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NASA TECHNICAL MEMORANDUM

(NASA-TM-78214) MATERIALS PROCESSING IN SPACE PROGRAM TASKS (NASA) 118 p HC A06/MF A01 CSCL 22A

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NASA TM-78214

MATERIALS PROCESSING IN SPACE PROGRAM TASKS

OFFICE OF SPACE AND TERRESTRIAL APPLICATIONS

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

DECEMBER 1978

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NASA

George C. Marshall Space Flight Center
Marshall Space Flight Center, Alabama

MSFC - Form 3190 (Rev June 1971)

MATERIALS PROCESSING IN SPACE

CATALOG OF TASKS

MATERIALS PROCESSING IN SPACE

INTRODUCTION

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

Additional effort will be devoted to identifying those special requirements which drive the design of hardware to reduce the risk in future developments. Examples of current hardware studies are high-gradient furnaces and heat pipes to take maximum advantage of the lack of convection; acoustic, electromagnetic and electrostatic containerless processing modules; and electrophoresis separation devices.

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

History

Materials Processing in Space was initiated with a few simple demonstrations of principles by astronauts on Apollo 14 and 16. These were followed by more extensively planned but limited experiments on Skylab in 1974 which provided the first strong evidence of improved crystal growth, the elimination of convection and other fluid effects in low gravity. On Apollo-Soyuz in 1975, many of the experiments were repeated with other materials and improved measurements. Also on that mission, electrophoresis experiments gave early indications of the possibility of improved separations of biological materials. In all, over fifty flight experiments or demonstrations of fundamental effects of weightlessness were completed. Since the end of the Apollo program, the flight opportunities for researchers have been limited to ballistic rocket flights providing about five minutes of weightlessness during the coasting phase of flight. About twenty-five experiments have been completed on Space Processing Applications Rockets (SPAR's).

A recent assessment was made of the program by a committee of the National Research Council on the Scientific and Technological Aspects of Materials Processing in Space (STAMPS). Recommendations stressed the need for more extensive ground-based research to serve as a support base for the evolution and assessment of investigations which would lead to a proper understanding of the role played by gravity in materials processes. Recourse to the weightless environment of space should be based primarily on the understanding and need in those specific cases identified from such a program. In addition, the first phase of the space flight program should be a demonstration of the new technology developed in the NASA program which should then be transferred to non-NASA entities for their use. The second phase, funded primarily by non-NASA users, should consist of a National Materials Laboratory in space to open the capabilities to all for a reasonable charge. Closer ties between the materials communities and NASA were recommended in the form of peer review of all proposals, both ground-based and space flight, and the periodic peer review of policies and plans. The Materials Processing in Space program has been restructured on the basis of these recommendations beginning with the earliest deliberations of the STAMPS Committee. An advisory committee has been formed to provide guidance in future program planning and policy making that is consistent with the spirit and principle of the STAMPS recommendations.

Program Strategy

The current program emphasis on fundamental processing science and technology in selected areas will continue for the period of this plan as the Materials Processing in Space program addresses problems of interest to the public and private commercial sectors which can be resolved by recourse to the space environment. During this phase of the program, the development and demonstration of current space technology for materials processing will be transferred, as appropriate, to non-NASA users. In order to assist this process, a Commercial Space Processing Task Team has been formed to resolve institutional constraints serving as disincentives to cooperative involvement. In addition, this team will serve as a single point of contact for interested parties and represent their interests within NASA.

Emphasis will be placed on the expansion of currently funded activities for both 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 by focusing ground-based activities in selected areas and by expanding support for current space flight investigations by forming facility experiment teams to provide advice and identify future involvement. At present, the three major facilities under development for Spacelab are the Fluid Experiments System, the Solidification Experiments System, and the Acoustic Containerless Processing Module.

Goal

The program demonstrates the capabilities of the space environment for materials processing to the scientific and commercial user communities and provides opportunities for independently funded users to exploit the space environment for processing related to their own needs.

Objectives

The program has been structured to achieve the following specific accomplishments:

- Demonstrate the ability to achieve control over process variables at levels not achievable on Earth

- Control thermal fields
- Increase compositional uniformity in crystals
- Lower defect concentrations in crystals
- Align internal structure of metal alloy systems
- Increase purity of optical glass systems
- Increase stability range of new glass systems
- Increase geometrical uniformity of glass microshells
- Reduce self-deformation and dislocation density of growing crystals
- Improve effectiveness of electrokinetic separation
- Produce large diameters of monodisperse polymer latex spheres

o Develop and demonstrate the capabilities of containerless processing techniques to handle and measure the properties of molten, reactive materials on which experiments cannot be performed in Earth-based laboratories

  - Viscosity and surface tension
  - Equilibrium and dynamic vapor pressures
  - Enthalpy, emissivity and temperature measurements
  - Phase equilibria studies
  - Enthalpy of solution and reaction
  - Processing of droplet arrays

o Demonstrate the nature of the vacuum achievable in space and its utility for extending the range of important experimental parameters in extra-high vacuum science as well as its potential for achieving novel materials processing capabilities

o Provide opportunities for independently funded scientific and commercial users to perform processing in the space environment

In addition to these activities, the program will also actively explore mission and hardware configurations which will reduce cost and provide sufficient power for experiments. These studies have already identified a free-flying mode of operation in combination with a power and support module which most effectively satisfies the program requirements. In the period 1980-84, these definition studies will identify a series of phase options to provide such flight opportunities and then proceed with preliminary designs.

Low-Gravity Flight Projects

Low-gravity research is provided by several means to scientists to carry on experiments to verify ground-based predictions:

a. Drop tube at MSFC (approximately two seconds of weightlessness)

b. KC-135 or F104 aircraft (up to 40 seconds of weightlessness in a Keplerian parabolic trajectory, available several times each year)

c. SPAR (five minutes of weightlessness in coasting flight after power cut-off; samples recovered after parachute descent); four more flights planned through 1980.

d. Materials Experiment Assembly (MEA) (up to five days in orbit in the Space Shuttle. MEA is an autonomous package which will support five experiments with a minimum of integration effort.

e. Spacelab Materials Processing Experiments (five or more days of weightlessness on the Space Shuttle); hands-on experiments operated by Mission or Payload Specialists in a laboratory environment. First flight planned for Spacelab 3.

f. Materials Experiments Carrier (MEC) will be an automatic, free-flying satellite to support a large number of experiments for three or more months; will use the 25KW Power Module for support.

Format

The key to the format for listing Materials Processing in Space program tasks in this catalog is as follows:

a. Title:

b. Performing Organization:

c. Principal Investigator:

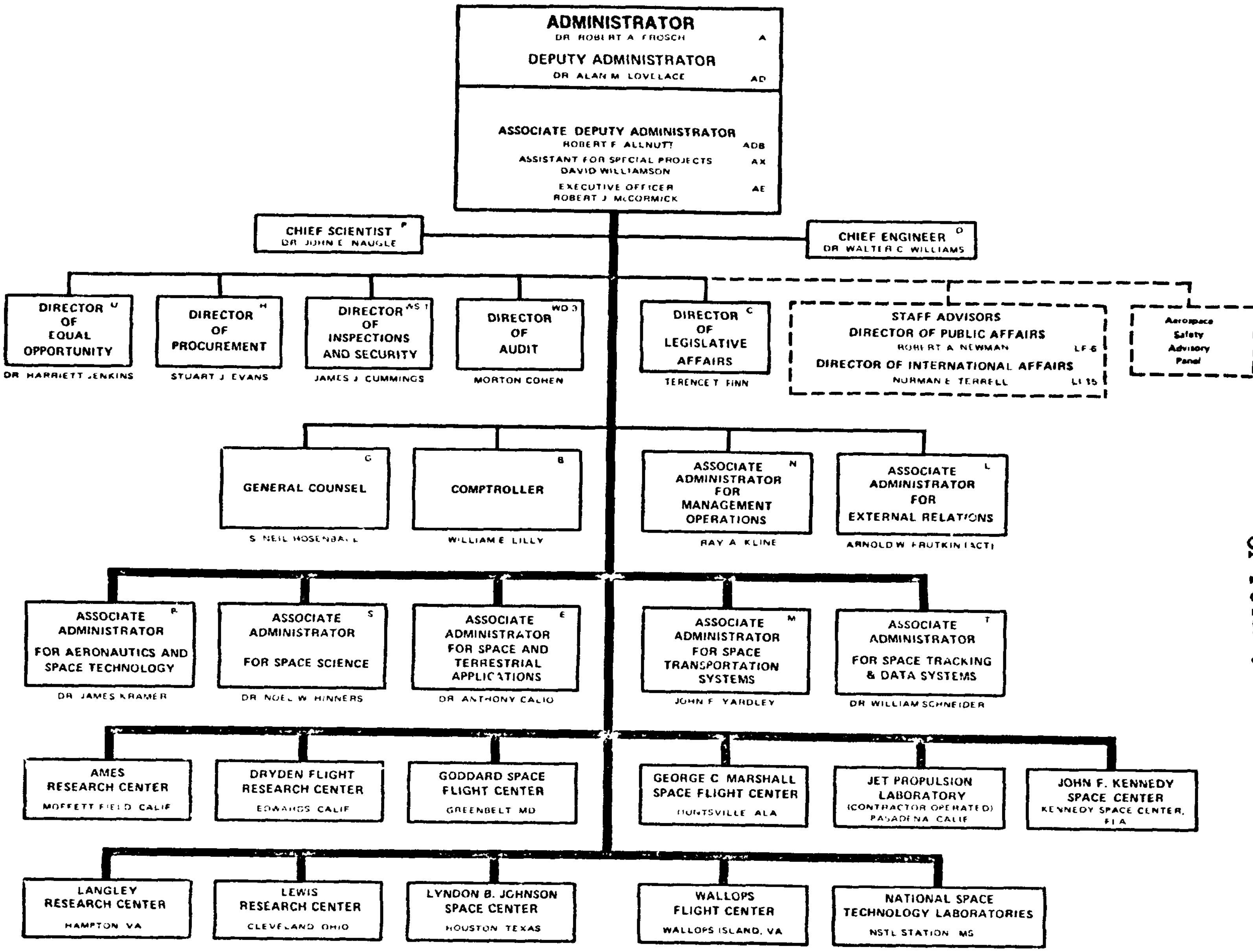
d. MSFC Contact (COR or Science Advisor):

e. Contract Number and Total Cost:

f. Abstract of Objectives, Approach and Results:

Program Organization

The NASA Materials Processing in Space program is administered by a Division Director and his staff in the Office of Space and Terrestrial Applications at NASA Headquarters, Washington, DC. The Division Director is supported directly in the management of the program by the George C. Marshall Space Flight Center's Materials Processing in Space Projects Office. That office depends upon the laboratories of the Marshall Space Flight, other NASA centers, other government agencies, universities and industrial laboratories for technical support.



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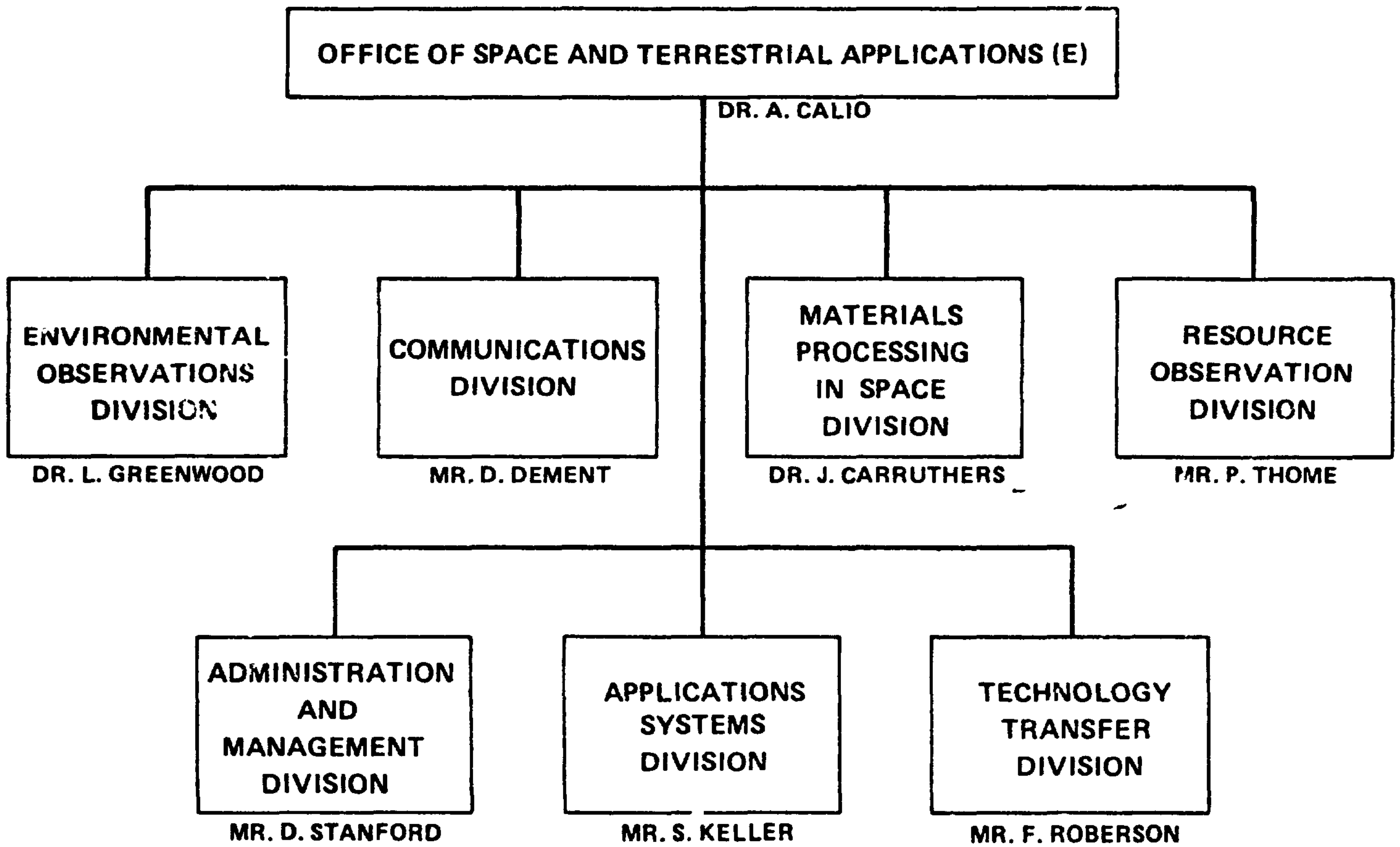

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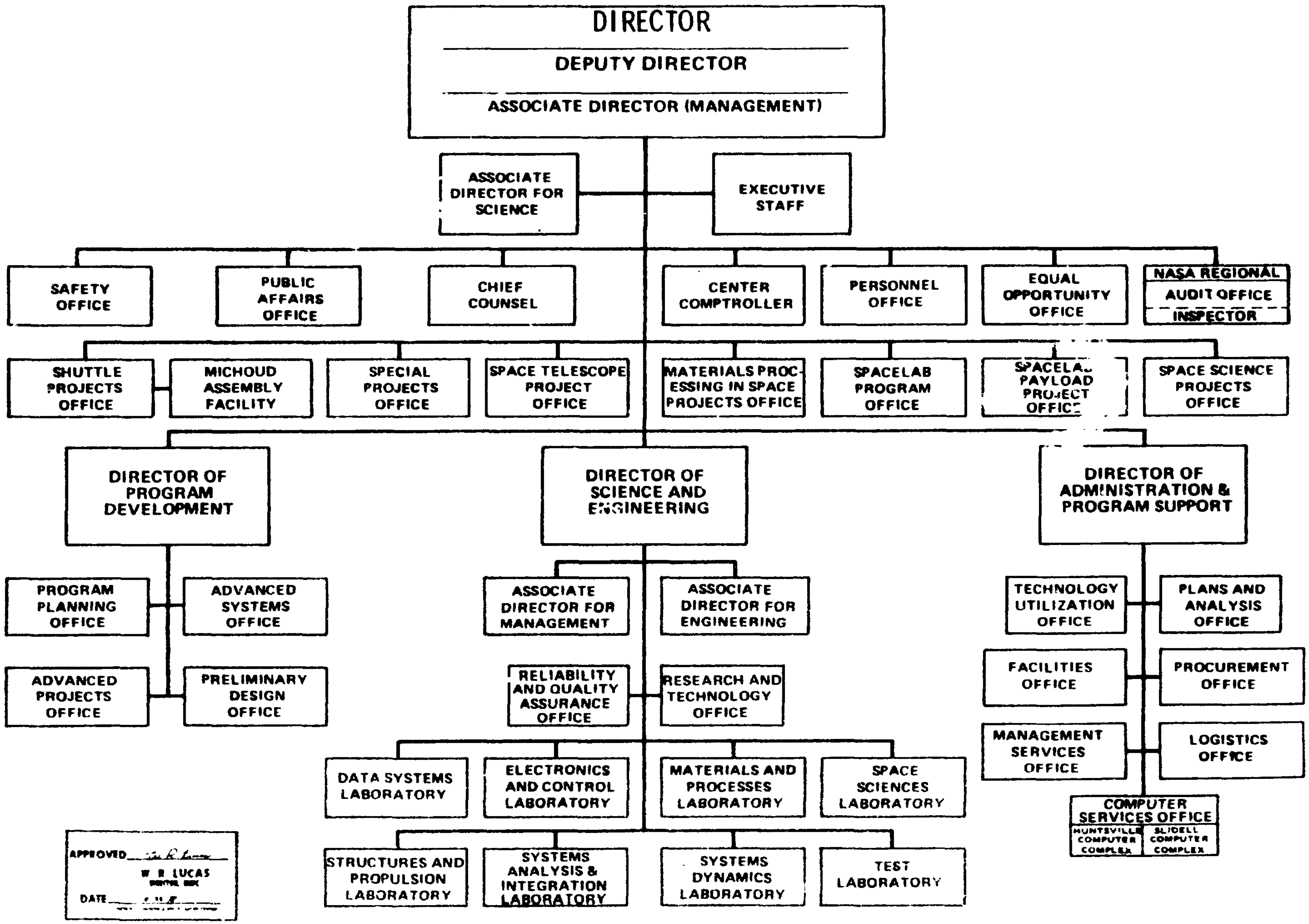

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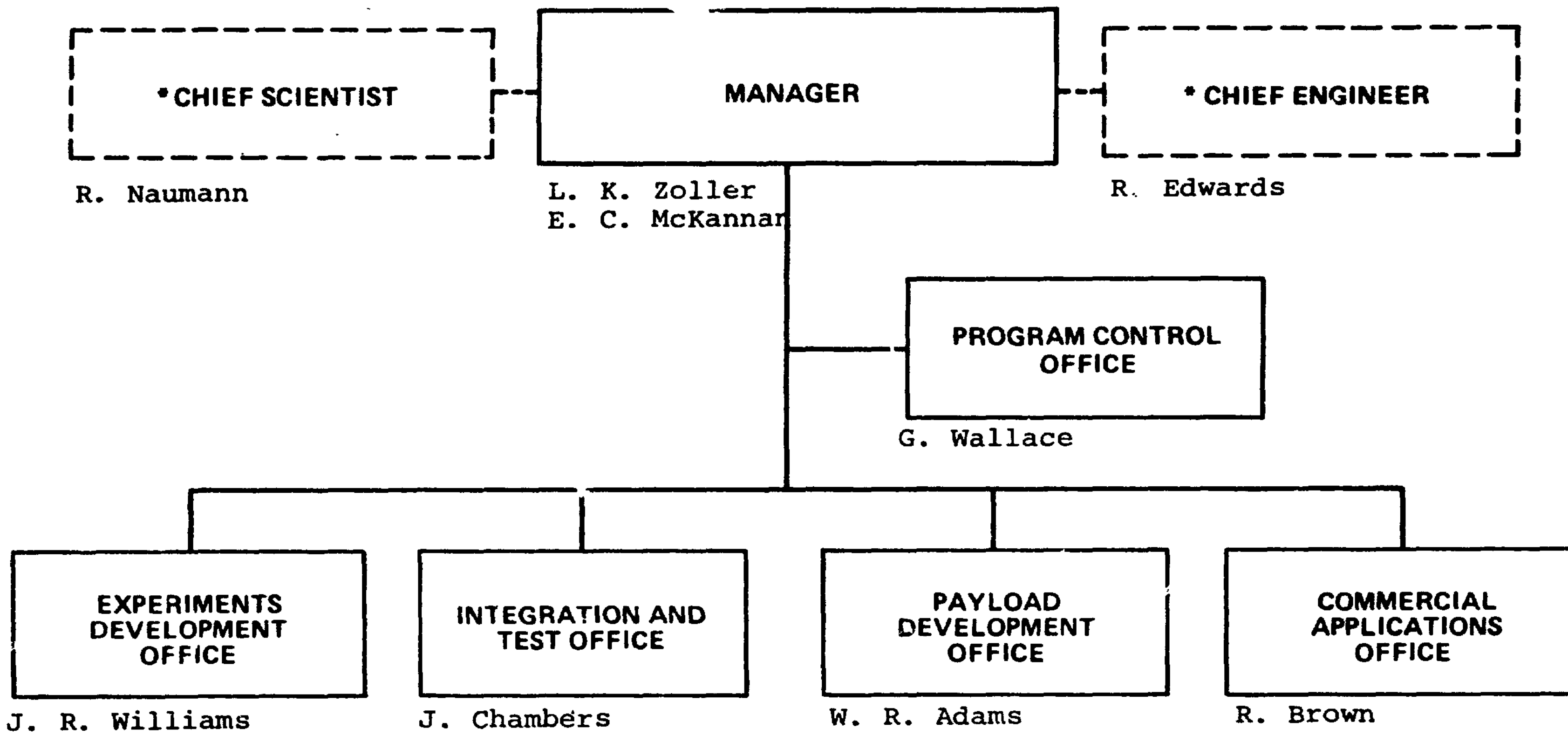
MARSHALL SPACE FLIGHT CENTER
MATERIALS PROCESSING IN SPACE PROJECTS OFFICE



* STAFFED BY SCIENCE AND ENGINEERING PERSONNEL

APPROVED W. R. Lucas
W. R. LUCAS
DATE 10-13-78

Semiconductor Materials Growth in Low-G (M77087)

NASA/Langley Research Center
Dr. R. K. Crouch
Dr. W. J. Debnam, Jr.
Mr. J. Williams, MSFC
Contract Not Applicable

This experiment will utilize the microgravity environment of space to investigate the growth of solid solution semiconductors. The specific objectives are to minimize or eliminate the segregation of constituents and to minimize the influence of thermal convection on the solidification process in the growth of single crystalline $Pb_xSn_{1-x}Te$. This particular material, like $Hg_xCd_{1-x}Te$, is a solid solution crystal, and the relative amounts of Pb and Sn can be continuously varied to give different band-gap energies. There is considerable demand for this material for use as infrared detectors to 14 microns. Present production techniques can produce only small crystals whose electrical properties cannot be precisely controlled. There is a demand for larger, highly homogeneous crystals for use as detector arrays for infrared imaging devices.

Three different growth processes will be considered: (1) a vapor phase sublimation for seeded growth, (2) a modified Bridgman growth in which polycrystalline aggregate is necked down to encourage growth of a single crystal, and (3) a modified Bridgman melt-back and regrowth. These experiments will be carried out in the furnace module on a Spacelab pallet.

It is difficult to grow crystals such as PbSnTe with a controlled composition because of the large difference between the solidus and liquidus lines on the phase diagram. Since PbTe solidifies first, macro- and microsegregation results. In zero-gravity conditions, macrosegregation is avoided because of the absence of sedimentation. In the absence of gravity-driven convection, a diffusion layer should build up in front of the solidification interface in which stead-state compositional uniformity is maintained. The absence of convective transport will allow much sharper gradients in the melt which are needed to prevent interfacial breakdown due to constitutional supercooling. In the absence of gravity, much longer zones can be maintained which allow a more nearly planar interface. In the vapor phase sublimation, the absence of gravity-driven convection provides a diffusion-controlled growth environment in which a uniform steady-state composition will be established and maintained. It is hoped that this will provide the proper stoichiometry for the desired composition.

Solid Solution Crystal Growth of PbSnTe

Jet Propulsion Laboratory (JPL)
Dr. M. Saffren
NASw-100 - $100K

The goal of this research program is to obtain high quality PbSnTe single crystal material for far-infrared (8-30μM) sensor arrays by investigating and exploiting the advantages of zero-gravity space processing.

The program objectives are to improve ground-based crystal-growth techniques; develop experiments for space processing based on the results of ground-based research; obtain crystals suitable for use in infrared charge-coupled device array development.

Immediate objectives are to study fluid-dynamic effects due to gravity in closed-tube vapor-growth of PbSnTe crystals and to define and initiate ground-based crystal growth experiments.

Growth of Solid Solution Single Crystals (M77069)

Marshall Space Flight Center (MSFC)
Dr. M. C. Davidson, MSFC
Dr. A. F. Witt, MIT
Dr. L. R. Holland, PRC
Dr. D. D. Schenk, BMD-ATC
Contract Not Applicable

The objective of this experimental program is to determine suitable growth conditions for the production of high-quality solid solution crystals such as $Hg_{1-x}Cd_xTe$. This material is particularly useful as infrared detectors in the 8 to 14 micron band. Many applications require larger crystals with much better homogeneity than are currently available. Particular emphasis in this study will be directed toward producing material with extremely high frequency response for wide bandwidth application. This requires very pure intrinsic material with carrier concentrations in the low 10^{14} cm^{-3} range.

An extensive ground-based program is underway to determine the effects of purity of starting materials, optimum growth conditions, and container material as well as the suitability of various growth techniques. Growth from the melt, from Te solvent zone, and solid state anneal are being considered.

Growth of HgCdTe is extremely difficult for a number of reasons; i.e., the high preferential segregation of CdTe with respect to HgTe, microscale fluctuations in growth rate which result in several compositional variances and contribute to interfacial breakdown, and the high vapor pressure of Hg which interferes with the liquid-solid interface in Te solvent zone growth.

Infrared Detector III - V Materials

Rockwell International
Dr. Gertner
NASw-100 - $15K

The goal is to obtain optical materials for high-performance infrared charge-coupled device sensor arrays, by investigating advanced approaches and exploiting advantages of space processing.

The project objectives are to improve ground-based crystal-growth techniques and to determine materials requirements for controlling and optimizing the active interface layers of the devices.

The specific objectives in the near term are to define ground-based experiments for InSb crystal growth; to perform initial ground-based experiments in InSb Czochralski/float-zone crystal growth; to define InCaSb crystal-growth experiments (float-zone); to procure InSb crystals and characterize oxide/InSb interfaces and stoichiometry of InSb surface as functions of the process; to examine the utility of ultrahigh vacuum deposition versus chemical vapor deposition techniques to compare the crystal-surface defects.

<u>Defect Chemistry and Characterization of (Hg,Cd)Te</u>

Honeywell
Dr. Dutt
Dr. Naumann, MSFC
Contract Being Negotiated

The objective is to improve the preparation (annealing and doping technology) as well as characterization of $Hg_{1-x}Cd_xTe$ alloys. It is necessary to carry out a comprehensive experimental and analytical study of the defect chemistry of (Hg,Cd)Te.

The thermodynamics of defect chemistry will indicate ways of achieving the following: improve the yield of n- or p-type material with desired level of carrier concentrations, obtain high doping levels, provide basis for an understanding of the diffusion mechanisms and the solubility of impurities, understand the self-compensation mechanism that is limiting the doping efficiency, identify the defect centers responsible for a low minority carrier lifetime in p-type.

Development of characterization tools for determine the compositional and electrical homogeneity will lead to an improved preselection criteria and improved control of the crystal growth.

The principal experimental means of data collection will consist of Hall measurements on annealed and quenched crystals for the study of defect chemistry. For characterizing the material, infrared spectroscopy will be investigated. Techniques such as Metal-Insulator-Semiconductor structures, p-n junction properties, and deep level transient spectroscopy will be used.

Observation of Interface Instability During Crystal Growth

Stanford University
Prof. W. A. Tiller
Contract Being Negotiated

This study utilizes a direct observation method of interface stability criteria in a convective environment. It will form the basis for future experiments under non-convective conditions. Through the use of a transparent-furnace the liquid-solid interface can be visually observed and the growth stability, velocity, degree of melt convection, and interfacial and bulk solute concentration measured directly during the growth process rather than afterwards.

To test the well-known stability equations of a particular material system, the relevant parameters which enter these equations will be measured including diffusion coefficients of the solute in the liquid, phase diagram and effective distribution coefficients for the solute, liquidus slopes for the chosen solutes, thermal conductivities and diffusivities for both liquid and solid, solid-liquid interfacial energy and its anisotropy and a study of interface attachment kinetics as a function of orientation. The system being studied is $CsCdCl_3$ with a copper solute.

The information gained from this study will have broad practical implications for cyrstal growth processing both on the ground and in space. A side benefit is that the results will have a marked impact on geological crystallization studies in magnetic systems which involve the breakdown of interfaces in faceted systems.

Advanced Methods for Preparation and Characterization of Infrared-Detector Materials

McDonnell Douglas Research Laboratories
Dr. Whitsett
Dr. Naumann, MSFC
Contract Being Negotiated

The objective is to quantitatively differentiate between intrinsic characteristics and effects of extraneous impurities and crystal defects and predict composition gradients for different crystal growth conditions of $Hg_{1-x}Cd_xTe$ alloys $(0<x<1)$.

Advancement of the technology of producing high-detectivity far-infrared-sensor materials ($Hg_{1-x}Cd_xTe$ alloys) requires control of purity, composition, and homogeneity, and a systematic study of the basic physical properties of the alloy system.

The approach is to theoretically model the electrical and optical properties of pure, homogeneous, single-crystalline $Hg_{1-x}Cd_xTe$ alloys and to verify the crystal growth kinetics.

It is essential that the intrinsic characteristics of the alloys be separable from effects caused by extraneous impurities and crystal defects through use of hyperpure Hg, Cd, and Te and the avoidance of impurities and crystal defects during melting and processing.

Marangoni Effect in Crystal Processing (MPS77D027)

Arthur D. Little, Inc.
Dr. A. A. Fowle
Dr. M. Davidson, MSFC
Prof. A. Witt, Massachusetts Institute of Technology
NAS8-32946 - $618K

One of the principal advantages of growing crystals in a weightless environment is the elimination of buoyant forces that give rise to thermal and solutal convective flows which produce unwanted fluctuations to growth rate and composition. There are, however, other mechanisms giving rise to flows that must be understood. Perhaps the most important non-gravity driven flow is the Marangoni effect which arises from variations in surface tension forces. This effect can be important in crystal manufacturing on Earth and will influence the optimum processing conditions in space. The present state of scientific knowledge regarding surface tension forces and their effect in the manufacture of crystals under any gravity conditions is inadequate. The initial space experiments use a float zone facility. In situ measurement of the temperature and velocity distributions within the melt will be made. Crystal samples will be returned to Earth for laboratory examination for segregation, growth behavior characteristics and defect formation. Comparison between the data from in situ measurements, theoretical predictions, and the results of crystal characterization will provide the scientific basis for quantifying the Marangoni effect in the crystal growth process.

Float Zone Experiments in Space (MPS770094)

Iowa State University
Dr. J. D. Verhoeven
Dr. F. A. Schmidt
Dr. E. D. Gibson
H-34328B - $130K

This experiment is planned to pinpoint the causes of convection in the low gravity environment and to allow design of space experiments which are free from convection. The analytical float zone crystal growth system will be utilized to perform these experiments.

Convection due to Marangoni flow (non-isothermal surface tension gradient induced flow) and volume changes upon solidification may be present under some conditions in space experiments. The conditions which produce this convection are at present not well defined. In float zone experiments the condition of the liquid surface (i.e. whether or not a solid film such as an oxide of even monolayer dimensions is present) may play a critical role in eliminating Marangoni convection.

The first objective of this experiment is to determine the extent of convection in float zone experiments conducted in space on semiconductors and metals, its dependence on temperature gradient, solute level, types of solute, and also to determine whether or not this convection can be eliminated by use of thin surface films upon the floating zone.

Float zone solidification may be used to prepare uniformly doped single crystals of reactive materials by the zone leveling technique. However, uniform axial and radial doping of semiconductors by float zone leveling have not been achieved in Earth-based experiments due to convection problems. Density driven convection is inherent to Earth-based float zone and Czochralski type experiments. Therefore, a second objective of these experiments is to determine whether or not one may sufficiently control convection in space float zoning to allow preparation of semiconductor crystals uniformly doped in both radial and axial directions.

Epitaxial Growth of Single Crystal Films (SPAR Experiment 74-45)

Rockwell International Science Center
Dr. M. D. Lind
Dr. R. L. Kroes, MSFC
NAS8-31733 - $103K

The objective is to grow an epitaxial film of gallium arsenide by liquid phase epitaxy in low gravity, and to compare it with films grown in normal gravity.

Convection and sedimentation affect the transfer of material and heat in liquid phase epitaxy and result in undesirable nonuniformities in growth rates and in the distributions of dopants or impurities, accompanied by strains and structural defects. Gallium arsenide substrate wafers mounted in a graphite slider are moved into contact with a high temperature (720°C) saturated solution of gallium arsenide in liquid gallium in a tubular resistance heated furnace. After one minute, the growth is terminated by retracting the slider. Films with improved compositional uniformity, less strain, and fewer structural defects are expected. Convectional techniques for characterizing semiconductor materials will be used to examine the space grown films and compare them to films grown in normal gravity.

Vapor Growth of Alloy-Type Semiconductor Crystals (M77082)

Rensselaer Polytechnic Institute
Prof. H. Wiedemeier
Dr. E. A. Irene, IBM
Dr. C. C. Wang, RCA
Dr. M. C. Davidson, MSFC
NAS8-32936 - $460K

The results of previous space experiments demonstrated the positive effects of microgravity on crystal morphology and led to the observation of unexpected transport phenomena. This experiment is concerned with the continued investigation of basic vapor transport and crystal growth properties of electronic materials. Solid solutions of these materials offer a wider range of electronic properties and can be designed for specific applications. The purpose of this investigation is to grow single crystals of alloy semiconductors by chemical vapor transport techniques in a microgravity environment.

The inherent partial pressure gradients of the system and the presence of gravitational forces on Earth cause convective interference with the transport and condensation processes. The negative effects of this interference on crystal perfection are known. In the absence of gravity-driven convection, crystals of better quality can be obtained.

Vapor Transport Mechanisms Using Laser Raman Probe Techniques

Athens State College
Dr. G. L. Workman
Dr. M. C. Davidson, MSFC
Mr. J. M. Zwiener, MSFC
NAS8-32920 - $78K

The objective is to analyze transport mechanisms involved in the growth of crystals by chemical vapor transport by use of Raman spectroscopy to probe the thermal and compositional environment in the growth process.

As a result of chemical vapor transport experiments performed in a microgravity environment on Skylab and ASTP an apparent discrepancy was discovered between observed growth rates and those predicted by diffusion theory. These flight experiment studies included several combinations of germanium compounds (GeSe, GeTe, GeS) transported using either germanium iodide or germanium chloride transport vapor. The transport rates were significantly greater than predicted by extrapolating ground based experiments at low pressures where convection is negligible or by diffusion calculations in which it is assumed that all chemical reactions take place at the source and the seed. In addition, it should be noted that the crystalline quality based on x-ray diffraction, etching and general growth morphology indicated that much higher quality crystals were grown in a microgravity environment than would be expected from diffusion controlled growth conditions. Additional chemical reactions may be taking place in the transport gas and that the heat of reaction may somehow drive a "thermo-chemical convective" process.

Measurements are being performed employing a laser Raman probe with various boundary conditions, transport agents, and source materials to determine if conditions exist which would significantly affect growth rates in low gravity environments. Stokes versus anti-Stokes intensities are assessed for the determination of local gaseous temperatures. Tubes containing I_2, GeI_4, and GeI_4 + Se have been run to obtain identifying Raman calibration spectra for the iodine, GeI_2, GeI_4, and Se in the gas phase. The Raman spectral band data has shown that GeI_4 in the gas phase exists in higher amounts as compared to the GeI_2 gas than expected.

Mercuric Iodide Crystal Growth for Nuclear Detectors (M77055)

EG&G Inc.
Dr. W. F. Schnepple
Dr. L. van den Berg
Dr. M. Schieber
Mr. J. Williams, MSFC
NAS8-32949 - $80K

The purpose of this investigation is to grow mercuric iodide crystals in a low-gravity environment to obtain higher perfection by taking advantage of diffusion-controlled growth conditions and by avoiding the problem of strain dislocations produced by the crystal's weight. This crystal has considerable practical importance as a sensitive gamma-ray detector and energy spectrometer that can operate at ambient temperature, as compared to presently available detectors that must be cooled to near liquid nitrogen temperatures. However, the performance of such crystals only rarely approaches the expected performance, presumably because of defects that limit the distance the carriers can drift before being trapped.

These crystals will be grown by vaporization and recondensation at approximately 120°C in a specially designed furnace. Provisions will be made to reverse the growth procedure if polycrystalline growth begins, which is a common problem. Experiments will be conducted prior to flight to optimize the growth process, to establish the best performance that can be obtained in the terrestrial laboratory, and to provide a basis for comparison with the low-gravity results.

There is good reason to believe that the growth of this crystal can be improved in a low-gravity environment. The absence of gravity-driven convection reduces fluctuations in the vapor density and temperature in the vicinity of the seed crystal. This better-controlled environment is conducive to uniform growth with far fewer defects, as demonstrated by Wiedemeier on Skylab. Secondly, this particular crystal is a layer type with weak bonding beteen slip planes, and it is believe that the high dislocation densities and strain fields observed in the terrestrially grown crystal originate from the weight of the crystal as it is supported at the growth temperature.

Crystal Growth in a Spaceflight Environment (M77100)

Jet Propulsion Laboratory
Dr. P. J. Shlichta
Dr. M. H. Leipold
Dr. C. H. Savage
Dr. W. R. Wilcox, Clarkson College
Mr. J. Williams, MSFC
NASw-100 - $172K

The experiment will determine whether the near-weightless environment of a Space Shuttle/Spacelab mission is sufficient for the growth of defect-free crystals and the extent to which residual and transient accelerations should be suppressed or minimized. In addition, data will be obtained on growth rate and crystal quality as a function of forced convection.

Crystals will be grown from transparent fluids at programmed cooling rates in controlled thermal geometries. A time-lapse movie camera will record a continuous cyclic sequence of schlieren, shadowgraph, and reflected-light images of the growing crystal and surrounding fluid. Analysis of these images will provide maps of the growth rate, fluid motion, and interface surface texture. These data will be compared with the distribution of defects and impurities in the recovered crystal and with the accelerational history of the space flight.

Solution Growth of Crystals in Zero Gravity (M77058)

Alabama A&M University
Dr. R. B. Lal
Dr. R. Kroes, MSFC
NAS8-32945 - $135K

Crystals will be grown by the low-temperature solution growth technique in a microgravity environment of the orbital Spacelab. The objectives are: (1) to produce structurally more homogeneous crystals free from inclusions of solution by eliminating convection transients, (2) to obtain data on mass and heat transport in a diffusion-controlled growth system, and (3) to confirm the advantages of a microgravity environment for solution crystal growth.

Triglycine sulfate (TGS) crystals will be grown in the multipurpose fluids facility extracting heat at a programmed rate through a seed crystal of TGS suspended on an insulated sting in a saturated solution of TGS. Variations in the liquid density, solution concentration, and temperature around the growing crystal will be studied using a variety of techniques such as optical absorption, schlieren, shadowgraph, and interferometric measurements. Growth in Earth gravity will be compared and correlated with the growth conditions in space.

Schlieren and shadowgraph images of crystals growing from solutions in unit gravity show massive convective flow because of thermal and concentration gradients as the solute is incorporated into the crystal. Generally, this is controlled by gently stirring the solution to keep uniform growth conditions. However, this uniformity can only be approximated, and small-scale inhomogeneities always exist which adversely affect the growth of the crystal. In a microgravity environment, it should be possible to eliminate convective flows and establish a diffusion-controlled transport of the solute to the interface. By extracting heat from the crystal in a controlled manner, it should be possible to maintain saturation at the growth interface, allow uniform growth, resulting in a high degree of crystal perfection.

Thermal Analysis of Bridgman - Stockbarger Growth

GAI, Inc.
Dr. Guest
Mr. Williams, MSFC
NAS8-33204 - $38K

The objectives of this study are to develop the thermal profile interface shape and position, and thermal gradients at the liquid-solid interface for a cylinderical sample in a Bridgman - Stockbarger crystal growth configuration. This analysis will be based on the alloys of HgTe and CdTe. The study will analyze various simplified models and develop suitable computer programs to model actual growth configurations. The program verification phase of the activity is nearing completion.

Determination of Crystal Perfection

National Bureau of Standards
Dr. M. Kuriyama
Dr. H. E. Burdette
Dr. R. Dressler, NASA Headquarters
H27954B - NA

The objective following the actual crystal growth on Earth is to help NASA establish methods for evaluating space-grown crystals with reference to nearly perfect crystals grown on Earth. For this purpose our intention is to perform Asymmeric (Double) Crystal Topography (ACT) on a real-time basis so that any laboratory can evaluate crystal perfection routinely with the same accuracy using an ACT camera. We have reported the crystal characterization work on nickel single crystal growth with emphasis on the interpretation of observed imperfection images including a new rule of image invisibility. Our activities on real-time imaging are aimed toward improvement of resolution and brightness.

Solidification Processes Involving Solutes

Rensselaer Polytechnic Institute
Prof. M. E. Glicksman
Dr. L. L. Lacy, MSFC
NAS8-32690 - $61K

The objective is to examine the effects of solute additions on the morphology and growth kinetics of dendrites and to determine the relationship of solute segregation and local equilibrium at the solid-liquid interface.

The addition of solutes to pure materials has a major influence in controlling the microscopic structure and consequently the properties of materials. An objective of space processing solidification experiments is the optimization of selected physical properties of materials by controlling the microstructure. This requires a quantitative understanding of the relationship of solute segregation and growth kinetics. This study together with a study on the influence of gravity-driven convection on the growth rate of dendrites forms the basis for a better understanding of the influence of solidification on the microstructural relationships in a wide category of materials for both structural and electronic applications.

Using high-purity succinonitrile as a solvent, various solutes will be added which either elevate or depress the equilibrium segregation coefficient. Optical observation and precision thermometry will be used to study the dendrite growth kinetics at various levels of undercooling. After performing studies on this model material, metal alloys will be investigated to establish the corresponding segregational phenomena in the interpretation of metallographic structures by using scaling laws.

The growth dynamics of dendrites growing at small levels of undercooling (0.1 to 1°K) have been shown to depend strongly on the direction of gravity since convective heat transfer is the dominant mode of heat transfer in dendritic crystals. The maximum growth rate occurs when dendrites grow in a downward direction parallel to gravity and the minimum growth rate occurs when the growth direction is upward or antiparallel to gravity. At larger undercooling ($\Delta T \gtrsim 1^{\circ}$K) the growth dynamics are controlled by diffusive heat transfer, which is independent of gravity.

Dendritic Solidification at Small Supercooling (SPAR Experiment 76-39)

Rensselaer Polytechnic Institute
Dr. M. E. Glicksman
Dr. L. L. Lacy, MSFC
NAS8-32425 - $120K

The objective of this experiment is o measure the dendritic growth velocity of a well characterized high-purity material in a low-gravity environment under conditions of small supercooling.

Dendritic solidification involves both a diffusive and convective heat transport process. Recent studies show that the diffusive component varies almost cubically with the supercooling whereas the convective component varies linearly. Consequently, under terrestrial conditions, dendritic growth tends to be dominated by diffusion at medium-to-large supercooling and by convection at low supercooling (i.e. $\Delta T \lesssim 1^{o}K$). This circumstance is unfortunate for precise experimental studies on the kinetics and morphology of growth since present theories on dendritic growth become inapplicable at small supercooling. In a low-gravity environment, convection would be greatly reduced, thereby extending to lower supercooling the useful range of experimentation.

This experiment will use a specially designed apparatus which will accurately control and measure the undercooling of a high-purity liquid (succinonitrile) to within $\pm$ 10 ^{o}mK at an undercooling in the range of 0.1 to $1^{o}K$. Crystal growth will be initiated after achieving quiescent conditions. The dendritic crystal growth will be photographed in two planes so that accurate quantitative measurements can be made to determine the morphology and growth velocities of the system.

Dendrite Remelting and Macrosegregation in Castings (SPAR Experiment 74-21)

Marshall Space Flight Center
Dr. M. H. Johnston
Ms. C. S. Griner
Mr. R. Parr
Contract Not Applicable

The objective is to investigate the effect of convection on the microstructure of metallic castings. Of particular interest are the mechanisms of dendrite multiplication and transport, secondary nucleation, and freckling.

Both thermal and solutal convection act to redistribute solute, break off dendrite tips, and transport these fragments throughout the melt forming new nucleation sites. The situation is vastly different in low-g where the primary driving force for the convective flow no longer operates. One way to investigate the effects of gravity driven convection is to compare two systems that are identical except for the presence of gravity. Also, this approach is useful for assessing the importance of non-gravity forces (if any) that are often masked by the more dominant gravity driven forces.

A transparent metal model system, NH_4Cl and H_2O, is used to study nucleation and growth processes, dendrite multiplication and transport, and grain morphology. Thermoelectric devices along the cuvette walls provide the cooling to produce solidification. Data is recorded by a 35 mm camera at 1 fps.

A cuvette with cooling from opposite walls was flown. Photographs have been made into movies comparing the 1-g and o-g processes.

Alloy Casting (SPAR Experiment 76-36)

Marshall Space Flight Center (MSFC)
Dr. M. H. Johnston
Mr. R. Parr
Contract Not Applicable

The objective is to investigate the factors affecting nucleation and growth in normal casting processes of metals as influenced by gravity induced convection.

This experiment is designed to investigate the similarities between metal model solidification results carried out in earlier SPAR flights (Experiment 74-21) and those expected from a metallic system. More specifically, the similarities of the two systems regarding convection induced dendrite fragmentation and their effects is of primary interest.

Three different alloys will be melted prior to flight, then frozen rapidly during low 'g' by a gaseous quench. It will be similar to the thermal configuration used with the metal model system flown on SPAR I (Experiment 74-21). The grain morphologies and macrosegregation will then be compared to appropriate ground base specimens.

Comparative analyses of flight and ground base tests may support the convection induced dendrite fragmentation process observed on SPAR Experiment 74-21. Close scrutiny of the metal system/metal model system results will substantiate the accepted technique of investigating metal solidification by direct observation of metal model systems.

Contained Polycrystalline Solidification in Low-G (SPAR Experiment 74-37)

Grumman Aerospace Corporation
Dr. J. M. Papazian
Dr. T. Z. Kattamis, University Connecticut
Mr. F. Reeves, MSFC
NAS8-31530 - $183K

The objective is to evaluate and understand the role of gravity-driven convection on the cast microstructures during solidification by examining the columnar - to - equiaxed transition in a cylindrical configuration with radial heat flow.

Cylindrical castings generally have a columnar grain structure oriented in the radial direction near the surface and an equiaxed grain structure in the interior. The mechanism for this transition has been the subject of several speculative theories. By performing the experiment in 1-g and 0-g environments, it is hoped to elucidate the role of gravity-driven convection in the formation of the equiaxed zone.

A transparent metal model system, NH_4Cl and H_2O, was chosen to allow the process to be studied photographically in real time. Four semicircular cells were used, permitting two different orientations of two cells which contained different concentrations of NH_4Cl. Cooling was accomplished by a flow of freon gas around the circumference of the cells. The solidification process was recorded on the ground and during the coast phase of the rocket.

Excellent photographs of dendrites breaking and the fragments circulating into the interior to nucleate the equiaxed structure have been obtained at 1-g. A flight on SPAR IV failed because of a cooling system blockage. A reflight on SPAR V returned useful data which is currently being analyzed.

Solutal Convection and Liquid Diffusion Coefficients

National Bureau of Standards
Dr. W. J. Boettinger
Dr. S. R. Coriell
Dr. F. S. Biancaniello
Dr. M. R. Cordes
Dr. R. Dressler, NASA Headquarters
H27954B - NA

This research is directed towards an understanding of solutal convection caused by the simultaneous concentration and temperature gradients present during the directional solidification of alloys. Included in this study is investigation of the effects of convection on the microstructure and chemical homogeneity of directionally solidified alloys and of the conditions under which undesirable effects can be eliminated.

The onset of convective and interfacial instabilities during the vertical directional solidification of a binary alloy is determined theoretically by means of a linear stability analysis. For values of the physical constants appropriate to the solidification of lead containing tin, the critical bulk solute concentration delineating the demarcation between stability and instability is calculated as a function of growth velocity. For concentrations less than the critical concentration, the system is stable. For growth velocities between 1 and 40 μm/s, the critical concentration increases with increasing velocity; for velocities greater than approximately 40 μm/s, it decreases with velocity.

The occurrence of solutal convection and its effects on the microstructure and macrosegregation of directionally solidified alloys, particularly off-eutectics, is investigated using lead-tin alloys. Lead-rich lead-tin alloys have been directionally solidified vertically upwards. During plane front solidification the concentration of less dense solute is increased in the liquid near the solid-liquid interface and there is the possibility of solutal convection.

Liquid Metal Diffusion in Solubility Gap Materials (SPAR Experiment 77-7)

Marvalaud, Inc.
Prof. R. B. Pond, Sr.
Mr. Don Reiss, MSFC
NAS8-33046 - $34K

The objective is to measure the diffusion rate of two liquid metals in one another for systems that exhibit a solubility gap.

There are difficulties in making diffusion measurements in liquid metal systems, particularly those exhibiting solubility gaps. Particles of the lower density phase that nucleate as the temperature is lowered precipitate out, yielding erroneous diffusion rate data. Rapid quenching minimizes this motion but takes the system out of equilibrium and may introduce uncontrolled convection which could alter the diffusion profile. The elimination of gravity-driven convection in a flight experiment will preserve the position of the precipitated particles, allowing more reliable measurements of diffusion rate to be made. This will provide a check on the validity of the rapid-quench method.

Ground-based experiments are being performed on Pb-Zn diffusion couples to establish the effects of convective mixing and the degree to which it can be controlled. A number of different combinations of temperature, time at temperature, couple diameter and orientation relative to gravity will be used. The samples will be subjected to metallographic examination after quench.

Agglomeration in Immiscible Liquids at Low-G (SPAR Experiment 74-30)

S. H. Gelles Associates
Dr. S. H. Gelles
Dr. L. L. Lacy, MSFC
NAS8-31534 - $244K

The objective of the experiment is to determine the effect of composition, cooling rate, and the presence or absence of gravitational forces on the structure of liquid-phase immiscible alloys, e.g., the aluminum-indium immiscible system.

Low-gravity processing of liquid-phase immiscible alloys could lead to unique microstructures and metastable phases not obtainable in the bulk on Earth. This experiment should lead to the determination of quantitative relations between the alloy structure and processing conditions including gravitational forces.

Pure indium and pure aluminum starting materials are enclosed in an alumina crucible cartridge. Four cartridges are provided which contain varying compositions of pure aluminum and indium. Prior to launch the samples are heated to 900°C and held until the low gravity portion of the flight is reached. The cartridges are then cooled through the immiscibility gap and the alloys are solidified in low-g to prevent the sedimentation of the more dense phase.

Liquid Miscibility Gap Materials (M77030)

S. H. Gelles Associates
S. H. Gelles
Mr. F. Reeves, MSFC
NAS8-32952 - $431K

The objective of this investigation is to study the evolution of microstructural features of alloys that contain a liquid phase miscibility gap. Of particular interest are the agglomeration and segregation that occur within the miscibility gap and the processes that occur at the monotectic and eutectic temperatures.

Many alloy systems have miscibility gaps in which two liquid phases are immiscible below the consolute temperature. Many of these alloys cannot be formed in bulk in Earth gravity because the density differences of the two liquid phases result in rapid separation when the melt is cooled through the miscibility gap. It is believed that removal of the buoyant forces by cooling such systems in a low-gravity environment will prevent this separation and allow the formation of these alloys.

The study of the behavior of immiscible alloys is an important theoretical problem in solidification physics that cannot be studied on Earth.

Unidirectional Solidification of Monotectic and Hypermonotectic Aluminum-Indium Alloys (SPAR Experiment 76-51)

French Atmoic Energy Commission, Nuclear Research Center of Grenoble
Dr. C. Potard
Mr. R. C. Ruff, MSFC
Contract Not Applicable

The objective is to study the solid structure formed during low-g solidification of monotectic and hypermonotectic Al-In alloys; to provide optimum thermal and composition parameter values for obtaining regular structures.

Metallic binary systems having an immiscibility gap in the liquid state form a broad family (about 350 systems) which has not been exploited because of the great difficulty of preparation on Earth. Experiments in space are necessary to determine if fine long-range dispersoid solid structures can be obtained in the absence of buoyancy forces.

Samples of monotectic (5 at % In) and hypermonotectic (10 at % In) composition will be solidified under conditions of both high and low termal gradients. The samples will be contained in silicon carbide coated graphite crucibles. It has been shown that SiC will preferentially be wetted by the aluminum-rich phase with the result that surface convection will be minimized. Also, the SiC will not act as a nucleation site or sink for the In-rich globules as they are formed.

The degree and uniformity of dispersion of the second phase will be measured. This will be compared to theoretical models.

Solidification Behavior of Al-In Alloys Under Zero Gravity Environment (SPAR Experiment 74-62)

Technical University Berlin
Prof. Ahlborn, University of Hamburg
Prof. Lohberg
Mr. M. Robi.. on, MSFC
Contract Not Applicable

The objective of this experiment is to solidify under a low-gravity environment an aluminum-indium alloy with a fine, uniformly dispersed second phase. The physical and electrical properties will be studied in the laboratory. Composition of the samples will be selected so that one sample will provide information concerning "spinodal decomposition."

Since the aluminum-indium system has an immiscibility gap in the liquid state, the two materials will separate into two interdispersed liquids upon cooling. Before solidification can occur in ground-based tests, the droplets of the higher specific gravity indium will sink and the two liquids thus separate. Agglomeration of the droplets will also be enhanced by thermal convection. Since the separation process is gravity-driven, it becomes necessary to process the aluminum-indium alloys in a low-gravity environment to produce an alloy with a fine, uniformly dispersed second phase.

Pure indium and aluminum will be enclosed in an alumina crucible cartridge. Two cartridges will be provided, one aluminum-rich and the other indium-rich. Prior to launch the samples will be heated to 900°C and held at that temperature until the low-gravity portion of the flight is reached. The cartridges will then be cooled and the alloys solidified. After recovery the samples will be studied to determine their physical and electrical properties as well as their microstructure.

Nucleation and Growth of Immiscible Phases

Marshall Space Flight Center (MSFC)
Dr. L. L. Lacy
Dr. Gary Nishioka
Contract Not Applicable

The study of immiscible organic liquids has four basic objectives: (1) increased understanding of nucleation and growth processes as an immiscible phase forms, (2) quantitative understanding of emulsion stability once a phase has formed but is still dispersed in the other phase, (3) understanding the effect of solidification on a disperse system, and (4) relating the above processes to the phase diagram.

By studying organic liquids we hope to gain a useful predictive knowledge of the kind of micro-structures that can be formed in low gravity where a basic understanding of critical phenomena is possible without the interfering effects of gravity. By studying the stability of emulsions in low gravity, fundamental knowledge of surface forces near the critical point can be obtained. An increased understanding of nucleation, growth, emulsion stability, and solidification as related to the phase diagram is needed.

Organic systems are being studied because their critical solution point is between 50-100°C, and they are transparent and can be studied by holography. Because holography can measure depth as well as length and breadth it is a powerful tool for the quantitative study of disperse systems. Important parameters such as surface tensions and activity coefficients are being determined to gain a quantitative understanding of behavior in the phase diagram.

Ultimate Coercive Strength of Sm-Co Magnets

Massachusetts Institute of Technology - Draper Laboratories
Dr. D. K. Das
Dr. J. McClure, MSFC

The objective is to produce high strength $SmCo_5$ magnets by removing impurities, particularly oxygen. Melting the Samarium and Cobalt in a levitation furnace in low gravity may yield significant improvements in purity and because using the high pumping speed of the space vacuum may reduce contamination during comminution and pressing of t' . $SmCo_5$ ingot. The work will proceed as follows: Verify that the improved coercive strength of plasma sprayed magnets is due to a reduction in oxygen content rather than due to a different grain size. Throughout the program attention must be paid to the grain size of the material since small changes in grain size can have large effects on the coercive force. Determine the impurity levels in SmCo ingots prepared by conventional techniques such as arc melting in a cold crucible, melting in a reducing atmosphere, melting in a crucible that does not introduce deleterious impurities or other techniques which show promise. Calculate the improvement in coercive force that will be obtained by levitation melting over other techniques. Determine whether $SmCo_5$ can be produced in Earth gravity by levitating in a gas cooled coil. Design and fabricate apparatus to comminute and press a SmCo ingot without exposing the powder to atmosphere. Either an inert gas or a vacuum will be used during this operation. Verify that the impurity level and the coercive force of magnets prepared in this apparatus are improved. Calculate the effect of comminution in a space vacuum. Determine the benefits of space processing $SmCo_5$ magnets.

Growth of Metastable Peritectic Compounds

Grumman Aerospace Corporation
Dr. D. J. Larson, Jr.
Dr. L. Lacy, MSFC
NAS8-32998 - $87K

This effort will attempt to produce an aligned peritectic compound such as Co_5Sm by first solidifying a metastable β-phase and then using a solid-state eutectoidal transformation to obtain an aligned magnetic structure for improved magnet performance.

Using the peritectic system Pb-Bi as a model, directional solidification studies will be carried out to determine the influence of thermal and solutal convection on the liquid-solid interfacial stability and the formation of a metastable peritectic phase. The results will be compared with solidification theory on peritectic phase formation. Convective effects will be studied by performing the experiments both parallel and antiparallel to the gravity vector. In the latter stage of the investigation, directional solidification studies will be performed on the Co-Sm system in attempts to produce an aligned magnetic structure of the peritectic compound, Co_5Sm.

Aligned Magnetic Composites

Grumman Aerospace Corporation
Dr. D. J. Larson
Dr. W. R. Wilcox, Clarkson College
Mr. F. Reeves, MSFC
NAS8-32948 - $163K

The previous space experiment resulted in a directionally solidified MnBi/Bi eutectic with a planar faceted/nonfaceted interface. The microstructure of the space grown ferromagnetic MnBi phase was considerably finer than identically processed terrestrial samples. Space-processed samples exhibited substantial improvements in intrinsic coercive strength, magnetic induction, and the static and dynamic energy products over the terrestrially processed samples. There are varying hypotheses to explain these results, and this investigation is designed to determine whether the origins of these improvements are gravitationally dependent. These experiments will ascertain whether the enhanced magnetic properties are unique to samples grown in space, or whether they can be duplicated on the ground by an optimization of the processing parameters, such as growth rate (R), thermal gradient (G), and composition (X).

Directional Solidification of Magnetic Composites (SPAR Experiment 76-22)

Grumman Aerospace Corporation
Dr. D. Larson, Jr.
Dr. R. G. Pirich
Dr. W. R. Wilcox, Clarkson College
Dr. J. McClure, MSFC
NAS8-32219 - $157K

The objective is to investigate the microstructure and magnetic properties of MnBi/Bi at different growth rates to determine what role convection plays in the directional solidification of this system.

An unusually high coercive strength phase at low temperature was discovered in the MnBi/Bi eutectic material directionally solidified on ASTP.* Although material processed in the laboratory has exhibited this high coercive phase at low temperatures, the values attained by the ASTP samples have never been duplicated for the same growth conditions. This experiment will provide additional data at high growth rates in order to investigate the effect further.

MnBi samples are loaded into quartz tubes 6mm in diameter by 30 cm long in the Automatic Directional Solidification Furnace. Thermocouples imbedded in the sample record the thermal history and growth rate. Magnetic characterization is performed to measure the high coercive phase at low temperatures. A matrix of values of composition, growth rate and gradient has been defined to systemmatically investigate the effect of convection on the directional solidification of the Mn/Bi system.

* Apollo-Soyuz Test Project

Foam Copper (SPAR Experiment 77-9)

Marvalaud, Inc.
Prof. R. B. Pond, Sr.
Mr. J. M. Winter, Jr.
Mr. D. A. Shifler
Mr. B. S. Tibbetts
Mr. Don Reiss, MSFC
NAS8-33021 - $31K

The objective of this experiment is to produce a copper foam with homogeneous porosity and a density less than a third of pure copper.

This experiment could lead to a new class of high-strength, low-density structural materials. Commercial applications for light, strong materials of this type are numerous. Replacement of solid metal structural members with metal foams could reduce vehicle weights, resulting in substantial reductions in fuel consumption. Since metal foams are formed by creating bubbles in a melt, the experiment will have to be done in the low-gravity environment of space to avoid segregation due to buoyancy. In space, the bubbles should remain at their nucleation sites until the melt solidifies, producing a homogeneous foam.

A sample consisting of copper, copper oxide, and graphite will be sealed in a metal cartridge and placed in the General Purpose Rocket Furnace. In microgravity, the sample will be melted, allowing the copper oxide and graphite to come into contact and react, producing carbon monoxide bubbles. The sample will then be cooled, producing a solid copper foam. The data obtained from this experiment should make it possible to design a recipe for producing copper foams of any desired density and bubble volume. It may be possible to extend these results to other useful alloys.

Solid Electrolytes Containing Dispersed Particles (M77012)

Arizona State University
Dr. J. Bruce Wagner
Mr. F. Reeves, MSFC
NAS8-32937 - $136K

In this investigation solid state electrolytes such as Ag halides with a uniformly dispersed second phase of fine Al_2O_3 and/or SiO_2 particles will be prepared in low gravity. It is known that the presence of a dispersion of this second-phase material consierably enhances the ionic conductivity of these electrolytes, but the exact mechanism is not understood. By varying the size and concentrations of the second-phase material, it may be possible to evaluate the relative importance of interfacial conduction, space charge effects, and possibly other ion conduction enhancement mechanisms if good control of the distribution of the dispersed particles can be maintained during solidification.

One of the difficulties involved in preparing such samples on Earth is that the dispersoids have a different density and tend to sediment when the matrix material is melted. In a low-gravity environment the buoyant forces responsible for this separation are absent and the initial homogeneity can be maintained through melting and solidification.

Containerless Processing Technology (SPAR Experiment 76-20)

Jet Propulsion Laboratory (JPL)
Dr. T. G. Wang
Dr. D. D. Elleman
Dr. M. M. Saffren
Dr. W. Oran, MSFC
NASw-100 - $101K

The objective is to study basic technology of containerless processing of materials in an acoustic chamber in space. The specific objectives initially are:

1. Determine the positioning capability of the acoustic system.

2. Determine the perturbation of drop shape oscillation due to g-jitter induced center of mass motion.

3. Determine the rotational capability of the acoustic chamber.

4. Determine the perturbation of drop rotation due to g-jitter.

In containerless processing, processes are carried out in a liquid state. The aim of these experiments is to gain a better understanding of the capabilities of manipulating liquids in a long-term zero-gravity environment. The effort will initially study the stability and manipulability of liquid drops at room temperature as a useful and cost effective step in the development of a high-temperature three-axis acoustic positioning system.

Fining of Glasses in Space

Jet Propulsion Laboratory
Dr. M. C. Weinberg, JPL
Dr. E. J. Hornyak, Owens-Illinois, Inc.
Mr. J. Williams, MSFC
Contract N/A - $94K

High-technology glasses must be produced virtually free from bubbles to be useful. However, fining of glasses in space may prove to be difficult since Stokes bubble rise will be virtually inoperative in the microgravity environment of the Space Shuttle. Thus, the plan is to investigate the feasibility of employing bubble dissolution as an effective bubble removal mechanism. This will entail finding the appropriate refining agent, refining agent concentration, and temperature to employ for a given fining situation.

Space Shuttle experiments will consist of two types: (1) single-bubble studies and (2) multi-bubble refining investigations. A single bubble may be introduced into a small, well-refined glass melt via a bubble injection technique. The position and size of the bubble will be monitored as a function of time via photographic methods. The chemical composition of the bubble may be determined by quenching the melt and analyzing the gas content of the bubble entrapped in the glass. Refining experiments will also be performed utilizing batch mixtures of raw materials. Preliminary gound-based studies will be employed to determine the best methods of sample fabrication, fining temperature, refining agent concentration, and other processing parameters. Bubble number and bubble radii will be monitored continuously.

Since in the 1-gravity environment of an Earth-based laboratory both convective flows in the melt and buoyant forces will contribute to bubble dynamics, only in the microgravity environment of space can one determine the role of gas diffusion effects at refining temperatures.

Physical Phenomena in Containerless Glass Processing (M77010)

Clarkson College
Dr. R. S. Subramanian
Dr. R. Cole
Mr. F. Reeves, MSFC
NAS8-32944 - $148K

The purpose of this experiment is twofold: (1) to develop novel techniques for mixing and fining glasses in a weightless environment and (2) to determine the feasibility of producing spherical glass shells with highly uniform wall thickness in space for the inertial confinement fusion program.

The experiments using molten glasses and model fluids will be carried out in a special furnace which uses a three-axis acoustic positioning device to manipulate the samples and to prevent them from contacting the furnace walls. A spot-heater will be used to introduce a thermal gradient to investigate thermocapillary mixing and thermal migration of bubbles. The centering of a bubble in the sample will be investigated by various mechanisms such as successive rotation about the three axes. Containerless processing of glasses in a weightless environment is a promising technological endeavor because problems of chemical reaction and nucleation at the container wall can be avoided. Also, the lack of buoyant forces on a bubble inside a thin-walled glass shell may allow more precise control of the wall thickness than can be accomplished by processing in normal gravity. The absences of gravitational forces may, on the other hand, adversely affect both the homogenization and fining (removal of gas bubbles) from the melt; therefore, it is necessary to consider alternate methods for accomplishing these operations in zero gravity.

A Thermochemical Study of Corrosive Reactions in Oxide Materials

National Bureau of Standards
Dr. H. S. Parker
Dr. S. Roth
Dr. C. D. Olson
Dr. E. R. Plante
Dr. R. Dressler, NASA Headquarters
H27954B - NA

The objective of this work is to understand reactions of alkali, iron and other oxides, particularly, in the very reactive liquid state. Another objective is to investigate the nature, extent and limitations imposed by the sample-container reaction and to investigate containerless techniques. The system $KFeO_2$-Fe_2O_3 has been examined over portions of the subsolidus region. The occurrence of a β-alumina structure-type at a composition $K_2O{:}5.75Fe_2O_3$ (85.2 mole percent Fe_2O_3) has been observed. Little corrosion of platinum by this composition at a temperature of 1475°C was detected. Vapor pressure measurements of $KFeO_2$ showed an equilibrium potassium pressure of the order of 10^{-4} atmospheres in the 1200°C range.

Dynamics of Liquid Bubbles (SPAR Experiment 77-18)

Jet Propulsion Laboratory (JPL)
Dr. T. G. Wang
Dr. D. D. Elleman
Dr. R. Nolen, KMS Fusion
Dr. W. Oran, MSFC
NASw-100

This is an experimental research program which will contribute to the understanding of containerless processing of fusion targets--microballoons-- in space. Specifically, it will study these aspects of containerless processing of fusion targets in space:

1. Determine the sphericity of a positioned liquid bubble.

2. Determine the efficiency of bubble centering by rotation and induced oscillation.

3. Study the natural resonance frequencies and damping mechanism of bubble oscillation.

4. Study the adiabatic expansion of liquid bubbles to better understand any physical processes limiting the shell thickness and bubble size.

Thermonuclear fusion is an extremely important scientific endeavor. One area of fusion research known as inertial confinement is attained by irradiating a target with extremely high intensity beams. The targets are hollow spherical shells, most often of glass, filled with deuterium and tritium. The requirements on the uniformity of the shell thickness and sphericity of the target are very severe and little is known about the formation of acceptable targets.

A simple but approximate model for bubble-shell centering may be the liquid bubble. Study of the centering of the inner gas bubble with the liquid-film container at <u>constant temperature</u> could be used to correlate viscosity effects, surface tension, gravity, centrifugal forces, and acoustic forces necessary to maintain the liquid bubble in position. A difficult task in studying glass shell formation directly is the high temperature needed to maintain glass viscosity in workable range. Therefore, the liquid bubble appears as a simple approximation for studying bubble-shell centering in the three-axis acoustic processing module.

Upgrading of Glass Microballoons

Bjorsten Research Laboratories, Inc.
Dr. S. Dunn
Mr. J. Johnson, MSFC
NAS8-33101 - $98K

The objective is to study, examine and perturb the processes and mechanisms involved in reforming glass microballoons to acceptable quality levels for laser fusion targets.

A collimated hole structure (CHS) will provide gas jet levitation and manipulation for exploring techniques to upgrade glass microballoons (GMB). Levitated GMB's will be heated and external pressure reduced to enlarge them. Spinning will also be accomplished to explore centrifugal concentering. Characterized GMB's will be used to permit assessment of upgrading experiments by comparing geometric, surface feature, and concentric changes.

This study is expected to produce new baseline information regarding CHS levitation and manipulation techniques and will determine the feasibility of uprating the GMB's by this method with implications for acoustic levitation.

Glass Shell Manufacturing in Space

KMS Fusion, Inc.
Dr. R. L. Nolen
Mr. J. Johnson, MSFC
NAS8-33103 - $227K

The objective is to study, model and understand the gravity dependent physical and chemical mechanisms involved in forming spherical glass shells. This will be accomplished by perturbing sphere forming mechanisms to determine the respective changes in characteristics of glass shells produced in free fall and levitation furnaces in a one-g environment.

Generation of a basic research data base to ascertain definable one-g technology of forming high-quality glass shells is required to predict the respective characteristics (e.g. size, surface features, concentricity, etc.) and practical feasibilities of formation of glass shell microspheres in a zero-g environment.

Ultrapure Glass Waveguides

Battelle Columbus Laboratories
Dr. Mukherjee
NASw-100 - $55K

The objective is to take advantage of the unique physical conditions existing in space for the preparation of ultrapure homogeneous glass for optical waveguides. The phenomena existing in an orbiting space vehicle are weightlessness, suppression of segregation due to density difference, and absence of gravity-induced convection currents.

Crucible-free melting excludes the last source of melt contamination from the crucible; and thus, the optical and electrical properties of the melt can be of superior quality. Hence, the containerless melting process in space could produce ultrapure (i.e., free from transition metals) highly transparent glass blanks. The application of glass for optical waveguides depends on two parameters; its transparency and its forming characteristics. The transparency, which can be achieved by avoiding contamination, permits signal transmission over long distances and the formation characteristics (i.e., workability) lead to easily fabricating fibers of any length. Hence, glass fibers for optical waveguides could be drawn from the ultrapure glass blanks prepared by the containerless melting in space.

Glass Formation (SPAR Experiment 74-42)

Rockwell International
Mr. Ralph Happe
Mr. Jerry Johnson, MSFC
NAS8-32023 - $214K

The objective is to form glasses of unique composition by use of containerless processing to avoid nucleation and crucible contamination.

It is desirable to explore selected materials which do not readily form glasses in the Earth environment but might be induced to do so in space. The projected combination of new and useful optical properties for such glasses appears attractive in developing advanced optical systems. The space environment will be unique in providing microgravity and containerless melting capability.

Containerless melting eliminates extraneous contamination by crucible wall materials and possible sites for heterogeneous nucleation during cooling. Higher melting point and corrosive melt materials can also be investigated using this technique.

An acoustic single axis levitator will be employed to position a specimen in a furnace. After the specimen is melted, the furnace will be turned off and the molten sphere allowed to cool in an unperturbed environment. In order to maximize the probability of glass formation, a three-constituent system of gallia, calcia, and silica will be used. The silica will slightly degrade the optical property but will enhance glass formation.

A binary gallia/calcia composition will then be processed.

Containerless Preparation of Advanced Optical Glass (M77095)

Rockwell International
Mr. R. Happe
Mr. J. Williams
NAS8-32953 - $624K

Melting of oxide glasses in space has the advantage that no container is required, eliminating reactions with the crucible. The absence of a mold for cooling would eliminate most heterogeneous nucleation sites, greatly enhancing the possibilities for glass formation. New glasses with interesting new combinations of optical properties (i.e., index of refraction, dispersion, partial dispersion, and transmission) might be produced.

The purpose of this investigation is to obtain experimental evidence of the scope of the anticipated new area of optical glasses that may be prepared with containerless melting and cooling in space.

Compositions with melting points below 1600°C will be melted in a containerless melting facility. In addition to sample composition, variables include effects of starting material preparation on final glass quality, effects of melting temperature and time on glass quality, and, perhaps, effects of various cooling rates on glass formation. Ground-based research will precede the flight experiments to determine appropriate compositions to be studied in space and investigate methods of starting material preparation. Processing parameters will be studied to serve as a comparison with flight experiment results. A post-flight evaluation will study the quality and property measurements on the returned flight samples.

Drop Tube Experiments

Marshall Space Flight Center (MSFC)
Dr. L. L. Lacy
Dr. M. Strongin, Brookhaven National Laboratory
Mr. J. R. Williams, MSFC
Contract Not Applicable

The objective is to investigate the effects of undercooling and low-gravity solidification on the microstructure and superconducting properties of metastable A-15 superconducting compounds such as Nb_3Ge and Nb_3Al, and to determine the feasibility of forming metastable Nb_3Ge in bulk form so that detailed physical property measurements can be made.

When molten metals are cooled without contact with crucible walls they can be undercooled by several hundred degrees. Supercooling can have a major influence on the microstructure and properties since it leads to the formation of non-equilibrium or metastable phases. Previous attempts to form Nb_3Ge have shown that this material does not exist in equilibrium form but can be prepared in metastable form by splat cooling. The present thin film forms of Nb_3Ge are not large enough to perform detailed physical property measurements. Nb_3Ge and Nb_3Al are excellent superconductors. Detailed physical property measurements on such materials can lead to a better understanding of why such alloys are superconductors.

This effort involves an investigation of techniques to undercool such alloys using a high-vacuum drop tube apparatus which provides for 2.6 seconds of low-gravity free-fall time. Molten samples of the alloys are prepared by the pendulant drop technique and allowed to undercool and solidify while free-falling.

A variety of Nb-Ge alloys (14 to 28 at % Ge) have been undercooled in the range of 200 to 500K and solidified in low-gravity. Spherical single crystals of pure niobium with diameters in the range of 2 to 5 mm have also been prepared by this technique. These samples have a diameter that is an order of magnitude larger than reported for other techniques. Property measurements are in progress.

Containerless Levitation Technology

Marshall Space Flight Center (MSFC)
Dr. W. A. Oran
Contract Not Applicable

The objective is to develop an understanding of state-of-the-art of containerless processing systems. The purpose is to understand thoroughly the operating principles and problems of the techniques, establish gravity imposed limitations of the techniques, to screen candidate materials for flight experiments, and to provide the best ground based technology against which flight results can be compared. We are currently developing a ground based containerless acoustic furnace (based on a St. Clair generator) which can process glass up to ~1000°C. We are also developing electromagnetic containerless processing systems for use on Earth. We plan to be able to specify an electromagnetic system which will be used with the drop tube and further develop some of the hardware. Specimens will be melted in the electromagnetic coil and cooled in the drop tube, thus eliminating contact with a container.

Electrostatic Control and Manipulation for Containerless Processing

Jet Propulsion Laboratory (JPL)
Dr. M. Saffren
NASw-100 - $200K

In this task techniques for an electrostatic method of positioning and manipulation of materials for containerless processing will be developed for material in both bulk and dispersed form.

This method obviates the difficulties associated with the previously developed acoustic and RF methods.

The long range objective of the task is demonstration of electrostatic containerless processing apparatus in Spacelab flight.

For bulk material, technical objectives include: development of a feedback system intrinsic for electrostatic control, determination of an optimum configuration of electrodes, development of a system to electrically charge the melt, and development of a method to rotate the melt.

Feasibility of concepts for an electrostatic positioning apparatus will be studied, and a design study utilizing laboratory breadboards will be initiated.

For dispersed material, an immediate objective is to determine the importance of suggested applications of an apparatus for the control and manipulation of dispersed material. Applications not requiring spaceflight will be studied using a laboratory apparatus.

Technical objectives are to develop a method for proper electric charging of the dispersed system, and determining the instabilities of the charged dispersion in an electric field.

Measurement of the High Temperature Properties of Tungsten

Rice University
Dr. J. Margrave
Dr. W. A. Oran, MSFC
NAS8-33199 - $20K

The experiment will measure the high temperature properties of tungsten by levitating and melting small pellets of tungsten followed by drops into a calorimeter. These experiments will help define the heat of fusion, heat capacity and other related thermophysical properties of tungsten at high temperatures. Tungsten is a scientifically interesting material since it lies at an extreme metal melting temperature and knowledge of its thermo physical properties is key in extrapolating/interpolating the properties of other materials. In particular, it is unknown whether the well-known Tomman rule can be used to calculate the heat of fusion of tungsten or whether tungsten will deviate significantly from predictions based on properties measured on lower melting refractory metals.

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

Harvard University
Prof. D. Turnbull
Dr. L. L. Lacy, MSFC
NAS8-32691 - $120K

The subject is the crystal nucleation behavior of certain molten alloys known to form glasses in rapid melt quenching (e.g. Au_4Si, Pb_4Si, and Cu-Zn). This characterization includes the measurement of the frequency of homogeneous crystal nucleation and its undercooling dependence and a determination of the ultimate undercooling at slow cooling rates for molten alloys which show the greatest promise of glass formation in massive form. Part of this effort involves a determination of the ultimate undercooling that can be achieved for pure molten metals such as Cu, Ni, and Pd and the grain structure which develops when crystallization is initiated at or near the ultimate undercooling.

This investigation involves the use of a short shot tower where small drops (5-40 μm) are allowed to supercool. Larger size samples (1 to 5 mm) will be prepared in the Drop Tube which is 32 meters long and provides for 2.6 seconds of free fall time. The crystallization temperature is determined by observing where the sample luminosity ("blick") increases due to recalescence at large undercooling. The materials are characterized by x-ray diffraction and microstructure.

Production of Bulk Metallic Glasses in Space (SPAR Experiment 74-49)

Drexel University
Prof. A. Lord
Dr. J. McClure, MSFC
NAS8-31972 - $329K

The objective is to explore containerless processes to produce metallic glass by severe undercooling while eliminating container induced nucleation sites.

When a metal solidifies, it crystallizes. Certain metal alloys, however, can be cooled so fast that the atoms are arranged in a more or less random fashion like the atoms in ordinary glass. Such disordered materials are termed amorphous and have very different properties from the same material in a crystalline state. Present techniques for fast cooling metals on Earth, require that the metal be in very thin ribbon so that heat can be extracted quickly. This space experiment will be an attempt to produce metallic glasses in bulk form by use of electromagnetic containerless techniques to deny nucleation sites during extreme undercooling. It is expected that the metal can be cooled below its usual melting point so that when freezing does take place, the liquid will be so viscous that the atoms in the liquid cannot rearrange themselves into a crystal. It is necessary to do this experiment in space because the high power needed to levitate this material on Earth would heat the metal so hot that it could not be solidified. The low gravity of space requires only a small amount of power to levitate and position the molten metal.

Continuous Flow Electrophoretic Separation (SPAR Experiment 77-E)

Marshall Space Flight Center (MSFC)
Dr. R. S. Snyder MSFC
Dr. G. V. F. Seaman, University of Oregon
Dr. D. A. Saville, Princeton University
Dr. A. J. Johnson, New York University Medical Center
NAS8-32609 - $88K
NAS8-32657 - $37K

The objective is to predict the performance characteristics of the continuous flow electrophoresis units employed at unit gravity and in a microgravity environment; to develop a general and realistic mathematical model of continuous flow electrophoresis taking into account the present state of knowledge; to project the magnitude of the advantages of microgravity processing; to separate biological cells under conditions that optimize both the resolution and condition of the separation species.

The available apparata for continuous flow experimentation on purified biological materials have been designed by essentially empirical methods. Continuous flow electrophoresis involves a large number of interacting phenomena. Our understanding of the basic processes that operate in such devices is incomplete and fragmented. Thus, the theoretical models so far developed for these apparata are of limited value for establishing the limitations of the devices operated under different conditions.

To provide the basic framework of theory and design concepts which will establish the optimal apparatus designs and their limitations on the ground and in space, a multidisciplinary group including a team of scientists representing fluid dynamics, separation processes and biology, has been assembled. Operation of a transparent electrophoresis chamber under carefully controlled experimental conditions will be compared to flight experiments using an electrophoretic separator designed for SPAR.

Laboratory results and theoretical models of space experiments will provide an understanding of continuous flow electrophoresis and the conditions required to yield high resolution, high throughput separations.

Electrophoresis Technology

Marshall Space Flight Center (MSFC)
Dr. R. S. Snyder
Mr. B. Nerren
Contract Not Applicable

The objective is to evaluate and analyze variations in apparatus and operating conditions for continuous flow electrophoresis. All apparata evaluated to date have been designed essentially by empirical methods. Laboratory experiments are being compared with mathematical models of the fluid dynamic behavior and data in the literature or being generated by other scientific groups participating in this project. These experiments will isolate the different sources of flow instabilities using simple chamber geometrics, extensive instrumentation and test particles. This integrated effort will yield a basic understanding for improving resolution and throughput of continuous flow electrophoresis on Earth for direct comparison with experimental results in space.

Automated Analytical Electrophoresis Apparatus

University of Arizona Optical Sciences Center
Dr. P. A. Bartels
Dr. R. S. Snyder, MSFC
NAS8-31948 - $235K

An automated electrophoretic microscope system has been developed to determine and display the spectrum of electrophoretic mobilities of biological cells in a given population suspended in a compatible buffer. An apparatus consisting of a microscope with a computer-controlled focusing device linked to a television camera and controlled by a computer has been built. The computer scans and locks onto the phase-contrast image of a migrating cell and measures its migration velocity, i.e., displacement as a function of time. The data are digitally encoded and stored for subsequent computer processing with all other known facts about the sample. The mobility distribution in a given sample is displayed as a histogram. The apparatus will be able to measure the mobilities of up to 500 cells in ten minutes. The estimate of mean mobility will be accurate to ± 0.5% over a mobility range 0.5 to 5.0 μm/sec/V/cm. The instrument is capable of collecting mobility data on non-pigmented cells in the size range of 0.5 to 25 microns in diameter.

Analysis and Modification of Free Flow Electrophoresis

University of Alabama in Birmingham
Dr. G. Sachs
Mr. P. H. Rhodes, MSFC
NAS8-32923 - $20K

The objective of this effort is to determine the optimum separation, biological throughput and operation stability of a Free Flow Electrophoresis Apparatus from Prof. Hannig which is available. NASA will supply test materials of measured electrophoretic mobility. The separations achieved with these particles at varying concentrations, flow rate and applied field will be obtained. The results of standard particle operation will be compared with biological cells available to the investigator. This effort is expected to indicate the best results for a modern commercial electrophoresis device on the ground.

Investigation of the Free Flow Electrophoretic Process

McDonnell Douglas Astronautics Company - St. Louis
Dr. R. A. Weiss
Dr. R. S. Snyder, MSFC
NAS8-33200 - $85K

Analysis of the free flow electrophoretic process requires that the influence of all physical phenomena on apparatus performance be determined. In order to study the effects of temperature, sample concentration and chamber geometry on the resolution and throughput of a soluble biological mixture, two new electrophoresis flow chambers were built, 60 cm and 120 cm long. The flow curtain thickness for each is 3 mm. We have begun to evaluate temperature effects in the curtain by measuring temperatures under various flow conditions and relating these measurements to a mathematical model of the temperature and flow fields.

Electrophoretic Cell Separation Based on Immunomicrospheres

Jet Propulsion Laboratory (JPL)
Dr. A. Rembaum
Dr. S. P. S. Yen
Dr. S. Margel
Dr. R. S. Molday, University of British Columbia
Mr. B. H. Nerren, MSFC
NASw-100 - $30K

Electrophoresis in space alleviates two major experimental problems: sedimentation of particles to be separated and thermal convection generated by Joule heating. This effort makes use of these advantages and offers the (1) tailoring of the electrophoretic mobility according to need and (2) a new dimension in electrophoretic separation of specific cell subpopulations based on immunological techniques.

The objective is to demonstrate that the cell separation of immunologically labeled cells is more efficient in space environment than on Earth.

The approach is based on labeling specific groups of cells with immunomicrospheres and isolating the labeled and unlabeled cells by means of electrophoresis or isoelectric focusing.

We have recently demonstrated the specific labeling of cell subpopulations, e.g., of murine B and T and human B lymphocytes, by means of immunomicrospheres, i.e., functional polymeric microspheres with antibodies covalently bonded to the functional groups on the surface of the microspheres. We propose to alter the electrophoretic mobility of cell subpopulations by means of these immunomicrospheres and demonstrate a successful separation of a specific cell subpopulation of human lymphocytes in hardware designed for space applications.

Electrophoresis of Whole Cells

University of Alabama in Birmingham
Dr. R. Chiovetti, Jr.
Dr. R. S. Snyder, MSFC
NAS8-33014 - $40K

The objective is to characterize the electrophoresis of whole cells under zero-g conditions and to investigate the factors responsible for the separation of cultured cells into subpopulations, using mean cell volume, profiles of plasma membrane proteins, mitotic indices and cell viability as the methods of investigation.

Preflight activities will include the following assays on synchronized and non-synchronized populations of human kidney fibroblasts: Mean cell volume, profiles of radioactively iodinated plasma membrane proteins and determinations of their molecular weights on SDS-polyacrylamide gels, mitotic indices and cell viability.

During flight, a series of parallel electrophoresis runs will be conducted at ground-based support facilities.

The post-flight phase will include assays as mentioned above which will be performed on the subpopulations of electrophoresed cells. In addition, aliquots of each subpopulation will be reinoculated into cell culture, and these parameters will be followed through several population doublings. The post-flight data will be statistically analyzed and compared to the pre-flight baseline values in an effort to explain the behavior of cells in electric fields under zero-g conditions.

Electrophoresis of Pancreatic Cells

State University of New York at Buffalo
Dr. C. J. van Oss
Dr. R. S. Snyder, MSFC
NAS8-32947 - $157K

The purpose of this experiment is to separate cells from the islets of Langerhans of the human pancreas at zero-g for the purpose of purifying and then culturing the insulin producing beta cells. Separation of the dispersed cells will be done by a zero-g electrophoresis device.

Insulin is used in treating diabetes mellitus which affects over 4,500,000 Americans. It ranks high on the list of diseases causing death and is a major contributor to heart attacks, strokes, kidney failure, peripheral vascular disease and blindness. Current means of treating diabetes represent only efforts to control the disease and at present there is no known cure. This experiment will lead to an understanding of the conditions required for maintenance and growth of human pancreas beta cells in vitro. Recently, progress has been made elsewhere in the synthesis of insulin from cell culture in vitro. However, the need for this experiment to aid in the understanding of the process is still relevant.

Null Lymphocyte Separation

Baylor College of Medicine
Dr. J. Twomey
Dr. D. Morrison, JSC
NAS9-15669 - $140K

Both experimental and clinical experience indicate the importance of the thymus to immunologic competence. Nude mice with congenital absence of thymic tissue undergo wasting, are susceptible to infections, and die prematurely. Humans with the DiGeorge syndrome, i.e., congenital absence of dysplasia of the thymus, also die from infection during infancy.

The role of the thymus may extend beyond immunologic maturation to the maintenance of immune competence throughout life. In mice effector, helper, and suppressor T-cell function declines following adult thymectomy. Curtailment of thymic hormone secretion with age-related thymic involution is probably a major factor in the waning of immunological vigor which occurs with advancing age. It is apparent that thymic hormone(s) has an important role in immunologic maturation, immunologic maintenance, and aging.

Thymic hormone assays have obvious clinical relevance. Such assays are necessary to correlate the status of thymic function with immunodeficiency.

The objective of this research is to make the bioassay for thymic hormone widely available. The bioassay requires the use of null lymphocyte indicator cells. The null lymphocyte cells currently used must be harvested from mice raised in a germ-free environment, which makes widespread use of the assay unfeasible. The objective is to provide an alternate source of these cells from conventional mice. The zero gravity of orbital flight may be useful in the development of this source.

The project includes: (1) separation of the null lymphocytes from the C3H mice by presently available techniques and verify the cells usefulness in the thymic hormone assay; (2) verification of the ability to freeze and store the C3H null lymphocytes without a significant effect on the sensitivity to the thymic hormone assay; (3) preliminary electrophoresis mobility studies on the null lymphocytes; (4) gradient density electrophoresis separation, and determination of mobilities of the fractions, and (5) thymic hormone assays on fractions from the gradient density electrophoresis.

The study should indicate whether or not electrophoresis in space may be required to isolate the null lymphocytes sensitive to low levels of thymic hormone.

Hormone Purification by Isoelectric Focusing in Space

University of Arizona
Dr. M. Bier
Dr. R. S. Snyder, MSFC
NAS8-32950 - $138K

The objective is to develop large scale purification of peptide-hormones by isoelectric focusing (IEF), taking advantage of the microgravity environment. Though aimed at peptide-hormones, the technology will be applicable also to other biomolecules, such as enzymes and isozymes, antibodies, vaccines, and at least some viruses.

Purification and separation methodology has given rise to a number of important industries involved in the preparation of purified biologicals, such as hormones, human blood proteins, and in more recent years, purified cell lines. The unique importance of the protein and peptide-hormones, such as human growth hormone, parathyroid hormone, oxytocin, vasopressin, is emphasized since they are all characterized by singular medical importance and inherently low supply. Hundreds of peptides are known to occur in the body, and most of these could have a medical application, if it were possible to isolate or synthesize them. The purification of both naturally occurring and synthetic peptide-hormones presents a number of difficult problems, and the successful development of space technology for their purification would have immediate widespread application.

IEF is the most powerful tool available for purification of proteins, but its output in ground-based instruments is limited. Microgravity is uniquely suited to overcome this limitation, and IEF can be scaled up. Two apparata will be developed, tested and compared: (1) continuous flow IEF and (2) recycling multi-membrane IEF. Neither concept is new and has been tried on the ground with limited throughputs because of gravitationally induced disturbances.

Countercurrent Distribution of Biologicals

University of Oregon Health Sciences Center
Dr. D. E. Brooks
Mr. B. H. Nerren, MSFC
NAS8-32353 - $50K

The objective is to evaluate the principle of field-driven phase separation in weightlessness and to determine the dependence of separation kinetics on electric field strength, droplet mobility and phase volume ratio. Countercurrent distribution of cells in two phase aqueous polymer systems is an extremely sensitive separation procedure but its use is limited to relatively small cells which do not sediment significantly in the time required for phase separation. These constraints are removed in space but an external driving force must be applied to induce the phases to separate since their density difference is inoperative. It has been shown that an applied electric field can supply the necessary driving force and biocompatible phase systems have been developed in which field-driven phase separation has been demonstrated.

Biosynthesis/Separations Laboratory

Johnson Space Center (JSC)
Contract Not Applicable

The Biosynthesis/Separations Laboratory supports the Materials Processing in Space studies on biosynthesis and cell separations for investigations into the production of high value pharmaceuticals which are very difficult or impossible to obtain on Earth with currently available technology.

The laboratory has both monolayer and suspension cell culture capabilities. Current research includes procedures for the obtaining of cell cultures, the growth and maintenance of continuous cell cultures, and the freezing and storage of cells. Procedures for growing cell cultures in suspension are being investigated. A continuous line of baby hamster kidney cells has been grown in suspension and the growth of cells on microcarriers is being pursued. A variety of beads were used as substrates for the attachment of cells. Good results have been obtained with baby hamster kidney cells; human kidney cells are being evaluated. Procedures for the analysis of biochemicals produced by cell cultures have been established. Fibrinolytic and colorimetric methods are being used routinely for the assay of urokinase. The production of urokinase in monolayers of human embryonic kidney cells has been demonstrated. Biochemical purification of secreted products on affinity columns is being developed. Procedures for the chromosome analysis of cell cultures (counting and karyotyping) have been established.

The Bioprocessing Laboratory supports other research involving cell separations and biomedical applications of electrophoresis. A laboratory model of a bioreactor for the continuous automated growth of cells in suspension will be operational and data collection of cells in suspension will be initiated. Studies will continue on the growth of human kidney cells in suspension. The requirements for a flight experiment will be established and the collection of ground-based data will be initiated. Ground-based technology for the growth of cell cultures in suspension will be constantly evaluated to optimize cell culture growth and product synthesis.

Tissue Culture in Space

Lyndon B. Johnson Space Center
Dr. B. J. Mieszkuc
Dr. R. S. Snyder, MSFC
Contract Not Applicable

The objectives of this investigation are to determine whether significant advantages in product production can be obtained from growing cells in the space environment and to fully characterize the growth of suspended tissue cultures in a low-g environment.

A space bioprocessing system will be designed and fabricated for the growth of tissue culture in low-g. This system will take advantage of the lack of sedimentation, buoyancy, and thermal convection in low-g. Ground studies will fully characterize a tissue culture system.

This study will determine whether there are any advantages to conducting tissue cultures/fermentations in the space environment. Conceivably, there could be a major breakthrough in the fermentation industry.

Space Biosynthesis System

Lehigh University
Dr. L. Nyiri
Dr. D. Morrison, JSC
NAS9-15619 - $145K

Large scale culture of mammalian cells to produce pharmaceutical products is dramatically compromised by sedimentation and inadequate transfer of gaseous oxygen. Experiments in low gravity revealed fluid behaviors which are different from those observed under terrestrial conditions; some of these appear to have advantages for cell culture. Also recent French experiments flown on the Soviet Salyout 6 mission indicate substantial increases in growth rate and cell size of eucaryotic cells grown under weightless conditions.

These studies are designed to (1) obtain data on the performance of cell culture vessel system elements and to define the biological oxidation process - the transfer of oxygen from gas to liquid and from liquid to oxidant and (2) determine the limits of ground-based technology using a preprototype reactor for studying enzymatic reactions and suspension cell cultures. The enzymatic conversion of glucose into gluconic acid is being used as a model to test the interactions between dissolved oxygen, glucose oxidase and glucose to determine the correlation between on-line operating sensors and the course of reaction in this particular bioreactor. Signals originating from the dissolved oxygen probe give information on the rate of reaction and the rate of oxygen transfer into the water under different experimental conditions. A computer model has been developed which can correlate theoretical and experimental data and which can be adopted for on-line, real-time monitoring of bioprocesses.

Current efforts include: replicate experiments to achieve high statistical accuracy of the sensor performance, the development and construction of a space bioreactor prototype, experimental testing of the prototype space bioreactor, and a project plan for the ground-based research to be completed before a flight experiment can be flown in a Shuttle/Spacelab mission. Follow on efforts are expected to include the design, construction, verification testing and flight test of a small space bioreactor to demonstrate the concepts and limitations of these new techniques using mammalian cells in culture which produce compounds of scientific or commercial importance.

Pituitary Cell Culture and Separation

Penn State University
Dr. W. Hymer
Dr. D. Morrison, JSC
NAS9-15566 - $170K

Human growth hormone (hGH) is in demand for the treatment of hypopituitary diseases such as dwarfism and the demand for hGH is far greater than the available supply. The problem could be solved by the *in vitro* production of hGH by cultures of human pituitary cells, however, the required techniques of cell culture and cell separation exceed present technology. This study will determine the limits of laboratory technology in the cultivation and purification of human somatotrophs and will assess whether gravity plays a role in establishing these limits.

Methodology has been developed to dissociate human pituitary tissue into single cell suspensions. A cell column perfusion system for studying the dynamics of hGH release and a highly sensitive radioimmunoassay for hGH has been established. No functional or morphological differences between cells derived from autopsy versus biopsy material have been detected thus far. Preliminary isoelectric focusing experiments with rat pituitary cells suggest that three cell populations focusing at pH's of 4.9, 5.25 and 5.6 are separable by this technique.

Investigations include: (1) an evaluation of different agents which augment the release of hGH from human pituitary tissue in a column perfusion system, (2) electrophoretic separation of the producing pituitary cells, and (3) comparisons with the use of density gradient electrophoresis for pituitary cell separation.

Kidney Cell Separations and Culture

Penn State University
Dr. P. Todd
Dr. D. Morrison, JSC
NAS9-15584 - $125K

During the ASTP Electrophoresis Technology Experiment (MA-011) the separation of urokinase producing human kidney cells was partially successful. The cells in fraction 15 produced 6-7 times more urokinase than ever before possible. However, the small number of cells returned from orbit limited the postflight analysis and precluded statistical analysis of the surface charge character of the cells producing higher levels of urokinase.

This effort includes: (1) development of optimum buffer systems, (2) viability tests, (3) ground-based research on electrophoretic mobilities, (4) development of standard cell culture methods and assays for urokinase, granulocyte conditioning factor, (5) study of a model kidney cell line to evaluate the best ground-based separations possible, (6) development of techniques to reduce the number of freeze-thaw cycles, (7) acquisition of the ground control data for comparison with cells returned from the electrophoretic separations carried out in microgravity, and (8) hardware verification and necessary experiment integration activities.

The effort will define the proper conditions for potential flight tests, determine the efficiency of this particular electrophoretic separation in space, and allow the pharmaceutical laboratories to determine the specific maintenance/culture characteristics of those kidney cells which produce products of commercial interest.

Carcinoembryonic Antigen (CEA)

Stanford Research Institute
Dr. J. Pincus
Dr. D. Morrison, JSC
NAS9-15463 - $94K

A glycoprotein antigen present on the surface of human colon carcinoma cells has been termed the CEA. It has been purified and a radioimmunoassay has been developed to detect it circulating in the blood. It is used by clinicians to measure CEA levels in colon cancer patients at different times after surgery as an aid in evaluating the patient's prognosis. The CEA is purified from hepatic metastases of colon carcinoma obtained at surgery. To obtain suitable amounts of CEA many individual tumor specimens must be pooled. This can be cumbersome, expensive, and each specimen may contain variable amounts of CEA. Therefore, pooled tumor tissue obtained from random surgical cases is far from satisfactory.

An alternative to using tumor tissue is to use colon tumor cells grown in tissue culture. Reproducible growth conditions can be obtained that allow for optimal production of CEA. However, cultured colon carcinoma cells must adhere to a surface to grow and growing large numbers of cells requires a large surface area. Current procedures and devices for growing cells on a large surface area in a small volume of media are not suitable for large scale commercial production.

This research is concerned with determining the possibility of growing a human colon carcinoma cell line (SK-CO-1) on microspheres to obtain increased yields of such cells, increased CEA production by the cells, and/or increased release of CEA into the culture medium. It is also concerned with identifying the limitations of using this system in unit gravity and how they may be overcome in a weightless environment.

The experiments performed to date have established that the doubling time and CEA content of SK-CO-1 cells grown on glass beads is similar to that of cells grown in monolayer culture. However, a larger amount of CEA is released into the medium when cells are grown on glass beads. Microspheres provide a means of obtaining a larger surface area per unit volume of culture medium, therefore, this approach to growing cells should be suitable for producing large amounts of CEA. However, further experiments are necessary to optimize the system and determine specific limitations of using this type of culture system in unit gravity.

Aggregation of Human Red Blood Cells (M77113)

University of Sidney, Australia
Dr. L. Dintenfass
Mr. J. Williams
Contract Not Applicable

The objectives of this investigation are: (1) to study aggregation of red blood cells in order to define the maximum size and morphology of aggregates of red cells under conditions of weightlessness, (2) to define the effect of various agents (fibrinogen, cholesterol, drugs, etc.) on the size of these aggregates in order to develop a new diagnostic test, and (3) to study viscosity of blood under high and low shear rate.

Anticoagulated blood samples at normal or lowered percentage of red blood cells and known content of cells, proteins, and lipids will be injected into the space contained by two flat parallel plates made of optical-quality glass. Blood preparations from normal donors and patients suffering from various diseases, e.g. multiple myeloma and other forms of cancer, will be studied. Measured amounts of agents such as fibrinogen, cholesterol, triglycerides, paraproteins, and snake venom preparations will be injected into the gap to observe two phenomena: (1) diffusion of the agent with corresponding increase/decrease of the size of aggregates, and (2) the effect of these agents as modified by the type of disease or type of ABO blood group.

The space environment offers the advantages of no fluid motion and weightlessness to determine the ultimate size of red cell aggregates. On Earth, gravity interfers with the measurement of aggregation because red blood cells, which are approximately 7 microns in diameter and have densities of approximately 1.09 g/cm^3, and their aggregates sediment rapidly.

Production of Large-Particle-Size Monodisperse Latexes in Microgravity (M77045)

Lehigh University
Dr. J. W. Vanderhoff
Dr. J. Micale
Dr. M. S. El-Asser
Mr. D. Kornfeld, MSFC
NAS8-32951 - $174K

The objective is the development of a process to produce monodisperse latex spheres in larger sizes than can be currently produced on the ground. Monodisperse latex particles have found a number of uses ranging from calibration standards for electron microscopy, light-scattering devices, and filters, to medical uses such as measuring pore sizes in membranes and sereological tests for a multitude of diseases. Monodisperse particles in the range from 2 to 20 microns a e not available because they are too large to be grown in production quantities by emulsion polymerization and they are too small to be sized by microsieving. Particles in this range are in demand for calibration of devices, particularly those used for counting blood cells and for various membrane sizing applications.

The difficulty in preparing particles larger than 2 microns on the ground is that the density of the particles changes during the process as the polymerization progresses. Since such particles are too large to be held in suspension by Brownian motion, they tend to "cream" during the early stages of growth and sediment during the later stages. This can be prevented by vigorous stirring or agitation, which tends to coagulate the mixture. These problems should be eliminated in a low-gravity environment since the buoyant forces are absent and the larger particles should stay in suspension more or less indefinitely.

Gravity Effects on Flame Inhibition

National Bureau of Standards
Dr. J. W. Hastie
Dr. Dressler, NASA Headquarters

This project has emphasized the development of new or improved techniques for the detailed molecular characterization of laboratory flames. This development has progressed very satisfactorily, with the following main achievements: (a) design and construction of a High Pressure Sampling Mass Spectrometer facility, suitable for transient species analysis in diffusion and particulate containing flames, (b) development of optical absorption spectroscopy of OH concentration and rotational temperature measurements in optically thick flames, (c) application of rotational Raman spectroscopy to flame temperature profile measurements, (d) Initiation of thermodynamic and chemical kinetic modeling predictions of flame temperatures, post flame species concentrations, and flame inhibition and promotion, (e) critical evaluation of new burner systems having potential utility as standard devices for interlaboratory comparison of flame diagnostic procedures, and (f) critical evaluation of various optically based flame temperature measurement techniques including OH-absorption, N_2-rotational Laser Raman, NaD-line reversal methods.

Contact and Coalescence of Viscous and Viscoelastic Bodies (SPAR Experiment 74-53)

Massachusetts Institute of Technology
Dr. D. R. Uhlmann
Ms. B. Facemire, MSFC
NAS8-31552 - $128K

The proposed investigation is intended to clarify and quantify the details of the physical processes whereby approximately spherical viscous and viscoelastic bodies contact and coalesce. The dependence of the flow field and geometry evolution upon parameters of recognized importance (including surface tension, materials rheology, particle geometry, applied stress, etc.) as well as upon parameters not previously recognized is to be determined.

The elucidation of the coalescence behavior (including initial contact) of viscous and viscoelastic fluids is important in materials processing, e.g. the thermal processing of phase separating glasses, the deformation processing of phase separated glass melts, the sintering of ceramic materials, the agglomeration of polymeric particles during melt processing, the solid processing of certain polymers (espcially PTFE), and the high pressure cold sintering of glassy polymers. The coalescence of molten droplets constitutes the mechanism for the formation of breccias; the present investigation would improve the accurcy of estimates of the thermal history of lunar breccias. An improved understanding of coalescence is important to the improved understanding of the rheology of suspensions, metal slushes and polymer solutions, and of certain biomedical problems involving agglomeration in multiphase flows. Coalescence is also a free boundary problem in hydrodynamics.

The necessity for gravity-free experiments arises from the fact that the principal interest in coalescence phenomena centers on situations in which gravitational effects are negligible, as with very small droplets; but under these conditions, it is almost impossible to study the flow fields of interest.

The acoustic levitation device will be used to position and maneuver drops of viscous fluids. These drops will contain tracer particles and will be made to coalesce in the field of view of cameras so that the flow fields can be observed. Initial contact, coalescence behavior and flow fields will be analyzed. This data will be used in addition to data from ground experiments to develop an understanding of the coalescence phenomenon.

The Interaction of Bubbles With Solidification Interfaces (SPAR Experiment 74-36)

Grumman Aerospace Corporation
Dr. J. M. Papazian
Dr. Ilmars Dalins, MSFC
NAS8-31529 - $194K

The objective is to observe the interaction of the solidification interface with bubbles and to observe the migration of bubbles in a thermal gradient.

In the absence of buoyant forces, the presence of bubbles may interfere with the processing of materials in low gravity. Also these interactions are of fundamental interest in the study of dispersive forces and of the effect of thermal Marangoni (surface tension) convection. This experiment is designed to investigate methods for control of bubbles in low-g processes.

An apparatus containing CBr_4 saturated with various gases was designed and constructed. Heaters hold the CBr_4 above the solidification temperature until the low-g portion of the flight, at which time one end is allowed to cool. Bubbles nucleate during the solidification process and are observed photographically.

In low "g", the solidification was dendritic which tended to trap the bubbles. Bubbles nucleating ahead of the front did not appear to move in the thermal gradient.

It has been suggested that trace impurities in the CBr_4 alter the surface tension dependence with temperature and prevent the thermal Maragoni convection from producing a net driving force in a thermal gradient. It is also possible that the bubbles reside on the walls of the container which prevents their motion.

Bubble Motion in a Thermal Gradient Under Zero Gravity Conditions (SPAR Experiment 77-13)

Clarkson College of Technology
Dr. W. R. Wilcox
Dr. H. D. Smith, Westinghouse
Ms. B. Facemire, MSFC
NAS8-33017 - $100K

The objective is to obtain data on bubble migration in a thermal gradient in the low gravity environment to refine existing theory for thermal fining of glasses in space and to analyze and compare observed motion with theory.

In the formation of glasses, bubbles become entrapped in the melting mixture. Removal of these bubbles, "fining," is necessary to render the glass transparent and to provide usable strength. Three basic methods are involved in fining: buoyant fining (the bubbles rise in the gravitational field), chemical fining (adding fining agents which reduce the amount of gas and/or increase the rate of diffusion of gas out of the glass), and thermal fining (migration of bubbles in an imposed thermal gradient to the glass surface). Proposals for the formation of improved or unique glasses in space will require a thorough understanding of fining in the absence of gravity. Migration of bubbles under a temperature gradient could indeed serve as a fining mechanism in zero-g for the production of bubble-free melts. Therefore, there is a need to have a quantitative understanding of the movement of bubbles in a thermal gradient.

Charged Drop Oscillations (SPAR Experiment 76-19)

University of Wyoming
Dr. C. P. R. Saunders, University of Manchester
Mr. W. Campbell, MSFC
NAS8-32486 - $27K

The objective is to determine the effect of electric charge on the vibrational frequency of water drops, to check Raliegh's theory for small drop oscillations; to determine the manner in which drops behave when they are charged sufficiently high for the surface energy to tend toward zero (surface charge counteracts surface tension).

Charged drop oscillations are important in rain cloud dynamics. Radar echos from clouds have been shown to contain a time varying component due to the vibration of raindrops, which are likely to contain varying amounts of charge. Drop oscillation frequency is dependent upon surface charge and drop radius. The intensity of the returned radar signal is a function of both drop size, and concentration, and so an independent assessment of drop size would permit cloud particle concentrations to be determined by radar.

In space the drop dynamics module provides a means of charging and acoustically levitating and exciting oscillations in a drop. This work cannot be done adequately on Earth. In labs, drop oscillation experiments involve suspending drops from strings or in a vertical air stream. Both of these techniques distort the drop shape and change oscillation frequency.

Liquid Mixing Experiments (SPAR Experiment 74-18)

Marshall Space Flight Center (MSFC)
Mr. C. F. Schafer
Dr. G. H. Fichtl
Mr. F. Reeves
Contract Not Applicable

The objective of these experiments was to characterize the sounding rocket environment with respect to residual accelerations which could lead to gravity-like body forces in liquid systems.

Metal samples were used in the form of cylinders with half of each sample being composed of pure indium and the other half being an In (80 wt%) - Pb (20 wt%) alloy (the samples were axially symmetric). These were enclosed in aluminum cartridges and placed in heater assemblies so that each sample was either parallel or perpendicular to a radius from the rocket payload axis. This insured that the density gradient in the samples was either parallel or perpendicular to the effective residual accelerations assuming that they would arise mainly due to residual rotation (spin) of the payload. The samples were melted and resolidified in low-g.

Flow was predicted using analysis which provided a Rayleigh number as a flow predictor, when the residual acceleration levels were on the order of 10^{-6}g. In addition, linear analysis indicated that perturbation growth times for these cases were on the order of the experiment duration.

Residual low-level accelerations (10^{-6} to 10^{-5}g) exist on the SPAR payload. These can lead to flow in liquids containing large density gradients in the few minutes of experiment time. Relatively simple analyses can be performed which can yield order of magnitude estimates of these flow effects. Also proper orientation of samples can minimize these effects.

Surface Tensions Variations With Temperature and Impurities

National Bureau of Standards
Dr. S. C. Hardy
Dr. S. R. Coriell
Dr. R. Dressler, NASA Headquarters
H27954B - NA

We have measured the surface tension of gallium in ultrapure helium atmospheres at room temperature using the pendant drop technique and have observed a time dependence which is more complex than that found in previous work. We have tentatively interpreted the observed time variation of the surface tension in terms of a bulk phase impurity which diffuses to the surface and desorbs. These preliminary investigations indicate the surface tension of pure gallium is near 800 mJ/m^2, a value about 10% higher than the equilibrium values previously reported.

Surface Tension Driven Convection Phenomena (MPS77D120)

Case Western Reserve University
Prof. S. Ostrach
Prof. J. Mann, Jr.
Mr. F. Reeves, MSFC
NAS8-33015 - $101K

This experiment is directed towards the study of the behavior of fluids in a reduced-gravity environment where buoyancy induced flows and sedimentation phenomena will be diminished. These effects offer attractive benefits of materials processing and crystal growth in space although there are also associated undesirable effects. Perhaps the most unique aspect of the reduced-gravity environment is that it offers the possibility of containerless processing of materials so that container reactions and interactions can be avoided. Liquids and molten metals with free surfaces are inherent to all containerless processes. The shape of the bulk fluid and its stability to changes must be predictable and the details of the surface flows and transport processes within the fluids over ranges of conditions must also be known.

Liquids with free surfaces behave much differently in a micro-gravity; an example is the driving force for fluid motion due to non-isothermal temperature induced surface tension gradients. Surface tension gradients due to concentration differences can also generate fluid motions. On Earth fluid motion forces due to buoyancy effects are dominant over the surface tension effects, whereas in space these surface tension effects predominate.

The research program is directed to measure and analyze non-isothermal surface tension driven flows and the associated transport phenomena, surface and bulk flows generated by surface tension gradients, excess concentration gradients and their coupling. Molecular theories of interfacial transport will also be studied to provide fresh insight into the transport occurring within molecular dimensions of the surface.

Space flight experiments are planned to be performed utilizing the Analytical Float Zone Crystal Growth facility. Materials to be studied in detail include transparent fluids, e.g. water, CBr, and the long chain hydrocarbons as well as for solutions of surfactants and water. In addition, pure metals such as gallium and mercury will be utilized in the experiments.

Nucleation and Growth of Immiscibles

Rensselaer Polytechnic Institute
Prof. S. Ross
Dr. G. Nishioka, MSFC
Mr. W. Witherow, MSFC
NAS8-33074 - $59K

The purpose of this experiment is to study mixed immiscible liquids in space. The specific objectives are: (1) to observe the nucleation of liquid drops in the immiscible region of a phase diagram, as influenced by the interfacial tension, the wettability of the nucleated particle by the matrix, and the degree of supersaturation or supercooling, (2) to measure the rate of coalescence of the nuclei due to frequency of collision, in which the constant of the Smoluchowski equation can be determined for the first time under conditions in which convection currents and buoyancy are eliminated, (3) to observe Ostwald ripening of the drops under conditions where molecular diffusion plays a significant part, and (4) to correlate the observed results with the information of the phase diagrams, with the purpose of generalizing the behavior observed.

By performing this experiment in space, convection currents and gravitational settling or creaming are eliminated. Thus, an immiscible system that is normally unstable and would separate quickly in one-g will be stable in space and allow measurements of the various parameters to be made. Using transparent fluids, the behavior of immiscible systems can be generalized and the theory obtained applied to space processing of glasses, ceramics, and metal alloys.

The experiment requires a method to instantaneously determine particle sizes down to a few microns everywhere in the test volume. A holographic system has been developed that will record and allow retrieval of the data for analysis. The holographic system is being tested in regard to making quantitative measurements from a hologram.

Uniform Dispersions by Crystallization Processing (SPAR Experiment 74-15)

Massachusetts Institute of Technology (MIT)
Dr. D. R. Uhlmann, MIT
Dr. B. Joiner, MIT
Dr. R. J. Naumann, MSFC
NAS8-31350 - $399K

The objective is to develop improved understanding of the interactions between second-phase particles and an advancing crystal-liquid interface and to develop a criteria for rejection/incorporation in terms of solidification rates for various particle characteristics.

One of the attractive prospects for materials processing in weightlessness is the possibility of solidifying melts with uniform dispersions of second phase particles. Such composites may have applications as solid state electrolytes with enhanced ionic conductions, oxide dispersion hardened alloys for use as turbine blades, etc. It is known that dispersion forces between the molecules in the melt tend to push particles ahead of the solidification front whereas drag forces tend to retard their motion. A critical growth velocity exists below which particles are pushed ahead of the solidification front and above which they are incorporated. A theory has been developed to predict the critical velocity as a function of particle properties, such as size, density, thermal conductivity, etc. The absences of gravitational sedimentation allows an experimental check to be made on the theory without extraneous frictional forces from the particles moving along the wall of the container.

The experiment consists of seven cuvettes containing camphor (which has an entropy of fusion similar to metals) and various sizes and composition of particles. During the low-g coast these cuvettes are melted back and directionally resolidified. One cuvette is photographed during the process to establish the growth rate.

Fluid Dynamics of Crystallization From Vapors

University of Utah
Dr. F. Rosenberger
Dr. A. Fripp, LaRC
Grant NASG-1534 - $101K

The experimental and theoretical work performed will help identify the accuracy to which physical properties need to be known before fluid flow characteristics of crystal systems can be predicted. With this understanding, the techniques developed will be applied to more complex systems.

Numerical methods are being applied to the coupled Navier-Stokes and mass transport equations for both physical and chemical transport in closed ampoules. Three-dimensional transport in a gravity field will be modeled.

Initially only materials that are easily handled and characterized (e.g., I and HgI_2) will be used to verify theoretical calculations. A laser-doppler anemometer will be utilized to measure fluid flow velocities. This unique procedure will allow flow measurements to be made without the perturbations on the fluid boundary conditions as caused by standard temperature probes.

Fluid Mechanics of Continuous Flow Electrophoresis

Princeton University
Dr. D. A. Saville
Dr. R. S. Snyder, MSFC
NAS8-32614 - $72K

The objective of this investigation is to furnish a basis for understanding the hydrodynamic characteristics of a rectangular separation chamber and their effects on the separation process. Particular emphasis is placed on the role buoyancy plays in establishing the basic flow and affecting its stability.

Hydrodynamics plays varied roles in the continuous flow electrophoresis of small particles, although one of the complicating factors is the role of buoyancy forces which can destabilize the flow or establish an unfavorable flow. To circumvent such problems it has been suggested that the apparatus be operated in a microgravity environment where, due to the reduced buoyancy forces, the chamber could be made larger and field strength increased. To achieve the required basic understanding we will:

a. Develop models to describe the flow and temperature fields;

b. Investigate the hydrodynamic stability of the flow field; and

c. Develop a model to predict electrophoretic separation efficiency.

Electrophoresis models which ignore the effects of temperature on transport properties can be misleading. In wide-gap machines operating in a 1-g environment, the steady-state axial velocity profile is unsatisfactory at modest field strengths insofar as electrophoretic separations are concerned when the device is operated with the volumetric flowrate fixed. Ostrach identified a buoyancy-driven feature which made downflow operation unsatisfactory. Upflow, which was once suggested as a means of overcoming the difficulty, turns out to be only marginally better for the cases studied. The difficulty arises from recirculating eddys which restrict the area available for separation. A microgravity environment suppresses and eliminates secondary flow of this sort.

Automation of Holographic System for Fluid Analysis

TAI, Inc.
Dr. R. Kurtz
Mr. J. R. Williams, MSFC
NAS8-33141 - $121K

The objective of this task is to develop a holographic optical Schlieren system breadboard and/or prototype device for measurement of density gradients in fluids, and to integrate this system with two experimental fluid test cells. The test cells provide an isothermal and a thermal-gradient environment for the fluid under study with appropriate windows for observation. Another part of the task is the automation of the sequencing of the system with appropriate operator controls. This system is useful in analysis of convection associated with crystal growth and solidification.

Space Vacuum Research Facility Modeling SVRF

Langley Research Center (LaRC)
Dr. L. Melfi
Mr. M. Page, MSFC
Contract Not Applicable

The objectives are to analyze a set of rarefied gas flow field configurations for a range of orbit heights, outgassing rates, Shuttle effluents, and engine firings and to determine from these data the density within the SVRF shield due to each gas source. These data will define a range of orbit parameters and Shuttle operation modes which will assure that the density within the molecular shield is sufficiently low.

The direct simulation Monte Carlo technique is being used to analyze the properties of the flow in the vicinity of the Shuttle. The flow field properties of interest are the density distribution within the flow field of the molecules from each source, the back scattered flux density distribution within the flow field due to each source and the column density associated with each source. These parameters will be determined for each flow field configuration and the results will be used to define the Shuttle attitude, orbit height range, molecular shield location, orientation and separation from the Shuttle which yield optimum shield performance.

Degassing Technology for Space Vacuum Facility

Marshall Space Flight Center (MSFC)
Dr. Ilmars Dalins
Mr. K. Taylor
Contract Not Applicable

The objective is to determine the most economical methods for obtaining outgassing rates of 10^5 molecules/cm^2/sec from candidate materials for the proposed space vacuum facility.

To approach the theoretical limit (10^{-14} Torr) behind an orbiting wake shield in a space vacuum facility, it is necessary to reduce the outgassing load from the structure to 10^5 molecule/cm^2/sec. This is possible with a high temperature insitu bakeout but such a procedure requires either a power consuming bakeout, or the launch of a pre-evacuated chamber that has been baked-out, on the ground. Both are very costly procedures in terms of weight and power. It may be possible to perform a pre-bake at high vacuum to eliminate the light gases such as H_2 and He that are trapped deep in the lattice and then allow the material to be re-exposed to ambient air. If the adsorbed molecules do not penetrate deeply into the structure, they can be removed by a short exposure to space vacuum. This would greatly simplify the process of designing and launching a space vacuum facility.

We are measuring the outgassing rates of various materials after extended insitu vacuum bakes at 950°C as a function of handling procedures such as re-exposure to various atmospheres. Argon ion sputtering will be evaluated as a cleaning technique. Various barriers and coatings will be evaluated to reduce the gas permeation. Depth profiling by Auger Electron Spectroscopy (AES) will be used to study these matters. Work on 304 stainless steel has shown that insitu vacuum bake at 950°C for 8 hours in 10^{-6} Torr reduced the outgassing rate from 3×10^8 to 2.3×10^6 molecules/cm^2/sec. This removes the hydrogen from the bulk of the material, which does not reenter from the ambient atmosphere unless the material is heated to elevated temperatures for an extended period.

Analysis of Degassing Techniques

McDonnell Douglas Astronautics Company - West
Mr. B. C. Moore
Mr. W. H. Stafford, MSFC
NAS8-33155 - $100K

The objectives of this study are to support Systems Engineering Studies of the Space Vacuum Research Facility by supplying and analyzing key ultra-high vacuum technology degassing procedures, and by providing data for engineering trade studies. We will develop and analyze methods of fabrication and degassing of the Research Facility and document this data in parametric form. The study will select materials of construction, define contaminants and their sources, define the lowest level of outgassing that can reasonably be attainable in orbit and identify any unique processing of materials that may be required.

Technology Demonstration Measurement for the Molecular Wake Shield

University of Texas at Dallas
Dr. J. Hoffman
Dr. W. Oran, MSFC
NAS8-32689 - $30K

One of the precursor activities to any space vacuum research facility or experiment would be the measurement of the atomic/molecular species present behind a spacecraft wake shield. We are studying modifying/refurbishing/calibrating a laboratory version of both the lunar mass spectrometer (which has measured pressures down to $\sim 10^{-13}$ torr) and other ion mass spectrometers to measure the rarefied shield environment. The result will indicate the technical feasibility of using ion mass spectrometers to measure gas density in the shield.

Space Vacuum Research Facility, User Requirements

Jet Propulsion Laboratory (JPL)
Dr. M. Saffren
Mr. M. Page, MSFC
NASw-100 - $282K

The objectives are to identify ongoing research impacted by the program; to establish a procedure for informing the vacuum research community about space opportunities; to identify environmental and operational information concerning ultra-high vacuum space facilities; to identify the evolution of logical sequence of experiments to be performed prior to flight of an "all-up" facility needed to obtain scientific and operational information for the design of the Space Vacuum Research Facility; and to identify the evolution of an ultra-high vacuum facility required to carry out those experiments.

Contacts will be made with the science community to solicit help in effecting a series of miniconferences of broadening participation, culminating in a full symposium. The program will lead to an Applications Notice (AN). The conferences will explore the desirability of an ultra-high vacuum facility in space and the classes of experiments that could be performed in it.

Electrotransport of Solutes in Refactory Metals

Iowa State University
Mr. F. A. Schmidt
Dr. O. N. Carlson
Dr. W. Oran, MSFC
H34328B - $30K

The objective of this investigation is to study the electrotransport behavior of metallic solutes in refractory metals. Metallic solutes migrate with the flow of electrons in thorium at significant velocities, unexpected from our present knowledge of transport phenomenon. Using the vacuum in space and low gravity, specific experiments will be made in an effort to better understand the behavior of these solutes so that maximum efficiency of the electrotransport refining process can be achieved. Electrotransport may be a good method of removing not only interstitial impurities but also metallic solutes which cannot be removed by any other refining process.

Preparation of Ultra-Pure Metals

University of California, Los Angeles
Dr. R. F. Bunshah
Dr. W. Oran, MSFC
NAS8-33115 - $67K

The objective is to prepare ultra high purity metals by space processing using an Ultra High Vacuum/Micro-g Space Processing Facility. The highest purity metals produced to date are quite impure (10 to 100 ppm atomic). It is expected that space purified metals will be purer by several orders of magnitude (about 1 ppb atomic impurity content). The experimental method will be a two-step process, vacuum melting followed by evaporation/condensation on a clean heated substrate to produce full density metal condensate. The rate of deposition will be very high, thus lending itself to short experimental times, compared to other purification techniques. Space processing vacuum facility furnishes a unique environment, i.e., very low pressures (10^{-13} to 10^{-14} torr), very high thruputs, i.e., almost complete removal of gases evolved from the sample environment and micro-g. Such an environment is not possible on Earth. Since the ambient gas impinging on the specimen is the principal contaminant, the importance of low gas pressures and high processing rate is evident for the preparation of samples of the highest purity. Initial candidates for purification studies will be selected from Beryllium, Chromium or Vanadium.

Efficient Solar Cells Made by Processing in Space

Iowa State University
Mr. F. A. Schmidt
Mr. H. R. Shanks
Dr. A. J. Bevolo
Mr. J. R. Williams, MSFC
H34328B - $154K

The objective is the development of efficient Schottky barrier solar cells (SBSC) utilizing the unique capabilities of the space vacuum. We will evaporate silicon onto various heated metallic substrates so that the Schottky barrier height, the polycrystalline grain size of the silicon layer, and the solar cell efficiency can be measured as a function of residual gas pressure. These studies will be performed in a low pressure space simulation chamber equipped with extensive in situ fabrication and analytical capabilities. These capabilities will be used to monitor those factors expected to influence the efficiency of the device, such as substrate work function and temperature, silicon layer thickness and deposition rate, and surface contaminants. After Phase I, that is the preparation of an efficient SBSC, Phase II will commence and have as its objective the design of a preprototype fabrication system that incorporates those features expected for the MSF.

Results expected from the study are the preparation of a high efficiency SBSC to demonstrate that such a device can be effectively produced by space processing. Space Processing of large arrays of photovoltaic materials could greatly aid in the solution of our nation's energy problems.

Ultravacuum Vapor Epitaxial Growth of Silicon

General Electric Company
Dr. C. A. Neugebauer
Dr. R. T. Frost
Mr. J. R. Williams, MSFC
NAS8-33121 - $100K

The objective of this investigation is to study heteroepitaxial growth of silicon monocrystalline or polycrystalline films which have large grain structures. Various substrates will be used; sapphire, tungsten or tantalum.

Ultra low pressures will enable slow film deposition times long enough to form possibly single crystalline films on certain substrates. The requirement for keeping the substrate at elevated temperatures for long periods of time at extremely low pressures in order to avoid contamination and maintain slow deposition rates presents difficulties with ground based systems.

Ultra-High-Vacuum Semiconductor Thin-Film Technology

Jet Propulsion Laboratory (JPL)
Dr. F. J. Grunthaner
Dr. B. F. Lewis
Dr. J. Maserjian
Mr. J. R. Williams, MSFC
NASw-100 - $80K

The objective of this program is to answer basic questions on the surface interactions of depositing semiconductor films in ultra-high vacuum (UHV), establish the process requirements and limitations of this technology and explore experimental opportunities in a space vacuum facility. The goal is to demonstrate that high-quality semiconductor films can be deposited on low-cost substrates under controlled surface and UHV conditions, focusing on the deposition of thin-film silicon solar cells on metal substrates.

The investigation emphasizes surface chemical interactions during the nucleation and growth stages of the process. Surface interactions are monitored, characterized and controlled with extensive surface analytical instrumentation, which include high-resolution x-ray, electron- and ion-stimulated spectroscopies to establish the critical stages of the process.

Optical Degradation of Thin Films

Naval Research Laboratory
Dr. W. R. Hunter
Dr. W. A. Oran, MSFC
H35835B - $7K

One of the precursor activities to any space vacuum research facility/experiment will be the measurement of atomic/molecular species. We are investigating use of trace quantities of oxygen, which is predominant behind a spacecraft wake shield and is known to be detrimental to many potential experiments. The measurement will involve an in-situ deposition of Aluminum behind the shield. The time history of the degradation of the reflectance will be recorded at several wavelengths. This effort will provide a technical assessment of the process for measurement of atomic oxygen.