In Situ Monitoring of Crystal Growth Using MEPHISTO

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A solidification experiment for a bismuth 1 atomic % tin alloy was conducted as a part of the Fourth United States Microgravity Payload (USMP-4) Space Shuttle mission aboard the STS-87 flight of *Columbia* from November 19 through December 2, 1997. The solidification experiments for this faceted alloy used an apparatus developed by Centre National d'Etudes Spatiales (CNES, Toulouse, France) and Commissariat à l'Energie Atomique (CEA, Grenoble, France) called MEPHISTO, which stands for Materiel pour l`Etude des Phenomenes Interessant la Solidification su Terre et en Orbit apparatus. The apparatus was developed for investigating directional solidification of metallic alloys and doped semiconductors. The research program was conducted with the collaboration of a multi-national team from the University of Florida, NASA-Lewis Research Center (NASA LeRC), the National Institute of Standards and Technology (NIST) in USA, CNES, Départemente d'Etudes des Matériaux at CEA, the Société Européene de Propulsion (SEP) in France, and the University of New South Wales in Australia.

The purpose of these experiments was to study the morphological stability (plane front to cellular transition) and the solidification/melting kinetics of a faceted material. During the previous space experiment it was found that the distance to the planar-cellular transition was not the same for neighboring grains (a bismuth 0.1 atomic % tin alloy) with different orientations. In one case a grain grew approximately 0.6 mm before breakdown, while a neighboring grain grew 12 mm before breakdown. These results suggest that kinetics and growth anisotropy have a marked influence on the stability of planar solidification in a faceted material. To extend our understanding of these effects additional experiments were performed aboard USMP-4 using a bismuth 1 atomic % tin alloy. The experiments involved repeated melting and solidification of three samples, each approximately 85 cm long and 6 mm in diameter. Half of each sample also included a 2 mm diameter growth capillary, to assist in the formation of single grains inside. One sample provided the Seebeck voltage generated during melting and freezing processes. Another one provided temperature data and Peltier pulsed demarcation of the interface shape for post-flight analysis. The third sample provided resistance and velocity measurements, as well as additional thermal data. The third sample was also quenched at the end of the mission to preserve the interface composition. The entire flight experiments were commanded and controlled via telemetry from the NASA Marshall Space Flight Center's Payload Operational Control Center.

Seebeck solidification and melting cycles were completed over a range of velocities as low as 2.67 millimeters per hour (mm/hr) to as high as 144 mm/hr. 150 mm of each sample has been directionally solidified with different velocities from 6.6 through 144 mm/hr. Peltier pulses were successfully performed in five regions of the Peltier sample. The resistance sample was quenched at the end of the mission. The Seebeck cycles were varied not only in velocity, but also in distance solidified or melted. We have recovered 450 mm of directionally grown samples under microgravity environment. The samples are currently being analyzed for microstructural and compositional variations as a function of growth conditions.

The Seebeck signals are also being analyzed as a function of growth parameters. It is found that the signals depend not only on the interface velocity and growth distance, but also on the structure of the solid forming behind the interface. These findings have also been verified by measuring solid-state Seebeck signals for the space-grown samples. The Seebeck voltage analysis has also included the solute build up during freezing, decay of the solute build up during holds, and comparison of solidification and melting cycles. Both the magnitude and the transient behavior of the Seebeck signal yield valuable insight into the growth and melting behavior of faceted interfaces. By appropriate normalization of the Seebeck signal, a relationship between the Seebeck signal and growth velocity is determined.