Analysis of Containerless Processing and Solidification Microstructures

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Objective and Application to Microgravity Knowledge Base

The main research objective is the evaluation and analysis of the undercooling and resultant solidification microstructures in containerless processing, including drop tube processing and levitation melt processing of selected alloys. The results are intended for use as an experience base for the design of space-based microgravity experiments.

Containerless processing in ground-based drop tubes simulates microgravity conditions via solidification of liquid samples under free fall conditions. The containerless environment removes a major source of impurities and heterogeneous nucleation sites, allowing for a large melt undercooling. This enhanced liquid undercooling exposes alternate solidification pathways, allowing for the formation of novel microstructures. Controlling the undercooling level provides some control of the operative solidification pathway and the resultant microstructure. The novel structures that may be produced in a ground-based containerless processing facility preview the wide range of possible materials processing experiments that may be conducted in a space-based laboratory. The results of the ground-based drop tube study will be used to identify critical experimental variables in microgravity processing and the analysis may be used to design and define the science requirements for extended duration space experiments.

Research Task Description and Progress to Date

The ground-based program represents a balanced experimental and analysis effort directed toward the investigation of drop tube and levitation containerless processing methods. The investigation focus is on the understanding and analysis of microstructural evolution during solidification of undercooled melts. The degree of liquid undercooling attainable in a laboratory scale (3 m) drop tube and levitation melting system can be altered through the variation of processing parameters such as alloy composition, melt superheat, sample size, and gas environment. The solidification behavior is evaluated through metallography, thermal analysis, and x-ray diffraction in conjunction with calorimetric measurements of falling droplets and a heat flow model of the processing conditions to judge the sample thermal history.

One of the main components of the investigation is the control of microstructural evolution in Ni-V and Co-Al alloys through the high undercooling levels provided by containerless processing. The study has provided new fundamental information on the control of nucleation. Solidification processing of eutectic alloys at high rates and large undercoolings has yielded varying degrees of solute trapping; but with very high undercooling typically only one of the solid phases forms partitionlessly. In contrast to this usual observation, high undercooling solidification of Ni-V alloys over a range of compositions has yielded a dual fcc and bcc phase partitionless structure. Due to the short solidification time available and the different nucleation and growth rates which may be expected for the two different phases, it is difficult to comprehend how this structure may develop at all if the kinetic competition leads to a sharp transition in phase selection. However, a

detailed cross-sectional TEM analysis of containerlessly processed Ni-V alloy samples reveals that the phase selection kinetics may not be as sharply defined as a thermodynamic transition. Several reaction pathways leading to the evolution of partitionless fcc and bcc phases have been identified, but most do not involve intermixed structures. The microstructural observations together with separate studies of highly undercooled bulk samples reveal the important role of a high heat extraction rate as well as a high undercooling in the development of novel dual phase microstructures. As a further part of the containerless processing analysis a heterogeneous nucleation kinetics model for quenched drops (mm) samples is under development and has provided new insight into nucleation controlled kinetics transitions as well as guidance for identifying the alloy constitution and processing conditions that favor dual phase partitionless reactions.

In order to examine the generality of the kinetics analysis approach established in the work on Ni-V alloys an analysis of phase selection is underway in the Co-Al system. High undercooling solidification of Co-Al alloys has also yielded a dual phase partitionless structure of fcc and B2(ordered bcc) phases. From TEM studies that are underway the microstructure evolution follows a similar pattern to that found for Ni-V alloys with the exception that an intermixed fcc and B2 duplex structure develops as a result of the solid state transformation of a portion of the B2 solidification product. Moreover, the analysis approach is being extended to allow for the construction of a processing map that will be essential in the design of a space experiment. This investigation contributes to the understanding of terrestrial solidification processing and demonstrates that microgravity processing methods can yield novel microstructures and phases that have not been observed with conventional processing approaches.

Another central component of the proposed program addresses the use of containerless processing together with an undercooled melt containing incorporated particles to develop a critical evaluation of solidification front-particle interactions. Combinations of nickel and copper with different reinforcement particles such as Ni-Ta_2O_5, Cu-Ta_2O_5, Cu-Al_2O_3, and Ni-TiC have been examined as model systems for different types of particle-melt interaction behavior. Significant melt undercooling in the presence of particles has been observed for these systems which even exceeds 200 °K in the case of Ni-Ta_2O_5. Experimentally, all stages of particle-solidification front interactions (i.e., pushing, entrapment, and engulfment) have been observed in the resulting microstructures. However, the existing models to describe the transition from particle pushing to entrapment or engulfment are not consistent with the observations. Moreover, the dependence of the incorporation behavior on particle size indicates that the simple assumptions derived for the case of directional solidification are not applicable for dendritic solidification. Here, not only the size of the particles, but also the characteristic length scales of the solidification front (i.e., the dendrite tip radius and the distance between the dendrites) have to be taken into consideration. Experiments are in progress to evaluate the different contributions carefully and to develop a better understanding of the critical velocity for particle engulfment.

Reliable evaluation of thermal history during containerless processing is essential. In the current studies, thermal measurement of a falling droplet is conducted using a calorimetric method. As a further aid, droplet solidification microstructures are used to establish the relationship between microstructural scale and level of undercooling as an *in situ* probe for sample thermal history. While these methods are useful, a direct thermal history capability is under consideration. Moreover, this ground-based experience will also be of value in the design of the science and hardware requirements for a space experiment.