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Microscopy & Microanalytical Support for NASA's Microgravity Materials Science Programs

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The Materials Science Program of NASA's Office of Biological and Physical Research (OBPR) has attacked fundamental problems of materials science as defined by an external advisory group, known as the Discipline Working Group (DWG). These have been:

- Nucleation and Metastable States
- Prediction and Control of Microstructures
- Crystal Growth and Defect Generation
- Phase Separation and Interfacial Phenomena
- Thermophysical Properties and Process Modeling.

While the program's primary objectives are science-based and despite the fact that 90% of these programs concentrate on pre-cursor and theoretical research on the ground, NASA demands that there be a microgravity rationale driving the research. With NASA's recent directive to focus on exploration, the program now includes work on:

- *In Situ* Fabrication and Repair (IFSAR)
- Materials for Radiation Shielding, and
- *In Situ* Resource Utilization.

High quality microscopy and microanalysis are essential for the success of both the fundamental and the new ventures. Examples will be given for some of these categories.

Nucleation and Metastable States refers to the study of the mechanisms of transformation of liquids to solids, often in an undercooled state, and to the formation and examination of novel structures such as quasicrystals and icosohedral phases. Among NASA's interest is the potential of levitating samples free of nucleating walls and hence obtaining high undercooling in quiescent melts. Microgravity allows levitating and positioning of samples

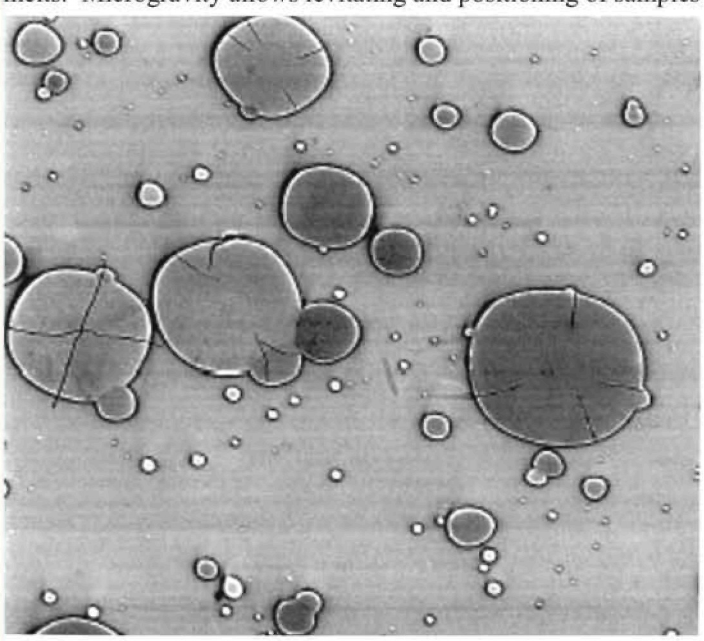

Figure 1. Supercooled glass in the Y_2O_3-Al_2O_3 system. This backscattered image shows the second liquid phase or "spheroid." Note that the crack propagates through the interface region into the matrix

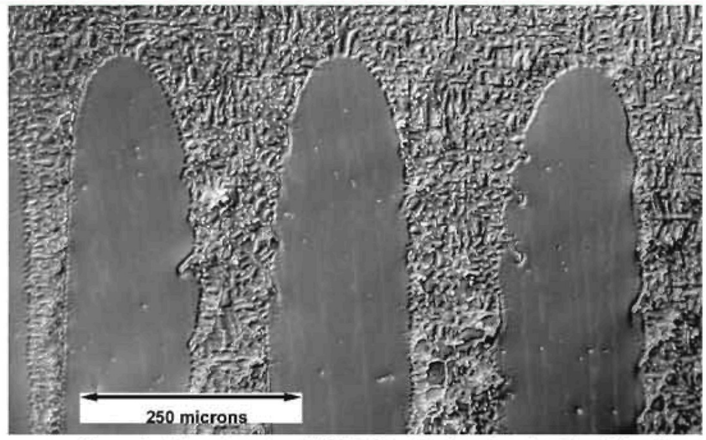

Figure 2. Directionally solidified Cu-Al alloy showing dendritic Al-rich phase in a (quenched) eutectic background. Photograph courtesy of R. Grugel, NASA.

to be done with low forces. On Earth, much work can be done in levitators (electromagnetic, acoustic, or electrostatic), but vigorous stirring and sedimentation still influence the material's behavior. Nonetheless, many interesting structures can be produced. An example is the supercooled glasses in the Y_2O_3-Al_2O_3 system, where there is a dramatic change in viscosity with temperature, as a result of the liquid changing from one to two phases (Figure 1) [1]. Electron microprobe results show that the two phases have identical compositions, demonstrating that this is a diffusionless transformation. A microgravity experiment was planned with the intent of examining the kinetics of the reaction in the absence of buoyancy and convection. These glasses have exceptionally high optical transmission and can be in optical and photonic devices, including high power lasers and surgical probes.

Prediction and Control of Microstructures. Microstructure is a principal factor in establishing the properties of a material, and so its control is a major goal of the materials scientist. As an example, the reduction of the number of grain boundaries will aid in increasing the strength and lifetime of a material during operation at elevated temperatures. While single crystal constitute one means of achieving this, it is often more economical to solidify materials directionally, particularly in multi-phase alloys. Such alloys often include a "mushy" zone, a zone of liquid and solid intermixed. Defects in the final structure will arise if the control parameters are unable to maintain a quiescent environment within the liquid.

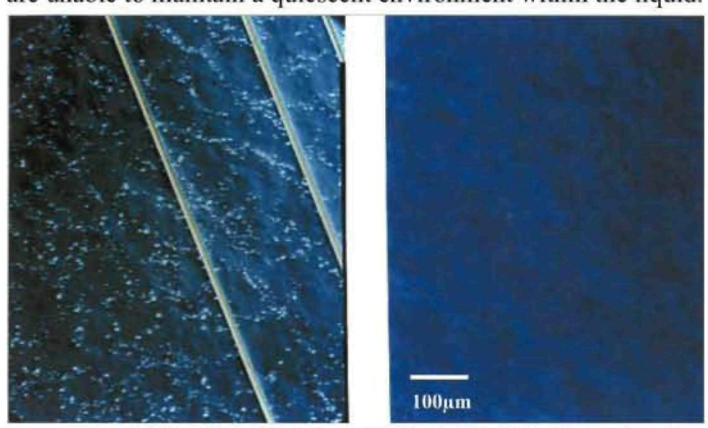

Figure 3. The effect of "detached solidification" in CdZnTe. On the left is shown a terrestrial grown crystal with an etch pit density of $10^4 cm^{-2}$, and twins propagating from the walls. On the right is a crystal grown on the Space Shuttle, where the materials has pulled away from the walls. There is only one etch pit in the filed of view representing a density of 800 cm^{-2}.

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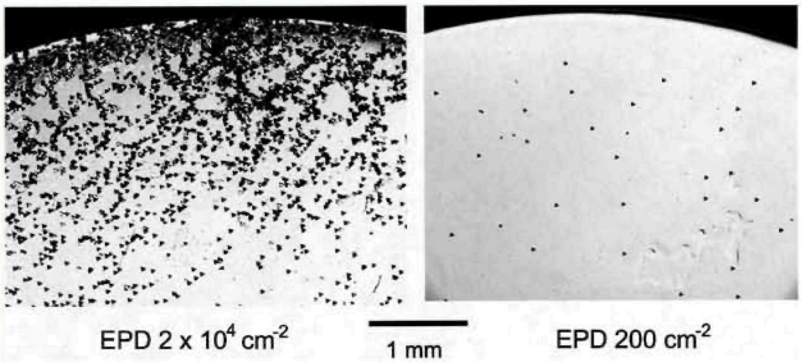

Figure 4. Quality of germanium-silicon alloy crystals is strongly affected by wall contact in directional solidification in an ampoule. The density of etch pits illustrates the effect of detachment.

NASA has studied the boundary condition under which some of these defects can arise. The mushy zone/dendritic structure shown in Figure 2 is of a quenched Al-Cu alloy solidified directionally, which emulates a high temperature alloy. Of importance here, is the control of the structure, and the conditions under which the structure changes from cellular to dendritic or the degree to which secondary arm dendrites form and trapped liquid leads to bulk imperfections [2]. Composition measurements at the dendrite tip and within the adjacent quenched liquid are used to establish how effectively the quenched structure represents the real-time solidification situation.

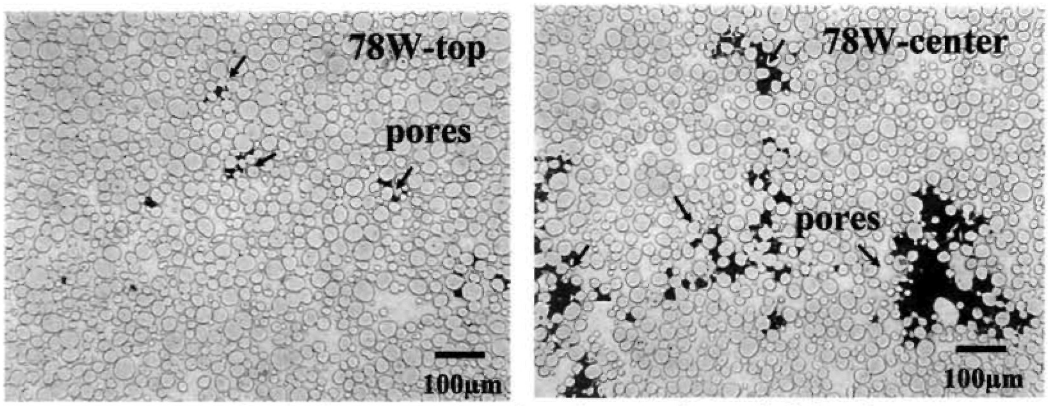

Figure 6. W particles (78%) in a Fe-Ni matrix identically processed on Earth (left), and in microgravity (right). Note the difference in porosity, and the size of the pores. The terrestrial sample is near the top, while the microgravity sample is in the center.

Crystal Growth and Defect Control. Growth of semiconductor crystals has been much studied by NASA in microgravity where the elimination of convective forces leads to an understanding of the nature of defects, including segregation and lattice defects. Another benefit of microgravity is that, provided that the molten charge does not produce a high vapor pressure, there is no hydrostatic pressure present on a solidifying sample. Thus, a solidifying sample can pull away from its containing ampoule wall with significant reduction in defect concentration. Figure 3 demonstrates the difference between CdZnTe grown on Earth and on the USML-1 Spacelab mission. Etch pits produced by a lactic acid-based etch reveal the density of line defects in the sample. In this case the reduction has been from $10^4/cm^2$ to a few hundred per sq cm. There is in fact only one triangular etch pit visible in the space grown sample. Figure 4 show some ground-based work on germanium-silicon samples where solidification against the wall has resulted in a high defect density [3]. By adjusting pressure balances it may be possible to achieve "detached" solidification with a much reduced defect concentration. The presence of such a high defect density leads to inferior electronic properties in what is becoming an important semiconductor material. The conditions of pressure control to produce 100% detachment are being investigated. This is an example of the development of knowledge gained through space flight experiments being applied to ground based research.

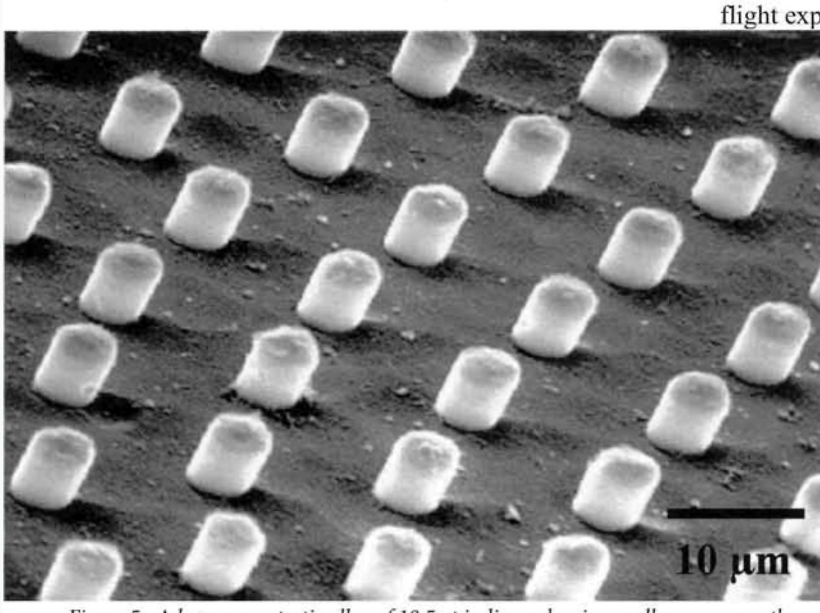

Figure 5. A hypermonotectic alloy of 18.5wt indium-aluminum alloy grown on the Life and Microgravity Sciences space mission. The structure shows aligned indium fibers in a In-Al eutectic background. Steady state growth has been achieved in microgravity.

Phase Separation and Interfacial Phenomena. The importance of the control of interfaces is shown in Figure 5. This is an example of an indium-aluminum alloy that includes a monotectic phase [4]. Unidirectionally cooled samples can form as two liquids with the rodlike structure shown here. In this case, the rods are indium and formed as liquid strands running through the material. Such structures can be tuned to give exceptional properties, as for instance with one superconducting phase within an insulator. Alternatively, one phase can be dissolved away to produce multiple tubes of controlled dimensions. The example shown was grown in space, where convection can be eliminated and the limiting conditions for producing such a well-behaved structure determined.

***In Situ* Fabrication and Repair.** Long duration space missions will require the use of natural resources where possible. In addition, the need arises to fabricate spare parts on a just-in-time basis. A strong candidate for materials parts fabrication is the use of liquid phase

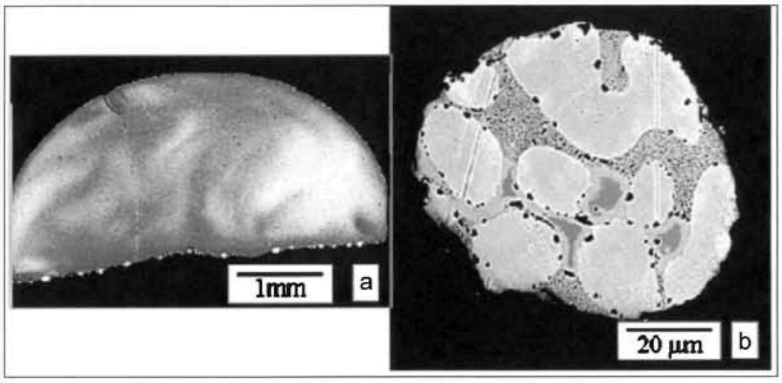

Figure 7. Mars simulant melted and quenched to a glassy bead. Figure 7a is a backscattered image showing Fe concentration, while 7b illustrates the P content of the FeP composite grain.

sintering (LPS). Recent research in microgravity by Randall German at Penn State University has determined the experimental boundary conditions that enable parts to be densely sintered and still maintain close engineering tolerances [5]. A potential problem with processing in microgravity is the ability to control or eliminate porosity in the absence of buoyancy. German has photographically catalogued the behavior of pores during the entire process of LPS. Figures 6a and 6b demonstrate pores in the ground-based and flight samples respectively. In planned International Space Station experiments the sintering process will be interrupted at intermediate times and temperatures to test the perceived history of sintering.

***In Situ* Resource Utilization.** The possibility of using natural resources, especially the surfaces of the Moon or Mars, entails much precursor work to evaluate chemical and physical properties. Possible uses include the fabrication of bricks for habitation, and the extraction of raw materials, from metals to oxygen to glasses. A glassy volcanic ash is used as a simulant for Martian soil. Preliminary work was reported recently that illustrates the type of work that may be anticipated [6]. The ash was melted in a series of experiments including a test with graphite present. Typical microstructures of the processed material are shown in the backscattered electron images, Figures 7a and 7b. These show a quenched basalt glassy bead. The image contrast is the result of Fe concentration. Figure 7b shows a FeP composite grain with decreased brightness representing increased phosphorus content of the FeP phases. In some of these tests spherules of iron were observed.

Current NASA research projects are available on the Office of Biological and Physical Research website [7]. ■

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