopic and x-ray diffraction methods, chemical etching, and electrical measurements. The ground-based studies are designed to optimize experimental parameters for future flight experiments.

The major tasks of ground-based studies of the epitaxial growth of  $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$  layers by chemical vapor transport reactions involve systematic investigations of the growth rate, morphology, homogeneity, and electrical properties of  $\text{HgCdTe}$  layers. These studies include measurements of the effects of substrate orientation relative to the density gradient, of temperature profile effects, and of transport agent pressure on the above properties. They are performed under horizontal and vertical stabilizing conditions with the goal to observe the effects of convective interferences on layer morphology and properties. The results of on-going ground-based studies are continuously evaluated and are used for the systematic improvement of growth parameters with the important goal to define optimum experimental conditions for the microgravity experiments of this system.

In addition to the experimental tasks, theoretical efforts involve the quantitative thermodynamic analysis of the system under investigation, the computation of fluid dynamic parameters, and the consideration of other possible effects on fluid flow and crystal growth under vertical, stabilizing and microgravity conditions. An important aspect of the theoretical effort is the further development and improvement of transport models for diffusion limited mass transport of simple and of multi-component,

multi-reaction vapor transport systems.

*Progress to Date:* Earlier accomplishments under this program include quantitative mass flux measurements and the analysis of crystal growth properties of bulk and layer-type crystals. These investigations demonstrate the effects of even minute fluid dynamic disturbances on the chemical and structural microhomogeneity of grown crystals. Dynamic microbalance techniques were employed, for the first time, to determine quantitatively the vapor pressure of Hg over  $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$  for different compositions within the homogeneity range. This work led to the direct in-situ determination of the Hg vacancy concentration and to the derivation of the enthalpy of vacancy formation for this important material. Work in progress is aimed at the refinement of these data. These results provide valuable information for the further elucidation of the mechanism of vacancy formation of  $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$ . Our recently developed thermodynamic model of the  $\text{Hg}_{0.8}\text{Cd}_{0.2}\text{Te}$ -iodine transport system has been extended to predict the composition of the transported crystals for different pressures and temperatures. Considering the complexity of the  $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$ - $\text{HgI}_2$  transport system, the theoretically predicted compositions are in good agreement with experimental observations for a wide range of compositions. This computational model is expected to be generally applicable for the prediction of mass transport properties in terms of mass flux and overall composition of transport products for the diffusion dominated vapor transport of ternary alloy-type materials. Such a predictive capability is of practical significance for the design, performance, and evaluation of crystal growth experiments on earth and in microgravity environment.

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



Alloy Undercooling Experiments in Microgravity Environment

Massachusetts Institute of Technology  
Professor Merton C. Flemings  
NAS3-24875 (NASA Contact: F. Harf, LeRC)  
March 1, 1988 - September 30, 1989

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

The scientific aim of the current and proposed modeling studies and ground-based experiments is to obtain baseline data and a baseline understanding of nucleation, growth, solidification, and structure of highly undercooled alloys on which comparable experiments will be conducted in space. Concomitantly, experimental techniques are being developed for performing and analyzing undercooling experiments in space and on the ground.

*Research Task Description:* The first alloy undercooling experiment in a microgravity environment was performed during the Columbia STS 61-C mission in January 1986. The results of the flight experiment have been published. The directly related ground-based experimental and analytical studies include thermal history measurement during the rapid recalescence and solidification of undercooled alloys, dendrite growth rate measurements, metallographic studies, and modeling of dendritic growth in undercooled melts. These results have been reported in published papers.

*Progress To Date:* Undercooling experiments have been performed using Ni-Sn, Fe-Ni, and Fe-metalloid alloys. New techniques have been developed for the study of undercooled melts containerless processing. A system for induction melting and solidification of iron and nickel base alloys with simultaneous temperature measurement has been developed. High speed cinematography has been utilized to observe the solidification of undercooled Ni-25%Sn alloy. Reheating curves measured during recalescence have been used to obtain dendrite tip velocity, fraction solid after recalescence, and other solidification features. Microstructural and microanalysis of samples produced were related to thermal and cinematographic results. Recent work has emphasized techniques for interrupted solidification studies and for video taping surfaces of samples during undercooling, recalescence, and final solidification.

The thermal data and cinematographic data have provided direct insight into the mechanism of solidification of undercooled metal alloys and have led to the development of detailed model of growth of alloy dendrites in undercooled melts. Measurements of dendrite tip velocity showed good agreement with predictions based on recent theories of steady state free dendritic growth. The maximum recalescence temperature has been found to be the temperature predicted by an adiabatic thermal balance combined with the equilibrium lever rule. Recalescence times have been found to decrease rapidly with increasing undercooling, and total solidification times have been found to decrease with increasing undercooling. Dendritic growth in undercooled melts can occur with a unique morphology of the growth front which has been described in the publications. The majority of the dendrite arms disappear during coarsening. The dendrite morphology undergoes a gradual and continuous microstructural transition with increasing undercooling. A detailed and comprehensive model was developed for processes occurring behind the dendrite tips during recalescence and subsequent solidification.

In order to understand the effect of composition and freezing range on the behavior of undercooled alloys, the results of experiments on low tin content Ni-Sn alloys have been compared to earlier results obtained in experiments with Ni-25%Sn alloys. Dilute alloys show significantly higher dendrite growth velocities, and interpretation of the growth models indicate high volume fractions of solid

form, initially, without segregation. Evidence has been found that the solid can be superheated during rapid recalescence and later rapidly remelt. The experimental work on glass coated Ni-Sn alloys has been completed.

Experimental work on Fe-Ni alloys (5%, 10%, and 30% Ni) has confirmed the growth models developed from observations of the Ni-Sn system. Fe-Ni alloys are being studied, in part, because of the narrow equilibrium solidification range. At high undercoolings dendrite growth velocities are very high and nonequilibrium solute partitioning is an important consideration. In addition, in the Fe-Ni peritectic system, a double recalescence phenomenon has been observed in alloys that normally would solidify with FCC austenite as the primary phase. Apparently, BCC delta ferrite nucleates preferentially, and the austenite nucleates after recalescence of the metastable delta phase.

Initial experiments on Fe-metalloid alloys have provided interesting observations. Experiments have been performed made with Fe-B, Fe-P, and Fe-Si alloys. These alloys are interesting because of their glass forming ability, because of their very low partition ratios, and because of their wide freezing ranges. Observed dendrite growth velocities in these alloys have been measured, and are observed to be much slower than in the earlier studied alloys. Recalescence times are one to three orders of magnitude slower. These alloys provide the potential for substantially increasing our understanding of dendritic and nonequilibrium solidification of undercooled alloys.

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Libera, M. R. and Shiohara, Y., "Analysis of Metastable BCC Phase Formation in Atomized Fe-Ni," in *Proceedings of MRS International Meeting on Advanced Materials*, May-June 1988 (in press).

### Isothermal Dendritic Growth Experiment

Rensselaer Polytechnic Institute  
Dr. Martin E. Glicksman  
Richard C. Hahn  
Shakeel H. Tirmizi  
Martin E. Selleck  
NAS3-25368 (NASA Contact: E. Winsa, LeRC)  
October 1988 - November 1989

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

*Research Task Description:* The Rensselaer Polytechnic Institute team is primarily concerned with the following aspects of the IDGE program: (1) engineering and development of the growth chamber for the flight experiment; (2) analysis and reduction of data accumulated during the flight experiment and during ground based testing; and (3) all "scientific" issues associated with the experiment.

*Progress to Date:*

#### Growth Chamber Design and Development

The major effort during the year 1988 was toward the design and testing of the flight growth chamber. Three units of the flight-type growth chamber (termed MOD I) were fabricated and tested. Preliminary results from these tests are quite encouraging. All aspects of the growth chamber performance are satisfactory and indicate that the growth chamber should perform adequately under microgravity conditions as well. No design changes are necessary, however, the present tests suggest several design modifications which would make the chamber perform optimally under terrestrial and microgravity conditions. The design modifications which we plan to incorporate in MOD II will be done keeping in mind flights 2 and 3 which are expected to follow within one year of flight 1.

Extensive tests were done on all the three MOD I chambers enabling us to critically test their performance and to suggest possible design modifications. These tests are summarized below:

##### 1) Plateau tests:

In-situ measurement of the melting point of the sample succinonitrile (SCN) is required during flight. The plateau tests showed that the thermistor assembly is capable of producing a melting point plateau which is flat and for a reasonably long duration to allow us to select the melting point accurately during flight.

##### 2) Recording gross melting and freezing:

Time-lapse video films were made of the SCN sample melting and freezing during plateau tests. These films help us understand better the growth chamber performance and suggest some design modifications. Films during melting show that gravity aids in providing a long-duration plateau. We then performed plateau tests with the growth chamber inverted, where gravity would be acting against producing a long-duration plateau. The resulting plateau under the inverted situation did produce an acceptable plateau, albeit for a shorter duration. This result showed that the chamber would produce an acceptable plateau under microgravity conditions as well.

### 3) Growth initiation (nucleation) tests:

Dendritic growth inside the chamber is initiated via a thermoelectric spot cooler assembly. We were able to initiate growth on command under all conditions of supercoolings of interest ranging from 0.05 to 1.0 C. Two sets of coolers are installed and we were able to induce consistent and reliable nucleation with only one set of coolers operating at 60% of its capacity. The spot cooler assembly thus has a safety factor greater than 2.0 and a complete redundancy as well.

### 4) Recording "free" dendritic growth:

Growth sequence of dendrites as they emerged out of the stinger to grow freely inside the chamber were recorded on video tapes. This was the most critical test of the performance of the growth chamber and the results were encouraging. We were successfully able to measure growth velocity of the emerging dendrites as a function of supercooling. Qualitative estimates of tip radii were also made.

From the above tests, we concluded that the growth chamber is performing satisfactorily under terrestrial conditions and that it should perform well under space flight conditions as well. Several design modifications are suggested as a result of the above tests. These modifications are aimed at making the growth chamber perform optimally under microgravity conditions. The suggested modifications are listed below:

- a) Reduction in overall volume of the growth chamber: this would be especially desirable for the flight where succinonitrile will be replaced by pivalic acid (PVA) as the model material for dendritic growth study.
- b) Window redesign: the present design is prone to leaks. New design would use SS 304 windows which can be welded to the body of the chamber (also made of SS 304) in a leak-free manner.
- c) Increase the window viewing area: video films on free dendritic growth suggest that the present 35mm photographic film format may not provide an adequate field-of-view at a magnification of 4x. A lower magnification of 2x is desirable and this would require the larger window viewing area.
- d) Thermistor probe design: we would like to place two thermistors inside the probe to make the temperature measurement inside the chamber. This would provide a redundancy in the SCN temperature measurement.
- e) Fill tube protection: MOD I has the somewhat fragile fill tube sticking out of the chamber. MOD II will provide some protection to this fill tube.
- f) Intermediate leak checking: we plan to helium leak check the chamber during several phases of fabrication as part of the quality assurance procedure.
- g) Spot cooler design: changes will be made to enable us test and install the spot cooler assembly as an independent unit.
- h) Stinger redesign: the stinger needs to be protected and the sequence of stinger welds need to be altered to ensure that it is free of metal particles generated during fabrication.

### Testing of the IDGE Prototype Flight Hardware

NASA Lewis is conducting extensive tests on the IDGE hardware. The entire experimental apparatus was subjected to vibration tests. All the key components, including the crystal growth chamber passed the NASA vibration test.

Extensive tests were carried out on the photographic system. A complete 72 hour experimental run, simulating the actual space flight experiment was carried out. The complete dendritic growth sequence including melting of the SCN sample, supercooling the bath, controlling the bath temperature, initiating dendritic growth, detecting crystal growth by means of the optic RAM and photographing the dendrite growth sequence on to 35 mm films, was done in an autonomous manner controlling the experiment by a computer using the software developed at NASA Lewis. The experimental data gathered during this test was analyzed by the RPI team. Preliminary indications on the performance of the IDGE setup are quite encouraging. Analysis of the photographic films using the analysis scheme developed at RPI suggest that some modifications are necessary to optimize the photographic system. Extensive tests, including optical ray tracing, photographing test targets etc. are being carried out at NASA Lewis in an attempt to further optimize the photographic system.

### Development of Telescience Capabilities

The IDGE will have limited telescience capabilities. Software is being developed to display the real-time data on user friendly computer screens. These "science" and "engineering" data screens will assist the IDGE investigators at the Payload Operations and Command Center to make judgements on the status of the experiment in real time and suggest possible changes in the experimental protocol. This will permit the most efficient means of conducting the space shuttle experiment.

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Casting and Solidification Technology (CAST)

University of Tennessee Space Institute  
Dr. Mary Helen McCay  
Dr. T. Dwayne McCay  
Dr. Robert Owen  
Ronald Porter  
Montgomery Smith  
NAS8-37292 (NASA Contact: R. C. Ruff, MSFC)  
December 1986 - December 1990

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

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

*Progress to Date:* A double optics system was assembled that provides confocal processing and particle tracking capabilities. The onset of connective instabilities within the inverted layer are being documented with this apparatus. A cuvette design that would accommodate shrinkage and expansion of the fluid was completed. An instrumented cuvette was assembled and operated under typical solidification conditions. Comparisons of concentration and temperature fields were made with optical and analytical predictions. A KC-135 flight rig was assembled incorporating a confocal optical system and VCR data recording. The advanced fluid thermal model has progressed continually and a graphics capability has been added so the solidification process can be viewed in real time as it is being calculated. Ground Control Runs have been completed on the FES hardware. The PI's are presently waiting for the opportunity to perform science runs in the Holographic Ground System.

Publications

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#### Presentations

McCay, T. D., McCay, M. H., Lowry, S. A. and Smith, L. M., "Convective Effects on Directional Solidification of a Simulated Metal Alloy," *27th ALAA Meeting*, Reno, Nevada, January 1988, AIAA Paper 88-0258.

### **3. FLUIDS, INTERFACES, AND TRANSPORT**



### Surface Tension Driven Convection

Case Western Reserve University  
Professor Simon Ostrach  
Professor Y. Kamotani  
NAG3-570 (NASA Contact: T.P. Jacobson, LeRC)  
January 1989 - October 31, 1989

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

*Research Task Description:* The experiment consists of a circular container (5 cm dia. and 5 cm deep) filled with silicone oil, heating systems, and a data acquisition system. The fluid free surface will be heated locally by a CO<sub>2</sub> laser or by a submerged circular heater placed at the center. The resultant temperature variation along the free surface will generate thermocapillary flow in the container. The flow field will be studied by a flow visualization technique and the temperature distribution along the free surface, which is important because it determines the driving force of the flow, will be measured by a thermography technique. The surface heat flux distribution, the heating level, and the static free surface shape will be varied to study their effects on the nature and extent of the flows. Two series of experiments are planned. In the first one, the basic thermocapillary flow will be studied and attempts will be made to obtain oscillatory thermocapillary flow. In the second series the oscillation phenomenon will be studied in detail because it is considered to be an important aspect of thermocapillary flow. Ground-based and drop tower experiments together with a numerical analysis have been made to provide base data and to insure that the operating condition and the configuration will lead to flows that can be reasonably observed and measured.

*Progress to Date:* The fabrication of the engineering model of the experiment and the design of the flight hardware are being done at NASA Lewis Research Center. The development of the diagnostic techniques (flow visualization technique, surface temperature measurement, etc.) are also being done at Lewis.

The supporting ground-based science work is being performed at CWRU. The study of oscillatory thermocapillary flow is being conducted using a small scale model of the test cell. The study of the oscillation mechanism and the scaling analysis of thermocapillary flow are nearly completed.

### Publications

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Kamotani, Y. and Lee, K. G., "Oscillatory Thermocapillary Flow in a Liquid Column Heated By a Ring Heater," PCH Journal, 1989 (in press).

## Presentations

Ostrach, S., "Thermocapillary Flow Experiment in Space" *Gordon Research Conference*, August, 1989.

Ostrach, S., "Flow Driven Surface Tension Gradient," *Carl Gunnard Johnson Colloquium Lecture*, WPI, Nov., 1989.

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

Massachusetts Institute of Technology

Professor Julian Szekely

Dr. O. J. Ilegbusi

Dr. J.-H. Zong

NAS3-25074 (NASA Contact: F. Harf, LeRC)

*Objectives:* The objective of this research is to develop an improved fundamental understanding of electromagnetic, heat flow and fluid flow phenomena in levitation melted specimens under both earthbound and microgravity conditions. The main motivation of this work is two fold: (1) a number of fundamental hydrodynamic and electromagnetic issues may be uniquely addressed in this manner, and (2) levitation melting is a key ingredient of many materials processing experiments in space, thus the present project provides an important support function for this effort.

*Research Task Description:* The current research pursues three complementary directions: (1) extensive computational work is being carried out to predict the electromagnetic force field, the velocity field and the temperature fields in electromagnetically-stirred (-positioned) metallic specimens. An important novel feature of this effort is that an allowance is being made for the behavior of free surfaces and free surface deformation; (2) experimental work has been carried out to measure and predict electromagnetically-driven flows in a molten Wood's metal due to the passage of current between two electrodes; and (3) calculations are being carried out to support a planned in-flight experiment aimed at measuring the viscosity of undercooled melts using a levitation melted specimen.

*Progress to Date:* Important milestones of the research include the following: (1) the development of a general methodology for computing electromagnetic force fields and the corresponding melt velocity fields in complex geometries; (2) the experimental measurement of electromagnetically-driven flows in Wood's metal melts, for both steady and fluctuating electromagnetic force fields; and (3) the development of a new initiative for measuring the viscosity of undercooled melts using levitation melted metallic droplets.

#### Publications

A. Murthy, J. Szekeley and N. El-Kaddah, "Experimental Measurement and Numerical Computation of Velocity and Turbulence Parameters in a Heated Liquid Metal System," Met. Trans. **19B**, 765-775 (1988).





#### 4. BIOTECHNOLOGY



Cell Partition in Two Polymer Aqueous Phases

Dr. Donald E. Brooks, Oregon Health Sciences University  
Dr. James Van Alstine, USRA  
Dr. J. Milton Harris, UAH

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

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

In low-g, phase emulsions demix to yield one phase floating like an egg yolk, surrounded by the phase which preferentially wets the container wall. Current research is aimed at controlling the rate of demixing and final disposition of the phases via both passive means (e.g., altered chamber geometry or polymeric wall coatings with different wetting properties) and active means (electrophoresis of the phase whose interfaces exhibit zeta potentials). In addition, variables such as interfacial tension, phase volume ratios and phase viscosity, are being studied to better understand their influence on demixing of the phases on both low-g and 1-g. Many of these variables were successfully investigated in another passive demixing Phase Partitioning Experiment (PPE) flown on STS-26. The demixing processes under study are relevant to a variety of demixing phenomena in materials processing. Further studies including the separating of test cell preparations, are anticipated for IML-1, Spacehab-1, and IML-2 missions.

## Publications

Van Alstine, J. M., Karr, L. J., Harris, J. M., Snyder, R. S., Bamberger, S., Matsos, H. C., Curreri, P. A., Boyce, J., and Brooks, D. E., "Phase Partitioning in Space and on Earth," in *Immunobiology of Proteins and Peptides IV* (M.Z. Atassi, ed.), Plenum Press, 1988, pp. 305-326.

Brooks, D. E., "Cell Partitioning in Two Polymer Phase Systems: Towards Higher Resolution Separations," in *Frontiers in Bioprocessing* (M. Bier, S.K. Sikdar, and P. Todd, eds.), CRC Press, 1988.

Boyce, J. F., Harris, J. M., Van Alstine, J. M., and Brooks, D. E., "Dextran Coated Glass Slides as a Model for Cell Surface Interactions with Aqueous Two Phase Polymer Systems," in *Advances in Separations Using Aqueous Phase Systems in Biology and Biotechnology* (D. Fisher and I.A. Sutherland, eds.), Plenum Press, 1989, pp. 239-247.

Van Alstine, J. M., Brooks, D. E., Sharp, K. A., Snyder, R. S., Karr, L. J., and Harris, J. M., "Novel Uses of Polymer Phase Partition Affinity Ligands," in *Advances in Separations Using Aqueous Phase Systems in Biology and Biotechnology* (D. Fisher and I.A. Sutherland, eds.), Plenum Press, 1989 (in press).

Bamberger, S. and Van Alstine, J. M., "Phase Partitioning in Reduced Gravity," *Progress in Low Gravity Fluid Dynamics and Transport Phenomena* (J.N. Koster and R.L. Sani, eds.), AIAA, 1989 (in press).

Van Alstine, J. M., Bamberger, S., Harris, J. M., Snyder, R. S., Boyce, J. F., and Brooks, D. E., "Phase Partitioning Experiments on Shuttle Flight STS-26," in *Proceedings of 7th European Symposium on Materials Sciences under Microgravity*, ESA SP-295, 1989 (in press).

## Protein Crystal Growth in a Microgravity Environment

University of Alabama at Birmingham

Dr. Charles E. Bugg

NAS8-36611 (NASA Contact: K. Johnson, MSFC)

November 1988 - October 1989

*Objectives:* The long range objective for this research task is to develop systematic and reliable techniques and hardware for growing protein crystals in space. Studies will be performed to evaluate the potential for enhanced protein crystal growth under microgravity conditions. Fundamental studies of protein crystal growth, both on the ground and in space will be performed in order to identify the major parameters that affect protein crystal growth. The fundamental studies will be used to define science requirements to be incorporated into advanced hardware that permit investigators to dynamically monitor and control the major parameters in protein crystal growth.

*Research Task Description:* This research program involves a multidisciplinary effort to produce protein crystals in space of sufficient quality and size to permit molecular structural characterization by x-ray crystallography, while simultaneously providing basic ground-based experimental and theoretical supporting research to develop a better understanding of protein crystal growth and to determine if gravity plays a limiting role in the growth process. Beginning with the Apollo program and extending into the Spacelab program, it has been demonstrated that the microgravity environment can provide stable growth conditions that can result in crystals with improved homogeneity and fewer defects. In this program, a variety of proteins will be crystallized on space shuttle flights over a three-year period. Optimum techniques for reliably growing protein crystals under microgravity conditions will be developed. Initially, emphasis will be placed on modified vapor diffusion techniques such as the "hanging drop method" that is widely used for ground-based studies in protein crystal growth. Long-range plans include development of new methods for growing protein crystals, based upon the experimental and theoretical studies performed as part of this research program. A major goal is to develop advanced hardware that will be available for use on the Space Station.

*Progress to Date:* Protein crystal growth hardware for use on the space shuttle was developed during the period between Challenger and STS-26. The hardware was constructed at Teledyne Brown Engineering Company, based upon the experience gained with four mid-deck flights in 1985 and 1986. The hardware includes a temperature control module, replaces a mid-deck locker, and contains three trays that permit 60 vapor diffusion experiments.

The hardware was flown in the mid-deck on shuttle flight STS-26 in September 1988. Three of the proteins included among the experiments on this flight produced crystals that were suitable for complete x-ray diffraction analysis. These three proteins are  $\gamma$ -interferon, porcine elastase, and isocitrate lyase. The proteins had been studied extensively in crystal growth experiments on earth prior to the microgravity experiments. Analyses of the space-grown crystals included collection of three-dimensional intensity data sets with electronic area detector systems. Comparisons of the microgravity-grown crystals with the best earth-grown crystals obtained in numerous experiments demonstrate that the microgravity grown crystals of these three proteins are larger, display more uniform morphologies, and crystals of these three proteins are larger, display more uniform morphologies, and yield diffraction data to significantly higher resolutions. Analyses of the three-dimensional data sets by relative-Wilson plots indicate that the space grown crystals are more highly ordered at the molecular level than their earth-grown counterparts.

The hardware was flown again on STS-29 in March 1989. Problems were encountered on this mission, resulting in showers of small crystals in nearly all of the experiments. Despite these experimental problems, crystals of  $\gamma$ -interferon, which were suitable for x-ray diffraction analysis, were obtained. Although these  $\gamma$ -interferon crystals were no larger than those obtained previously on earth,

they confirmed the finding from STS-26 that the microgravity-grown crystals diffract to higher resolution than earth-grown crystals, and display relative-Wilson plots that indicate enhanced molecular order.

Along with the hardware development program, a number of basic studies in protein crystal growth have been completed. Particular emphasis has been placed on methods for detecting initial nucleation events, using laser light scattering and other optical techniques. As part of this project, investigators at The University of Alabama in Huntsville have determined the phase diagram for crystallization of lysozyme. Other work supported under this contract at Georgia Institute of Technology has led to the development of an automated system for dynamic control of vapor diffusion rates. These studies will be combined together to define hardware and techniques for dynamic control and monitoring of protein crystal growth parameters.

#### Publications

DeLucas, L. J., Smith, C. D., Smith, H. W., Senadhi, V-K, Senadhi, S. E., Ealick, S. E., Carter, D. C., Snyder, R. S., Weber, P. D., Salemme, F. R., Ohlendorf, D. H., Einspahr, H. M., Navia, M. A., McKeever, B. M., Nagabhushan, T. L., Nelson, G., McPherson, A., Koszelak, S., Taylor, G. Stammers, D., Powell, K., Darby, G., and Bugg, C. E. "Protein Crystal Growth in Microgravity," Science **246**, 651-654 (1989).

DeLucas, L. J., Smith, C. D., Smith, H. W., Senadhi, V-K, Senadhi, S. E., Ealick, S. E., Carter, D. C., Snyder, R. S., Weber, P. D., Salemme, F. R., Ohlendorf, D. H., Einspahr, H. M., Navia, M. A., McKeever, B. M., Nagabhushan, T. L., Nelson, G., McPherson, A., Koszelak, S., Taylor, G. Stammers, D., Powell, K., Darby, G., and Bugg, C. E. "Protein Crystal Growth Results for Shuttle Flights STS-26 and STS-29," J. Cryst. Growth, 1989 (in press).

## **5. COMBUSTION SCIENCE**





Scientific Support for an Orbiter Middeck Experiment on Solid Surface Combustion

Mississippi State University  
Professor Robert A. Altenkirch  
Dr. S. Bhattacharjee  
Mr. Jeff West  
NAS3-23901 (NASA Contact: S. Olson, LeRC)  
December 19, 1984-December 20, 1992

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

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

An experiment has been designed for the Middeck of the Space Shuttle to aid in understanding the process of flame spreading in the absence of a buoyancy-driven flow.

A chamber of approximately  $0.035 \text{ m}^3$  in volume is to contain either a thin sample of a cellulosic material or a thick sample of polymethylmethacrylate and an oxidizing environment  $\text{O}_2$  and  $\text{N}_2$ . Samples will be ignited at one end, and the ensuing flame spread process will be filmed. The spread rate can be determined from the films, and surface and gas--phase temperatures just above the surface will also be recorded. A matrix of eight experiments to be carried out on the Middeck has been identified. These data will help to clarify the mechanism of forward heat transfer in the low--gravity flames.

The experimental apparatus has been constructed at NASA's Lewis Research Center and tested in the Drop Tower facilities. Methods of data reduction and theoretical analyses of the reduced-gravity flame spread problem have been developed and are being refined. Results to date show that measured spread rates over thin cellulosic fuels are less at microgravity than for downward spread in normal gravity. Theoretically predicted spread rates not accounting for any effects of radiation heat transfer are generally higher than what is measured. Inclusion of surface reradiation in the modelling reduces the predicted spread rates and brings them more in line with experiment. Gas--phase radiation provides a feedback mechanism to the solid and a loss mechanism to the environment such that the effect of gas--phase radiation on the flame spread rate is rather complicated. Consideration of gas--phase radiation results in predicted spread rate trends that qualitatively compare favorably with experiment.

*Progress to Date:* Computational methods for determining gas--to--solid heat flux from experimental solid--surface temperature data for thin solid combustibles have been developed and applied to experimental drop tower results. The computational model is now able to account for finite--rate gas--phase and solid--phase chemistry, surface reradiation, and gas--phase radiation. In considering gas--phase radiation, an assumption is made that the radiation fed back to the surface and absorbed is approximately equal to that reradiated such that the net effect of gas--phase radiation is to provide a mechanism of heat loss to the environment.

## Publications

Altenkirch, R.A. and Vedha-Nayagam, M., "Opposed-Flow Flame Spread and Extinction in Mixed-Convection Boundary Layers," Twenty-Second Symposium (International) on Combustion, The Combustion Institute, Pittsburgh, pp. 1495--1500, 1989.

Bhattacharjee, S., Altenkirch, R.A., Srikantaiah, N., and Vedha-Nayagam, M., "A Theoretical Description of Flame Spreading over Solid Combustibles in a Quiescent Environment at Zero Gravity," Comb. Sci. & Techn., 1989 (submitted).

## Presentations

Vento, D., Zavesky, R., Sacksteder, K., and Altenkirch, R., "The Solid Surface Combustion Space Shuttle Experiment Hardware Description and Ground-Based Test Results," *27th Aerospace Sciences Meeting*, Reno, NV, January 1989, AIAA Paper 89-0503 (also NASA TM 101963).

Altenkirch, R.A., "Flame Spread over Solid Fuels," invited presentation at the International Microgravity Combustion Workshop, NASA Lewis Research Center, Cleveland, OH, January 25-26, 1989.

Bhattacharjee, S. and Altenkirch, R.A., "The Effect of Surface Radiation on Flame Spread in a Quiescent, Microgravity Environment," *Central States Section/The Combustion Institute Meeting*, Dearborn, MI, April 03-May 3, 1989.

Bhattacharjee, S. and Altenkirch, R.A., "Radiative Effects in Opposed-Flow Flame Spread over Thin Fuels," presented at the *Eastern Section/The Combustion Institute Meeting*, Albany, NY, October 30-November 1, 1989.

**C. PHYSICS AND CHEMISTRY EXPERIMENTS (PACE)**



Precise Viscosity Measurements Very Close to Critical Points

National Institute of Standards and Technology

Dr. Robert F. Berg

Dr. Michael R. Moldover

C-32001-K (NASA Contact: R.A. Wilkinson, LeRC)

October 1, 1988 - September 30, 1989

*Objectives* Our objective is to accurately measure the viscosity of a pure fluid near its liquid-vapor point. The space experiment will be the fourth of a series of tasks which are: (1) theoretical studies, (2) critical viscosity measurements of binary liquid mixtures, (3) critical viscosity measurements of pure fluids in 1-g, and (4) measurements of pure fluids in low gravity.

*Research Task Description:* Near the critical temperature  $T_c$  the viscosity  $\eta$  is predicted to diverge as:

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

This functional form had been verified for binary mixtures near their consolute points but with apparent exponents  $\gamma$  as low as the theoretical value (0.032) to as high as 0.042. In pure fluids near their liquid-vapor critical points measurements of  $\gamma$  were even more uncertain, in part because of gravitational stratification near  $T_c$ . The task is to improve the measurement of  $\gamma$ , first on earth and then in a microgravity environment.

*Progress to Date:* We have developed a unique series of viscometers devoted to critical point measurements with both low frequency and low shear operation in a thermostat with sub-millikelvin thermal perturbations. Their application to four binary mixtures narrowed the range of the experimental exponent to 0.040 - 0.044, a result inconsistent with theory. We have also measured the viscosity of CO<sub>2</sub> and xenon near their critical points. The xenon measurements will be an essential input for analyzing the results of the Zeno microgravity light scattering experiment. Our preliminary analysis of the carbon dioxide and xenon data incorporates crossover theory and 1-g stratification effects. The results are in agreement with our binary mixture data and not theoretical predictions.

We measured the vibration sensitivity of the existing 1-g viscometer in order to specify vibration isolation requirements for a possible Shuttle flight. The experimental results and theory show that vibration isolation will be a serious engineering challenge: Near the operating frequency of our viscometer, the ambient acceleration must be less than  $4 \times 10^{-8} \text{ g/Hz}^{1/2}$ .

We are investigating the feasibility of a magnetically suspended, oscillating sphere viscometer through a collaboration with the University of Virginia. We have obtained the first complete hydrodynamic description of this viscometer's behavior, thus enabling us to optimize its design, improve its accuracy, and adapt it to nearly critical fluids.

In cooperation with A. Wilkinson of NASA (LeRC) and R. Gammon of the University of Maryland we organized an international workshop on "Equilibration Near the Critical Point" which was held on March 16-17, 1989 at NIST. This workshop provided the first forum for a topic of great practical importance for low gravity critical point experiments and scientifically interesting in its own right. Several scientific papers stimulated by the workshop are in preprint form. The workshop also stimulated a critical point equilibration experiment, scheduled for IML-1. We have contributed to the design and fabrication of the initial cells, filled several cells with SF<sub>6</sub> at critical density, tested the cells' upper temperature limit, and tested for contaminants leaching from the magnetic stirrer in the cell.

## Bubble-In-Liquid Mass Transport Phenomena in a Reduced Gravity Environment

University of Toledo  
Professor Kenneth J. De Witt  
Chain-Nan Yung  
Jonathan L. Brockwell, Union Carbide Technical Center  
NAG3-334 (NASA Contact: A.T. Chai, LeRC)  
November 15, 1983 - January 1, 1989

*Objectives:* The long term objective of the experiment is to observe the dissolution of an isolated gas bubble which is initially of specified size and composition in a thermostatted solvent liquid of known concentration in the reduced gravity environment of earth orbit. The critical radius phenomenon is utilized to maintain the bubble in a state of unstable equilibrium. Subsequent bubble dissolution from the stable critical state is initiated by a step increase in the liquid pressure. A numerical model has been developed which simulates the bubble dynamics under reduced gravity conditions and can be used to analyze bubble behavior and to determine transport parameters by comparison with the experimental data. The results will yield information that will have application to many problems in physical chemistry and technology.

*Research Task Description:* The experiment involves the observation and measurement of dissolution and movement of an isolated gas bubble. Feasibility of bubble injection and deployment has been demonstrated in both normal gravity using mutually buoyant fluids and under reduced gravity conditions in the NASA Lear jet. Use of the critical radius phenomenon for bubble initialization has been demonstrated to be feasible. Subsequent bubble dissolution or growth from the stable critical state is initiated by a step increase or decrease in the liquid pressure. The justification for conducting the experiment in space is the elimination of large-scale buoyant bubble motion and free convective mass transfer effects that occur in normal gravity. These effects generally mask the molecular mass transfer and interfacial contributions to the dissolution process, and their elimination would allow a more fundamental understanding of the molecular mass transfer and interfacial efforts to be obtained.

*Progress to Date:* Work completed during the current period of performance involved experimentation in two areas: measurement of parameters governing the critical radius phenomenon and demonstration of bubble generation and deployment in microgravity. The critical radius phenomenon has been verified in normal gravity for the CO<sub>2</sub>-toluene system in a prototype experiment package constructed at LeRC. Two injectors have been tested to satisfy bubble injection and deployment. The first injector relies on gas inertia for bubble generation and deployment, and has been successfully tested on the Learjet. The second injector uses gas inertia to generate the bubble and uses an additional liquid push to deploy the bubble. This concept has been successfully tested using mutually buoyant fluids in normal gravity. The numerical simulation has been developed to the stage where bubble movement due to residual g-level can be predicted, along with the resulting increase in the dissolution rate. A design team at LeRC has successfully developed the necessary equipment and imaging devices to run the experiment. The Science Requirements Document has been completed and the Conceptual Design Review is imminent.

### Publications

Yung, C. N., De Witt, K. J., Brockwell, J. L., McQuillen, J. B., and Chai, A. T., "A Numerical Study of Parameters Affecting Gas Bubble Dissolution," J. Colloid & Interface Science **127**, 442-452, 1989.

### Presentations

Yung, C. N., De Witt, K. J., and Brockwell, J. L., "A Numerical Study of the Dissolution of a Slow Translating Gas Bubble," *PhysicoChemical Hydrodynamics Conference*, MIT, June 1989.

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

University of Oregon  
Dr. Charles E. Swanson  
Dr. Russell J. Donnelly  
Benjamin You  
Dennis Kessler  
(NASA Contact: Daniel D. Elleman, J.P.L.)

**Objectives:** The objective of this research is to measure finite size effects in the isobaric expansion coefficient,  $\alpha_p$ , near the lambda transition,  $T_\lambda$ , in liquid helium. Finite size effects are manifested as a rounding of the divergence in thermodynamic functions near a critical point as the correlation length increases towards the system size. We can thus test renormalization group theory predictions, universality assumptions, and boundary conditions.

We will measure  $\alpha_p$  for liquid helium confined between parallel plates. The eventual experiment will measure  $\alpha_p$  over a range of temperatures very near  $T_\lambda$  (both above and below), a range of pressures, and a range of plate separation distances. In order to measure finite size effects very near  $T_\lambda$  or with a very well characterized macroscopic geometry, we need a plate separation which would result in an extremely inhomogeneous sample in the earth's gravitational field; a microgravity environment eliminates this problem.

**Research Task Description:** We will measure the dielectric constant of helium confined between parallel plates as a function of temperature. Using the Clausius-Mossotti relation, we can calculate the density and thus the expansion coefficient of liquid helium. The measurement method involves observing the change in resonant frequency of a reentrant superconducting L-C cavity with liquid helium contained between the plates of the capacitor. Our current apparatus, loaned to us by Craig Van Degrieff of NBS and Mike DiPirro (now at NASA Langley) who designed and built it but never successfully operated it, is suitable for work at only one pressure (essentially SVP). Before flight, we intend to have the ability to work at the full range of liquid pressures.

Our apparatus has three superconducting capacitors in a single superconducting niobium structure. Two cavities are for the primary measurement, one with a  $5\mu$  gap in which to see finite size effects and the other with a  $50\mu$  gap for a control. The third cavity is a gas thermometer. The experiment itself will consist of determining the resonant frequencies of the three cavities at various temperatures (indicated by the gas thermometer cavity).

The immediate research task is to make the measurements as intended by Van Degrieff and DiPirro, that is by determining the resonant frequency of the three cavities as the temperature changes slowly. A further task is to investigate various methods of determining the resonant frequency and to change the apparatus to allow pressure control.

**Progress to Date:** The NBS apparatus probe has been reinforced and the probe is in working order. The cavities have not been consistently working, however. Several attempts to run the apparatus with a stimulus/response measurement of the resonance have always found at least one cavity failing. We have been able to resuscitate the cavities upon warming by heating to  $250^\circ\text{C}$  and blowing He gas through or by immersing in an ultrasonic 10 molar HCl bath. We are not sure why the cavities failed or why these attempts revived them.



We are currently designing a new apparatus which will allow us to service the cavities in a more straightforward fashion. We do not know if this can be done without sacrificing the mechanical stability inherent in the NBS design. Until this new apparatus is available, we will continue with attempts to get the old apparatus working, with more care to keep possible contamination from the cavities. We hope that this can be accomplished without necessitating a clean room.

We have been able to characterize the thermometer at low temperatures, and should be able to get 10 nK resolution with the Q at  $10^5$ , as it was for our last successful run. More recent changes in coupling suggest that the Q can be higher, although thermal instabilities obfuscated the results.

In summary, we have been unsuccessful in resurrecting the old NBS apparatus, although encouraging signs are often given. We are designing and beginning construction of a similar but more flexible apparatus, but will run with the NBS apparatus in hopes of some success until the new one is available.

### Satellite Test of the Equivalence Principle

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

**Objectives:** The objective of this research is to test the equivalence of inertial and passive gravitational mass in an earth-orbiting satellite. Preliminary work and technology development is being done in a ground-based version of the apparatus that could test equivalence principle to about one part in  $10^{12}$ . Recent studies show that the satellite version of this experiment should have a sensitivity better than one part in  $10^{17}$ . The apparatus might also be used in a search for composite dependent short-range forces.

**Research Task Description:** The requirements for an orbital experiments have been well defined by studies using the earth-based apparatus. The experiment consists of comparing the motions of two concentric cylindrical test masses suspended in precision superconducting magnetic bearings and free to move along their common axis. The materials are chosen according to the best theoretical likelihood of an effect, and the masses, weighing one to ten kg each, are plated with superconductor to make them uniformly controllable. A position detector based on SQUID magnetometers measures both differential and common mode motion of the masses. This makes an accelerator sensitive to  $10^{-14}$  cm/sec<sup>2</sup> or less. The remainder of the apparatus controls the positions of the test masses and isolates them from disturbances. Essential features include a drag-free spacecraft with acceleration levels of about  $10^{-11}$  g/ $\sqrt{\text{Hz}}$ ; a mass positioning servo which uses the acceleration from the earth's gravity gradient to center the masses on each other within 0.2 Å; a charge measurement and control system to limit the effects of cosmic ray charging; a method of controlling liquid helium sloshing; and superconducting shields for electromagnetic isolation.

We expect to be able to compare at least six test masses in a single six-month mission. The spacecraft would be 300 kg free orbiter in a 450 km or higher orbit. A version of this experiments performed on Shuttle would be severely limited by vibrations and drag; extreme care, together with some modification to the Shuttle attitude control system could reach no better than  $10^{-14}$ . The free orbiter should reach  $10^{-17}$  and will be limited mostly by gas pressure effects and residual acceleration in the drag-free system.

An interesting possibility exists of using the equivalence principle spacecraft in a sensitive test for short-range composition-dependent forces. For this it is necessary to fly a heavy spacecraft such as Shuttle on a slow, close pass near the experiment. This provides a source of the interaction. The experiment could detect less than  $10^{-4}$  of the gravitational force between itself and Shuttle, placing a significant restriction on the size of these forces.

### Publications

Worden, P. W., "Measurement and Control of Disturbing Forces on an Equivalence Principle Experiment," in *Proceedings of International Symposium on Gravitational Physics*, Guangzhou, China (P. Michelson, ed.), 1987.

Worden, P. W., "Almost Exactly Zero: The Equivalence Principle," *Near Zero: New Frontiers of Physics* (J.D. Fairbank, B.S. Deaver, C.W.F. Everitt, P. Michelson, eds.), W.H. Freeman & Co., 1988, pp. 766-783.

### Critical Fluid Light Scattering (Zeno)

University of Maryland  
Professor R. W. Gammon  
Dr. J. N. Shaumeyer  
NAG3-849, NAS3-25370 (NASA Contact: R. Lauver, LeRC)

*Objectives:* The objective is to measure the decay rates of critical density fluctuations in a simple fluid (xenon) very near its liquid-vapor critical point using laser light scattering and photon correlation spectroscopy. Such experiments are severely limited on earth by the presence of gravity which causes large density gradients in the sample. The goal is to measure fluctuation decay rates with 1% precision two decades closer to the critical point than is possible on earth, with a temperature resolution of three microKelvin. This will require loading the sample to 0.1% of the critical density and taking data as close as 100 microKelvin to the critical temperature ( $T_c = 289.72$  K). The minimum mission time of 100 hours will allow a complete range of temperature points to be covered, limited by the thermal response of the sample. Other technical problems have been addressed such as multiple scattering and the effect of wetting layers.

*Research Task Description:* We have demonstrated the ability to avoid multiple scattering by using a thin sample (100 microns), and a temperature history which can avoid wetting layers, a fast optical thermostat with satisfactory microcomputer temperature control and measurement, and accurate sample loading. There remain the important engineering tasks of mounting the experiment to maintain alignment during flight and using vibration isolation to prevent Shuttle motions from distorting the sample.

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

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

*Progress to Date:* The preparation of the Zeno flight experiment moved to the hardware contract stage at the beginning of this period. The design evolved and was presented at the Preliminary Design Review in October. The design features now two photomultipliers and a correlator card capable of processing two autocorrelation functions so that forward and backward scattering measurements can be done simultaneously. This new correlator design by K. Schaetzel was demonstrated with our scattering experiment in April. A new concept for active, inertial isolation was developed and demonstrated by John Sandercock. This vibration isolation system has become part of the Zeno experiment design. A workshop was organized with A. Wilkinson (LeRC) and M. Moldover and R. Berg (NIST) on "Equilibrium Near the Critical Point" in March. Following this meeting the suggestion of A. Onuki that temperature changes were greatly speeded up by adiabatic effects has lead to three manuscripts by the Zeno group, by Onuki, Hao and Ferrell, and by Zappoli, Bailly, Garrabos, Le Neindre, Guenoun and Beysens all showing some version of this effect in calculations. The startling conclusion is that near the critical-point temperature changes can occur to within 1% of their final value in seconds and this gets faster the closer the sample is to the critical point. A first video demonstration of this effect was made which we are now trying to improve.

## Publications

J. N. Shaumeyer and R. W. Gammon, "Optimizing Fitting Statistics in Photon Correlation Spectroscopy," in Photon Correlations Techniques and Applications Proceedings (J.B. Abbiss and A.E. Smart, eds.), OSA, 1989.

B. M. Law, R. W. Gammon, and J. V. Sengers, "Dynamic Light Scattering from Long-Range Density Correlations in a Nonequilibrium Liquid," in Photon Correlations Techniques and Applications Proceedings (J.B. Abbiss and A.E. Smart, eds.), OSA, 1989.

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H. Boukari, J. N. Shaumeyer, M. E. Briggs, and R. W. Gammon, "Critical Speeding up in Pure Fluids," Phys. Rev. Lett., submitted.

## Presentations

Gammon, R. W., "Dynamic Light Scattering from Critical Xenon on the Space Shuttle," *March American Physical Society Meeting*, St. Louis, March 20-24, 1989.

Gammon, R. W., "Dynamic Light Scattering from Critical Xenon on the Space Shuttle," *VIIth European Symposium on Materials and Fluid Sciences in Microgravity*, Oxford, September 10-15, 1989.

B. M. Law, P. N. Segre, R. W. Gammon, J. V. Sengers, "Rayleigh Scattering from a Nonequilibrium Liquid in a Steady-State Temperature Gradient," *American Physical Society Spring Meeting*, Baltimore, May 1-4, 1989.

H. Boukari, M. E. Briggs, J. N. Shaumeyer, and R. W. Gammon, "Observation of Long-Wave Oscillations in a Strongly Stratified Compressible One-Phase Critical Fluid," *American Physical Society Spring Meeting*, Baltimore, May 1-4, 1989.

M. E. Briggs, H. Boukari, J. N. Shaumeyer, and R. W. Gammon, "High Resolution Location of the Critical Point of Fluids in the Presence of Gravity," *American Physical Society Spring Meeting*, Baltimore, May 1-4, 1989.

J. N. Shaumeyer, H. Boukari, M. E. Briggs, R. W. Gammon, "Multiple Times to Equilibrium in One-Phase Critical Xenon," *American Physical Society Spring Meeting*, Baltimore, May 1-4, 1989.

### Convection as a Feasibility Study for a Microgravity Experiment

University of Texas at Austin

Dr. E. L. Koschmieder

NAG3-393 (NASA Contact: J.A. Salzman, LeRC)

September 13, 1989

*Objectives:* The objectives of the planned set of experiments is to establish an experimental data base in order to assess the feasibility and outcome of surface tension driven convection experiments in microgravity. In particular we want to establish the critical temperature gradient required for the onset of convection and the form of the pattern of convection. The second objective of these experiments is to provide sufficient information to compare the experimental results with the theory of surface tension driven convection by Pearson, which theory assumes zero gravity.

*Research Task Description:* This effort is the experimental investigation of surface-tension driven convection under supercritical conditions. While the previous studies were concerned with pattern formation under just critical conditions, which are determined by Pearson's theory, we will now investigate the flow at temperature differences larger than the critical temperature difference. We do this following a recommendation of the PACE Science Review Board. If a surface tension driven convection experiment is made in microgravity then one should use this unique opportunity and extend the experiment into the supercritical range, although Pearson's linear theory is not applicable and no theory exists as yet to predict the outcome of such experiments. From our experience with buoyancy driven Rayleigh Benard convection it appears that it will not be easy to develop a nonlinear theory of surface tension driven convection. The experimental results of supercritical surface tension driven convection in microgravity will therefore be a most useful guide for future theoretical investigations.

The second task of the current set of experiments is the investigation of time requirements for convection experiments in microgravity. This is a matter of practical concern. The time required for a convection experiment to be in equilibrium is the so-called relaxation time, of which there are two, the vertical and the horizontal. While the vertical relaxation time can be easily accommodated on the shuttle, the horizontal relaxation time can be quite long in containers of a couple of inches diameter. The purpose of the planned investigations is to find ways to reduce the horizontal relaxation time to practical time-spans.

### Publications

Koschmieder, E. L. and Prahl, S. A., "Surface Tension Driven Benard Convection in Small Containers," J. Fluid Mech. (accepted).

## Critical Transport Phenomena in Fluid Helium Under Low Gravity

Duke University  
Dr. Horst Meyer  
Dr. Robert P. Behringer  
NAG 5-379 (NASA Contact: S. Castles, GSFC)  
October 1, 1988- July 31, 1989

*Objectives:* These have been outlined in some detail in our 1988 Program Description Document, and will only be repeated here briefly. The objectives are to measure the shear viscosity  $\eta$  in  $^4\text{He}$  or  $^3\text{He}$  near the liquid-vapor critical point  $T_c$  and near the tricritical point ( $T_t = 0.87\text{ K}$ ,  $X_t = 0.67$ ) in  $^3\text{He}$ - $^4\text{He}$  mixtures where  $X$  and  $T$  are the  $^3\text{He}$  concentration and  $T$  the temperature. The viscosity diverges at these points, but the earth's gravity produces a rounding of this divergence, because of the induced density gradients in the fluid layer. In the absence of gravity, this rounding should disappear and the predicted divergence should be observed until frequency effects (from the measuring torsional oscillator) will produce another rounding closer to the transition. To this end, the viscosity of liquid helium mixtures is systematically measured near the superfluid transition line that terminates at the tricritical point, and also at lower temperatures where there is a first-order phase transition. Such a study, not previously performed, will determine whether or not an experiment under nearly zero gravity on a space flight is feasible given various constraints, and if so, whether the effort and cost are justified.

*Research Task Description:* The viscosity is measured by means of a torsional oscillator operating in a continuous mode at the frequency of 150 Hz, as described in the 1988 Report. The horizontal fluid layer in our present cell, the third one constructed in this program, is 0.03 cm high with a diameter of 5 cm, and is contained in a thinwalled beryllium-copper cell that oscillates around its axis with an extremely small amplitude detected electronically. The oscillator becomes damped by the viscous drag and a viscosity change of 3 parts in  $10^4$  can be detected. The temperature is controlled to a few microdegrees. The experiment consists in automatically taking viscosity data for a given mixture as a function of temperature in small increments as the transition is approached. The apparatus measures the product  $(\rho\eta)$  where  $\rho$  is the density and which has to be determined separately for each mixture. Combination of the two measurements then finally gives the viscosity  $\eta$ .

*Progress to Date:* In the last ten months we have analyzed all our  $(\rho\eta)$  data on helium mixtures between 1.2 and 4.2 K, with special emphasis on the region near the superfluid transition, and furthermore we have taken density data and obtained  $\eta$ . A detailed paper has been written on these experiments and their analysis. Close inspection of the viscosity of pure  $^4\text{He}$  very near the superfluid transition  $T_\lambda$  has shown an unexpected behavior. Our data indicate that the slope  $d\eta/dT$  does not diverge at  $T_\lambda$ , contrary to a previous determination and to very recent predictions- but passes over a maximum slightly below  $T_\lambda$ . If correct, this would be a rather

In parallel with the writing of the paper and tabulating the data, we have installed a  $^3\text{He}$  evaporator stage and associated cryogenic tubing in our cryostat, and associated external vacuum plumbing, to extend the temperature range of our viscosity measurements down to 0.7K. Our program calls for detailed measurements with a number of mixtures in the region of the tricritical point,  $0.75 < T < 1.0\text{ K}$  and  $0.55 < X < 0.75$ , necessary to get a complete mapping of the viscosity. This will permit a general survey study of the mixture system in this region and pinpoint the "tricritical", as separated from the "background" behavior. We have installed the new composite torsional oscillator cell in which the density can be measured simultaneously with the product  $(\rho\eta)$  and we have tested and made progress in eliminating various problems. Advances have also been made in developing a new software for the new MacII computer for automatic data acquisition and analysis.

At present, we have underway for several weeks the stringent test to verify the behavior of  $\eta$  for pure  $^4\text{He}$  near  $T_\lambda$ . Preliminary results, not completely analyzed, indicate a good performance of the equipment.

A presentation of our mixture data has been made at the 1989 spring meeting of the American Physical Society in Baltimore. Besides the paper written on the viscosity of helium mixtures, a long review paper, also sponsored by NASA, has been written at the invitation of the editors of the International Union of Physica and Applied Chemistry (IUPAC) series. This paper, describing experimental techniques for measurements in fluids, has been submitted for publication.

#### Publications

Howald, C., Wang, S., and Meyer, H, "Shear viscosity of  $^3\text{He}$ - $^4\text{He}$  Mixtures," Bull. Am. Phys. Soc. **34**, 1182 (1989).

Meyer H., "Experimental Techniques for Transport Measurements in Fluid Helium at Low Temperatures," *Transport Properties in Fluids* (A. Nagashima, J.V. Sengers and W.A. Wakeham, eds.), in press.

Wang, S., Howald, C., and Meyer, H., "Shear Viscosity of Liquid  $^3\text{He}$ - $^4\text{He}$  Mixtures near the Superfluid Transition," (submitted).

## Studies in Electrohydrodynamics

Princeton University  
Dr. D. A. Saville  
NAG3-259 (NASA Contact: R. Balasubramanian, LeRC)  
February 1982 - April 1990

*Objectives:* The purpose of this work is to develop and test models of electrohydrodynamic processes involving fluid interfaces. Particular attention is given to the behavior of fluid globules in systems with poorly ionized solutes at high (applied) field strengths. An ad hoc model (the leaky dielectric) described by G.I. Taylor and J.R. Melcher will be expanded so as to include electrokinetic effects and put it on a firm theoretical basis.

*Research Task Description:* Extant theories which account for the details of physico-chemical processes associated with charged interfaces deal exclusively with low field strengths and fully ionized solutes. The model now used to describe processes at high field strengths--the leaky dielectric--omits consideration of electric double-layers, adsorption at interfaces, and chemical processes involved in the dissociation and recombination of solute species. Thus, even though the model depicts some features associated with bulk fluid motion faithfully, it fails to give a comprehensive picture.

The research involves several tasks: (i) construction of a mathematical model describing low field strength electrokinetics with fluid globules wherein the interface is permeable to ions and the electrolyte contains poorly ionized solutes, (ii) extension of the model described in (i) to high field strengths, (iii) adaptation of the model to oscillating fields and (iv) experiments involving oscillating fields with fluid globules to test the theory. The experiments will take advantage of a micro-gravity environment to overcome problems associated with sedimentation and buoyancy driven convection arising from Joule heating.

*Progress to Date:* The theoretical work is now largely complete for steady fields. We first studied the low field strength electrophoresis of drops with interfaces permeable to ions to gain insight into the more difficult problem of high field strength electrophoresis. The mathematical model developed accounts for: (i) weak electrolytes, (ii) charge regulation at the interface, (iii) interfacial tension gradients at the interface, (iv) surface transport of ions within the interface, and (v) the permeability of the interface to ions.

The second theoretical problem we attacked concerned the inception of field dependent mobility, i.e., the point at which the mobility is no longer proportional to the field strength. This was solved using singular perturbation methods.

The central problem in this research is to describe the high field strength behavior of fluid globules. Using the knowledge gained in solving the problems described earlier, we constructed an asymptotic solution to describe the behavior of an uncharged or charged drop in a strong electric field when the drop is large compared to the Debye thickness. Here the flow and stress fields near the interface differ from those computed according to the leaky dielectric model which ignores electrokinetic effects.

The sample cell and high voltage electronics have been designed, constructed, and assembled for experiments involving a drop (free or pendent) in a liquid. Output from a high voltage (DC) power supply is converted to a sine wave and fed to the two electrodes. We can vary the frequency from 0 to 100 Hz; voltage across the electrodes ranges from 0 to 15 kV. A CCD camera (Ikegami) is used to image the drop. Since the last report the cell and cell holder were redesigned and rebuilt to furnish a more stable platform. In addition we can now view the drop from the top and

We have succeeded in producing prolate and oblate deformations in steady and alternating fields using different neutrally buoyant drops suspended in a silicone fluid. Current efforts are focussed on the



use of high voltage probes to investigate the field structure between the electrodes to insure a known homogeneous field at the position of the drop. We are also setting up apparatus to measure the interfacial tension, dielectric constant and conductivity of the test liquids. Although many tasks remain to be completed we believe we have the essential elements of a superb system.

#### Publications

Saville, D. A. and Baygents, J. C., "The Migration of Charged Drops and Bubbles in Electrolyte Gradients: Diffusiophoresis Physicochem. Hydrodyn. 10, 543-560 (1988).

Saville, D. A. and Baygents, J. C., "The Circulation Produced in a Drop by an Electric Field: A High Field Strength Electrokinetic Model," in *Proceedings of the Third International Colloquium on Drops & Bubbles*, 1989 (in press).

### Mechanics of Granular Materials

University of Colorado at Boulder,  
Professor Stein Sture  
NAS8-35668 (NASA Contacts: I.C. Yates, MSFC)  
November 2, 1983 - October 1, 1989

*Objectives:* The Mechanics of Granular Materials (MGM) research effort is aimed at understanding the constitutive behavior of granular cohesionless materials subjected to very low intergranular or effective stress states.

*Research Task Description:* In the past year a versatile three invariant analytical constitutive model for predicting the response of granular materials has been developed and implemented in a nonlinear finite element code. The model is based on conventional isotropic hardening incremental (flow) plasticity and accounts for pressure sensitive dilatancy over a wide stress range including near zero mean effective stress. An implicit (Backward Euler) integration scheme termed the Generalized Closest Point Projection Method is used to integrate the constitutive equations. The scheme is unconditionally stable and robust in the sense that finite load (stress or strain) steps can be handled within the solution of boundary value problems. Conventional axisymmetric and prismatic specimens have been analyzed subjected to boundary conditions that exist in laboratory apparatus and body forces that exist in a terrestrial laboratory, as well as specimen response under simulated zero-gravity conditions. It is shown that the presence of gravity has a significant influence on response at confinement stress levels ranging up to 15 kPa for specimens that have a typical size of 10 cm (cube). The capabilities of the nonlinear analysis scheme has been verified on laboratory experiments involving both heterogeneous and homogeneous deformation and stress fields.

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Runesson, K., Larsson, R., and Sture, S., "Characteristics and Computational Procedure in Softening Plasticity," J. Engr. Mech. 115, 1628-1646 (1989).

Macari-Pasqualino, E. J., "Behavior of Granular Materials in a Reduced Gravity Environment and under Low Effective stresses," Ph.D. Dissertation, University of Colorado at Boulder, August 1989.

Schipperleit, T. M., "Stress-strain and Dilatancy Behavior of a Dense Sand at Very Low Effective Stresses," M.S. Thesis, University of Colorado at Boulder, December 1988.

## **D. FACILITIES**



Microgravity Materials Science Laboratory

NASA Lewis Research Center  
Thomas K. Glasgow  
In-House

This laboratory was established to provide to researchers from industry, academia and government access to specialized equipment and experienced personnel to aid in development of space flight concepts, experiments, and hardware. The laboratory is equipped with a number of unusual experimental and computational facilities and staffed by engineers from several disciplines and technicians familiar with microgravity practice.

In the Microgravity Materials Science Laboratory (MMSL) investigators are encouraged to take the first steps toward defining space flight experiments for later performance on the space shuttle or on the space station. The laboratory may also be used in preparation for experiments to be conducted in other ground based, reduced gravity environments such as those provided by drop tubes, aircraft, or rockets. The computational facilities may be used to model the expected fluid flow and heat transfer in microgravity experiments. Another use of the Laboratory is for post flight investigations. The most notable example of this type of analysis was the post flight exposition of the convective effects in 3M's DMOS experiment. Work on DMOS and follow on modelling was essentially completed in 1988. One interesting finding was that even completely reversed accelerations can cause convective mixing and that even low accelerations, e.g.  $10^{-6}$  g, can cause bulk flow.

Attention has been given more recently to the PVTOS experiments. The MMSL sponsored preliminary study of physical vapor transport of large gas molecules in a thermal gradient was completed. This study was conducted by Professors Rosner and Keyes of Yale. The most important finding was that sidewall creeping flow of gasses, previously ignored in PVT and CVT, may contribute significantly to the total flow both on earth and in space. The modelling was performed using an assumption of axisymmetry; this preliminary study should be extended to full three dimensional modelling not possible under the time and funding constraints of the initial work. Professors Rosner and Keyes have submitted a proposal for this extension to Code EN for review. In house work has begun on the definition of a system to measure concentration and temperature noninvasively in the vapor of a PVTOS experiment.

In conjunction with Westinghouse Corporation, the salt solidification furnaces of the MMSL were used to grow crystals of lead chloride and lead bromide. Both are opto-electronic materials potentially important for upcoming generations of computers and communication devices. Another industrial interaction was with Brimrose Corporation, manufacturers of laser guidance equipment. Brimrose was awarded a phase one SBIR contract to study means of observing convection in crystal growth. They concentrated on high resolution thermography.

Other organizations working with the MMSL include GTE, Rockwell, Rensselaer Polytechnic Institute, Case Western Reserve, Michigan Tech, University of Akron, Cleveland State, University of Kentucky, and Ohio State University. Discussions have been conducted with researchers from the Naval Surface Weapons Center concerning microgravity crystallization of high temperature semiconductors.

The MMSL facilities have been used by Francis Chairamonte of the Lewis Space Experiments Division for precursor work pertaining to his Learjet solidification project. He is investigating the formation of voids during solidification in microgravity; his real interest is in the influence of voids on heat transfer in practical space systems such as heat storage devices.

Telescience has been under investigation in a joint project with RPI. The major effort in this

area has been devoted to the creation and testing of software required to link process controllers and video cameras to computers. Preparations have been made to control a glass melting furnace remotely.

The MMSL has had a very active involvement in two ATD projects, Laser Light Scattering Instrument and High Temperature Furnaces. The Laser Light Scattering project has generated considerable interest for advancing to flight experiments, brought together many of the world's most active developers of this technology. There was general agreement that the most important pieces of hardware required for a compact, efficient, space flight version of the instrument will be available for testing. The ADT project scientist, Bill Meyer, has been coordinating the specification of new compact lasers, new optical systems, special optical fibers, efficient, fast, solid state photon counters, and compact dedicated correlator computers.

High Temperature Furnace technology has been under development in house and on grant. The in house effort has concentrated on a modular approach to building efficient furnaces, including transparent furnaces, and on devising means of garnering real data for inclusion into furnace design and control models. A related effort has been the development of multizone control software necessary for flexible control of complex thermal profiles. On grant we have been investigating the feasibility of actively controlling advancing solidification interface position and shape by the use of directed energy sources.

While the original MMSL concept was to provide access to ground based functional duplicates of space flight hardware we have found the greatest use of lab provided facilities has been in the area of computational modelling. Therefore we have used a significant portion of our total funding to provide the equipment, software, and support staff in this area. At the request of Mark Lee of Code EN the MMSL has presented a plan to shift half of its work to a peer reviewed basis over a period of three years.

The Microgravity Materials Science Laboratory in addition to its computational facilities has equipment for observation of salt solidification and physical vapor transport, for planar front and dendritic growth from the liquid, for studying convection in mixing of liquids of slight density differences, for glass melting and solidification, for hot stage microscopy, for bulk undercooling, for levitation of conductive melts, and for solidification isothermally and in a gradient. Several presentations of MMSL capabilities are given each year at different symposia to inform the community of its availability. The laboratory also serves an educational role not only for the summer students typically employed but also for touring teachers and students, many receiving their first introduction to microgravity concepts.



### Ground-Based Research Facilities

NASA Lewis Research Center  
Jack Lekan  
In-House

The ground-based reduced gravity facilities at the Lewis Research Center continued to operate at their highest level of utilization in 1989. The number of tests conducted, operational experiment packages, and researchers continue at an all-time high. The 2.2 Second Drop Tower surpassed its 9,000 test drop as a record number of over 1000 drops were performed over the last year. The 5.18 Second Zero-Gravity Facility has now completed over 2200 research drops. The Learjet also supported the most programs ever. Fifteen visiting scientists and students participated either independently or jointly with NASA investigators in the performance of NASA-sponsored research.

As stated above, over 1000 research drops were performed in the 2.2 Second Drop Tower in 1989 in support of twenty-two programs involving reduced gravity research on combustion and fluids. This facility continues to enhance its technological capabilities as initiatives have been undertaken in the areas of fiber optic data transmission and combustion diagnostics. The Zero-Gravity Facility is operating at full utilization. Twelve programs were supported and 173 test drops were performed. Some of the experiments that were performed in the Lewis reduced-gravity facilities in 1989 include: Pool Boiling, Burke-Schumann Diffusion Flames, Smoldering Combustion, Spacecraft Fire Safety, Gas Diffusion Flames, Droplet Combustion, Pool Fires Combustion, Surface Tension Driven Convection, Two-Phase Flow, and Solid Surface Combustion.

The ground-based facilities have continued to play a vital role in the Microgravity Science and Applications Program as they provide the baseline, normal gravity and reduced gravity data needed to support a large number of broad-based in-house and sponsored programs. The facilities are utilized to: execute ground-based science programs; perform precursor tests to define space experiment science requirements and conceptual designs; and also to perform tests for space experiment technology development and verification. The research performed in these facilities enhances the value and success of space experiments. It is anticipated that their importance will not diminish in the foreseeable future.



**E. CENTERS OF EXCELLENCE**



Center for Separation Science

University of Arizona  
Dr. M. Bier  
NAGW-693

The Center for Separation Science continues to serve as a center of excellence for NASA and the U.S. biotechnology industry in matters related to microgravity science and applications. The Center's primary expertise is in electrophoresis, where it has developed a new technology for protein purification based on recycling isoelectric focusing. Several instruments developed at the Center are now in commercial production. Most widely accepted is the Rotofor, marketed by BioRad of Richmond, CA. This apparatus mimics microgravity and was developed as a result of the 1984 and 1988 space electrophoresis experiments. The Center continues to maintain its leading role in mathematical modelling and computer simulation of electrophoretic transport processes, capillary electrophoresis and two-dimensional electrophoresis.

A new fundamental program on cell fusion in microgravity is lead at the Center by Dr. David Sammons. It is a collaborative project with German scientists, participating in the D-2 space mission. Other international collaborative projects involve Center scientists and French researchers in Toulouse, Prof. Wagner in Saarbrucken, Germany, Prof. Righetti in Milano, Italy, and Dr. Thormann, in Bern, Switzerland. Dr. Ryu, an assoc. prof. of chemical engineering from Korea is spending his second year at the Center.

The Center is in the process of greatly expanding the scope of its activities at no cost to NASA, through support by the University's resources. As part of this expansion, Dr. Jerker Porath, an eminent professor from the University of Uppsala, Sweden, has joined the Center on a half-mile basis. Prof. Porath is one of the most prominent world figures in separation science due to this discoveries of gel permeation, affinity and metal ion chromatographies. Other specialists in program in phase partitioning has already brought striking results, illustrating the importance of thermodynamics in transport processes.

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Center for Low-Gravity Fluid Mechanics and Transport Phenomena

University of Colorado

Professor D. R. Kassoy

Professor R. L. Sani

NAGW-951 (NASA Contact: R.K. Crouch, HQ)

September 1, 1986 - September 30, 1989

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

The Center has initiated research activity in the following areas:

(1) *Mass Transport in Vapor Phase Systems*

D.R. Kassoy, Department of Mechanical Engineering

A. Herczynski, Postdoctoral Research Associate

B. Zappoli, CNES, France

The main purpose of the proposed study is to model thermally generated transient bulk motion in gravity-free systems in order to understand the fluid dynamics of CVT and PVT experiments. While considerable attention has been given in recent years to other physical mechanisms that drive non-buoyant convection in fluids (e.g. Marangoni effects in reduced gravity), little is known about the mass motion induced by localized thermal expansion arising from transient heating in compressible gases.

(2) *Droplet Collisions and Coalescence in Microgravity*

R.H. Davis, Department of Chemical Engineering

J.R. Rogers, Graduate Student

X. Zhang, Graduate Student

S. Yiantsios, Postdoctoral Research Associate

D.O. Frazier, NASA Marshall Space Flight Center

Theoretical work on macroscopic coalescence phenomena is directed at developing the population dynamics model for gravity-induced, Marangoni-induced, and Brownian-induced coalescence, incorporating improved collision efficiencies into the model, incorporating spatial segregation into the model, and supporting the proposed experiments. Theoretical work on microscopic coalescence phenomena is directed at completing the calculation of two-sphere mobility function for axisymmetric and nonaxisymmetric motion of drops, predicting coalescence times and collision efficiencies for two interacting drops, and studying the effects of interfacial tension, internal viscosity and van der Waals forces on deformation and film drainage between two drops.

(3) *Rapid Solidification of Undercooled Melts*

D.R. Kassoy, Department of Mechanical Engineering

A. Norris, Graduate Student

The objective of this research is to use first principles to predict solidification rates and solidification zone structure in terms of parameters appearing in the nucleation kinetics laws and governing energy

equation. Clearly, to understand the solidification zone, it is necessary to include the nucleation process in the mathematical models and examine length scales on the order of the solidification zone. This coupling of the nucleation rate laws and the continuum equations will describe heat release as a function of temperature, composition and material properties.

(4) *Computer-Aided Analysis of the Flow, Transport and Stability of Systems with Free and/or Moving Interfaces*

R.L. Sani, Department of Chemical Engineering  
A. Alshamian, Graduate Student

The modeling effort and associated development of state-of-the-art computational tools described herein is focused on extending a state-of-the-art model with very general free surface capability to include the assessment of the linear stability of the system to *three-dimensional perturbations*. While, in general, the full three-dimensional simulation of these complicated systems is not feasible even in today's supercomputing world, the linear stability assessment can be done and, in fact, we believe is the logical next step beyond two-dimensional simulations.

(5) *Interaction of Liquid Droplets with A Solidification Front*

W.B. Krantz, Department of Chemical Engineering  
D.B. Thiessen, Graduate Student  
D.O. Frazier, NASA Marshall Space Flight Center

This research concerns the formation of microstructures in a class of materials known as miscibility-gap alloys. The phenomenon of interest is the interaction of liquid droplets in the melt with an advancing solidification front. Such droplets are observed to be pushed by the solidification front until they reach a critical size by diffusional growth or coalescence with other droplets. This droplet-pushing phenomenon limits the degree of fineness achievable by going to a low-gravity environment and using acoustic mixing of the melt. A mathematical model is being developed which will predict the critical droplet size as a function of solidification rate and material properties without arbitrary parameters. This model is being validated with quantitative experiments using a unique apparatus, the gradient-stage microscope, available at NASA Marshall Space Flight Center.

(6) *Numerical Simulation of Benard Convection with Gravitational Modulations*

Sedat Biringen, Department of Aerospace Engineering Sciences  
L.J. Peltier, Graduate Student  
G. Danabasoglu, Graduate Student

Effects of a modulated gravitational field on buoyancy-driven convection are numerically simulated in rectangular cavities and in three-dimensional boxes. Sinusoidal as well as random excitation fields are considered. Spectral methods are developed for the solution of three-dimensional enclosed flows.

(7) *Thermal Instabilities in Low-Prandtl Number Liquids*

J.E. Hart, Department of Astrophysical, Planetary and Atmospheric Sciences

Experiments in annular containers and in rectangular boxes of various aspect ratios were completed. When the layer of mercury is differentially heated between two vertical conducting boundaries at different temperatures, a traveling or stationary wave oscillation appears as the temperature difference is increased. Measurements on the bifurcations to multiply periodic and chaotic states were made.

(8) *Laboratory and Theoretical Models of Planetary-Scale Instabilities and Waves*

John E. Hart, Department of Astrophysical, Planetary and  
Atmospheric Sciences  
Juri Toomre, Department of Astrophysical, Planetary and  
Atmospheric Sciences

The Geophysical Fluid Flow Cell (GFFC) experiment is an implementation of the idea in which fluid is contained between two rotating hemispheres that are differentially heated and stressed with a large a-c voltage, generating an artificial radial gravity.

The GFFC flew on Spacelab III in May, 1985. Data in the form of global Schlieren images of convective patterns were obtained for a large variety of configurations. These included situations of rapid rotation (large Taylor numbers), low rotation, large and small thermal forcing (i.e. a range of Rayleigh numbers based on the electrostatic gravity and the statically unstable radial temperature contrast), and situations with applied meridional temperature gradients.

Our group has been involved with the analysis and interpretation of the GFFC-85 data. We have also developed television displays of convection data and for near-realtime interactive experiments. These experiments, on the transition to global turbulence, the breakdown of rapidly rotating convective planforms (banana cells) and other phenomena are proposed for the United States Microgravity Laboratory (USML-1) aboard the shuttle in 1992.

(9) *Manufacturing Spherical Shells under Microgravity Conditions*

C.Y. Chow, Department of Aerospace Engineering

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

(10) *Modeling and Experiments on Fluids Systems with g jitter*

P.D. Weidman

Computational methods are used to model g-jitter effects in a thermosiphon heat loop. Ground-based g-jitter experiments are designed to measure effects on convection in fluid-filled boxes as well as those with a free surface.

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

The Center fosters extensive interaction with scientists and engineers in other universities, in government laboratories, and in industry. Cooperative research projects are being developed and long-term visits have been arranged. The Center seeks the broadest possible participation by members of both the national and international lowgravity communities.

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**APPENDIX A**  
**MSA ORGANIZATION LIST**



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