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SUMMARIES OF EARLY MATERIALS PROCESSING IN SPACE EXPERIMENTS

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George C. Marshall Space Flight Center
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TABLE OF CONTENTS

		Page
I.	INTRODUCTION	1
II.	DROP TOWER EXPERIMENTS	3
	Solidification of Liquid Miscibility Gap Alloys Under Free Fall (Drs. L. L. Lacy and G. Otto)	4
	Surface Tension-Driven Flow in a Weightless Fluid (Dr. S. Ostrach)	5
III.	PRE-SKYLAB EXPERIMENTS	7
	Heat Flow and Convection Experiment (T. C. Bannister and Dr. P. G. Grodzka)	8
	Composite Casting Experiment (I. C. Yates)	10
	Electrophoresis Demonstration (Dr. R. S. Snyder)	11
IV.	SKYLAB EXPERIMENTS	13
	Zero-Gravity Flammability (J. H. Kimzey)	14
	Metals Melting (E. C. McKannan and R. M. Poorman)	16
	Exothermic Brazing (J. R. Williams)	17
	Sphere Forming (Dr. J. Larson)	18
	Vapor Growth of IV-VI Compounds (Dr. H. Wiedemeier, F. C. Klaessign, S. J. Wey, and E. A. Irene)	20
	Immiscible Alloy Compositions (J. L. Reger)	22
	Radioactive Tracer Diffusion (Dr. O. A. Ukanwa)	24
	Influence of Gravity-Free Solidification on Microsegregation (Dr. J. T. Yue and Fred W. Voltmer)	26

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TABLE OF CONTENTS (Continued)

		Page
	Seeded, Containerless Solidification of Indium Antimonide (Dr. H. U. Walter)	28
	Whisker Reinforced Composites (Drs. T. Kawada and S. Takahashi)	30
	Steady State Growth and Segregation Under Zero Gravity: InSb (Prof. A. E. Witt, Prof. H. C. Gatos, M. Lichtensteiger, M. C. Levine, and C. J. Herman)	31
	Directional Solidification of InSb-GaSb Alloys (Drs. W. R. Wilcox, J. F. Yee, K. Sarma, M. Lin, and Sanghamitra Sen)	33
	Metal and Halide Eutectics (Dr. A. S. Yue and J. G. Yu)	35
	Copper-Aluminum Eutectic (E. A. Hasemeyer)	36
V.	SKYLAB DEMONSTRATION EXPERIMENTS	37
	Ice Melting (Drs. L. L. Lacy and G. Otto)	38
	Diffusion in Liquids (Barbara Facemire)	39
	Liquid Floating Zone (Dr. J. R. Carruthers)	40
	Immiscible Liquids (Drs. L. L. Lacy and G. Otto)	42
	Liquid Films (W. Darbro)	43
	Rochelle Salt Growth (Dr. I. Miyagawa)	44
	Deposition of Silver Crystals (Dr. P. G. Grodzka)	45
	Fluid Mechanics (O. Vaughan, B. Facemire, and Dr. S. Bourgeois)	46
	Charged Particle Mobility (Dr. D. M. Bier)	48
VI.	APOLLO-SOYUZ EXPERIMENTS	49
	Electrophoresis Technology (Drs. R. E. Allen and G. H. Barlow)	50

TABLE OF CONTENTS (Concluded)

		Page
	Electrophoresis (Dr. K. Hannig)	52
	Crystal Growth (Dr. M. D. Lind)	54
	Surface-Tension-Induced Convection (Drs. R. E. Reed, W. Uelhoff and H. L. Adair)	56
	Monotectic and Syntectic Alloys, (Drs. L. L. Lacy and C. Y. Ang)	58
	Interface Marking in Crystals (Prof. H. C. Gatos, Prof. A. F. Witt, M. Lichtensteiger, and C. J. Herman)	60
	Zero-G Processing of Magnets (Dr. D. J. Larson)	62
	Crystal Growth from the Vapor Phase (Dr. H. Wiedemeier, H. Sadeek, F. C. Klaessing, M. Norek, and R. Santandrea)	64
	Halide Eutectic Growth (Dr. A. S. Yue, C. W. Yeh, and B. K. Yue)	66
	Multiple Materials Melting (Metals) (L. I. Ivanov, V. N. Kubasov, V. N. Pimenov, K. P. Gurov, E. V. Demina, Yu. S.Malkov, and V. M. Lazarenko)	67
	Germanium-Silicon Solid Solutions (V. S. Zemskov, V. N. Kubasov, I. N. Belokurova, A. N. Titkov, I. L. Shulpina, V. I. Safarov, and N. B. Guseva)	69
VII.	APOLLO-SOYUZ DEMONSTRATION EXPERIMENTS	71
	Chemical Foams (Dr. P. G. Grodzka)	72
	Liquid Spreading (Dr. S. Bourgeois)	73
	Capillary Wicking (A. F. Whitaker)	74
VIII.	SUMMARY OF WHAT WAS LEARNED	75

I. INTRODUCTION

As the Materials Processing in Space program is approaching a degree of maturity in the Shuttle/Spacelab era, it is instructive to reflect back on earlier experiments that contributed to our present state of knowledge. This is especially valuable for new investigators developing space experiments for the first time. Many times the questions that arise in the development of various types of experiments have already been addressed, allowing the experimenter to avoid some of the pitfalls of his predecessors. Even experienced investigators sometimes tend to forget some of the lessons taught by the early experiments.

The purpose of this document is to provide a summary of these early experiments conducted on Apollo, Skylab, and the Apollo-Soyuz Test Project (ASTP). These summaries are given in terms of the objective, approach, and results together with references to the publications resulting from the experiment to which the reader may refer for more details than are provided here. In reporting the results no attempt has been made to do an in-depth critical analysis of the experiment; however, the results stated herein are put in context with our current understanding of the experiments and reflect the consensus of the scientific community active in the program.

This document deals only with the experiments through the Apollo-Soyuz program. Since then, additional experiments have been carried out on various sounding rocket programs such as the Space Processing Applications Rocket (SPAR). These are summarized in "Description of Space Processing Applications Rocket (SPAR) Experiments," NASA TM-78217, January 1979.

II. DROP TOWER EXPERIMENTS

The following experiments were conducted in drop towers at the Marshall Space Flight Center (MSFC) and the Lewis Research Center (LeRC) as precursory to the development of spaceflight experiments. Although many tests were performed in the drop towers that were essential to developing experiment apparatus or verifying experiment concepts, the experiments listed have yielded publishable scientific results.

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SOLIDIFICATION OF LIQUID MISCIBILITY GAP ALLOYS UNDER FREE FALL

Drop Tower Experiment

Investigators: Dr. L. L. Lacy, MSFC
Dr. G. Otto, University of Alabama in Huntsville

The objective of this investigation was to investigate the solidification of alloy systems that exhibit a liquid phase immiscibility gap, with the hope of producing fine dispersions. Such dispersion alloys can be expected to possess unique electrical and mechanical properties. They cannot be prepared from the melt terrestrially because the immiscible phase rapidly separates because of the density differences as the melt is cooled below the consolute temperature.

Samples of Ga-50 At % Bi were monogenized above the consolute temperature and rapidly quenched during free fall in the 90-m MSFC drop tower. The experiment apparatus was protected from aerodynamic forces by a drag shield which was initially accelerated by small thrusters. Various quench rates were used to examine the effect of quench rate on microstructure. Typical quench rates were 5×10^3 K/sec.

Fine uniform dispersions of Ga-rich particles in a Bi matrix were obtained in the free-fall solidification. Sizes ranged from 1 to 5 microns, depending on the quench rates. Control samples solidified under normal gravity exhibit massive separation between the Bi and Ga. The resistivity of the sample as a function of temperature exhibits a very unique behavior, quite different from the pure substances and from the 1-g control sample. This is attributed to the unique microstructure obtained by low-g solidification.

References

Lacy, L. L. and Otto, G. H.: Electrical Resistivity in Ga-Bi Solidified in Free Fall. AIAA Journal, Vol. 13, February 1975, pp. 219-220.

Lacy, L. L. and Otto, G. H.: The Electrical Properties of Zero Gravity Processed Immiscibles. AIAA Paper 74-208 (74A18853), February 1, 1974.

Otto, G. H.: Immiscible Alloys-Ground Based Studies. NASA CR-120796, June 1974.

SURFACE TENSION-DRIVEN FLOW IN A WEIGHTLESS FLUID

Drop Tower Experiment

Principal Investigator: Dr. S. Ostrach
Case Western Reserve University

The object of this investigation was to obtain experimental data on surface-driven convection in the absence of gravity-driven flows.

A teflon-lined cylindrical plexiglass dish (10 dia × 3.16 cm high) was filled with distilled water. A few particles of methylene blue dye were sprinkled on the water. As the larger particles sank, the dye trails acted as flow indicators. A temperature gradient was imposed on the surface by a radiant heat source as the apparatus was dropped in the 500-ft drop tower at LeRC. The resulting surface-tension driven flow was recorded by two 10-mm high-speed Milikon cameras.

Flow was observed within microseconds after the heater started to heat the surface. The surface was pulled away from the heater region by the surface tension gradient at the rate of approximately 3 cm/sec. No flow was observed beyond 3 cm from the heater. The return flow penetrated the bulk of the fluid with a velocity on the order of 1.0 mm/sec.

The control experiment at normal gravity exhibited reduced surface tension-driven velocities and a much larger boundary layer near the free surface. This results from the coupling between the surface tension- and gravity-driven flows. This illustrates that surface tension-driven flows can induce significant convection in a low-g environment.

References

Ostrach, S.: Convection Phenomena at Reduced Gravity. Material Science in Space, ESA SP-114, European Space Agency, September 1976, pp. 41-56.

III. PRE-SKYLAB EXPERIMENTS

The experiments summarized in this section mark the beginning of the Materials Processing in Space Program. These experiments were performed in an ad hoc manner on available flight opportunities. The long trans-Earth coast of the Apollo spacecraft returning from the Moon provided such opportunities.

The experiments were of necessity very restricted. There was very limited room in the Apollo Command Module, and the experiments had to fit into a small suitcase — like a carry-on package. The experiments had to be self-contained and could not be integrated into the spacecraft power and data system. The primary purpose of the experiments was to test concepts, demonstrate techniques, and to gain experience in carrying out space processing experiments.

Despite these limitations, several rather interesting experiments were carried out which provided valuable insight into fluid behavior and solidification phenomena in a spacecraft environment.

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HEAT FLOW AND CONVECTION EXPERIMENT

Apollo Demonstration Experiment

Principal Investigators: T. C. Bannister, MSFC
Dr. P. G. Grodzka, Lockheed

The heat flow and convection experiment had several objectives: (1) to determine to what extent contributions from residual vehicle accelerations and nongravity-driven convection affect heat transfer; (2) to dramatize the fact that convective flow can occur in the absence of gravity; and (3) to study the onset of unstable surface tension-driven convection in the absence of buoyancy-driven convection.

A special experiment package was developed that contained a thin flat convection cell with a uniformly heated bottom, a cylindrical gas conduction cell with a stub heater in the center, and a cylindrical tube liquid cell with a heater at one end. Liquid crystals indicated the temperature by color changes that were recorded photographically. This package was carried on Apollo 14 and 17.

The convection cell contained a 2-mm layer of Krytox oil. The bottom was uniformly heated, and the top surface was free. Difficulty in maintaining a uniform thickness was experienced on Apollo 14 because the surface tension caused the oil to creep up the sides of the cell. The cell was modified on the Apollo 17 flight, and a moderately uniform layer was maintained. Bernard cells were observed due to the fact that the warmer fluid beneath the surface would have a lower surface energy if it displaced the fluid at the surface. The onset of the stability was analyzed and compared to the stability under unit gravity.

The heat flow experiment on Apollo 14 was conducted with CO_2 in the gas cell and a sugar solution in the liquid cell. The heat flow was 10 to 30 percent larger than predicted from pure conductive heat flow. This was attributed to crew-induced disturbances. On Apollo 17 Ar was substituted for the CO_2 in the gas cell, and Krytox was substituted for the sugar solution. Marker particles were added for flow visualization. Considerably more care was exercised in preventing crew disturbances. The heat flow this time agreed with predictions based on pure conduction. No evidence of flow was observed in the fluid with marker particles.

References

Bannister, T. C., et al.: Apollo 17 Heat Flow and Convection Experiments Final Data Analyses Results. NASA TM X-64772, July 1973.

Grodzka, P. G. and Bannister, T. C.: Heat Flow and Convection Experiments Aboard Apollo 17. Science, Vol. 187, January 1975, pp. 165-167.

Bannister, T. C., Facemire, B. R., and Grodzka, P. G.: Heat Flow and Convection Demonstration (Apollo 14). NASA TM X-64735, March 1973.

Grodzka, P. G. and Bannister, T. C.: Heat Flow and Convection Demonstration Experiments Aboard Apollo 14. Science, Vol. 176, May 1972, pp. 506-508.

COMPOSITE CASTING EXPERIMENT

Apollo Demonstration Experiment

Principal Investigator: I. C. Yates, MSFC

The objective of this experiment was to investigate the possibility of forming various composite materials with large density differences from the melt. The composites cannot be formed from the melt on Earth because of density segregation.

Two types of materials were investigated. The first group contained dispersed components such as fibers, particles, and gases mixed into a molten matrix of In-Bi and solidified. The second group consisted of immiscible mixtures of sodium acetate and paraffin. These samples were processed in a special low-temperature furnace during the translunar coast of Apollo 14.

The space processed samples did not exhibit the separation of phases experienced by the ground control samples. However, the distribution of the dispersed phase was not as uniform as expected. The paraffin-sodium acetate mixture formed a fairly uniform in situ composite.

References

Yates, I. C., Jr.: Apollo 14 Composite Casting Demonstration Final Report. NASA TM X-64641, October 1971.

Apollo 14 Composite Casting Demonstration. NASA CR-61369, Final Report, Arthur D. Little, Inc., Cambridge, Massachusetts, August 20, 1971.

Stuerer, W. H. and Kaye, S.: Preparation and Evaluation of Apollo 14 Composite Experiments. NASA CR-61368, August 1971, pp. 3-1 to 3-8.

ELECTROPHORESIS DEMONSTRATION

Apollo Demonstration Experiment

Principal Investigator: Dr. R. S. Snyder, MSFC

The early electrophoresis experiments were intended to test the concept of using low-g to prevent unwanted convective flows from Joule heating in static-column, free-flow electrophoresis and to identify problems that may be encountered with bubble formation, nongravity-driven flows, and other possible problems that might be encountered in space electrophoresis.

Special small self-contained electrophoretic separators were developed as carry-on experiments for Apollo 14 and 17. The Apollo 14 apparatus had three columns containing a mixture of red and blue dyes, human hemoglobin, and DNA extracted from salmon sperm. An improved version was flown on Apollo 17. It had two columns containing mixtures of 0.2 and 0.8 micron polystyrene latex particles.

The Apollo 14 experiment suffered from poor photographic data. It was confirmed that the red and blue dye was transported along the tube, but the shape of the sample band was severely distorted by electroosmotic flow. The Apollo 17 experiment achieved a slight separation between the latex spheres, but again the sample bands were severely distorted by electroosmotic flows. These experiments provided the impetus to develop special coatings to lower the zeta potential and eliminate such flows in future experiments.

References

Snyder, R. S.: Electrophoretic Demonstrations on Apollo 16. NASA TM X-64724, November 1972.

Snyder, R. S., et al.: Free Flow Particle Electrophoresis on Apollo 16. Separation and Purification Methods, Vol. 2, 1973, pp. 259-282.

McKannan, E. C., Krupnik, A. C., Griffin, R. N., and McCreight, L. R.: Electrophoresis Separation in Space — Apollo 14. NASA TM X-64611, August 1971.

IV. SKYLAB EXPERIMENTS

Skylab provided the first opportunity to carry out dedicated spaceflight experiments in materials processing. A total of five experiments were conducted in crystal growth and six experiments were conducted in metallurgical processes. The objectives, approach, and results are summarized here.

The state of development of space processing apparatus was rather primitive at the time. The furnace, for example, was limited to 1000°C and had only one heated zone. A heat leveler provided a 4-cm isothermal region. Heat flow was along the axis to a heat extractor 8 cm from the heat leveler, which provided a gradient region. Crystal growth and directional solidification experiments were carried out in this region. Solidification was controlled by lowering the temperature in the hot zone. This resulted in a continuously variable growth rate and gradient in the sample. Containerless techniques using acoustic and electromagnetic positioning were under development at the time, but had not progressed far enough to be used on Skylab.

One important aspect provided by Skylab was the long duration of the mission which allowed experiments to be repeated after the first results were evaluated. This was used to considerable advantage by a number of the Principal Investigators whose experiments were performed during Skylab III. The samples were returned and analyzed to the extent that various possible improvements in the experiment procedure became apparent in time to be incorporated in Skylab IV.

The most complete reference to these experiments is found in the Proceedings, Third Space Processing Symposium: Skylab Results, Vol. 1, NASA M-74-5, Marshall Space Flight Center, June 1974. Summary reviews of the Skylab experiments are found in the papers by C. A. Lundquist, "Skylab Experiments on Metals," and "Skylab Experiments on Semiconductors and Alkali Halides," Processing and Manufacturing in Space, Proceedings of a Symposium held at Frascati, Italy, ESRO SP-101, European Space Research Organization, July 1974. Reviews of the crystal growth experiments are found in J. R. Carruthers' paper, "Crystal Growth in a Low Gravity Environment," J. Crystal Growth, Vol. 42, pages 379-385 (1977), and in R. L. Parker's paper, "Results of Crystal Growth in Skylab (and ASTP)," Crystal Growth and Materials, (ed. E. Kaldis and H. J. Scheel) North-Holland Publishing Company, 1977, pages 852-886.

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ZERO-GRAVITY FLAMMABILITY

Experiment M-479

Principal Investigator: J. H. Kimzey, Johnson Space Center

Of great concern in the design of a spacecraft is the flammability of the materials used in its construction. Extensive testing of the flammability of these materials had been done on the ground, but it was recognized that combustion processes would be quite different in a low-g environment where there is no convective flow to bring oxidants to the flame or to remove combustion products. The zero-gravity flammability experiment was primarily devoted to verifying the adequacy of the procedures used to test the materials and to confirm theoretical expectations of flame behavior in a diffusion-controlled environment.

Some 37 experiments were conducted using 6 materials: aluminized mylar, nylon sheet, neoprene-coated nylon fabric, polyurethane foam, teflon fabric, and paper. The primary objective was to note the extent of surface flame propagation and flashover to adjacent materials. All tests were performed in the work chamber of the M-512 Materials Processing Facility. The atmosphere was 65 percent O_2 and 35 percent N_2 at 5 psia.

In the low-g environment, the flames were completely different in appearance from those on Earth, as one might expect. Instead of the teardrop shape formed by the rapidly rising combustion products, the flames in low-g were corona-like, surrounding the fuel. Since diffusion is the only process bringing in oxygen and removing exhaust products, burning rates are considerably slower, except for the case of thin, highly flammable material in which the flame spreads rapidly over the surface. Nonmelting materials, such as paper, tend to be self-extinguishing. Porous materials, such as polyurethane foam, are thoroughly impregnated with oxygen and tend to burn vigorously. An interesting spherical smoke pattern was noted when the fire burned itself out.

Several methods of extinguishing fires were investigated. Simply evacuating the chamber is quite effective. However, the flow produced by such a process feeds fresh oxygen into the flame and causes it to burn more intensely until a sufficiently low pressure is reached and combustion can no longer be sustained. Water is effective in extinguishing flames in low g provided its application is controlled and the quantity is adequate. Again, the disturbance caused by the application may produce a larger fire.

Reference

Kimzey, J. H.: Zero-Gravity Flammability. Proceedings of the Third Space Processing Symposium: Skylab Results, Vol. I, June 1974, p. 115.

METALS MELTING

Experiment M-551

Principal Investigator: E. C. McKannan, MSFC
Co-Investigator: R. M. Poorman, MSFC

The objectives of this experiment were to (1) study the behavior of molten metal in low gravity with particular attention to the stability of the molten puddle and its interface with the solidified melt, (2) characterize metals solidification in low gravity with regard to grain size, orientation, and sub-grain patterns as might be effected by the difference in convection during solidification, and (3) determine the feasibility of joining and casting metals in space.

The experiment was performed in the Skylab materials processing facility, using the electron beam gun as a source of heat for melting. Disc-shaped specimens of varying thickness were mounted on a motorized mechanism which provided rotation of the discs with respect to the fixed electron beam. Discs were fabricated from three metals: stainless steel containing chromium and nickel (type 304), a high-strength aluminum alloy (2219-T87), and pure (99.5 percent) tantalum. Initially, the sharply focused electron beam cut through the thinnest portion of the disc. As the disc rotated, the thickness gradually increased to provide partial penetration. Eventually the beam was defocused to allow a large molten pool to form and solidify.

The experiment demonstrated the feasibility of electron beam welding, cutting, and melting in the low-gravity environment of space. The only significant difference noted between the Earth-based and the low-g results was the formation of the finer, more equiaxed grains in the low-g sample. Apparently the strong flows associated with the electron beam melting cause breakage of dendrite arms, which results in a multiplication of nucleation sites. In 1-g, these small grains fall to the bottom of the melt and initiate columnar growth, whereas in low-g the grains remain distributed throughout the melt and initiate an equiaxed structure.

References

McKannan, E. C. and Poorman, R. M.: Metals Melting Experiment. Proceedings, Third Space Processing Symposium: Skylab Results, Vol. I, NASA M-74-5, Marshall Space Flight Center, June 1974, pp. 85-100.

Garland, J. G. and McKeown, D.: Metallographic Assessment of Skylab Experiment M551. Processing and Manufacturing in Space (Proceedings of a Symposium held at Frascati, Italy, March 1974), ESRO SP-101, July 1974.

EXOTHERMIC BRAZING

Experiment M-552

Principal Investigator: J. R. Williams, MSFC

The objectives of the Skylab brazing experiment were to evaluate the feasibility of brazing as a tube-joining method for the assembly and repair of hardware in space and to investigate the mobility, mixing, and capillary behavior of molten braze alloy in low gravity.

The Skylab braze specimen consisted of a tube, a sleeve designed to slip over the tube to provide a specific clearance or gap, tapered spacer inserts designed to wedge between the tube and sleeve to fix the clearance uniformly around the tube, and braze alloy rings which snapped into grooves near each end of the sleeve. This specimen was then surrounded by thermite mixture and insulation to form an experiment assembly. Four such assemblies were prepared for brazing on Skylab: two with pure nickel tubes and sleeves, and two made from stainless steel. The braze alloy was composed of 72 percent silver, 28 percent copper, and 0.2 percent lithium by weight.

The Skylab brazing experiment verified that brazing is feasible in space. Because there is no gravitational force in space to act in opposition, gaps in braze joints were filled much more readily by capillary flow of the molten braze alloy. In one of the Skylab specimens, a joint with a gap of 0.5 mm was successively brazed, and it is assumed that greater gaps could be tolerated. On Earth, gaps 10 times smaller must be maintained to ensure a completely brazed joint. Therefore, gap tolerances for fabrication in space could be relaxed considerably; and many joints, which on Earth would have to be welded, could be made by brazing.

Some unexpected results, unrelated to braze quality, were also recorded in the form of different interactions between liquid and solid metals in space and on Earth. Under identical conditions of exposure in the two environments, the braze alloy apparently dissolved nickel much more rapidly in space than on Earth. Similarly, copper from the braze alloy penetrated solid stainless steel more extensively in space. This unanticipated behavior has not been explained. A full appreciation of its significance must await the opportunity for additional experimentation on liquid-solid metal interactions in space.

Reference

Williams, J. R.: Exothermic Brazing. Proceedings, Third Space Processing Symposium, Vol. I, NASA M-74-5, Marshall Space Flight Center, June 1974, p. 33.

SPHERE FORMING

Experiment M-553

Principal Investigator: Dr. D. J. Larson
Grumman Aerospace

This experiment represents a first attempt to perform containerless melting and solidification in space. In such a process, castings of pure metals and alloys could be produced without the contamination that results from interactions with molds and containers. It was anticipated that supercooling (cooling below the normal freezing point without solidification) could occur because the free-floating molten metal would not be in contact with the container walls that provide many of the nucleation sites for the initiation of solidification. As a result, extremely fine-grained microstructures and uniformity of alloy constituent distribution could be attained.

Droplets of metal were formed by electron beam melting of drops on a pedestal. The droplets were detached and allowed to solidify while floating freely in the test chamber. Two wheel assemblies were prepared, each containing 14 samples. The samples consisted of pure nickel, nickel alloyed with 1 weight percent silver, nickel with 12 weight percent tin, and nickel with 30 weight percent copper. Specimens were approximately 6 mm in diameter after solidification. Nickel and nickel alloys were selected for the experiment because solidification theory for the face-centered cubic crystal structure exhibited by these materials is the most advanced.

None of the samples appears to have solidified completely before contact with a wall, as was evident by flat surfaces found on the samples. Also, there was no method for determining when solidification occurred or estimating the degree of undercooling attained.

Typical in the Skylab processed specimens were the three distinct regions of solidification. One region apparently resulted from heterogeneous or localized nucleation at the pedestal base or at unmelted solid adjacent to the base, and solidification progressed upward. A second region appeared as a spherical cap at the top of the figure and possibly resulted from homogeneous or general nucleation on the liquid surface. From this cap, solidification progressed laterally on the surface and radially inward. The third region was the last to solidify, the central portion of the sphere, where the surface is pocked with shrinkage porosity caused by the volume constraction associated with the relatively rapid transformation from liquid to solid of the major portion of metal in the sample.

Reference

Larson, D. J.: Sphere Forming. Proceedings of the Third Space Processing Symposium: Skylab Results, NASA M-74-5, Marshall Space Flight Center, June 1974, p. 101.

VAPOR GROWTH OF IV-VI COMPOUNDS

Experiment M-556

Principal Investigator: Dr. H. Wiedemeier
Co-Investigators: F. C. Klaessig, S. J. Wey, and E. A. Irene
Rensselaer Polytechnic Institute

This experiment was designed to study the growth of semiconductor materials by chemical transport reactions using a vapor transport agent in 1-g and in low-g during two Skylab missions. The low-g environment was utilized to reduce convection which produces turbulent flow in the vapor mass in 1-g, and results in nonuniformity of the growing crystals. It was anticipated that the mass transport rates could be measured and that differences in the crystal morphology between the low-g and 1-g grown samples could be determined.

A vapor transport agent of GeI_4 was used with GeSe and GeTe samples. The reactions occurred in sealed quartz ampoules (15 cm long with 1.37 cm inside diameter) which were placed in the multipurpose gradient furnace. During the SL3 mission a temperature of 520°C was established at the hot end of the ampoule with the cool end temperature of 420°C and a nearly linear gradient between them. In the SL4 mission these corresponding temperatures were 412°C and 346°C. After achieving these temperatures, the samples were soaked for 33 and 34 hours, respectively, with cool-down periods of 12.5 and 7 hours following. Three samples were processed simultaneously, both in the ground-based and flight experiments, consisting of two GeSe samples with different quantities of GeI_4 to vary the pressure, and a single GeTe sample.

The investigator reported that X-ray diffraction analysis indicated no difference in lattice constants between the comparable 1-g and low-g grown crystals. The mass transport rate in low-g was reported to be several times greater than predictions based on a diffusion-only model. This was interpreted to mean that transport modes exist which are ignored in conventional transport analyses. It was later suggested that these modes may relate to the thermochemistry of the gas phase reaction and to secondary effects of the temperature gradient. In the flight samples, the crystal faces were reported to be more smooth, and with better edge definition, than those grown in 1-g. Thermal etching analysis indicated an order of magnitude lower defect density in the flight samples than in 1-g samples. The investigator suggests that the internal consistency of the results obtained for the two materials, two different temperature gradients, and the different transport agent pressures strongly supports the reported results.

The higher than expected growth rates are not yet completely understood and were the subject of continued research in the Apollo-Soyuz experiment MA-085.

References

Wiedemeier, H., et al.: Vapor Growth of IV-VI Compounds. Proceedings, Third Space Symposium: Skylab Results, Vol. 1, NASA M-74-5, Marshall Space Flight Center, June 1974.

Wiedemeier, H., et al.: Crystal Growth and Transport Rates of GeSe and GeTe in Microgravity Environment. Journal of Crystal Growth 31, 1975, pp. 36-43.

Wiedemeier, H., et al.: Crystal Growth by Vapor Transport of $GeSe_2$ and GeTe and Transport Mechanism of GeTe. Journal of Crystal Growth, Vol. 13/14, 1972, pp. 393-396.

IMMISCIBLE ALLOY COMPOSITIONS

Experiment M-557

Principal Investigator: J. L. Reger
TRW Systems Group

One potential technique for preparing fine in situ dispersions that is possible only in space is the solidification of an immiscible system. Many metallic systems exhibit a miscibility gap in their phase diagram which means that certain compositions cannot be solidified directly from the melt because as the temperature is lowered into the immiscible region, the two liquids separate like oil and water, and quickly unmix because of the density differences of the two fluids. At low-g it should be possible to maintain a fine suspension of the immiscible materials during solidification. The objective of this experiment was to investigate the possibility of preparing such alloys by isothermal and directional solidification.

One isothermal ampoule contained an alloy consisting of 76.85 weight percent gold and 23.15 percent germanium, selected because it exhibits almost complete solid state immiscibility. The other contained a mixture of 45.05 percent lead, 45.06 percent zinc, and 9.89 percent antimony and is characterized by immiscibility below a certain temperature in the liquid state. Above this "consolute" temperature the liquids are miscible, and a single-phase homogeneous solution is formed. The gradient ampoule was filled with an alloy of 70.20 weight percent lead, 14.80 percent tin, and 15.00 percent indium. This alloy was selected to determine if the tin phase could be preferentially oriented by directional solidification. The ampoules in the isothermal portion of the furnace were melted at a temperature above the consolute temperature of the lead-zinc antimony alloy and allowed to soak for a sufficient time to allow complete mixing of the elements by diffusion. The gradient ampoule was fixtured so that the cold end would not melt. This afforded the opportunity to compare, in one specimen, material solidified in space and on Earth.

The experiments showed some interesting microstructural modifications when the alloys were solidified in a low-g environment. In general, low-g solidified specimens showed more homogeneous structure but did not have the fine, uniformly dispersed structure that was expected. There was a slight apparent increase in magnetic coercive strength found in the Pb-Sn-In alloy directionally solidified in low-g. A secondary superconducting transition was observed in the Pb-Zn-Sb sample solidified in space which may result from the fine dispersion of Pb in the Zn-Sb matrix. The Au-Ge samples processed in space exhibited superconductivity of 1.5 K while the ground control samples did not. This indicates the presence of a different phase that formed in the flight samples.

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RADIOACTIVE TRACER DIFFUSION

Experiment M-558

Principal Investigator: Dr. A. C. Ukanwa
Howard University

This experiment was designed to utilize the reduction of convection in the low-g environment to determine the self-diffusion coefficient for liquid zinc while also quantifying any effects disturbing the diffusion process.

The M-518 furnace was used to melt Zn with Zn-65 tracer and soak the samples at 775°C for an hour and allowed to cool. Identical sets of three rods of Zn were prepared for ground and flight use. Sections of the tracer material were joined to one end of each of two rods and inserted between the halves of a third. The specimen ampoule consisted of a tantalum tube with solid tantalum end caps lined with carbon. The solidified samples were machined into transverse sections which were then cut into shavings and heat sealed in plastic envelopes. The gamma ray intensity was then determined for each envelope, and normalized Zn-65 concentrations were plotted as a function of section location on the zinc cylinder.

The investigator reported that the flight samples had unique wrinkled surfaces after melting in space, which may have been caused by solidification shrinkage and/or surface perturbations induced by the samples contacting the carbon liner walls of the tantalum capsule. He noted that in the flight samples, there occurred well-defined axial Zn-65 concentration gradients which were well modeled by the one-dimensional unsteady-state Fick's law of diffusion in the absence of convective currents. Zinc-65 concentrations in samples melted in unit gravity were homogeneous. The marked decrease in Zn-65 movement in liquid zinc in the near-zero gravity of space was thought to be caused by the absence of gravity-induced convective mixing. The relatively uniform radial Zn-65 distribution observed in the flight samples were taken to indicate that convective mixing was negligible in space. In the flight samples an average diffusion coefficient was found to be 4.28×10^{-5} cm^2/sec at 550°C, which is 50 times less than the effective mass diffusivity produced by convective mixing in 1-g. It was determined that an effective convective velocity of only 4.16×10^{-4} cm/sec would be needed to produce this 50-fold increase in the value of the mass diffusivity for liquid zinc; hence, the investigator notes critical importance of even slight perturbations to the pure diffusion process.

Reference

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INFLUENCE OF GRAVITY-FREE SOLIDIFICATION ON MICROSEGREGATION

Experiment M-559

Principal Investigators: Dr. J. T. Yue and
Fred W. Voltmer
Texas Instruments, Inc.

This experiment was designed to utilize the low-g environment for growth of semiconductor material crystals with reduced solute microsegregation compared to 1-g solidification results.

Three sets of germanium crystals were prepared continuing, in each set, specimens doped with gallium, antimony, and boron in concentrations of approximately 8×10^{16}, 0.4×10^{15}, and 2×10^{15} atoms/cm^3. Two sets were used as ground control samples. A gradient freeze method was employed in the vertical and horizontal directions in these sets. A heat leveler produced a constant temperature over about 6 cm from the hot end of the furnace which was brought to 1000°C in a 3-hr warm-up period to remelt a portion of the crystal and soaked at that temperature for approximately 2 hours. The hot end was cooled at 0.6°C/min to produce slow resolidification. Microsegregation characterization of the slow growth portion of the crystals was conducted.

On the basis of a comparison of radial resistivity profiles of the three Ga-doped crystals, the investigators reported improved compositional uniformity of the space-grown crystal on the macroscale and the microscale. By taking variations of resistivity in the radial direction as a measure of the gross macrosegregation fluctuation, they conclude that the space-grown crystal shows a sixfold improvement over the 1-g crystals. They contend that microsegregation in space is reduced by two- to fivefold in the bulk and eightfold near the surface for the Ge crystals doped with Ga. This is also based on the comparison of resistivity fluctuations in the samples.

No extensive analysis was reported for the Sb- and B-doped crystals since they showed contamination effects large enough to interfere with the electrical properties of the samples. These contaminants, Mn, Cu and Fe, may have entered the melt from the stainless steel tube containing the graphite cylinder with the sample enclosed. The investigators suggested that the G-doped crystals were sufficiently highly doped so that electrical interference from the contaminants was negligible.

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SEEDED, CONTAINERLESS SOLIDIFICATION OF INDIUM ANTIMONIDE

Experiment M-560

Principal Investigator: Dr. H. U. Walter
University of Alabama in Huntsville

This experiment was designed to investigate the feasibility of containerless processing of single crystals in low-g to avoid the problem of contamination of the melt by crucible material. It was intended that unidirection, steady-state solidification could be achieved in the low-g environment through reduction of convective effects. Information needed for developing the production of homogeneously doped semiconductor materials was to be obtained.

Two sets of cartridges containing three samples each were processed during two Skylab missions. The samples consisted of Czochralski grown crystals of InSb mounted into a graphite base located in the cold end of the multipurpose gradient furnace. From this support, the sample extended through the gradient section of the furnace into a hemispherical cavity in the hot zone. A temperature gradient was established which initiated melt back which adhered to the seed crystal and was to have formed a near spherical shape without contacting the cavity walls. The crystals ranged from heavily doped (10^{19} Se atoms/cm^3) to undoped. The melt was soaked 60 min at 653°C prior to cooldown at 0.6°C/min.

A combination of volume change during solidification and menescus effects from the contact angle between the melt and solid caused the resolidified crystal to grow into a teardrop shape instead of a sphere. The unanticipated length caused contact with the wall of the growth chamber near the end of the growth. Well-developed facets were observed on the free samples. During the first mission, a vehicle maneuver produced a ring-shaped mark on the sample, marking the position of the growth interface at the time of the maneuver. From this, a growth rate of 12 mm/hr was estimated. Reflection X-ray topography analysis revealed no grain or lattice misorientation in the space-grown samples. Only the very end portion of the space-grown crystals was reported to exhibit irregularities, and these areas were in contact with the mold. The average dislocation density was approximately $10^2/cm^2$, and this density decreased linearly in the center section of the space-grown crystals. The sample that was doped with Se was reported to be homogeneously doped in the center section only. Striated solute distribution was observed in the first and last portions of this crystal. The cause of these striations is not understood. Essentially no-fluid-flow effects were reported, and there was no indication of convective interference in the growing crytals in low-g.

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WHISKER REINFORCED COMPOSITES

Experiment M-561

Principle Investigator: Dr. T. Kawada

Co-Investigator: Dr. S. Takahashi
National Research Institute for Metals,
Tokyo, Japan

This experiment was directed toward preparing a high-density, uniform dispersion of SiC whiskers in a Ag matrix from the melt. The starting material was prepared in the conventional manner, using powder techniques with 2 to 10 volume percent SiC whiskers. The powder grains averaged 0.5 μ in diameter, and the whiskers were 0.1 μ in diameter and 10 μ in length.

After the mixture was compacted and sintered at 900°C in a H_2 atmosphere, it was hot pressed. The samples were then soaked above the melting point of Ag for 5 hr. Recognizing the fact that there was no mechanism such as Stokes bubble rise to remove the remaining voids in low-g, a spring-loaded plunger was contained in the ampoule to compress the mixture at a pressure of 60 kg/cm^2, which was considerably above the calculated 19 kg/cm^2 required to crush the voids between the particles and the whiskers.

The flight and ground control samples exhibited a densification during melting, but some voids were found in all of the samples. The distribution of the whiskers was fairly uniform in the flight samples, whereas they tended to cluster near the top of the sample in the ground control tests. A corresponding uniformity in microhardness was found in the flight sample, whereas the ground control samples equaled the hardness of the flight samples near the top where the whiskers tended to congregate. Elsewhere the hardness was significantly diminished. It was also found in bend load tests that the low-g samples did not exhibit brittle fracture, as did the ground control samples, but instead showed large ductility. This is attributed to the more uniform distribution of whiskers.

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Takahashi, S.: Preparation of Silicon Whisker Reinforced Silver Composite Material in a Weightless Environment. AIAA Paper 77-195, January 1977.

STEADY STATE GROWTH AND SEGREGATION UNDER ZERO GRAVITY: InSb

Experiment M-562

Principal Investigator: Prof. A. E. Witt

Co-Investigators: Prof. H. C. Gatos, M. Lichtensteiger, M. C. Levine, and C. J. Herman
Massachusetts Institute of Technology

This experiment was designed to study the process of partial melting and resolidification of crylindrical single crystals of InSb that were Te-doped, Sn-doped, and undoped. The low-g environment was utilized to reduce growth rate fluctuations and gravity-induced convective stirring which leads to variable dopant concentration on a micro as well as a macro scale. It was intended to produce semiconductor crystals of high chemical uniformity and structural perfection and to evaluate the influence of low-g in attaining these properties.

Ground-grown sample crystals were machined and etched to fit heavy wall quartz ampoules for use in the multipurpose longitudinal gradient furnace. About one-half of the sample crystals were melted and allowed to regrow using the unmelted half as a seed. Approximately 2 hr were required for the back-melt, and this was followed by a 1-hr soak to achieve thermal equilibrium in the system and homogenization of the melts. A cooling rate of 1.17°C/min for 4-hr after regrowth initiation was followed by passive cooling to ambient temperature. In Skylab IV, a second set of the samples, intended for backup purposes, was used in an unscheduled second experiment. This second experiment was essentially the same but also included interruption of the controlled cooling rate by a second 60-min soak period. It was intended to obtain time reference markings in the crystal to obtain data on transient segregation-growth rate dependence. A mechanical shock perturbation was also introduced during the growth period to provide a time marking.

The investigators found that all regrown crystals conformed to their expectations based on ideal steady state growth and segregation predictions. Growth rate fluctuation was effectively eliminated; and steady-state, diffusion-controlled growth conditions were achieved after a few millimeters, resulting in very uniform dopant distribution throughout most of the sample. The Te-doped melt, however, did not wet the quartz wall as did the other samples but solidified with a free surface configuration. Narrow surface ridges formed, which provided the only contact with the container walls. The investigators concluded that surface tension effects in space remained localized on the surface and did not affect growth and segregation in the bulk. The mechanical shock perturbation was found to cause a localized increase in dopant segregation.

The investigators have extended this research in the ASTP experiment MA-060, using other semiconductor materials to demonstrate the growth of crystals of high uniformity in composition and structure.

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DIRECTIONAL SOLIDIFICATION OF InSb-GaSb ALLOYS

Experiment M-563

Principal Investigator: Dr. W. R. Wilcox

Co-Investigators: Drs. J. F. Yee, K. Sarma, M. Lin, and Sanghamitra Sen
University of Southern California

This experiment was designed to utilize the low-g environment to reduce the gravity-driven effects which prevent the preparation of high-quality bulk crystals of Group IV and III-V semiconductor alloys. In 1-g melts, convection leads to crystal inhomogeneities, breaking of dendrite arms, local variations in interface stability, fluctuations in freezing rate, and other undesirable effects. This experiment attempts to determine the role of gravity in these effects.

Polycrystalline and dendritic feed ingots containing GaSb and InSb were mixed in a carbon-coated silica tube at over 900°C and then cooled in air. Compositions were chosen at 10 percent, 30 percent, and 50 percent InSb. After removal, the samples were placed in a carbon-coated 8 mm I.D. silica tube with graphite plug and a quartz wool spring at each end. These ampoules were backfilled to 10 Torr with helium. Three samples were processed in the gradient furnace during each of two Skylab missions, horizontally on Earth, and vertically on Earth with the hot end of the furnace on top. The hot end temperatures of 960°C and 1020°C for the two flight experiments were sufficient to melt approximately half of each ingot. After a 16-hr soak, cooling at 0.6°C/min was initiated. The processed samples were cut longitudinally, sandblasted, and observed using oblique polarized incident lighting. The void volume and the numbers of grain boundaries, twin boundaries, and microcracks were determined at 1 mm intervals along each of the 12 ingots.

The investigators reported that although the initial value of the ratio of temperature gradient to freezing rate (G/V) was sufficient to avoid constitutional supercooling, as the ingot cooled, the temperature gradient decreased, resulting in an increase in the freezing rate. Eventually, constitutional supercooling occurred in all ingots; hence, local inhomogeneities developed in all samples. With the six Earth-processed ingots and one of the space-processed ingots there was sufficient bireflectance to reveal the grains and twins by use of slightly uncrossed polarizers. Analysis of the samples showed that defect densities were less for the space-processed samples; however, the investigators indicate that sample contamination may account for this. They suggest that in low-g, small particles randomly distributed throughout the melt cause twins when the growing interface reaches them,

whereas on Earth these particles would tend to settle on the interface and to impact it repeatedly when carried by a convective stream and produce the greater twinning they observed. Bubble densities, grain boundaries, and microcrack frequencies were also reported for the samples. Gas bubbles were more uniformly distributed in the space-processed ingots, but the investigators point out that a reasonable vacuum would eliminate them completely. A wide variety of grain sizes was noted; however, no clear implications were drawn. The tendency to form microcracks increased with increasing InSb content but showed no systemmatic dependence on gravity.

In continuing analysis of the samples by the principal investigator and others, electron microprobe data have been obtained from the transverse sections which show variations in indium concentration that indicate the presence of radially symmetric convection conditions in the melts during solidification.

References

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METAL AND HALIDE EUTECTICS

Experiment M-564

Principal Investigator: Dr. A. S. Yue

Co-Investigator: J. G. Yu
University of California at Los Angeles

This experiment was designed to utilize the low-g environment fiberlike structure of NaF during solidification of a NaF-NaCl eutectic. It was intended that the microstructures of the eutectics solidifying unidirectionally in low-g and 1 g could be compared and that their optical and other relevant characteristics could be assessed. The electric, thermomangetic, optical, and superconducting characteristics of continuous fiberlike eutectic microstructures would be strongly anisotropic, suggesting various solid state device applications.

Ingots of NaF and NaCl eutectics were melted in a gradient furnace in a graphite container held by stainless steel and copper ampoules in low-g and 1 g. The eutectics solidified directionally and their microstructures, fiber orientations, and optical properties were compared.

No reaction was observed between the samples and their graphite containers. The NaF fibers in the low-g samples were reported to be regularly spaced and parallel to the growth axis, as were 1-g vertically grown fibers. The low-g samples, however, showed significantly improved continuity of the fibers over the vertically grown 1-g samples. Transmittance data were reported for the NaCl-NaF samples grown in low-g, vertically in 1 g, and nonvertically in 1 g. Some improvement in transmittance was observed in the low-g sample compared to the vertically grown 1-g sample. A 5 to 10 percent improvement was reported across the region from 2.5 to 18 μm which increases up to 30 to 40 percent in the 8 to 10 μm region. The 1-g sample grown nonvertically was quite low in transmittance compared to the other samples, showing negligible transmittance of wavelengths below 7 μ.

This work was continued in the Apollo-Soyuz experiment MA-131 where LiF-NaCl eutectic was employed.

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COPPER-ALUMINUM EUTECTIC

Experiment M-566

Principal Investigator: E. A. Hasemeyer
MSFC

This experiment investigated the effect of gravity on the directional solidification of eutectics to determine if improvments in rod or lamellae continuity and structure could be obtained by reducing convective flow. The choice of Cu-Al was dictated by furnace capability and by the fact that this system has been extensively studied and is a well-known model system for eutectic solidification.

The samples were prepared by directional solidification on the ground, sheathed in graphite, and sealed in standard stainless steel cartridges. They were melted back in space and directionally solidified by lowering the temperature while maintaining a thermal gradient.

An inspection of the specimens after processing revealed that the diameter of each specimen solidified in space was reduced in the regrowth region, giving the specimen an hourglass shape. This effect was not observed in specimens processed on Earth under otherwise identical conditions. The reduced diameter was caused by failure of the alloy to wet its graphite container. In the absence of gravity, surface tension was the predominant force in shaping the liquid mass, and the liquid withdrew from the container walls in a natural attempt to minimize its surface energy. This opens the possibility for surface tension-driven convection which could have affected the solidification.

Comparison of the flight and ground control samples shows little, if any, difference in the microstructure, indicating that gravity-driven convection plays no significant role in the generation of faults and terminations of the lamellae. Unfortunately, there is no way of obtaining time correlation between the observed faults and acceleration events on the Skylab to determine if the faults corresponded to g-spikes or other events that could produce flow.

Reference

Hasemeyer, E. A.: Copper Aluminum Eutectic. Proceedings of the Third Space Processing Symposium: Skylab Results, Vol. I, NASA M-74-5, Marshall Space Flight Center, June 1974, p. 457.

V. SKYLAB DEMONSTRATION EXPERIMENTS

The long duration of the Skylab missions and the proficiency developed by the astronauts in performing their planned experiments provided some free time during the latter part of the mission. A number of fairly simple demonstration experiments were suggested by various personnel associated with the program. The demonstrations were carried out mostly using equipment on-board the Skylab, although some special apparatus was carried up on Skylab IV.

The demonstration experiments served several purposes. First, they were used to dramatize the peculiar effects of weightlessness and provide graphic teaching aids to students as well as to scientists planning space experiments. Secondly, they provided valuable experience in testing experimental concepts and in handling fluids in weightlessness. Thirdly, they served to elucidate various fluid processes and in some cases provided quantitative as well as qualitative data on fluid behavior in the spacecraft environment.

These experiments are summarized in T. C. Bannister's papers, "Skylab Science Demonstrations," Proceedings, Third Space Processing Symposium: Skylab Results, Vol. 1, NASA M-74-5, Marshall Space Flight Center, June 1974; "Post Flight Analysis of Science Demonstrations," Scientific Investigations of the Skylab Satellite, Progress in Astronautics and Aeronautics, Vol. 48, (Ed. M. Kent, E. Stuhlinger, and S. Wu) AIAA, New York, 1976; and S. Ostrach's paper "Skylab Science Demonstrations," Processing and Manufacturing in Space, ESRO SP-101, (Proceedings of a Symposium at Frascatti, Italy, 25-27 March 1974), European Space Research Organization, July 1974.

ICE MELTING

Science Demonstration

Investigators: Dr. L. L. Lacy, MSFC
Dr. G. Otto,
University of Alabama in Huntsville

This science demonstration was designed to dramatize the effect of low-gravity on melting and solidification phenomena. The liquid/solid interface was observed and the melting time and melting rate were determined.

In this demonstration, a cylinder of ice was frozen on a cotton swab in a pill dispenser bottle that was 6.3 × 2.5 cm. After the ice had been in the freezer for 1 day, it was removed from the pill bottle and mounted in front of the 35-mm camera. Periodic photographs of the ice melting were made. The rate of ice melting in zero gravity without buoyancy convection and the shape of the liquid on the unmelted ice portion were determined. As water formed, surface tension tended to make the layer spherical. Consequently, the thicker layer over the larger surface area (cylindrical surface) tended to insulate the ice, whereas more heat was able to enter at the ends, so that these melted first. This effect, together with the absence of convection in the surroundings, resulted in the melting time being about six times as long as on Earth. It was concluded by the investigators that 87 percent of the heat transfer was by radiation and 19 percent by conduction. There was no apparent heat transfer by convection. (The experiment was shielded from the circulating cabin atmosphere driven by fans by means of a baffle.) At the conclusion of the ice-melting experiment, a large globule of water remained attached to the retaining stick. It was decided to utilize this to observe surface tension effects by the addition of surface-active solutions. First, a small drop of soap solution was added to the large water globule with a syringe. After studying the effects, a small drop of grape juice was added in a like manner. The surface of the globule was seen to retract vigorously when touched by either the soap solution or grape juice. Rapid fluid motion was seen by bubble and grape color movement for some moments after the addition of the surface-active solutions. A mixture of soap solution and air was then injected directly into the globule from the syringe. Small bubbles formed which persisted inside the globule. The cluster of air bubbles eventually became critical in number (size); additional injection then resulted in bubbles popping out of the globule.

Reference

Otto, G. and Lacy, L.: Observations of the Solid/Liquid Interface in Low-Gravity Melting, AIAA Paper 74-1243, 1974.

DIFFUSION IN LIQUIDS

Science Demonstration

Investigator: Barbara Facemire, MSFC

The purpose of this demonstration was to dramatize the diffusive mixing of liquids in the absence of gravity-driven convection. A plastic tube 12 cm long and 2 cm in diameter was filled with water from a syringe. A few drops of instant tea (about 7 times normal concentration) were carefully placed at one surface of the tube, which was periodically photographed over a period of 3 days. The tea in the center of the tube diffused in a distance of about 2 cm in 45 hr, illustrating how slow diffusive mixing tends to be. One unexpected effect was that the tea did not diffuse as rapidly along the wall, possibly because of some electrostatic repulsion between the wall and the molecules of tea.

A significant observation in this experiment is that combined effects of vehicular motion, crew motion, thruster firings to dump angular momentum from the accumulation of gravity gradient torques, and other sources of acceleration or g-jitter integrated over 3 days did not produce any discernible mixing.

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LIQUID FLOATING ZONE

Demonstration Experiment TV101

Principal Investigator: Dr. J. R. Carruthers
Bell Research Laboratories

The purpose of this demonstration was to simulate an important technique for growing crystals in space by means of a floating zone and to investigate the stability of such a zone under static and dynamic conditions.

An apparatus was constructed from parts on-board Skylab. It consists primarily of a pair of socket wrench extensions supported by four camera mounts to form a sort of lathe. Thin aluminum discs were fixed to the ends of the socket wrench extensions with double-coated marking tape to form the end caps for the floating zone. Water was used as the test liquid, and various amounts of soap and other additives were used to vary the surface tension and viscosity. The aluminum discs were coated with grey tape treated with acetone to reduce the contact angle of the water. The outer edges of the disc were coated with Krytox oil to prevent the water from wetting the entire disc. Rotation was provided by means of a twine wrapped around the wrench extensions. This is one of the most outstanding examples of resourcefulness in developing an experiment from scratch during a manned space flight.

It was also found that nonrotating zones could be stretched approximately 5 percent beyond the theoretical limit (L, an equal to circumference) for stability predicted by Rayleigh. Beyond the stable length the zone is no longer cylindrical but assumes the shape of an unduloid, which apparently increased the stability.

The rotational stability was investigated under a number of conditions. For sequences involving the single rotation of only one bounding disc, the zone assumed an axisymmetric, bottle-shaped, deformation possessing stability limits in general agreement with theory. For sequences involving equal rotations of both bounding discs, an unexpected nonaxisymmetric or "C-mode" instability was encountered for zone lengths approaching 2/3 πR. The liquid zone resembles a jump rope and is relatively insensitive to the rotation velocity of the disc once initiation occurs. The mode is self-amplifying and possesses extremely long decay times after the rotation of the discs has ceased. Such instabilities are not found in conventional floating zones on Earth because of the shorter zone lengths imposed by hydrostatic pressure. These short zones are more susceptible to the axisymmetrical capillary wave instabilities. An understanding of the stability of liquid zones is crucial to the development of space experiments designed to take advantage of the extended zone length possible in low gravity.

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Klett, M. G.: Analysis of Skylab Liquid Floating Zone Experiment. NAS8-27015, LMSC-HREC TM D3906913, February 1975.

IMMISCIBLE LIQUIDS

Demonstration Experiment TV102

Principal Investigator: Dr. L. L. Lacy, MSFC

Co-Investigator: Dr. G. Otto
University of Alabama in Huntsville

The objectives of this experiment were to evaluate the stability of finely dispersed immiscible liquids in the low-g environment and to observe the coalescence and growth of the immiscible droplets.

The apparatus consisted of a clip containing three vials (6.3 × 1.2 cm) containing mixtures of Krytox oil and 25, 50, and 75 percent water. A small brass nut in each vial aided in mixing as the vials were shaken. A grid of black lines on white cardboard behind the vials provided a measure of the clarity of the mixtures.

When the experiment was performed on the ground, a maximum of only 10 sec was required to separate all of the mixtures. The 75 percent oil mixture is the slowest because the viscosity of the oil slows the motion of the suspended water droplets. The mixtures remained remarkably stable in space. After 10 hr (36000 sec) in the Skylab environment, no change could be detected. Apparently the g-jitter was not sufficient to cause agglomeration, and the fact that the g forces were random prevented the low-level accelerations from causing significant sedimentation or creaming.

References

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LIQUID FILMS

Demonstration Experiment TV103

Principal Investigator: W. Darbro, MSFC

The purpose of this experiment was to investigate shape and stability of liquid films in the absence of gravitational distortion or sagging.

A variety of two- and three-dimensional shapes were fashioned from safety wire that was found on-board. Films were formed by clipping the shapes into plain water and water containing a small amount of shower soap.

Initially, sizable films could be made without soap but could not later be reproduced, possibly because the wire became oily from handling. Stability was greatly enhanced because of the absence of sagging. Films laster three to ten times longer than in the ground control experiments. The most fascinating aspect of the experiment was the shape of the film produced in the three-dimensional cubical frame. When the frame was withdrawn from the water, it contained a cube of water. When some water was shaken out, a smaller cube remained supported by a web of 12 films connecting it to the frame. Eventually, as more water was shaken out, the central cube collapsed to a film, reproducing the classical plateau configuration.

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ROCHELLE SALT GROWTH

Demonstration Experiment TV105

Principal Investigator: Dr. I. Miyagawa
University of Alabama, Tuscaloosa

The objective of this experiment was to investigate the growth of crystals by precipitation from an aqueous solution in a low-g environment. This eliminates the solutal convection which normally acts to maintain solute concentration in the growth region. In low-g, diffusion is the dominent mechanism that controls the transport of solute.

The experiment was performed in a 4-in. diameter food can with a transparent lid. Rochelle salt powder and a larger seed crystal were dissolved in the can while it was being heated on the food heating tray. After the seed was three-fourths melted, the heat was removed and the can was wrapped in towels to provide a slow cooldown. During storage in zero g, the seed crystal slowly regrew as the can cooled back down to cabin temperature over a period of 2 days, after which the towels were removed. Two weeks later, Astronaut Pogue removed the seed crystal from the solution to observe the results in space. The solution contained many small Rochelle salt crystals that had nucleated from solution. Pogue described the solution as "slushy" and the nucleated crystals which he saw as being "Micalike." The seed crystal, which was returned to Earth, had regrown in the form a plate about 2.6 cm long on each side and about 5 mm thick. The crystal was broken into three parts during handling in space. The seed crystal is polycrystalline, containing at least five crystals. A striking feature of this crystal is the existence of 0.1-mm diameter cavities, many of which are up to 4 mm in length oriented along the optical axis. Cavities having a geometry such as that seen in the space-grown crystals have not been seen in crystals grown on the ground. The five or more component crystals are oriented with their axes parallel. On Earth, the orientation is usually random. According to Miyagawa, this unusual arrangement of the component crystals suggests the presence of a long-range molecular force of attraction between crystals. A small part of the crystal is almost completely free of defects and appears extremely good optically.

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DEPOSITION OF SILVER CRYSTALS

Demonstration Experiment TV 106

Principal Investigator: Dr. P. G. Grodzka
Lockheed Missiles and Space Company

The objective of this experiment was to elucidate the effect of reduced gravity on electrochemical displacement and crystal growth reactions.

A previously prepared coil of copper wire with scored notches to expose the bare copper was placed in a vial of 5 percent silver nitrate solution. A slow chemical reaction between the copper and the solution produces silver crystals. In a normal gravity environment, these polycrystals are tree-shaped dendrites. In space, long polycrystals (dendrites) were formed, with small arms which could be described as elongated dendrite structures. It should be noted that vibration during re-entry dislodged most of the crystals from the wire. The crystals that were returned from the Skylab experiment were more powdery or less compact than crystals similarly grown at 1, 2, and 5 g. Crystalline compactness was found to be a direct function of g level: the greater the g level, the more compact the crystalline mass. The increasing crystalline compactness as g level is increased is related to the vigor of natural convection accompanying crystal deposition. Apparently, at 5 g, vigorous convection would make the rate of crystal deposition more likely to be determined by inherent crystal deposition kinetics. In the normal zero-g case of Skylab IV, the rate of crystal deposition was controlled by the slow rate of mass diffusion. For this particular system this resulted in a highly dendritic structure, with the final product being fragile and powdery. It has been suggested that such structures may serve as good catalysts because of their large surface-to-volume ratio.

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FLUID MECHANICS

Demonstration Experiment TV 107

Principal Investigator: O. Vaughan, MSFC

Co-Investigator: Ms. B. Facemire, MSFC
Dr. S. Bourgeois, Lockheed

The objective of this investigation was to provide an educational film depicting fluid phenomena in a free fall environment, emphasizing the role of surface tension in the absence of containers and gravitational distortion.

A series of experiments were performed using water droplets and various agents for providing color and for altering their surface tension. A 16-mm movie camera was utilized to record the data. Quantitative measurements have been made from the film. These included the frequency and damping of oscillations of different-size liquid droplets, the approach velocity and frequency of oscillation for the droplet coalescence demonstrations, and the deformation of drops during the rotation and breakup demonstration.

Spheres of water with and without soap were caused to oscillate by pressing against their sides with plungers from a syringe and quickly releasing. The observed frequency could be related to the surface tension according to Rayleigh's theory. The relationship between damping time and viscosity was not in as good quantitative agreement with theory, perhaps because the first order theory of Lamb assumes small oscillations. This demonstrates a technique that can be used for obtaining high temperature physical properties measurements for reactive materials.

The coalescence of two impacting liquid spheres was observed. The water droplets were colored with strawberry and grape drink so that the internal mixing could be visualized. One of the striking observations was the relatively small amount of mixing associated with the rather violent oscillations produced by the collision process.

The injection of air into liquid globules demonstrated the effect of compressible air in damping oscillations. This test also demonstrated the feasibility of forming hollow, thin-walled liquid spheres. Several syringes of air were injected into a water globule, forming a single sphere of air inside the liquid globule. A process such as this could be used to manufacture large fuel containment shells for use in inertial confinement fusion research.

Perhaps the most striking demonstration concerned the rotational stability and fissioning of a liquid sphere. A free floating globe of water was spun-up with a piece of string. The sphere first deforms as an oblate spheroid at low rotational speeds. At high rotational rates, the body becomes "dumb-bell" shaped and eventually fissions into two spheres. A similar process is thought to be responsible for the formation of double stars.

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CHARGED PARTICLE MOBILITY

Demonstration Experiment TV 117

Principal Investigator: Dr. D. M. Bier
Veterans Hospital, Tuscon, Arizona

The goal of the Skylab electrophoresis experiment was to determine if the use of low-g could alleviate the convective mixing and sedimentation problems inherent in the one-g process to achieve protein separation comparable to gel techniques and to see if larger particles such as cells could be separated by this process.

A special free-flow electrophoretic separator with two static columns was developed for this experiment. One column contained human red blood cells and the other contained a mixture of proteins, ferritin and hemoglobin. A mixture of two buffers was used, one with anions having greater mobility than the sample, and one with anions of less mobility than the sample. This variation of electrophoresis, known as isotachophoresis has the advantage that the interface between the sample and buffer is self-sharpening and is capable of very high resolution provided convective mixing can be avoided.

The attempt to separate the proteins was not successful. It is conjectured that a bubble formed in the electrode chamber of this column, blocking the current flow. The column containing the red blood cells produced better results. A bubble was initially observed in the column, but was dislodged by the astronaut gently tapping the instrument. The cells moved as expected when the current was applied, but the front was distorted into a bullet shape primarily because of electroosmotic flow.

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VI. APOLLO-SOYUZ EXPERIMENTS

The joint U.S.-Soviet Apollo-Soyuz Test Project (ASTP) provided a second long-duration, manned flight opportunity to carry out material processing experiments. The relatively short time between Skylab and the ASTP mission and the limited space, power, and mission time-line restricted the types of experiments that could be carried out on this mission. The ASTP mission did provide a valuable opportunity for several of the Skylab investigators to upgrade and repeat their earlier experiments to confirm their results or to explore different aspects of their findings. Also several additional investigators were brought into the program with new experiments.

The detailed results of these experiments are reported in (1) Apollo-Soyuz Test Project, Preliminary Science Report, NASA TMX-58173, Johnson Space Center, February 1976; (2) Apollo-Soyuz Test Project: Composite of MSFC Final Science Report, NASA TMX-73360, Marshall Space Flight Center, January 1977; and (3) Apollo-Soyuz Test Project — Summary Science Report, NASA SP-412, Johnson Space Center, 1977.

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ELECTROPHORESIS TECHNOLOGY

Experiment MA-011

Principal Investigator: Dr. R. E. Allen, MSFC

Co-Investigator: Dr. G. H. Barlow, Abbot Labs

The objective of the electrophoresis technology experiment was to demonstrate the feasibility of free-flow electrophoresis in a static column by using the low-g environment to suppress the convective mixing associated with the joule heating. In addition, a number of technical difficulties had to be resolved, such as sample deployment and retrieval, maintaining sterility, and sample viability and suppression of electroosmotic flows.

A special column electrophoretic apparatus was developed with the associated electrode chambers, power supply, and controls. A special coating was developed to lower the zeta potential of the walls of the column to suppress electroosmotic flow. Samples consisint of three types of red blood cells (rabbit, horse, and human), human lymphocytes, and fetal kidney cells were frozen into thin sample insertion discs. After electrophoresis was accomplished, the column was frozen in place with a thermoelectric device and transferred to an LN_2 dewar for return to Earth.

The columns containing the human lymphocytes malfunctioned because of a clogged electrode rinse line. The red blood cells were separated with sharp and well-defined boundaries, indicating that no significant flows were acting. The kidney cells were separated into a number of fractions and returned to Earth maintaining 25 percent viability. Enhanced production of urokinase, erythropoietin, and granulocyte conditioning factor were found in the separated cell fractions, which hints that separation according to cell function was accomplished.

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ELECTROPHORESIS

Experiment MA-014

Principal Investigator: Dr. K. Hannig
Max Planck Institut fur Biochemie,
Munich

One of the limitations of continuous flow electrophoresis on Earth is the necessity of confining the flow to a very thin chamber to remove the heat generated by the current flowing through the buffer and prevent convective instabilities resulting from thermal gradients. This limits the throughput of the device by requiring a very small sample stream and introduces distortions from wall effects which degrade the resolution of the device. By operating such a device in space, it should be possible to greatly improve both the resolution and the throughput of continuous flow electrophoresis. The purpose of this experiment was to investigate and evaluate the increase in sample flow and sample resolution achievable in space.

A special continuous flow electrophoresis system was developed for use in ASTP. The flow chamber was 28 mm wide and 3.8 mm thick. The separation took place over the 180 mm electrode length. The applied voltage was 60 V/cm. The buffer flow was varied, reaching a maximum of 0.275 cm^3/sec. The sample flow ranged from 1 to 5 cm^3/hr. The samples included rat bone marrow cells, mixtures of human and rabbit erythrocytes, rat spleen cells, and rat lymph node cells mixed with human erythrocytes as markers. The separation was monitored by photo diode array illuminated by an ultraviolet halogen lamp. No attempt was made to collect the separated samples.

The flow systems apparently functioned as planned; however, an unexpected increase in brightness of the halogen lamp that provided the illumination for the ultraviolet detector caused the detector elements to saturate. Thus they were only able to detect when more than one cell passed through their field of view. This greatly limited the amount of useful data, but the events detected appeared to be roughly consistent with the expected operation of the system.

The reason for the anomalous brightness of the lamp is not completely understood. In ground tests the lamp functioned normally. It can only be surmised that the lack of convective flow in the low-g environment influenced the operation of the lamp and caused it to put out more light. This effect obviously should be considered in future flight experimemts.

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CRYSTAL GROWTH

Experiment MA-028

Principal Investigator: Dr. M. D. Lind
Rockwell International
Science Center

The objective of this experiment was to investigate the growth of single crystals of insoluble substances by a process in which reactant solutions are allowed to diffuse toward each other through a region of pure solvent. The low-g environment was used to eliminate the sedimentation of the growing crystals and reduce the convection effects wihout introducing the complications of the gel growth technique. In 1-g the sedimentation reduction by gel technique also tends to isolate the growing crystal and limits the size attainable. Contamination by the gel is also problemmatic.

The three crystals grown in low-g were calcium tartrate, calcium carbonate, and lead sulfide. Each crystal was grown in a pair of reaction chambers, slightly different in size, in a water compartment with two reactant solution compartments attached on opposite sides. In addition to the geometry differences in the pair of reaction chambers, the pH was adjusted to 1.0 in one reactor and 0.5 in the other by addition of HCl. Color photographs were made every 12 hr after the valves were opened to allow the reactant solutions to enter the growth chamber.

The calcium tartrate and calcium carbonate reactions went to completion before splashdown. The lead sulfide crystals were still forming at splashdown and continued to form sediment in 1-g. Some of the calcium tartrate crystals were 5 mm long, which is longer than gel-produced crystals in 1-g. More of these crystals were plate-shaped, rather than the usual prism-shaped. The investigator offered no conjecture as to the reason for this phenomenon. Many of the calcite crystals were about 0.5 mm in edge length and rhombohedral in shape. Their appearance was reported to be very similar to 1-g natural and synthetic crystals. Birefringence was also exhibited by the low-g grown crystals of calcite. The investigator noted that most of the calcite crystals adhered to the reactor wall; hence, a better choice of reactor chamber material could have been made. It was further reported that the lead sulfide experiments, while less successful, both resulted in discrete single crystals with dimensions as large as 0.1 mm. Since the reaction which produced these crystals did not go to completion, the investigator suggested that a longer reaction time or a higher temperature to increase the solubility of the reactants may result in larger crystals.

Greater variation in cabin temperature (60° to 75°F) was experienced than anticipated, and the investigator reported that solubilities of the reactants and crystalline products were strongly enough temperature dependent to adversely affect the crystal growth. It was further reported that nucleation in all samples was excessive. The investigator suggests that in the refining of the experimental parameters to produce less nucleation and larger crystals, precise temperature control and longer growth times probably are the main improvements which should be made.

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SURFACE-TENSION-INDUCED CONVECTION

Experiment MA-041

Principal Investigators: Dr. R. E. Reed

Co-Investigators: Drs. W. Uelhoff and H. L. Adair
Oak Ridge National Laboratory,
Oak Ridge, Tenessee

The purpose of this experiment was to investigate compositional induced surface tension-driven convection in wetting and nonwetting containers in a low-g environment. Specifically, it was attempted to determine whether or not the no-slip boundary condition applies to nonwetting systems in the absence of the hydrostatic pressure forcing the liquid against the container wall.

Lead-gold alloy (0.05 atomic percent gold) was pressure-bonded to lead to create a liquid diffusion couple. Samples enclosed in ampoules lined with steel and graphite, anticipated to be wettin and nonwetting, were subjected to two diffusion temperature profiles peaking at 923 and 723 K. Temperature gradients within the ampoules were minimized to isolate effects due to compositional variations in surface tension. An identical set of the graphite wall ampoules was given opposite orientation with regard to heat flow direction so that the relative effects of melting/solidification on the gold concentration profile could be determined. Gold concentration profiles were obtained by autoradiography analysis of the sample sections following neutron bombardment to produce α-emitting 198Au.

Analysis of the low-g samples showed that the gold moved about one-half the distance in the low temperature ampoule as in the high temperature ampoule, as would be expected from diffusion theory. Some distortion of the diffusion profile was observed near the walls, and this distortion seems to depend on the sample orientation in the furnace. This suggests that either volume change or segregation effects during solidification may have been partially responsible for this effect. No difference was observed between samples processed in the steel and graphite ampoules. Apparently the melt did not wet the steel as expected.

The ground control samples showed almost complete mixing when processed in the vertical destabilizing orientation (with the heavier gold alloy at the top). In the stable configuration (gold alloy at the bottom) substantial, but not complete, mixing was observed, probably because of the radial gradients.

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The investigators conclude that Marangoni convection offers the best explanation for the observed distribution, and therefore the "no-slip" boundary condition apparently does not apply to nonwetting materials in low-g. However, a detailed analysis was not presented to support this assertion. Such a conclusion must therefore be regarded as premature. However, the bulk migration of Au in the Pb appeared to be almost completely diffusion controlled in the space processed samples. No trace of Au was found away from the diffusion region, indicating negligible mixing resulted from the combination of vehicle accelerations and the various nongravitational effects.

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MONOTECTIC AND SYNTECTIC ALLOYS

Experiment MA-044

Principal Investigators: Dr. L. L. Lacy, MSFC
Dr. C. Y. Ang, The Aerospace Corporation

The low-g environment was utilized by this experiment to minimize buoyancy and convective influences which in normal gravity prevent adequate synthesis of material systems in which significant specific gravity differences exist. Microstructural homogeneity and stoichiometry of the semiconducting compound AlSb and phase segregation effects for the immiscible binary Pb-Zn were studied. Aluminum antimonide was chosen because of its potential use in solar cells of much greater efficiency than silicon. The Pb-Zn mixture was chosen because, like aluminum and antimony, large specific gravity differences prevent adequate 1-g mixing.

Three cartridges, each containing two ampoules with a sample of each of the two material systems, were heated, soaked, and cooled in the ASTP multipurpose furnace. The polycrystalline AlSb samples were soaked isothermally at 50 K above the melting temperature (1353 K) for 1 hr after a 3-hr 20-min heat-up period. The Pb-Zn samples were 20 atomic percent Pb and underwent the same heat-up and soak periods, soaking at a temperature 40 K above the consolute temperature of the binary (1068 K). The cool-down period was 6.5 hr. A variety of assessment techniques were employed to characterize the samples, including metallography, quantitative microstructural analysis, scanning electron microscopy/energy dispersive X-ray analysis, electrical resistivity measurements, X-ray diffraction, chemical analysis, and ion-microprobe mass analysis.

The principal investigators conclude that the liquid-state homogenization of polycrystalline, multiphase AlSb in low-g produces major improvements in macroscopic and microscopic homogeneity, showing 4 to 20 times less of the unwanted secondary phase than in 1-g. Very small amounts of an Al-rich phase are found only along parts of the grain boundaries in the flight samples, whereas the 1-g samples show major grains of Al-rich or Sb-rich phase. Their diffusional and liquid-state homogenization analysis indicates that gravity-induced convection can severely complicate the homogenization of 1-g melts, inducing compositional and microstructural inhomogeneity during solidification. The investigators contend that other unique binary systems which are difficult to synthesize on Earth because of gravity-induced effects may be advantageously processed in space.

The investigators observed incomplete liquid-state mixing of Pb and Zn in spite of the homogenization temperature 40 K above the miscibility gap commonly accepted. In the flight samples, a dispersion of fine particles of Pb in the Zn matrix was observed, but the bulk of Pb remained in its original position. On the basis of this observation, the investigators suspected that a significant inaccuracy may exist in the commonly accepted phase diagram for Pb-Zn. Recent ground-based research (Trahan and Lacy) supports this assertion, indicating that the accepted immiscibility curve between 20 and 70 atomic percent Zn is as much as 20°C too low. Other recent experiments indicate that a much longer soak period than realized is required to homogenize immiscible materials. It is, therefore, quite probable the initial melt was never completely mixed. A quasi-solid-state diffusion of Pb into Zn with a diffusion coefficient of 2.4×10^{-6} cm^2/sec was indicated from the analysis of the samples.

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INTERFACE MARKING IN CRYSTALS

Experiment MA-060

Principal Investigator: Prof. H. C. Gatos

Co-Investigators: Prof. A. F. Witt, M. Lichtensteiger, and C. J. Herman
Massachusetts Institute of Technology

This experiment is an extension of the investigators' indium antimonide crystal growth experiment on Skylab which demonstrated that growth rate fluctuations could be eliminated and steady state diffusion-controlled growth maintained in a low-gravity environment, resulting in an improvement in homogeneity. A novel electric pulsing system to mark the interface was utilized to determine the growth rate during the solidification process. Other objectives included the determination of dopant segregation, investigation of possible nongravitational convection phenomena, and the comparison of wetting phenomena between 1-g and low-g.

Cylinders of Ga-doped Ge were heated in the MA-010 furnace to 1393 K on the hot end so that Ge was melted down to approximately 3.5 cm from the cool ends of the 10-cm cartridges, and soaked for 2 hr. The temperature was lowered for approximately 1 hr at 2.4 K/min and then switched off. Helium injection occurred 1 hr later for rapid cooldown. A technique was introduced for marking the crystal surface by sending a pulse of electricity every 4 sec through the growing crystal into the molten sample. The Peltier cooling at the interface caused a fluctuation in growth rate, which produced an increase of dopant on a microscopic scale. Slices of the crystal were etched to reveal fine lines marking the crystal interface at each pulse occurrence.

In the postflight analysis, the investigators note that the Ge crystal slipped easily from the quartz tube, indicating that wetting did not occur in low-g, although in 1-g wetting takes place. The investigators suggest that this wetting inversion in space-grown crystals may drastically reduce contamination of the melt by their confinement materials. The growth rates of the low-g and 1-g crystals were virtually identical, assuming a value of about 9.5 μm/sec after about 2.5 cm of growth. Development of a modified segregation theory, which considers the existence of initial growth rate transients, has been initiated by the investigators as a result of data obtained from this experiment. Dopant concentration increases steadily over about 1.5 cm from the regrowth interface but does not reach the anticipated steady-state prediction. This behavior is not fully understood, but it appears that asymmetrical

thermal gradients arising from the three furnaces in the module are most likely responsible for the variations in growth rate and dopant distribution. The investigators insist, on the basis of their analysis of the dopant segregation and compositional homogeneity of the samples, that no time-dependent convective flows occurred in the Ge melts even though there was little or no contact between the melt and the container. This result illustrates the importance of establishing a thermal configuration producing planar or near-planar solidification fronts to achieve radial compositional homogeneity during crystal growth under diffusion-controlled, near zero-g conditions.

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ZERO-G PROCESSING OF MAGNETS

Experiment MA-070

Principal Investigator: Dr. D. J. Larson
Grumman Aerospace Corporation

The purpose of this experiment was to investigate the effects of reduction of gravitationally dependent elemental segregation and convection in the solidification of high-coercive-strength magnetic composites in low-g.

The three sets of three samples were processed simultaneously in the MA-01 furnace, each furnace cartridge containing three sample ampoules. One ampoule containing Bi and Mn (each 50 atomic percent) was located in the hot end of the furnace and isothermally brought to 1348 K, held there 45 min, allowed to cool to 673 K over 10.5 hr, and then cooled quickly by helium insertion. The other two ampoules within the cartridges were subject to lower temperatures and longitudinal temperature gradients. In the ampoule containing a Cu-$(CuCo)_5$ Ce mix, the peak temperature was 1323 K, but variable gradients around 30 K/cm were experienced. A peak temperature of 1123 K was reached in the sample ampoule containing a mix of 97.8 percent Bi and 2.2 percent Mn with gradients of 10 to 80 K/cm.

The array of MnBi crystals processed isothermally apparently resulted from edge-to-center gradients and produced no unusual magnetic effects. The Cu-$(CuCo)_5$ Ce reacted with the boron nitride crucible walls and solidified in crystals extending inward from the walls. Again, no unusual magnetic effects were observed. The directionally solidified flight samples showed considerably different microstructure from the ground control samples. The MnBi rods were smaller in diameter and were circular in cross section rather than chevron-like. Some difference was also noted in the lattice parameter. The most remarkable difference was an improvement of approximately 60 percent in coercive strength. In fact, the space processed samples appear to have a coercive strength higher than any previously recorded for MnBi processed in the as-grown state. It is still not completely clear why the flight sample differed in microstructure and magnetic performance, but recent ground-based experiments are beginning to suggest an interesting possible explanation. For example, it has been recently found that the MnBi eutectic composition was slightly different than presumed. The flight experiment was actually slightly off-eutectic (Bi-rich). In the ground control sample the convective stirring apparently caused a continuously variable amount of the MnBi compound to be solidified, which charged the rod diameter along the sample. A steady state growth was apparently achieved in the diffusion controlled flight sample which

resulted in smaller than normal rod diameters which may have been more nearly the optimum size for elongated single domains. Also, the gradient freeze growth utilized in the ASTP furnace subjects the solidified material to a much longer high-temperature soak than the conventional stockbarger technique. This heat treatment has been found to promote the formation of the magnetic phase which exhibits high coercivity at ambient temperatures.

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CRYSTAL GROWTH FROM THE VAPOR PHASE

Experiment MA-085

Principal Investigator: Dr. H. Wiedemeier

Co-Investigators: H. Sadeek, F. C. Klaessig, M. Norek, and R. Santandrea
Rensselaer Polytechnic Institute

This experiment, like the Skylab experiment M-556, was designed to study the growth of semiconductor crystals by chemical transport reactions using a vapor transport agent in a low-g enviornment. Using the same basic experimental procedure with different transport gases, the investigators wished to confirm their previously reported result of improved crystal morphology in low-g vapor growth and to study the unexpectedly high mass transport observed in the M-556 experiment.

Three stainless steel cartridges for the MA-010 furnace contained sealed quartz tubes with sample materials held in place by thin silica rods. $GeSe_{0.99}Te_{0.01}$ and the transport agent GeI_4 occupied one ampoule. The other two ampoules held GeS and $GeS_{0.98}Se_{0.02}$ with different quantities of $GeCl_4$ transport agent. In one of these ampoules argon was added to elevate the pressure by about a factor of three. The other two ampoules were at slightly less than 1 atmosphere pressure. After a 2-hour heat-up period, the hot end was an isothermal region of approximately 1.5 cm at 604°C followed by a nearly linear gradient toward the cool end at 507°C. These temperatures were held constant in low-g and 1-g experiments for 16 hr prior to cooldown.

No difference was found in the lattice parameters and the orientation of the native growth faces of the crystals formed in low-g and 1-g. Based on their analysis of the deposition patterns, the investigators concluded that the turbulent flow characteristic of 1-g growth did not exist in the low-g environment. The majority of the 1-g grown crystals were reported to have ragged edges, twinning, and frequently stepped faces. The low-g grown crystals have smoother surfaces and better-defined edges. An average density of etch pits for the space-grown crystals was reported to be one or two orders of magnitude less than the crystals grown in 1-g. As in the earlier M-556 experiment, the improvements noted in the structural and chemical homogeneity of the low-g grown crystals are attributed to the reduced convective turbulence and interference with the transport process. In addition to the confirmation of greater mass transport rates in low-g than predicted, the investigators suggest that the flux results indicate the existence of thermochemically induced convection in reactive solid-gas phase systems. They suggest that this "thermochemical" convection may be overshadowed by gravity-driven convection in 1-g.

The principal investigator is continuing to study the vapor growth of crystals of semiconductor materials in Shuttle experiment M77082.

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HALIDE EUTECTIC GROWTH

Experiment MA-131

Principal Investigator: Dr. A. S. Yue

Co-Investigators: C. W. Yeh and B. K. Yue
University of California at
Los Angeles

The growth of the LiF fibers in NaCl was investigated in this experiment. The principal investigator's Skylab M-564 experiment had produced long fibers of NaF in NaCl when a mix of the two was melted and directionally resolidified in low-g. Well-aligned, continuous fibers of LiF could be used in fiber optics work for transmission of infrared light 3 to 12 μ in wavelength. Convective disturbances in 1-g can produce defects in the fibers which limit their usefulness. The low-g environment, by reducing the convective effects, could lead to the production of fibers having more desirable characteristics.

Samples were prepared using 99.96 percent pure NaCl and 99.99 percent pure LiF to produce eutectic ingots, 29 percent weight LiF, by melting and solidification in graphite crucibles and an argon atmosphere. Samples were held in stainless steel graphite-lined ampoules with an evacuated expansion cavity at one end. The multipurpose gradient furnace was used in low-g to produce melt-back and resolidification of three samples simultaneously. Another set of three samples was processed in 1-g under equivalent temperature conditions. The hot end temperature reached 1293 K while the cool end remained below 1073 K, the melting point of NaCl, with a gradient of about 50 K/cm. The controlled cooling rate was 0.6 K/min. The fibers grown were analyzed to determine length, diameter, spacing, and optic properties.

The investigators reported that fiber length in portions of the low-g samples showed a many-fold increase over their 1-g fibers. Transmittance of the low-g fibers was reported to exceed that of the 1-g fibers by several fold over most of the wavelength band from 4 to 10 μm. Improved image transmission was also noted. The fibers diameters and interfiber spacing of the directionally solidified low-g and 1-g fibers were reported to show no significant differences.

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MULTIPLE MATERIALS MELTING (METALS)

Experiment MA-150 (Part 1)

Principal Investigators: L. I. Ivanov, V. N. Kubasov, V. N. Pimenov, K. P. Gurov, E. V. Demina, Yu. S. Malkov, and V. M. Lazarenko
Institute for Metallurgy, USSR

This experiment was designed to utilize the low-g environment to reduce gravity-driven segregation effects in the synthesis of compound materials of significantly different specific gravity. It was also designed to investigate the mutual diffusion and formation of intermetallic phases as a result of interaction of a meltable matrix (Al) and hard, refractory inclusions (W). The melting and solidification of powdered Al was also to be investigated in low-g. Additional studies were made on microgravity melting and solidification of copper aluminum (CuAl) eutectic alloy as it interacts with Al, as well as on the diffusion and formation of intermetallic phases in the tertiary system of solid W, solid rhenium and liquid Al.

Both flight and ground-based samples of all types were melted in the multipurpose furnace. The systems of W-Al and W-Al-Re were soaked at peak temperature for 1 hr prior to cooldown. All samples underwent controlled cooldown followed by passive cooling and a final He injection.

The investigators reported that the kinetics of diffusion and phase formation in the solid W (WRe alloy)/liquid Al diffusion area was approximately the same for both ground-based and flight samples. This conclusion was suggested by the similarity in geometrical characteristics of the compared diffusion layers as well as by the analogous phase composition and distribution of the diffusion layer constituents which they observed. The low-g diffusion and phase formation processes for the W-Al, however, were reported to exhibit several differences from the W-Al-Re system. In the binary system, the WAl_5 phases are thinner than in 1-g, are needle-shaped, and have a lower distortion angle; the WAl_{12} phase has a crystalline faceting. In the case of the tertiary system, the distinguishing features were reported to include a higher porosity at the diffusion layer/WRe alloy interface, and the formation of phases rich in Al. The CuAl eutectics and powdered Al low-g samples showed no appreciable difference from their Earth-processed counterparts according to the investigators. Melting of the powdered Al suggested to the investigators that slight excess of temperature above the Al melting point reduces the possibility of individual particles being fused together in a low-gravity orbital environment.

Germanium-silicon solutions were also investigated in experiment MA-150. These results are reported in the MA-150 (Part 2) summary.

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GERMANIUM-SILICON SOLID SOLUTIONS

Experiment MA-150 (Part 2)

Principal Investigators: V. S. Zemskov, V. N. Kubasov,
I. N. Belokurova, A. N. Titkov,
I. L. Shulpina, V. I. Safarov,
and N. B. Guseva
Institute for Metallurgy, USSR

This part of the MA-150 experiment on melting and directional crystallization of an antimony (Sb)-doped germanium (Ge) silicon (Si) solid solution was designed to study the possibility of using microgravity conditions for obtaining solid solution monocrystals with uniformly distributed components. Solid solution monocrystals obtained in a microgravity environment in the absence of buoyancy forces and thermal convection were expected to have a more homogeneous and even distribution of components compared to those obtained under normal gravity conditions. It was anticipated that the absence of convectional mixing and the occurrence of mass transfer in the melt, caused by diffusion only, would create conditions for stationary crystal growth and would provide a compositional segregation consistent with these conditions. Also, in the absence of uncontrollable convective mixing, laminar distribution of components in a monocrystal is not likely to occur.

Antimony-doped Ge-Si solid solutions were chosen for studying these processes because the distribution coefficient of Si in Ge is greater than unity and that of Sb is less than unity. This enables investigation of the behavior of components having different distribution coefficients. Also, because Sb-doped Ge-Si solid solution is a semiconducting material, the precision techniques that are widely used in studying semiconducting materials can be used for a detailed investigation of component distribution in crystal volume and for analyses of defects in crystal structure.

Cylindrical ingots of Sb-doped Ge-Si solid solution were made of monocrystals obtained by pulling from a melt which was continually fed with Si. These samples were inserted into ampoules, placed in the gradient zone of the multipurpose gradient furnace, and heated. The gradient along the ampoule was 30 to 40 K/cm during the molten state and from 10 to 40 K/cm during the cooldown phase. The peak hot and cool end temperatures were intended to be 1323 K and 973 K. A partial melt-back was planned, followed by directional solidification.

All the flight samples melted completely; therefore, the crystallization in low-g did not occur under the expected ideal stationary growth and segregation conditions. The temperature at the cool end of the ampoule, where the seed was intended to be, was reported to be greater

than 1211 K. Regeneration of the seed occurred in the low-g samples, but polycrystalline structure resulted. The investigators report that convective mixing was negligible in the low-g environment and that the graphite ampoule walls were not wet by the molten samples.

Part 1 of this experiment, dealing with melting of multiple materials (metals), is reported separately.

References

Ivanov, L. I.; Kubasov, V. H.; Pimenov, V. N.; Gurov, K. P.; Demina, E. V.; Malkov, Uy. S.; and Lazarenko, V. M.: Multiple Materials Experiment MA-150. Apollo-Soyuz Test Project — Summary Science Report, NASA SP-412, Vol. 1, Johnson Space Center, 1977, pp. 501-538.

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VII. APOLLO-SOYUZ DEMONSTRATION EXPERIMENTS

The purpose of these demonstration experiments was to utilize the low-gravity environment to study various interfacial phenomena, such as chemical foaming, spreading of liquids, and capillary wicking in the absence of gravitational forces which tend to interfere with the measurements.

The experiments are summarized by R. S. Snyder, "Science Demonstrations," Apollo-Soyuz Test Project, Preliminary Science Report, NASA TMX-58173, Johnson Space Center, February 1976.

CHEMICAL FOAMS

ASTP Science Demonstration

Principal Investigator: Dr. P. G. Grodzka. Lockheed

The objective of the chemical foaming experiment was to investigate the stability of a liquid foam in the absence of liquid draining from thin liquid walls. It was also postulated that the increased stability and surface area might influence the rate at which chemical reactions take place.

The stability of a foam was investigated by shaking a mixture of water, ethyl alcohol, and thymol blue indicator. This produces a pink foam which contrasts sharply with the gold of the bulk liquid, allowing the dissipation of the foam to be observed by the data acquisition camera. The reaction rate experiment utilized a series of reactions between sulfite, sodium metabisulfite, formaldehyde, and phenol phthalein. This reaction produces excess OH^- to react with the indicator only after all of the bisulfite ion has reacted. Therefore, after the components are vigorously shaken to mix them and to produce the foam, a sudden color change occurs after a delay of some 30 sec required for the reaction to go to completion. This delay was to be measured in the flight experiment and compared with the ground control experiment to determine if there is any difference in reaction rates.

Unfortunately, the 16-mm camera was badly out of focus, which precluded obtaining any definitive data on the dissipation of the foam. The reaction rate data were also lost for the same reason, although the astronauts' description indicated a unique morphology of the reaction in the flight samples. Flights in the KC-135 indicated somewhat faster reaction rates in the low-g tests than in the ground control tests.

LIQUID SPREADING

ASTP Science Demonstration

Principal Investigator: Dr. S. Bourgeois, Lockheed

The object of the liquid spreading experiment was to investigate the spreading of liquids over solid and liquid interfaces and to measure the shape of the spreading liquid and the rate of spreading.

Small amounts of oil and/or water were injected onto the inside bottom of a transparent cube. One surface of the cube had been treated to be nonwetting. This surface faced the camera. Red and blue dyes were added to the oil and water to increase the visibility.

Only one experiment was carried out and, again, the poor quality of the photography did not allow a definitive analysis of the shape or spreading rate to be made. The oil spread uniformly up the walls of the box and collected along the corners to minimize the exposed surface area. The addition of water on the oil surface produced a repulsion of the oil, resulting in an equilibrated configuration of water droplets surrounded by an oil film.

CAPILLARY WICKING

ASTP Science Demonstration

Principal Investigator: A. F. Whitaker, MSFC

The wicking experiment was designed to illustrate wicking action in a weightless environment. Also, it was hoped to determine the efficiency of transfer and wicking rates of stainless steel wicks used for fluid management in spacecraft.

Three stainless steel wicks and one nylon cloth wick were used simultaneously with two fluids; a water-soap solution, and a silicone oil. The stainless steel wicks were a 325 X 2300 Dutch twill weave, a 200 X 200 Plain weave, and a 200 X 600 Plain Dutch weave. The nylon wick was added for comparison.

Wicking of both oil and water proceeded much faster in the ASTP than anticipated on the basis of ground tests and KC-135 flight tests. The liquid was observed to rise along the corner formed by the Teflon support back and the mesh. Since Teflon is not normally wetted by the fluids used, this behavior was unanticipated and serves to illustrate how unexpected fluid behavior can occur in weightlessness.

VIII. SUMMARY OF WHAT WAS LEARNED

In the area of crystal growth the significant findings are as follows:

1) Control of macrosegregation in melt growth has been demonstrated. It was shown that a steady state diffusion layer can be established over a short growth distance, resulting in a macroscopically uniform dopant distribution over most of the length of the boule. The distribution of dopants in the Witt and Gatos Skylab experiment resembles the classical text book description of diffusion-controlled growth.

2) Elimination of microsegregation due to growth rate fluctuations has been demonstrated. This indicates that it may be feasible to grow some of the higher composition alloy semiconductors such as HgCdTe and PbSnTe from the melt without interfacial breakdown resulting from uncontrolled growth rate fluctuations and under conditions where defects resulting from the growth process may be eliminated.

3) Seeded containerless growth has been demonstrated. Extremely flat surface facets were formed, indicating that the crystalline ordering forces dominated over surface tension forces. Elimination of the container also resulted in a substantial reduction of strain-induced defects.

4) Crystals grown by chemical vapor transport in low-g showed improved growth habit, surface morphology, and lower defect density than those grown on Earth. These differences imply a more uniform growth environment in the absence of gravity-driven convection.

5) Observed growth rates by chemical vapor transport in low-g were substantially higher than expected on the basis of extrapolation of laboratory data from the low pressure regime where convective effects were thought to be insignificant. This indicates a fundamental lack of understanding of the transport mechanisms and their dependence on gravity-driven flows.

6) The ability to produce and maintain extended floating zones was demonstrated. Predicted rotational liquid surface instabilities were confirmed experimentally, and an unexpected nonaxisymmetric instability mode was discovered.

7) There was no evidence of surface tension-driven flows in some of the high-temperature melts that apparently had free surfaces.

In the field of metallurgy, the significant findings are as follows:

1) Welding and brazing can be performed in space. Voids due to trapped gases were effectively prevented. The observed microstructure of weldments in low-g was different from that observed in unit gravity and, in fact, was contrary to what was expected. This is probably due to the absence of sedimentation of the dendrite arms broken off by the flow induced by the e-beam.

2) A number of experiments were designed to produce composites with uniformly dispersed second phases. It was shown that buoyancy effects were effectively eliminated but that close attention must be paid to sample preparation, elimination of gases, and control of solidification rates in order to achieve uniform distributions.

3) Unexpected agglomeration of some immiscible metallic systems occurred during solidification. This indicates that there are strong driving forces for droplet growth or coalescence in the Al-In system, for example, which act in the absence of appreciable buoyancy and are much too fast to be a diffusion effect. One hypothesis at present is that this massive agglomeration is a constitutional effect.

4) Attempts to understand some of the results obtained in the flight experiments have prompted the reexamination of published phase diagrams of several systems. Several refinements were established; e.g., the previously accepted value for the consolute temperature of Pb-Zn was low by 20 K and the eutectic composition of Mn-Bi was found to be 2.6 atomic percent Mn and 97.4 atomic percent Bi.

5) The low-temperature, high coercive strength magnetic phase of MnBi has been studied in detail and a new ordering effect at high magnetic intensities has been discovered. The enhanced magnetic performance of the MnBi samples processed in space is now thought to be the result of finer rod dimensions of the MnBi phase obtained by directionally solidifying an off-eutectic composition that remained uniform in the absence of convective stirring.

In the area of fluid phenomena there appears to be considerable confusing and conflicting data, indicating that there is still much to be learned about the behavior of fluid in a low-gravity environment. The present understanding of these phenomena is summarized as follows:

1) Although a few of the experiments were apparently disturbed by vehicular accelerations, crew activity, or inadvertant crew contact, most of the experiments did not seem to be adversely influenced by the residual g-levels of Skylab and ASTP. There was no evidence of major bulk flows from the random acceleration environment associated with the manned missions, in which the accelerations tend to average to zero.

2) Anomalous wall contact behavior was observed in a number of solidification experiments in which the solid pulled away from the wall

even though the liquid presumably wet the container. In one case the type of dopant atoms present in trace quantities seemed to influence this behavior. Whether such effects result from different wetting behavior in the absence of hydrostatic pressure, the presence of trapped gases, or from volume change phenomena during solidification is not clear.

3) There is still much to be learned about the importance and control of surface tension-driven flows in low-g. The Heat Flow and Convection Experiment flown on Apollo demonstrated unstable surface tension-driven flow in an oil film with a free surface with a destabilizing perpendicular thermal gradient. Surface tension-driven flows resulting from concentration gradients were suggested to explain the observed mixing in the Reed experiment on ASTP, although not conclusively demonstrated. However, there was no evidence of surface tension-driven flows in the crystal growth experiments in which the solid pulled away from the wall.

4) Some anomalous effects were noted in the diffusion experiment of Ukanwa and in the simple Skylab demonstration experiment using a mixture of tea and H_2O. Although the expected diffusion profile was obtained near the center of the sample, this profile was distorted near the walls as though the diffusive transport was retarded.

5) Long-term stability of a fine dispersion of oil and H_2O was demonstrated. This indicates that low-level residual accelerations or other effects do not cause significant agglomeration in an isothermal environment.

6) Free column electrophoresis was successfully demonstrated. Methods for controlling nongravitational flows due to electroosmosis were developed.

In many cases these first experiments were constrained by available resources: development time, flight facilities, power, on-orbit processing time, processing environment, etc. Ambiguities still exist because many of the results were unanticipated and could not be investigated with sufficient detail with the available experiment designs and instrumentation. However, these experiments have provided an extremely valuable first step in the learning process and form an essential background for the next generation of space experiments.

APPROVAL

SUMMARIES OF EARLY MATERIALS PROCESSING IN SPACE EXPERIMENTS

By Robert J. Naumann and E. Darby Mason

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

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