

Crystal Growth Using MEPHISTO

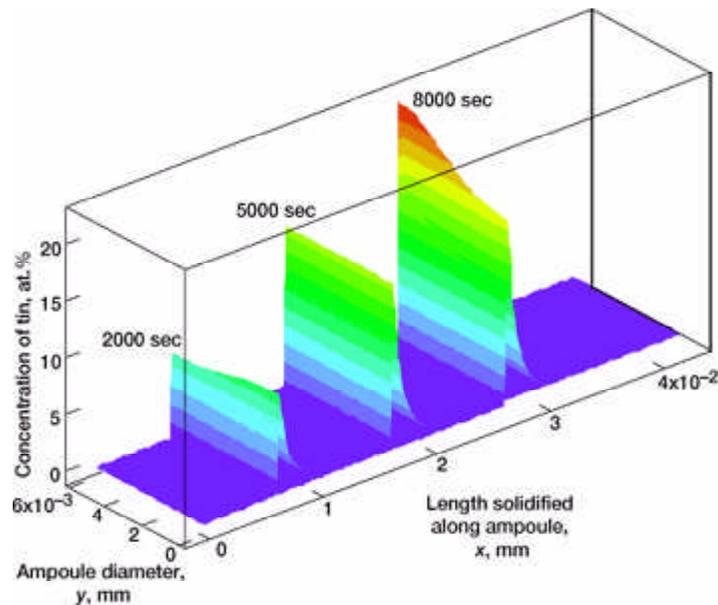
The shuttle flight experiment "In Situ Monitoring of Crystal Growth Using MEPHISTO" was accomplished during STS-87 as part of the fourth flight of the United States Microgravity Payload (USMP-4), which was flown from November 19 to December 5, 1997. The data returned from that flight are just now beginning to yield quantitative results. This project is an international collaboration: the furnace system known as MEPHISTO was built in France by CNES (French National Space Agency) and CEA (French Atomic Energy Commission); the principal investigator, Prof. Reza Abbaschian, is from the University of Florida at Gainesville; and numerical and analytical modeling support includes collaborators from the University of New South Wales, Australia, the University of Wisconsin at Milwaukee, the National Institute of Standards and Technology, and the NASA Lewis Research Center. MEPHISTO is a French acronym that translates into English as *Materials for the Study of Interesting Phenomena of Solidification on Earth and in Orbit*. Since this was the fourth flight of the MEPHISTO furnace, the experiment is referred to as MEPHISTO-4.

MEPHISTO-4 was a directional solidification experiment that studied the liquid-to-solid transformation of bismuth alloyed with tin. Directional solidification is a freezing technique common to the processing of the electronic materials used in integrated circuits and detectors, such as silicon and germanium. When liquids are frozen on Earth, they must be cooled. The cooling causes stirring because of density variations in the liquid (ref. 1). This stirring, known as natural convection, influences the quality of the resulting solid. During freezing, regions of high and low concentrations of tin are created. This introduces another important phenomenon: diffusion, or the movement by molecular action of matter from regions of high concentration to regions of lower concentration. In MEPHISTO-4, it is tin that diffuses from the high-concentration region in front of the solid-liquid interface to more distant low-concentration regions. Finally, the concentration gradients of tin also cause density variations, and hence are another source of stirring of the liquid on Earth. The interplay among temperature, concentration, convection, diffusion, and solidification becomes extremely complex, and a complete understanding of solidification is beyond the present state of the art.

In MEPHISTO-4 the microgravity environment is being used to greatly reduce convection effects in the solidification process. In this way, preliminary theories describing solidification in terms of diffusion-only can be examined, and the effects of diffusion can be more readily assessed than is possible on Earth, where natural convection swamps the more delicate diffusion phenomena. When theories that include only diffusion have been tested using the flight data, more advanced theories that include both diffusion and convection can be developed and tested using both flight and Earth-based data.

All pertinent aspects of the MEPHISTO-4 experiment were monitored and controlled remotely through NASA's Payload Operations Control Center located at the NASA Marshall Space Flight Center. To monitor all the parameters needed for the study of solidification, three samples were processed identically and simultaneously in the

MEPHISTO furnace: one used the Seebeck technique (refs. 2 and 3) to measure the temperature at the interface between the solid and liquid; the second employed a pulse of electricity to mark the shape of the solid/liquid interface; and the third measured the rate of solidification by monitoring the electrical resistance of the sample. This third sample was also quenched at the end of the experiment to freeze the concentration gradients built up in the liquid near the solid/liquid interface. As part of the analysis of the flight sample, this quenched concentration profile will be measured and used to calculate a diffusion coefficient for tin in liquid bismuth. The diffusion coefficient is an important parameter in the analyses and theories of solidification. In the MEPHISTO furnace, several experiments can be done through the repeated melting, solidifying, and remelting of the samples. Due in part to the results of the numerical modeling teams (refs. 4 and 5), MEPHISTO-4 operations were extended and continued through nearly the entire 16-day mission. The flexible and responsive performance of the furnace, the optimization through numerical modeling of the experiment, and the dedication of the principal investigator's teams enabled about twice the number of experiments to be accomplished during flight than were originally planned.



Length solidified along the ampoule, x , versus the ampoule diameter from bottom to top, y , and concentration of tin. No radial segregation is shown after 5000 sec of growth, indicating diffusion dominance; radial segregation is shown after 8000 sec because of solutal convection.

An example of the numerical modeling results is shown in the figure (ref. 6). The modeling enabled the team to estimate the level and effects of convection during flight. It also determined how long we needed to wait between solidification runs for the high concentrations of tin at the solid/liquid interface to be reduced to low levels by diffusion and convection, thereby allowing our mission time to be optimized.

After the USMP-4 mission, the MEPHISTO hardware was shipped to the French support contractor SEP (Société Européenne de Propulsion) for checkout and removal of the

samples. The samples and flight data are now being analyzed by the University of Florida team. MEPHISTO-4 is scheduled to be completed, with its final report given to NASA, by April 1999. Mission and flight data results were recently summarized (ref. 7) and include measurement of the average diffusion coefficient of tin in liquid bismuth of 3.5×10^{-5} cm²/sec.

Find out more:

<http://zeta.grc.nasa.gov/expr/insitu.htm>

<http://cfm.mech.unsw.edu.au/>

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Special recognition: Best poster in session, 11th International Heat Transfer Conference, Korea, 1998