COMPOUND SEMICONDUCTOR GROWTH IN A LOW-G ENVIRONMENT

(Re-fight of AADSF, USMP-3 experiment)

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SCIENCE REQUIREMENTS DOCUMENT

List of Symbols

g	Gravity level or body force
k	Segregation coefficient
m	Slope of liquidus line from the phase diagram
z	Solid length of crystal
C_S	Composition of the solute
D	Solutal diffusion coefficient
G_S	Solutal gradient in the melt
G_T	Axial temperature gradient
R	Growth rate
Z	Total length of crystal

SUMMARY

The growth of the alloy compound semiconductor lead tin telluride (PbSnTe or LTT) was chosen for a microgravity flight experiment on the Advanced Automated Directional Solidification Furnace (AADSF), on the United States Microgravity Payload-3 (USMP-3). This document describes two additional experiments, again using the AADSF, to grow PbSnTe on USMP-4.

The primary objective of this series of experiments is to detect what effects the low gravity environment has on convective mixing during the growth process. The properties of an array of devices made from PbSnTe are dependent on the ratio of the elemental components. Compositional uniformity in the resultant crystal is only obtained if the liquid is quiescent during growth.

The PbSnTe growth experiment on USMP-3 was launched in February 1996. During that flight, three separate crystals were grown in a single, segmented ampoule. The crystals were grown in series, each in one 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.

This new work will grow two sets of three crystals, again in the segmented ampoules. It will use the orientation that produced the most favorable growth on USMP-3 which preliminary inspection indicates is the hot-on-top configuration. The variables, this time, will be ampoule translation rate, thermal gradient, 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 nucleation procedure is studied by using both seeded and un-seeded growths and tests the influence of the evolution of latent heat on initial growth.

While the immediate objective of these experiments is to grow PbSnTe and establish its fundamental growth properties, another, more important objective is to gain a better understanding of the mechanisms involved in generalized crystal growth, particularly those affected by gravity. This information will not only help produce better quality materials on Earth, but also help define future efforts of crystal growth in space and lead the way to more extensive materials science research.

PROBLEM DESCRIPTION
Microgravity:

The reason for using a microgravity environment for crystal growth is that gravitational forces present on earth produce perturbations in nutrient supply and heat distribution that adversely affect the resultant crystal quality. These perturbations are caused by buoyant convection driven by gravity.

In zero gravity, there is no buoyancy to cause convection in the molten crystal material. However, true zero gravity does not exist even on a spacecraft. In space, the movement of the astronauts, aerodynamic drag on the spacecraft, physical displacement from the center of mass of the spacecraft, and thruster firings each cause gravity-like accelerations. Instead of true zero gravity, the net accelerations present on the spacecraft are reduced to the point that they are measured in units of one millionth of normal gravity, or microgravity.

The first flight experiment in this series was in the MEA furnace on STS 61A in October 1985. It was shown that a high degree of convection was still apparent at the low growth rates used in that experiment. The convective instability may have been exacerbated because the crystal axis and the steady state gravity vectors were (estimated) to be perpendicular and the growth rate was not well controlled. Analysis of this flight sample served as a basis for continued ground based research and the development of the AADSF flight experiments.

Lead tin telluride was also grown on Space Lab "J" by Kinoshita and Yamada[1]. Due to the inability to accurately predict the extent of melt back in their experiment, their seed was over 40% of the total length of the crystal. This subsequently reduced the growth length to a point where it is difficult to determine the effect of convection by looking at the resulting compositional profile. Figure 1 shows Kinoshita's data with our calculations for the two extremes of mixing in the liquid. Using their stated growth rate, and the resultant crystal length, fig. 1 illustrates the difficulty in seeing the difference between diffusion controlled and convection controlled crystal growth for this case.

Segmented Ampoule:

As mentioned before, the USMP-3 AADSF experiment used a single segmented ampoule to grow three separate PbSnTe crystals in series. A similar design will be used on USMP-4. The ampoule, represented in fig. 2, allows each of the three crystals to grow with different conditions without affecting the outcome of the others. For USMP-3, each crystal was grown identically except for the orientation of the ampoule with respect to the residual gravity vector.

Unfortunately, using the three cell ampoule decreases the total crystal length. It also decreases the percentage of the crystal grown where the growth rate nearly matches the translation rate. A long crystal will obtain a nearly thermal steady state growth region sandwiched between two end effect thermal and compositional transient regions. Using the three cell sample decreases the length of any steady state region, however, it provides growth conditions that are nearly thermally equivalent for each of the three cells. Most important, it keeps the composition for the three different regions separated, so that each crystal will have identical starting compositions. Each cell is long enough to reach compositional steady state before the liquid diffusion tail reaches the end of the ampoule.

Figs. 3 and 4 show the calculated growth rates and interface shapes for PbSnTe crystals grown in both the one cell and three cell configurations with a translation rate of 1 cm/hr. As a rough approximation of diffusion controlled growth, the growth rates and interface shapes were calculated assuming a constant composition and melt temperature of 900EC.

Fig. 3 shows that the growth rate is very similar in each of the three cells for the same growth conditions. Fig. 4 shows the calculated interface shape curvature is substantially the same for the three crystals, except for the beginning of the first. If the experiments were completed by stopping and starting growth of a single longer crystal, the growth conditions for each orientation would not be equivalent. The interface shapes, growth rates, and melt composition would all be different.

Lead Tin Telluride:

Lead tin telluride is an alloy of PbTe and SnTe. The technological importance of PbSnTe lies in its band gap versus composition diagram which has a zero energy crossing at approximately 40% SnTe. This facilitates the construction of long wavelength (>6 µm) infrared detectors and lasers. The properties and utilization of PbSnTe are the subject of other papers.[2,3]

PbSnTe is amenable to study because it is easily compounded, it has a relatively low vapor pressure, and it is miscible with the same crystal structure for all compositions. There is also existing, though limited, literature on its growth and properties. The nominal starting composition for this work is 20% SnTe and 80% PbTe that produces a bandgap to match the long wavelength atmospheric window.

PbSnTe is also interesting from a purely scientific point of view. It is, potentially, both solutally and thermally unstable due to the temperature and density gradients present during growth. Density gradients, through thermal expansion, are imposed in directional solidification because temperature gradients are required to extract heat. Solutal gradients occur in directional solidification of alloys due to segregation at the interface. Usually the gradients vary with both experiment design and inherent materials properties.

In a simplified one dimensional analysis with the growth axis parallel to the gravity vector only one of the two instabilities works at a time. During growth, the temperature in the liquid increases ahead of the interface. Therefore the density, due to thermal expansion, is decreasing in that direction. However, the phase diagram shows that the lighter SnTe is preferentially rejected at the interface. This causes the liquid density to increase with distance away from the interface.

Coriell et al.[4] have shown that the two opposing density gradients cannot be readily balanced to stabilize the flow. Moreover, both experiments[5,6,7,8] and numerical analyses[5,9] have demonstrated that radial thermal gradients will start fluid motion long before the onset of convection predicted by a one dimensional model. Hence, there will always be convection in the liquid.

Bridgman Growth:

Bridgman crystal growth offers the opportunity to independently fix the temperature gradient and interface position with respect to the furnace. However, these parameters cannot be varied without bound. There are temperature limits on the furnace and ampoule as well as limits imposed by the growth process. An excellent review of recent advances in Bridgman growth has been given by Favier.[10]

The limits imposed by the growth process are primarily concerned with maintaining an initial solutal translation zone of reasonable length and preventing interfacial instability. Second order problems are the maintenance of interface shape control, and thermal strain in the solid.

Due to the size of the AADSF, a sufficiently high growth rate is required to achieve steady state composition within 20 to 30 millimeters of the start of growth. The furnace must then be controlled to produce an axial thermal gradient in the melt that is sufficient to maintain a stable interface. If the growth is diffusion controlled, the growth distance needed to get within 1% of compositional steady state, i.e. uniform, growth is[11]

$$z_{ss} = 5D/kR \ . \qquad \textbf{(1)}$$

However, the permissible growth rate is limited by the fundamental phenomena of interface breakdown. The short form of the equation for preventing breakdown is[12]

$$\frac{G_T}{R} > \frac{C_s}{D}\frac{(1-k)}{k}|m| \ . \qquad \textbf{(2)}$$

Eq. 1 shows that a short initial transition zone requires a high growth rate while eq. 2 shows that a high growth rate requires a large thermal gradient to avoid interfacial breakdown. Increasing either the axial thermal gradient or the growth rate increases the density gradient and the mixing in the liquid. Consequently, it can be seen that there is a trade off between growth rate, temperature gradient and the degree of mixing for a given gravity level.

Ground Based Research

Ground based research efforts have been manifold and intensive yet all of the factors necessary for complete knowledge to design the flight experiment are still not known. The earliest efforts were in setting baseline crystal growth[13,14,15,16,17] parameters and measuring thermophysical properties[13,18,19]. Subsequent efforts were on furnace characterization,[20,21] development of melt-solid interface measurement techniques,[22,23,24] and measurements thereof.[24,25] Other ground based efforts have been in measurements of both steady[5,6, 18, 19] and periodic[26,27] fluid flow.

Numerical modeling has been part of this effort from the beginning[28,29] and will continue to be so. To help extend from simple models to actual crystal growth systems, a heat transfer measurement device[30,31] has been designed and used to measure ampoule/furnace interactions.

USMP-4 Experiments:

The hypotheses for the experiments described in this document are part of our peer reviewed program. The interaction of growth rate, the diffusion boundary layer, and the convective driving forces were clearly part of our initial proposal as seen by, "The objectives of this experiment are to ... minimized the influences of thermal convection on the solidification process in the growth of PbSnTe" and the "...variations in the boundary layer thickness....".[32] The investigation of this effect was later revalidated in the Schreiffer review as, "However the best idea at present is to conduct a series of crystal growth experiments in which the growth rate is varied between runs. ... Hence the attainment of compositional steady state will be controlled by both the configuration of convection cells and the relative velocity of the fluid and the interface."[33] This experiment on USMP-4 is part of a cohesive effort to fully characterize the cause and effect of gravity driven convection on the growth properties of this material.

The experiments will examine the interaction of growth rate and the convective driving force found in the microgravity environment of low Earth orbit. Eq. 1 shows that a high growth rate is needed to obtain steady state growth quickly. Eq. 2 shows that the ratio of axial thermal gradient over the growth rate must be kept high to avoid interfacial breakdown. The experiment will of course control the ampoule pull rate, but in the proposed range the actual growth rate is related to the pull rate.

Reduced gravity levels on Earth orbit will not eliminate convection although the convective forces will be greatly diminished in the microgravity environment. Therefore, all a crystal grower can do is to design the experiment to minimize the effect of the residual microgravity and maintain a growth rate such that the interface movement (hence the movement of the diffusion boundary layer) is fast relative to the fluid velocity. The optimum alignment of the gravity vector and density gradients was the subject of the USMP-3 experiment.

In the USMP-4 experiment the dichotomy of the effects of growth rate are investigated. High growth rates create steep concentration gradients, which exacerbates convection. With low growth rates, the lower concentration gradients extend the diffusion tail away from the quiescent boundary layer near the interface and into the thermal convection streamlines. Hence low growth rates will not only allow more time for mixing per unit length of crystal growth but also expose more of the diffusion tail to the thermally induced convection cells. The bounds of potential growth rates are determined by the crystal length to compositional steady state on the slow end and the need to maintain interfacial stability on the high end. We will investigate growth rates of one half and twice that used in USMP-3.

Other variables to be investigated on the USMP-4 experiments are thermal gradient and nucleation procedure. The change in thermal gradient has the obvious effect of changing the temperature dilatation contribution to the convective driving force. For the growth of one of the cells we will reduce the gradient to approximately one half that used used in USMP-3 (as well as the five remaining the cells in USMP-4), and we will use our standard translation rate of 1 cm/hr. This combination will produce the same G_{Th}/R ratio as in the 2 cm/hr growth.

The nucleation procedure is studied by using both seeded and un-seeded growths. As the supercooling before nucleation in PbSnTe is low and repeatable (ref 19), the USMP-3 experiments were un-seeded growths. After the observation of recalescence we allowed a solutal diffusion period to re-homogenize the liquid. In the USMP-4 set of experiments we will have one seeded growth and one growth in which we will continue translating after onset of recalescence. This latter growth is affectionately called the Keep Chugging experiment. The primary purpose of the seeded growth experiment is to avoid the release of heat and subsequent, although transitory, temperature inversion part way into the growth of the crystal. Whereas the primary purpose of dispensing with the solutal diffusion period is to avoid time for bubbles to coelesce at the seed-liquid interface as observed on the USMP-3 samples.

The crystals grown in this research will be analyzed for both structural perfection and compositional homogeneity. The compositional profile is the most sensitive measure of convection during growth and will be measured by a wavelength dispersive electron microprobe. The effect of melt stabilization on crystal structural properties are more subtle than that on composition. However, the crystals will be examined for preferred growth orientation, grain size, grain substructure and dislocation content as well as for general perfection and pore/void content.

Thermal calibration of the AADSF is a vital component of flight preparation. The furnace, developed by Marshall Space Flight Center, has two primary heating zones separated by an insulation zone that contains a heat extractor plate. Calibration with an instrumented sample was performed with subsequent experimental verification using germanium growth and interfacial demarcation. This data was then used as "ground truth" for subsequent numerical modeling using PbSnTe thermophysical parameters. Experimental data and concomitant modeling allows a more detailed thermal description of the isotherms within the sample than is normally available.

Growth Method and Identification of Materials System for USMP-4.

The growth material will be the same as for USMP-3: lead tin telluride (LTT). LTT is a pseudo-binary alloy of lead telluride and tin telluride. The growth method will be Bridgman crystal growth with the angle between the residual gravitation vector and the growth axis determined from the results obtained on USMP-3.

The AADSF:

The processing requirements for this experiment are consistent with the capabilities of the AADSF. The three axis acceleration data and all pertinent furnace data will be required from Shuttle operations. The sample ampoule will be instrumented with thermocouples and this data would be recorded for both real time monitoring and subsequent analysis.

The preferred AADSF configuration is the 2-2-2 configuration used on USMP-3. This configuration has twice the thickness of the cold zone insulation, heat extractor plate, and hot zone insulation than the original AADSF design (1-1-1) used for the USMP-2 flight. The 2-2-2 configuration has been shown, using extensive ground tests, to provide the most flexibility in choice of the respective hot and cold zone temperature settings without significantly altering the achievable thermal gradient. However, should another configuration be chosen, the ground test results, already available, will be used to choose suitable temperature settings.

Sample Translation Rate:

The translation rates determined for the experiment, along with the crystal length and thermal soak time, will determine the total time needed to complete the experiment. The translation rates will fall in the range of 0.5 to 2 cm/hr. For sample crystal lengths of approximately 6 cm, this gives bounds of 3 to 12 hours of growth time required for each individual crystal.

Required AADSF Zone Temperatures:

Since the solidification temperature of LTT is between 870-905EC, depending on composition, the AADSF hot zone must be set at a temperature higher than this range. For the USMP-3 experiment, the hot zone was 1150EC, while the cold zone was 525EC. We anticipate using these same temperatures during the USMP-4 experiment except for the low thermal gradient experiment where we expect that the cold zone will be raised to 700-750 C and the hot zone reduced to 1000-1050 C. These temperature setting are easily obtainable in the AADSF and should not cause any problems during use.

Desired Gradient Zone Length:

The desired gradient zone length is consistent with the length obtained in the AADSF 2-2-2 configuration. Other configurations could probably be accommodated, but would be less desirable.

Orbiter Microgravity Constraints:

Since the microgravity environment is the key to the whole experiment, it is obvious that the P.I. would prefer the lowest possible disturbance level possible at all times when growth is occuring. Another advantage the segmented ampoule has over a continuous ampoule is that the microgravity time can be broken into segments. For example, the crystals in the USMP-3 experiment were only grown during crew sleep periods. A similar procedure may be possible on USMP-4 in order to provide both the optimum microgravity environment, and minimize constraints on the crew.

Concluding Remarks

We have proposed a study of the effect of gravitational body force on the convective properties of alloy compound crystal growth as modified by both reduced gravity and the translation rate. We have chosen a material that clearly illustrates these effects.

We will investigate the growth properties as functions of both gravity orientation and growth rate. We have proposed both a strong experimental program and a strong supporting numerical analysis program.

This proposed work and the work in an accompanying proposal featuring magnetohydodynamic damping in the melt[34] will complete the set of experiments on this exciting material.

KEY PERSONNEL

Archibald Fripp (Langley Research Center) is the Principal Investigator on this project. He has 15 years experience with the Microgravity Science program and 28 years experience with materials processing in general. Dr. Fripp will spend approximately ten months a year on this project.

William Debnam (Langley Research Center) is an integral part of the electronic materials research effort at Langley with thirty years experience. He is responsible for laboratory operations at Langley and for the ampoule design and preparations for both ground based work and the flight experiments.

William R. Rosch (NRC Fellow at Langley Research Center) has 7 years experience in microgravity crystal growth at both Clarkson University and at the Langley Research Center. Dr. Rosch is responsible for thermal transport measurements and analysis.

Ranga Narayanan (Professor of Chemical Enginering at the University of Florida) has 19 years experience in computational fluid dynamics of flows in both Microgravity and on Earth. Dr. Narayanan is responsible for computational fluid dynamics work in research.

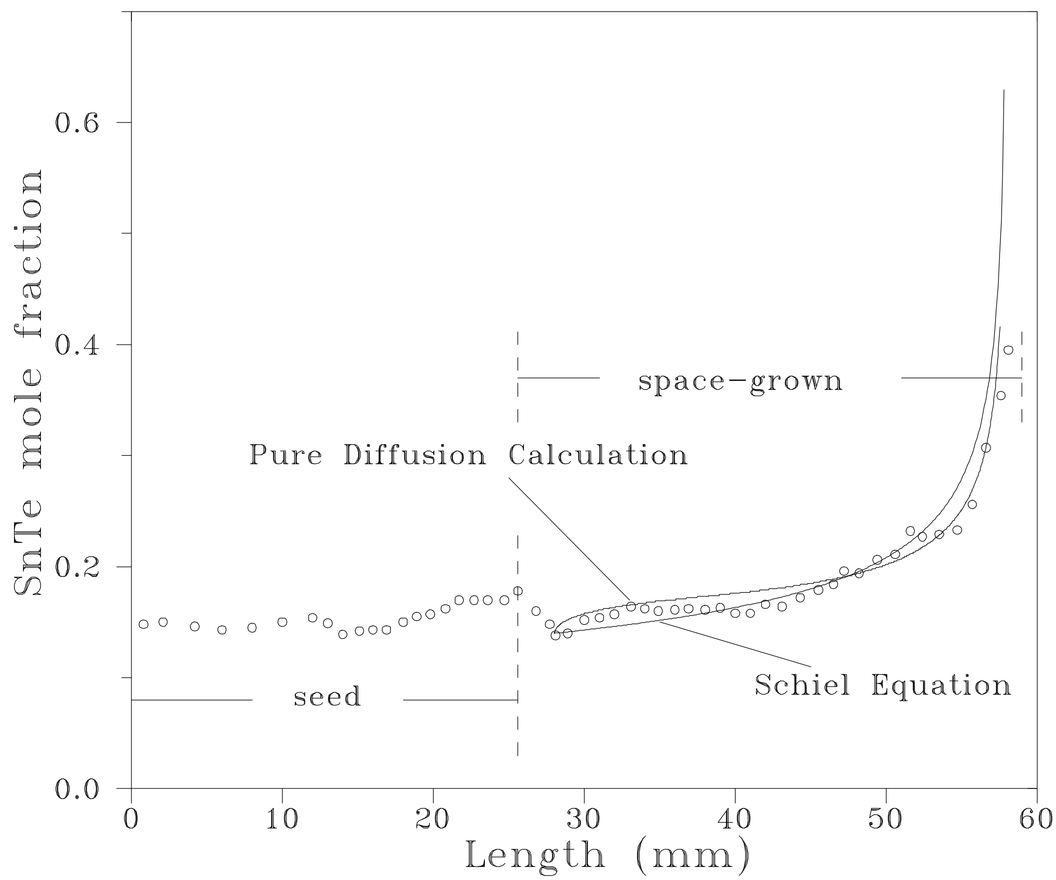

Figure 1 Comparison of Kinoshitra's experimetal result with the Schiel equation and a pure diffusion calculation.

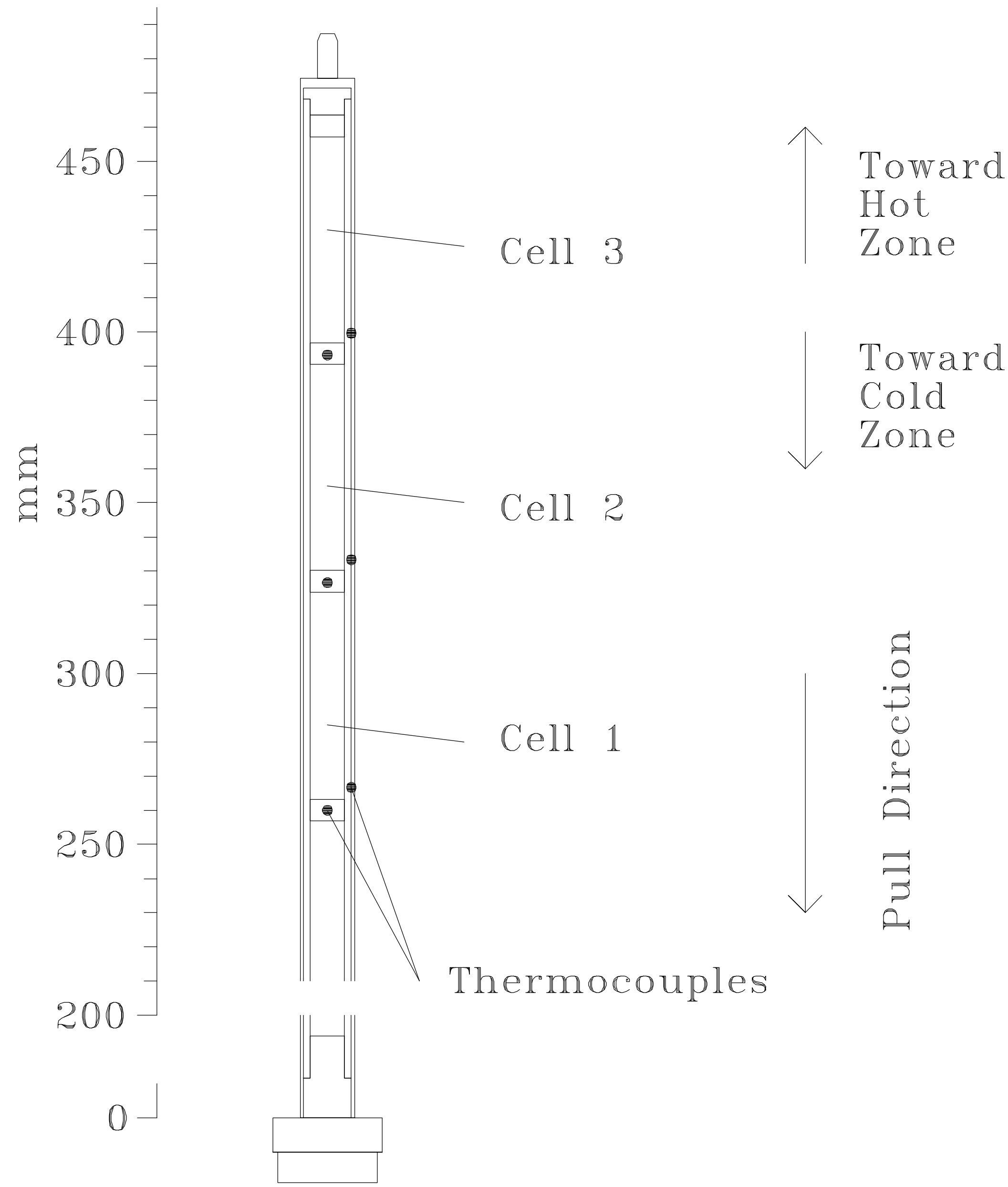

Figure 2 A typical segmented ampoule showing the three cells, and the location of the sample thermocouples. Growth is initiated by inserting the three cells into the hot zone and then slowly translating the sample into the cold zone.

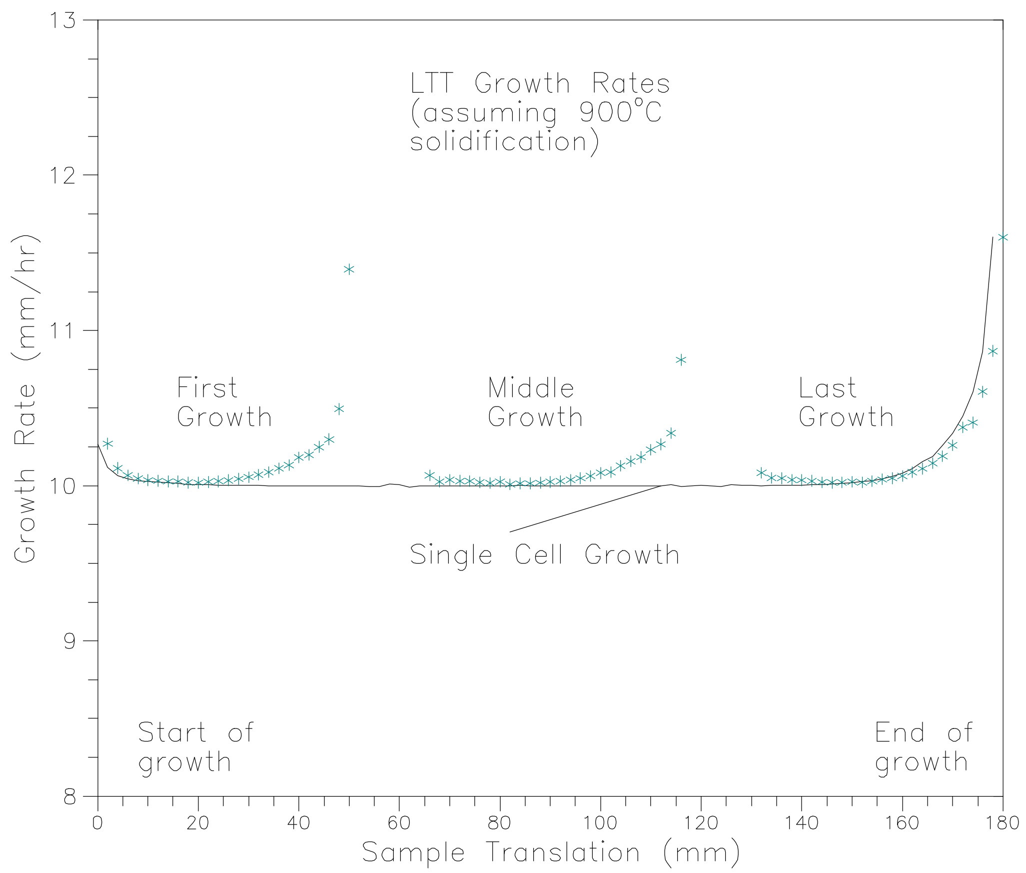

Figure 3 Growth rate comparison for an amoule with a single long cell, and a segmented ampoule with three cells. The calculations used the properties of lead tin telluride, and assumed a 900 degree solidification temperature.

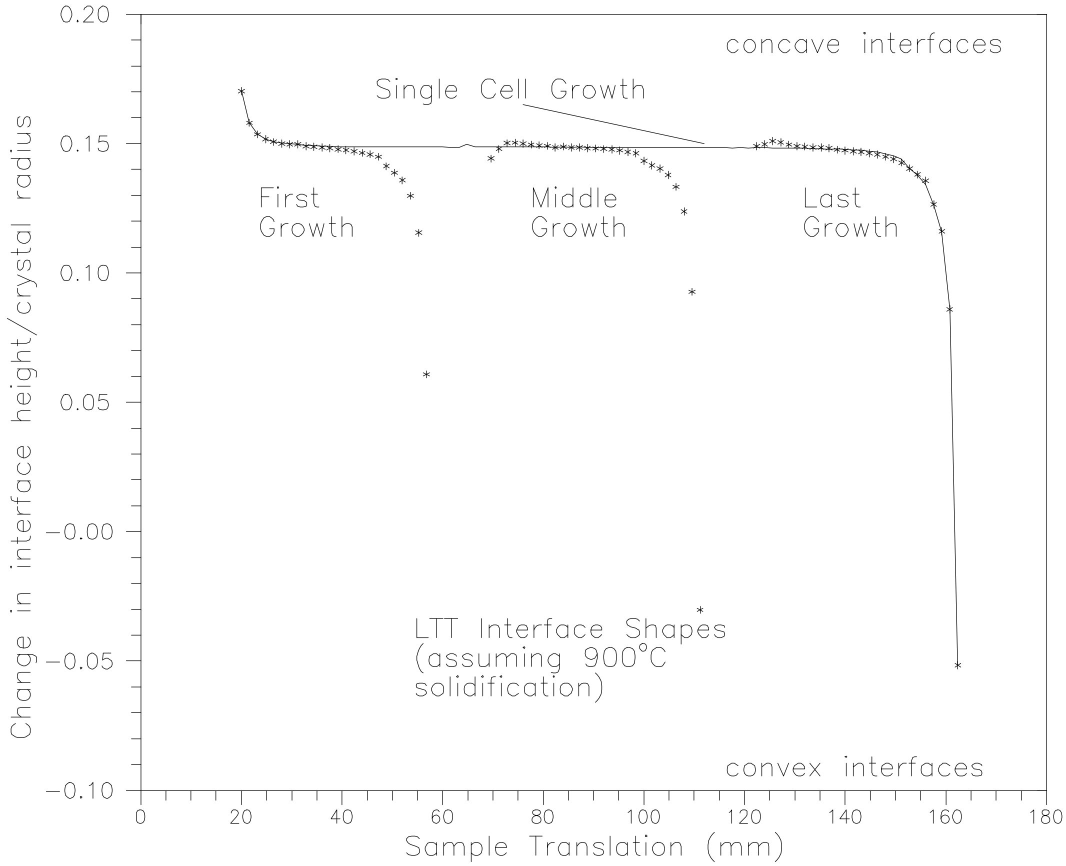

Figure 4 Comparison of the interface shape measurement for a one cell and segmented cell ampoule. Positive values indicate a concave interface while negative values indicate a convex interface.

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