Microgravity Growth of PbSnTe

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Summary

The growth of the alloy compound semiconductor lead tin telluride (PbSnTe) was chosen for a microgravity flight experiment in the Advanced Automated Directional Solidification Furnace (AADSF), on the Third United States Microgravity Payload (USMP-3) Space Shuttle flight in February 1996 and on the Fourth United States Microgravity Payload (USMP-4) Space Shuttle flight in November 1997.

The objective of these experiments was to determine the effect of reduction in convection, during the growth process, brought about by the microgravity environment. The properties of devices made from PbSnTe are dependent on the ratio of the elemental components in the starting crystal. Compositional uniformity in the crystal is only obtained if there is no significant mixing in the liquid during growth.

The PbSnTe growth experiment on USMP-3 consisted of three separate crystals grown in a single segmented ampoule. The crystals were grown in series, one in each of the three primary orientations with respect to the residual gravity vector. The growths on USMP-3 were roughly analogous to hot-on-top, cold-on-top, and horizontal growth.

The work on USMP-4 was to grow two sets of three crystals, again in segmented ampoules. The hot on top orientation was chosen for all growths. The variables, this time, were to be ampoule translation rate, thermal gradient, internal pressure, and nucleation procedure. The growth rate, which is related to the translation rate, is a key growth parameter under control of the experimenter. Higher growth rates produce steeper solutal gradients but less penetration of this vital diffusion zone into the convecting fluid flow. Thus, the growth rate presents a dichotomy of effects; a high growth rate produces a steeper concentration gradient while a low growth rate allows the diffusion tail to extend into the thermal convection cells. The change in thermal gradient has the obvious effect of changing the temperature dilatation contribution to the convective driving force. The internal pressure, at elevated temperatures, was adjusted by the amount of excess tellurium in the compound, and it was thought that it may affect pore formation in the crystals. The nucleation procedure was studied by using both seeded and unseeded growths and tests the influence of the evolution of latent heat on initial growth.

We designed a set of nine experiments in three different ampoules to measure the effect of the gravitational body force on the convective properties of alloy compound crystal growth as modified by reduced gravity and other crystal growth parameters. As is often the case, especially in new and difficult experimental arenas such as found using the microgravity laboratory, nature may have her way with even the best laid plans of human endeavor and can rend total havoc with strategies such as ours.

Ampoule #1 processed without any problems that were relayed to the ground. Recalescence was observed in cells 1 and 2, and due to failure of the uppermost

thermocouples (not a surprising event due the operating limits of the 20 mil diameter sheaths) was not expected to be observed in cell 3.

The observations, as seen on the ground, for the second ampoule were not so gratifying. On this part of the experiment, anomalies were observed in the sample thermocouples during the initial melting of the samples. When control thermocouples failed on the furnace booster heater, a cell (ampoule) failure and leakage from one of the lead tin telluride samples was suspected. To protect the two experiments already processed, the furnace drive was sent to the store position (full insertion), and growth was started in a gradient freeze manner by selectively powering down the different furnace zones. The experiment was terminated when the main heater control thermocouples failed.

The actual anomalies were only identified during sample retrieval at the Kennedy Space Center in February 1998. Clearly, the anomaly first occurred in ampoule #1, not the third ampoule. Cell 1, of ampoule #1, was intact and cell 2 was broken with approximately one third the length of the crystal still in the broken ampoule. The Inconel cartridge was swollen along its length starting near the cold end of cell 1, which was heated to 1000 °C; the swelling increased at the beginning of cell 2, where the temperature had increased to 1150 °C; and then the cartridge appeared ripped apart in the vicinity of the ampoule breakage. The remainder of the cartridge and ampoule were deposited in the furnace and caused the observed problems during the space processing of ampoule #2.

The ampoules and furnace reside at the Marshall Space Flight Center (MSFC) awaiting deposition by the Anomaly Resolution Team. The two ampoules, still in their cartridges, have only been examined by computer aided tomography, x-rays, and photography. Efforts are underway at both the Langley Research Center and MSFC to understand and recreate the anomaly.