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CASTING AND SOLIDIFICATION TECHNOLOGY (CAST)

Directional Solidification Phenomena in a Metal Model at Reduced Gravity

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The CAST experiment will study the phenomena that occur during directional solidification of an alloy, e.g., constitutional supercooling, freckling, and dendrite coarsening. The reduced gravity environment of space will permit the individual phenomena to be examined with minimum complication from buoyancy driven flows.

Constitutional Supercooling

In recent years a series of Space Processing Applications Rocket (SPAR) and KC-135 aircraft experiments have been flown to study the effects of gravity on the solidification of first, a metal-model (28 NH_4Cl-H_2O)[1,2] and then metal alloys (Sn-15Pb, Sn-3Bi, Al-4.5Cu, MAR-M246 and PWA 1480)[3-6] In the first metal-model experiment (bi-directional solidification) four [110] dendrite arrays nucleated and grew outward from the container walls filling the entire crucible. No crystallites appeared ahead of the interface, implying that in the absence of gravity, the forces were not present that would cause dendrite fragmentation and movement in the liquid. The second (unidirectional solidification) experiment also began [110] growth at the cooled wall, but several [100] crystallites appeared and grew ahead of the interface. In each instance the low-gravity arrays grew significantly slower than one-g ground runs. In the two metal-model experiments, the growth rates were the same (R, cm/min) but the temperature gradients differed by a factor of 7 (70° C/cm for flight one and 10°C/cm for flight two). This gives constitutionally super-cooled regions of 0.5 cm and 3.8 cm, respectively. Based on theories of the columnar to equiaxed transition in castings 7-9, several interpretations can be made from these results, the principal two being that gravity driven fluid flow is responsible for the melting off of dendrite arms and their transport into the melt, or a non-convection related constitutional supercooling phenomena encourages nucleation ahead of the interface. Rather than distinguishing between the two possibilities as was intended, the SPAR experiments were inconclusive.

A third explanation was the occurrence of residual flows from the de-spin of the sounding rocket. If these were still present when the second experiment began freezing, they could account for crystallites moving into the fluid. This hypothesis was tested in KC-135 and F104 low gravity aircraft experiments[10,11] using shadowgraph, interferometry and Schlieren techniques. These optical techniques enabled the experimenters to observe both the depletion layer adjacent to the growth interface and the presence of convection plumes. The flight profile of the KC-135 causes a sample to experience one to two g's before entering a 20 sec. period of low gravity. During unidirectional solidification studies growth plumes were established during the initial high g and flow rates on the order of 1cm/min were seen prior to inception of the low (.01 g) gravity period. These plumes dampened and began to diffuse 10 seconds after entry into low gravity, indicating that the predicted [12] damping times for these experimental cases must have been inaccurate. Further thermal data from F104 flights supports the concept that damping times in these materials are rapid. It, therefore, is implausable that the nuclei were carried ahead of the interface by residual flows.

Experiments with metal systems on SPAR produced similar results.[3,4] The Sn - 15Pb alloy solidified with large equiaxed grains in contrast to the more columnar plus small equiaxed grains obtained from one-g and centrifuge solidification. The Sn - 3Bi alloy also solidified initially with large grains on SPAR, but the final region surrounding the shrinkage cavity froze with small equiaxed grains. It is not known if these small grains formed due to constitutional supercooling nucleation, a gravity independent flow as a result of the shrinkage cavity, or gravity driven convection due to the sounding rocket leaving the low-gravity conditions. The controversy remains concerning the formation of grains ahead of an interface.

The investigators feel that this controversy can be resolved by a more thorough study of solidification in microgravity. The extended times available on Spacelab will allow experiment conditions that cover a range of growth rates, temperature gradients and, hence, constitutionally supercooled regions without the confusing presence of gravity driven fluid flow. The solute and thermal fields associated with the dendrite growth front will be measured and compared to a theoretical (computational) model.

Freckling

The ammonium chloride-water system is used to study the occurrence of thermo-solutal convection and pluming as it would occur in metal systems such as steel castings,[13,14] the superalloys[15] or others,[16,17] since it has a density inversion at the interface due to rejection of water-rich solute. This phenomena is often called freckling and tends to limit the range of compositions for many alloys processed on earth since it causes localized segregation and small equiaxed grains in the final casting. The plumes contain cooler liquid with a different composition from the surrounding region and as they traverse through the dendrite forest creating a channel, they carry crystalline fragments which appear as trails of equiaxed grains in the final ingot. The region of the freckles, therefore, has a different composition (and melting point) and crystalline morphology from the remainder of the ingot.

Freckling was first studied systematically in the metal-model NH_4Cl-H_2O[15] and found to depend on thermal diffusivity, density inversion, solute diffusivity and viscosity. Several investigators[15,16] have attempted to define criteria for the width and stability of the inverted layer. One criteria is based on thermal properties and the other on concentration effects. Since neither criteria includes both the effect of latent heat and segregation simultaneously, they grossly underestimate the solidification conditions (growth rate and temperature gradient) that are necessary to eliminate freckling. The presence of latent heat increases the size of the segregation generated inverted layer at a critical growth rate. This is not apparent from the earlier two criteria. Consideration of the combined effects decreases the growth rate for stability in 28 NH_4Cl-H_2O by two orders of magnitude.[18]

In the proposed flight experiment, the size and characteristics of the layer as a function of growth conditions will be measured using holography and compared with present theories. For the minimum temperature gradient (2°C/cm) the layer will decrease with increasing growth rate until a critical R is reached upon which the layer will increase due to latent heat effects. For the maximum temperature gradient (28°C/cm), the layer will decrease monotonically with increasing R since the critical R will never be reached due to FES limitations.

Freckling has generally been thought to begin within the mushy zone below the dendrite tips. A recent study,[17] however, also on NH_4Cl-H_2O, suggests that the channels for freckling originate at the dendrite front and spread. Since optical techniques such as Schlieren and interferometry easily delineate the plumes, the FES system is a powerful tool for studying this phenomena. The size of the inverted layer can be as large as 1cm under the proposed experiment's low temperature gradient and growth conditions. In ground based experiments the layer becomes unstable long before it reaches its maximum size with the result that events within the layer are difficult to study. During the IML flight the layer can reach a large enough size that its characteristics, such as internal pluming, can be resolved.

Dendrite Coarsening

Dendrite coarsening is a phenomena that occurs on the micro-scale and is primarily responsible for final dendrite arm spacings by causing the dissolution and shrinkage of smaller arms and the growth of larger arms. It is a function of local solidification time, and temperature and concentration gradients.

The SPAR experiments[1-4] and KC-135 flights[5,6] have shown a gravity-related coarsening effect on the secondary dendrite arms. Each alloy system showed greater arm spacings for the low-gravity solidification. In the instance of KC-135 flights, the arm spacings increased in low-g, decreased in high-g and then increased again when the next low-g parabola was flown. Theories on dendrite structure[19,20] suggest that by changing the surrounding concentration field and effective diffusion length, the perturbation frequency and, hence, the dendrite arm spacing is affected. In the case of low-gravity, the diffusion length would increase and, therefore, so would the arm spacings.

A more recent KC-135 experiment[5] on the superalloy PWA 1480 has shown the same physical results for the primary arm spacings (e.g. spacing increases as gravity level decreases). Review of current theories[21,22,23] suggests that these spacings are related to temperature gradient and growth rate or concentration gradient. This would drive the material toward smaller arm spacings in reduced gravity which is contrary to the experimental results. This was the first study of low-gravity primary arm spacings, and it suggests that there may be a subtle effect which has not yet been identified.

It is planned that once the solidification process itself has been modelled for the microgravity environment, the coarsening of the dendrite arms will be studied. This will be accomplished using a magnification lens attachment to the FES and is proposed for a subsequent flight.

Method and Approach

The study will proceed systematically with a matrix of nine temperature gradients and growth rates that will encompass a range of density inverted layer sizes and constitutionally supercooled regions. Based on earlier KC-135 results, the temperature gradients are expected to be different in low-gravity for the same test parameters, so two non-solidifying control samples will be run at identical conditions to two of the matrix runs to evaluate those differences. To produce more rapid solidification fronts, two runs will be processed in which the fluid is cooled below its freezing point and solidification initiated by a cold thermal pulse.

Holograms will be taken during each of these growth runs. In this way, various optical techniques can be used through post-flight reconstructions to determine concentration and thermal profiles, observe perturbations in the inverted layer, and distinguish nuclei that form ahead of the interface.

Since NH_4Cl-H_2O has been extensively used for similar studies, it has been chosen for these experiments. A two component system is required in order to model alloy solidification and investigate freckling phenomena. The present investigators have characterized NH_4Cl-H_2O optically in addition to accummulating other significant property data. Compared to other available metal models (e.g. succinonitrile with solute), NH_4Cl-H_2O is the superior medium available.

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