

Experimental Measurement of Solutal Layers in Unidirectional Solidification

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An experimental study to evaluate and characterize the effects of inverted density layers during the directional solidification of metal alloys is described. The temporal history of the inverted density layers in front of a dendritic interface for directionally solidified ammonium-chloride 72-wt-% water is presented based on photographic data taken using a laser schlieren system and compared with predicted values. Data are given for both 1- and low-g solidification runs and the stability of the layer is discussed. The reduction of buoyancy effects is shown to be significant and the impact of reducing gravity level on freckling in metal alloys is speculated.

Nomenclature

| | |
|-----------|---|
| C_o | = bulk concentration of solute, wt % |
| C_l | = solute concentration in fluid, wt % |
| D | = mass diffusivity, cm^2/s |
| G | = temperature gradient, $^\circ\text{C}/\text{cm}$ |
| k | = partition ratio |
| m_l | = liquidus slope, $^\circ\text{C}/\text{wt %}$ |
| R | = growth rate, cm/s |
| T | = temperature, $^\circ\text{C}$ |
| x' | = distance ahead of solidification front, cm |
| \hat{x} | = inverted layer thickness |
| ρ | = fluid density, g/cm^3 |

Introduction

IT is well known¹ that convective phenomena strongly influence the quality of castings. Segregation effects arise due to both interdendritic and bulk flow and generally limit the achievable properties. Directional solidification has been used to confine the segregation to a small portion of the casting but there is still serious detrimental convection, driven primarily by thermal and solutal density differences. The superalloys are among the alloy systems particularly subject to these effects due to their characteristic lower-density region ahead of the solidification front that occurs due to preferential rejection of the lighter species. Since the resultant segregation is density driven, i.e., a buoyancy effect, it has been suggested² that multicomponent materials subject to these phenomena could be processed in the near-zero gravity environment of space to eliminate convective-driven segregation effects. In space the inverted-density layer ahead of the interface would still exist, but the extraordinarily low Grashof numbers would indicate no convection would occur. Thus, convention-generated segregation could be minimized, possibly even eliminated.

In solidification of metal systems in-situ diagnostics are impossible. Transient effects must be studied by using a quench and expensive postexperiment metallurgical analyses. A metal model material permits direct optical access into the melt and thus direct observation of thermal and concentration fields in real time by classical optical approaches based on refractive

index changes of the material. Ammonium chloride and water can be used to model metal alloy solidification since it freezes in a very similar manner. In the work described here a series of experiments were conducted at both 1 and $10^{-2}g$ to examine the low-density layer at the solidification front and relate the observed layer size to theoretical values, and to study its effect on initiation and intensity of convection.

Background

Metal Model Simulation of Solidification in Alloys

Since the early paper by Jackson and Hunt,³ transparent "metal-models" have been used as metal analogs. Ammonium chloride and water, which is one of these systems, has been used primarily to study the occurrence of freckling, a thermo-solutal convection phenomenon, as it would occur in steel castings,^{4,5} the superalloys,⁶ and other metal systems,^{7,8} since it has a density inversion at the interface due to rejection of water-rich solute during the solidification process.

The growth front of the ammonium-chloride water system is generally morphologically unstable, i.e., dendritic. As the ammonium-chloride-rich dendrites freeze out of the solution, they reject a lighter water-rich (compared to the bulk solution) liquid into the surrounding region. The interdendritic mushy zone is, therefore, comprised of a water-rich liquid surrounding the dendrite stalks. Figure 1 is a schematic representation of the solute concentration fields for both dendritic and planar interfaces. During planar growth the rejected solute forms a layer ahead of the growth front that decreases exponentially from a maximum at the interface to the bulk liquid composition. During dendritic growth the concentration of solute (water in the ammonium-chloride water system) increases from the dendrite tips back through the mushy zone until it reaches its maximum adjacent to the completely solid material. Classical treatments⁹⁻¹¹ of segregation in the mushy zone assume that the concentration of solute reaches its minimum value, the bulk composition, at or very slightly ahead of the dendrite tips. Experimental studies for materials that reject a heavier liquid, such as dendritically solidified aluminum-copper alloys,¹² show to the contrary a high solute concentration ahead of the dendrite tips that decreases exponentially for several millimeters until reaching the bulk composition. In the absence of convection the light-layer fluids, such as the superalloys and ammonium chloride-water, should behave similarly.

Some investigators^{6,13} have attempted to define criteria for the width and stability of the low-density layer and there have been a few attempts to experimentally characterize it.¹⁴⁻¹⁶ In general, the experiments have simply substantiated its existence, and there has been no correlation between measurements and predictions. The present work seeks to combine

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theoretical estimations of the layer thickness with results from laboratory optical measurements at both Earth and reduced-gravity levels.

Convection in Directional Solidification

The subject of convection in solidifying systems has been given increased attention in the last few years¹⁷⁻²⁰ in the realization that control of the fluid flow is necessary to optimize materials' properties of the solidified sample. If one is to minimize fluid flow, then horizontal gradients, both thermal and concentration, must be near zero. The vertical (with respect to the gravitational field) temperature and concentration gradients theoretically do not necessarily give rise to a convective field, i.e., the fluid flow equations possess a trivial solution if the density ratio between solid and liquid is one (a typical assumption). This is true even if both thermal and solutal fields provide for an increasing (with height) density profile.

In the system considered here, a positive vertical solutal gradient is always present ahead of the interface since the partition coefficient k is less than one and the solute (water) is the lighter component. Thus, from consideration of concentration only, a lighter fluid sits under a heavier fluid and a fundamentally unstable situation is possible. This can be overcome if a large enough positive temperature gradient (assuming increase in volume with temperature) exists within the fluid. This simple criterion for convective stability, that density decreases with height in the fluid, is, of course, not correct, but has been used in the static sense to assess effects of conditions and fluid parameters on initiation of convection.^{14,21} The resultant convection due to combined temperature and concentration gradients is generally termed thermosolutal convection. In fact, a statically stable condition can be unstable and one statically unstable can be stable. An example of the latter case is the thermal profile that provides a lighter fluid under a heavy fluid. This fundamentally unstable situation will not break down until buoyancy is sufficient to overcome the viscous forces, i.e., when one exceeds a critical Rayleigh number. For the former case one can examine a monotonically decreasing (with height) density gradient consisting of a positive temperature gradient and a decreasing solutal gradient. This is the situation under consideration for ammonium chloride and water. If the fluid has a relatively low Prandtl and a relatively high Schmidt number, then it can still be convectively unstable. If a particle of fluid is distributed upward by some mechanism, it will tend to continue upward as follows. The concentration of the particle will be rich in the light solute but may still be more dense than its upward neighbor because it is cooler. Once it moves upward a small amount, the heat conduction quickly brings the particle to the surrounding temperature, whereas the slow mass diffusion has a minimal effect. Thus, the particle now finds itself compositionally lighter than its neighbors and thus will continue to rise. Thus, this seemingly statically stable situation is actually unstable to an upward perturbation.

The mass diffusion layer one would expect to find in front of a solidifying interface is strongly influenced by convection. The layer is disturbed by the motion and in many instances takes part in the overall motion. Since the solidification front-movement velocity is partially dictated by the diffusion of fresh material through this layer, and the motion itself depends upon solidification velocity, the two are strongly coupled. The analysis of the convection field and its influence on the front velocity is a long-term goal of this research.

Experiments

Experimental Techniques

A combined schlieren and Mach-Zehnder interferometric system²² was designed and built for use with a directional solidification apparatus. A schematic of the optical system is shown in Fig. 2. The directional solidification apparatus consisted of an optical-quality quartz cuvette that contained a saturated ammonium chloride 72-wt-% water solution and a

top and bottom thermoelectric to provide the desired temperature profile as well as the heating and cooling. In selected experiments, thermocouples were placed at one-half centimeter intervals in the fluid as additional temperature monitors. Black and white photographs or video can be taken of both the schlieren and interferometry.

Experimental Procedure

The solidification sequence consisted of preheating the cuvette and location to 45°C for a 30-min period. The desired temperature gradient was then established across the cuvette. After a 15-min holding time, the top and bottom thermoelectric devices were ramped down at rates to provide a selected growth rate and constant-temperature gradient. Temperature measurements were taken in the fluid and at the surfaces of the thermoelectrics. Schlieren photographs were taken in sequence at 1-min intervals for ground-based runs and 1-s intervals for low-gravity runs.

The aircraft that flew the low-gravity experiments is a modified KC-135 whose cargo area has been dedicated to experimental use. During a typical mission, the aircraft flies between 30 and 40 parabolas, each of which yields up to 30 s of approximately $1 \times 10^{-2} g$'s alternating with up to 2 g 's during the 1.5 min pullout. For these experiments the solidification was directionally solidified continuously during the alternating low-gravity and pullout time periods.

Analysis

Diffusion-Driven Layer

The ammonium-chloride 72-wt-% water system under study is dendritic for all the conditions considered in this investiga-

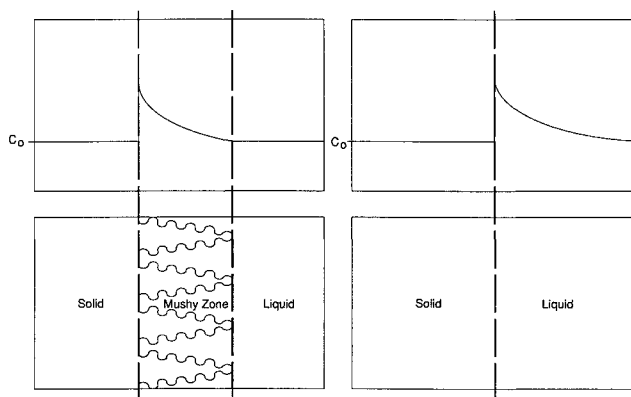


Fig. 1 Schematic representation of solidification fronts and solute concentration during steady-state dendritic and planar growth (assuming complete diffusion in the solid).

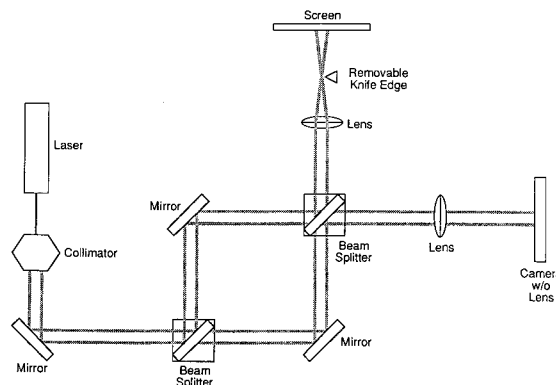


Fig. 2 Laser schlieren and Mach-Zehnder interferometric system.

tion. The solute-rich layer in front of the dendrite tips (interface) should, therefore, be very small. The steady-state distribution of solute ahead of the dendrite forest is given by¹

$$C(x') = C_o \left[1 - a \exp\left(-\frac{R}{D} x'\right) \right] \quad (1)$$

$$a = \frac{DG}{m_l R C_o} \quad (2)$$

For a planar interface system the steady-state solute concentration ahead of the interface is given by

$$C(x') = C_o \left[1 + \frac{1-k}{k} \exp\left(-\frac{R}{D} x'\right) \right] \quad (3)$$

Thus, the relative size of the two layers is driven by the coefficient of the exponential term. For small k ($k = 0.004$ in $\text{NH}_4\text{Cl}-72\text{H}_2\text{O}$) the planar layer would be much larger than the dendritic layer. However, the small k leads to the large levels of supercooling that dictate planar interface breakdown.

The steady state is in reality not realizable for most growth rates of ammonium-chloride 72-wt-% water and the diffusion layer size and profile must be determined from the time-dependent equations. The equation that represents the solute profile ahead of the interface in its time-dependent form is given by¹

$$\frac{\partial C_l}{\partial t} = D \frac{\partial^2 C_l}{\partial x'^2} + R \frac{\partial C_l}{\partial x'} \quad (4)$$

with boundary conditions

$$\frac{\partial C_l}{\partial x'} + \frac{R}{D} (1-k) C_l = 0$$

for $x' = 0$ and all t ;

$$C_l = C_o \text{ at } t = 0 \text{ for } x' > 0,$$

and

$$C_l = C_o \text{ at } x' = \infty \text{ for } t > 0$$

Smith et al.²³ developed a solution to Eq. (4) for constant growth rate conditions. This was not used in this work, however; rather, the equation without constant coefficients was solved using a fully conservative control volume approach.²⁴ The calculated solute concentration fields were then combined with the density variation from a specified temperature gradient to determine the density fields and the inverted density layer thickens.

Separation of Thermal and Solutal Effects

In a nonuniform media a light beam is turned in the direction of increasing index of refraction that generally means toward the region of higher density or lower temperature. A schlieren system measures this deviation by focusing the beam to a spot and then using a knife edge to eliminate all but the deviant beam. Thus, a bright image can be produced on a screen from regions that have greater index refraction (or density). Regions of decreasing index of refraction (or density) can be imaged by the same method but by reversing the knife edge.

The contrast in a schlieren system is proportional to the first derivative of the index of refraction that is a function of concentration and temperature. In the present study of ammonium chloride-water the dependence on temperature is negligible since both the low linear gradient ($15^\circ\text{C}/\text{cm}$) and the small layer size (< 0.04 cm) mean that the index of refraction could only vary by as much as 0.0018. The dependence on concentration is an order of magnitude greater.

For the purposes of comparison of measured layer sizes with those calculated based on Eq. (4), temperature effects due to

temperature gradient were ignored and the bright band seen ahead of the growth interface is assumed to be created solely by the concentration variation of the depicted (low-density layer).

Results and Discussion

Diffusion Layer Development

Figure 3 is an example of a typical schlieren photograph taken during a ground-based (1 g) run. The inverted layer is clearly indicated by the bright area just above the dendrite forest. A series of these photographs taken at selected intervals was used to determine the average location of the forest interface and the density inversion layer thickness as a function of time. The edge of the inverted layer was judged to be that point at which the bright layer began to fade into the black region above it (see Fig. 3). The solidification of ammonium chloride 72-wt-% water was studied for several growth rates and imposed temperature gradients. The $15^\circ\text{C}/\text{cm}$ gradient was typical and the results for that case are most inclusive and, thus, will be presented.

Figure 4 is a plot of the inverted density layer size as a function of time for two measured interface growth rates: 0.1 and 0.3 cm/h. The two data sets are seen to be nearly identical over the first 12 min of growth (even though one interface has grown three times as far) and each begins to approach a steady value. The faster growth-rate case then shows a sudden decrease in layer (inverted density) size at approximately 18 min. This decrease in layer size is initiated when the layer becomes unstable and begins to convect upward, i.e., plume. At 1 g all cases are found to eventually plume. Table 1 gives values of the plume inception time for three growth rates. The limited data show a significant increase in plume inception time with reduc-

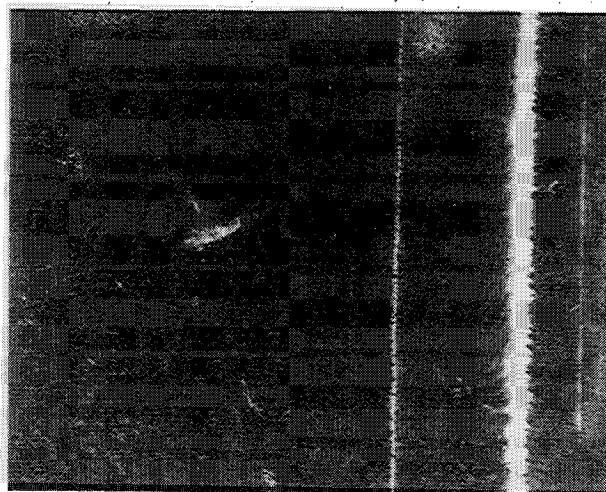


Fig. 3 Schlieren photograph of dendritic growth front.

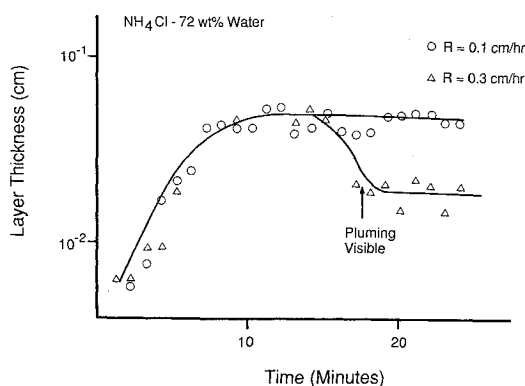


Fig. 4 Experimental inverted layer size as a function of time for two growth rates.

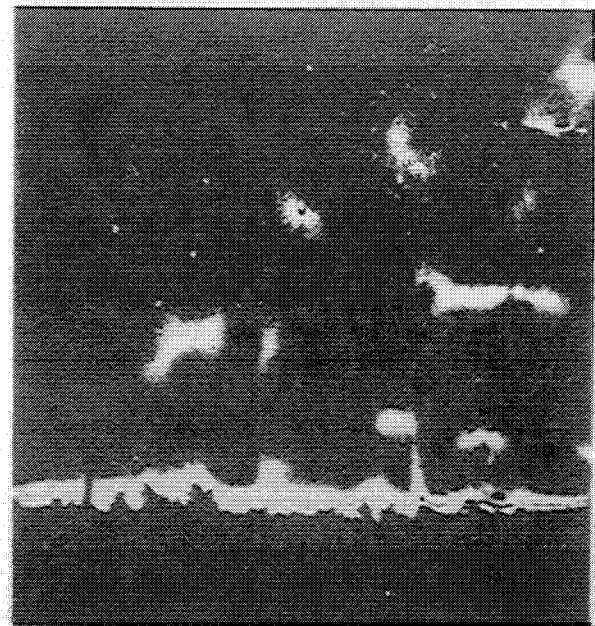
tion in growth rate. This is consistent with the expected behavior when one considers only the theoretical rate of growth of the layer. The experiments show something different, however: that the layer grows at the same rate for both growth rates. An explanation for why the slower rates do not plume at the same time remains to be formulated. The layer sizes and solutal gradients are very similar and thus the solutal Rayleigh numbers exceed critical at approximately the same times.

The low-gravity experiments conducted on the aircraft failed to provide an indicator of why the same-size layers at low R do not plume. In the low g the Rayleigh numbers were less than critical and all convection stopped. Unfortunately, the 30 s of low gravity are not sufficient to permit pluming to occur at the low-growth rates. In between low- g sessions the aircraft provides a high- g environment that makes study of low- g convective processes difficult due to the intense fluid stirring. Figure 5 gives a sequence of schlieren photographs taken during one set

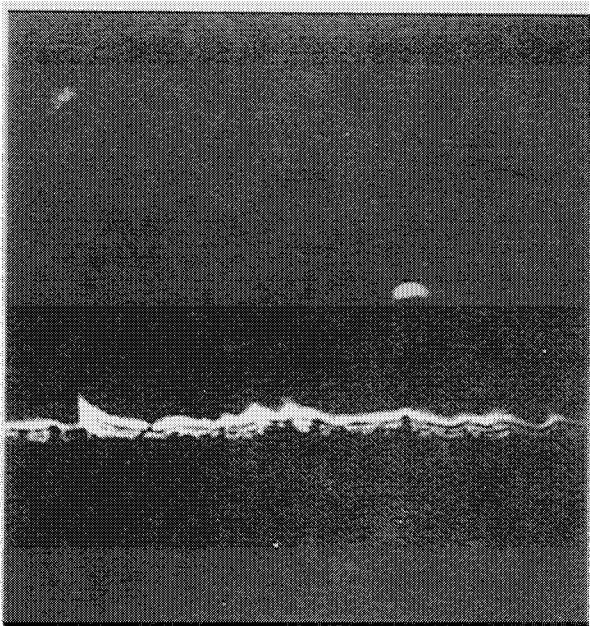
of parabolas. At 10 s the gravity level is about 1; the convection is seen with portions of the layer being lifted above the interface. At 53 s the low- g parabola is just beginning; the plumes from the high- g time period are slightly visible but are dissipating quickly. Near the end of low g (71 s) the layer is very well behaved and no indication of convection is noted. By 96 s the pullout begins and the excessive g loading leads to very vigor-

Table 1 Time to plume as a function of growth rate

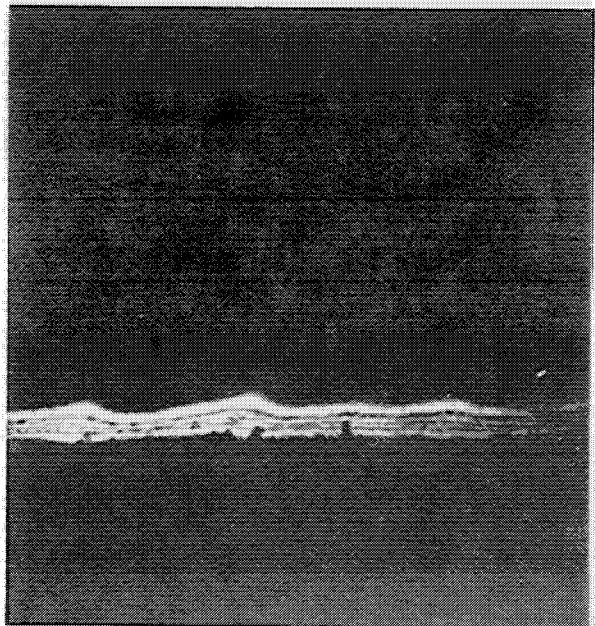
| Pluming time, min | Growth rate, cm/h |
|----------------------|----------------------|
| 1 | 1.1 |
| 18 | 0.3 |
| 41 | 0.108 |



10 Seconds



53 Seconds (low g start)



71 Seconds



96 Seconds (pullout)

Fig. 5 Schlieren photographs of KC-135 solidification sequence ($G = 15^{\circ}\text{C}/\text{cm}$, ammonium-chloride 72-wt-% water).

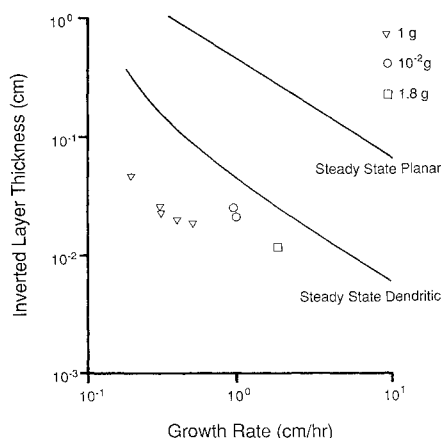


Fig. 6 Theoretical and experimental inverted layer size as a function of growth rate for ammonium-chloride 72-wt-% water ($G = 15^\circ\text{C}/\text{cm}$).

ous pluming, the layer is breaking down, and the intact portions shrink considerably. The layer does not appear to reach a steady-state thickness but is growing throughout the low-g period. The high-g cycle then disrupts layer growth so significantly that basically all must begin again. A long time-duration low-gravity period would permit the layer to develop fully and answer the question of its stability. Thermosolutal convection cannot be directly related to Rayleigh number and thus an actual measurement would be most welcome.

An additional point about the high-g layer sizes should be noted. The measured layers reach steady state much more quickly than indicated by the transient calculations. In fact, the slow rates appear to become steady in a few minutes when they should require several hours. Since the diffusion layer size is over an order of magnitude smaller than the momentum layer, this is not explained by convection. If the layers were able to reach steady state, the layer sizes would be anticipated to be similar to the momentum layer. The discovery of the reason for reaching steady state so swiftly and the small sizes will require further study.

Effect of Growth Rate on Layer Size

The data showing the relationship between growth rate and layer size are plotted in Fig. 6 along with the steady-state predicted values for planar and dendritic growth fronts. The experimental data lie below the planar and dendritic predictions. The planar theoretical curve decreases almost linearly with growth rate whereas the dendritic case and the data decrease in a noticeably nonlinear fashion. The nonlinearity is due to the dependence of the solute concentration ahead of the dendrite interface on the growth rate as shown by Eq. (1). As the growth rate decreases, the water concentration immediately ahead of the interface increases. This leads to a thicker inverted layer.

As discussed earlier, plumes occur during the growth process and can act to decrease the size of the layer. With the gravity level varying from 1.8 to 1.0 to 10^{-2} , the solutal Rayleigh number varies accordingly from 12,000 to 6500 to 65. Therefore, pluming should always occur during pullout, as the layer approaches 0.03 cm in 1 g and never in the low-gravity period. The data show that the layer sizes increase in that order, being smallest for the pullout and largest for the low-g cases (but not yet reaching the theoretical values).

Conclusions

This work represents the initial effort in a long-term study for the directional solidification of segregating alloys using optically transparent metal model materials. The details of the diffusion layer and its involvement in thermosolutal convection and their effect on solidification rate and alloy composition are the primary concern. In this investigation using both ground-

based and aircraft-based solidification systems, the following conclusions were drawn:

1) The size of the inverted density layer measured for dendritic growth is well below that expected from theory. The experimental layers apparently reach a steady-state limit that must somehow be influenced by convection. The low-g runs do provide larger layer sizes but are still below theoretical values. Calculation of the momentum layer does not indicate that convection should limit the diffusion layer size so significantly.

2) The time necessary for the layers at 1 g to reach steady state is significantly shorter than predicted. The low-g data runs were too short to ascertain whether steady state was being approached for those cases.

3) The plume inception time decreases with growth rate and appears to be driven by some mechanism other than the layer thickness above the interface. The best correlation is with growth time, indicating that the total height of the mushy zone may be the most significant parameter. The low-g runs did not plume and, in fact, showed convective fields to be indiscernible.

4) Long time period low-g runs are necessary to characterize the diffusion/inverted density layer in these materials. Measurements using schlieren/interferometric methods are viable for this purpose.

5) The reduction of convective effects in the low-g environment appears to be reasonable for solidification processes and could be used to eliminate segregation in alloys due to freckling.

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