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Influence of Gravity on the Directional Solidification of Indium Antimonide

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Undoped, Te-doped and Se-doped InSb were directionally solidified in the laboratory, in the KC-135 aircraft, and in two centrifuges. The melt never pulled away from the ampoule wall during solidification in the KC-135 aircraft during parabolic flight maneuvers designed to produce weightlessness. Gas bubbles on the wall moved slightly toward the solid when the freezing interface contacted them. An undoped ingot grown in the centrifuge at 9 *g* ($g = 9.81$ m/s^2) consisted primarily of a single grain containing a number of twins. The microstructure of the ingots depended on the *g* level and on doping. With Se doping microcracks occurred about 7 *g*, but they never occurred without doping, with Te doping in the KC-135, or at 1 *g*. Impurity striations were not found in ingots solidified up to 10 *g*, indicating that the freezing rate did not fluctuate. Centrifuge processing almost completely eliminated the bubbles normally present on the outer surface of InSb ingots.

Introduction

UNEXPECTED results have been frequently obtained in crystal growth experiments, especially when performed in space. Many of the space-processing results have not yet been convincingly explained because measurements and observations were not possible with the apparatus available and/or materials being grown. One such unexpected and strange behavior is discussed in the next paragraph.

Consider the directional solidification of a melt contained in an ampoule that is not completely full, i.e., there is a gas space in the ampoule. On Earth, gravity holds the melt in contact with the ampoule wall as it solidifies, resulting in a crystal surface with the same topography as the ampoule wall. The usual fused silica (quartz) ampoule has a smooth inner surface and hence the resulting crystal has a smooth outer surface, with the outer diameter differing from the inner diameter of the quartz ampoule only by thermal contraction. The results from space experiments were often different. Directional solidification in space has often produced ingots with a slightly smaller diameter than the containing ampoule (after correcting for thermal contraction).

Skylab contained a gradient freeze furnace constructed by Westinghouse. In each run, three samples were heated at one end and cooled passively at the other. Programming down the heater caused solidification, with the temperature gradient in the material decreasing with time and the freezing rate increasing. In one set of experiments, Wilcox et al.[1] obtained three InSb-GaSb alloy ingots smaller in diameter than their carbon-coated quartz ampoules.[1,2] The ingots' surfaces were wavy. A second set of experiments gave only smooth surfaces in contact with the ampoule walls. The second set of experiments was run with a higher initial heater temperature.

Witt et al.[3,4] directionally solidified tellurium-doped indium antimonide in the Skylab furnace built by Westinghouse. A single crystal rod was melted back partially and refrozen. For about two diameters, the resolidified ingot necked in from the seed crystal so that it had a smaller diameter. The diameter then expanded to fill the ampoule. There were irregularly spaced miniature ridges on the surface of the crystal. These ridges were about one-thousandth of an inch high and contacted the ampoule wall. The width of these ridges increased down the crystal, and in the last centimeter became irregular and branched out.

Similar results were obtained by others in Skylab, the Apollo-Soyuz Test Project (ASTP), and in various Soviet spacecraft.[5–7] In many of these experiments, the reduced contact portions of the ingots also had on their surface irregular ridges that were in contact with the ampoule walls. Furthermore, Soviet authors claimed that the crystal quality was superior when the crystal had little contact with the ampoule wall.[8] They also claimed that a smaller ingot diameter was obtained only when the level of acceleration was very low.

The usual explanation for the above phenomenon has been that the melt is not in contact with the ampoule wall during solidification, because it does not wet the ampoule wall. The exact meaning of the phrase "does not wet the ampoule" is unclear but presumably means that the melt has a high contact angle (>90 deg) on the surface of the quartz ampoule. This has also led to proposals to use ampoules with noncircular cross-sections to further reduce contact of the ampoule by the melt in space.[9,10]

In previous space experiments, directional solidification had always been performed in opaque furnaces. For example, in the Skylab and the ASTP experiments, directional solidification was performed in an opaque furnace built by Westinghouse.[1-5] There was no provision for viewing the ampoule during solidification and hence the actual behavior of the melt and its interaction with the freezing interface was unknown. Sen and Wilcox[9] made the first attempt to observe the behavior of non-wetting liquids inside a cylindrical ampoule. Experiments were performed in the KC-135 aircraft during parabolic flight maneuvers giving about 20 s of low gravity. The liquid never lost contact with the ampoule walls. Sometimes large gas bubbles formed on the surface of the ampoule walls, and sometimes the liquid separated into two or more columns. A theoretical treatment was used to give the limit of stability of gas bubbles before the liquid would break into separate columns.[11] It is worth noting that Chiaramonte et al.[12] performed directional solidification of aluminum in both normal and reduced gravity aboard the KC-135 aircraft using a real-time radiographic viewing system.

The influence of gravity on the directional solidification of InSb is not complete until the other end of the gravity spec-

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trum is studied, namely high gravity. Crystal growth in high gravity has sometimes resulted in ingots with higher crystallographic perfection than those grown on the ground or in space.[13-20] Each material exhibited a particular g level at which crystals of higher crystallographic perfection were obtained. This g level was not only characteristic of each material, but also depended on the centrifuge used. Crystal growth can be performed in furnaces, either in a thermally stable environment (hot zone on top of the cold zone) or in a thermally unstable environment (cold zone on top). For example, Regel et al.[20] grew Ag-doped PbTe and $Pb_{0.83}Sn_{0.17}Te$ crystals under high gravity using two different centrifuges in a thermally stable environment.[13,14] In crystals grown at a particular g level, the dopant concentration profile through most of the crystal was uniform, as expected in the absence of convection. Ag-doped PbTe solidified in an 18-m arm centrifuge had a constant Ag concentration along most of the crystal at 5.2 g, while the Ag concentration was constant at 2.0 g in a 5.5-m arm centrifuge. Zhong et al.[15] obtained single crystals of GaSb during growth in a centrifuge of 7-m arm length at 3, 6, and 9 g. The axis of the furnace was perpendicular to the arm of the centrifuge. As the acceleration level was increased the impurity striations became weak and indistinct, thereby improving the homogeneity of the impurity distribution. Wiedemeier et al.[17] performed physical vapor transport experiments on GeSe in the presence of 2 atm argon at 1, 5, and 10 g. The mass transport rate increased only slightly with g when a thermally stable environment was used. However, in a thermally unstable environment, the mass transport rate at 10 g was nearly an order of magnitude larger than the anticipated $g^{1/4}$ dependence. Bárczy et al.[18] grew Al-Al_3Ni eutectic in a centrifuge at different gravity levels between 1 and 8 g in a thermally stable configuration. They obtained a cellular eutectic microstructure for eutectic and off-eutectic compositions. The interlamellar spacing was a maximum at 4.5 g. Regel et al.[20] solidified Al-Cu eutectic from 1 to 7 g in a thermally stable environment. The lamellar spacing of the Al-$CuAl_2$ decreased with increasing g. One would expect such a behavior if convection was reduced. The Al-Cu eutectic grown at 5 g had a perfect plate-shaped structure and the Cu concentration was constant along the length of the ingot.

Muller et al.[16] grew GaSb in a 1.5-m arm length centrifuge by the travelling heater method in a thermally unstable environment. At 20 g they obtained a tenfold increase in the rate at which inclusion-free single crystals could be grown compared to the normal laboratory conditions of 1 g. Muller et al.[16] performed directional solidification experiments of Te-doped InSb in a thermally unstable environment and in a horizontal zone melting configuration. Dopant striations were eliminated when the centrifugal acceleration was increased beyond a critical value that depended on the thermal boundary conditions.

Research on materials processing at high gravity is important for two reasons, namely, scientific interest and technological/commercial applications. At present there is no scientific explanation for obtaining optimal homogeneity and perfection of crystals at a particular acceleration level. Perhaps materials equal in quality to those grown in space can be produced commercially in a centrifuge.

The first objective of the present research was to directly observe the solidification of a poorly wetting melt in a partially filled ampoule under low gravity. The second objective of this research was to determine the influence of gravity on the microstructure of InSb.

Experiments

Ampoule Preparation

Ground and KC-135 flight ampoules were prepared with two different configurations, cylindrical and triangular. Cylindrical ampoules were of two different diameters, 8 mm o.d. × 5 mm i.d. and 11 mm o.d. × 9 mm i.d. The inner width of each side of the triangular ampoule was 7.9 mm. All ampoules were 68.5 cm long, made of fused silica, and had a cylindrical quartz plug sealed 18.4 cm from the base of the ampoule. The InSb feed ingot rested on this plug.

The ampoules for the centrifuge experiments were 16 mm o.d. and 12 mm i.d., with a conical bottom end. The ampoules were 10.5 cm long and made of fused silica. All ampoules were cleaned thoroughly using the procedure described in Ref. 21. The InSb alloy was prepared by premixing pellets of indium and antimony (99.9999% pure) in stoichiometric amounts in a cylindrical compounding ampoule 11 mm o.d. × 9 mm i.d. The material was melted, directionally solidified, and removed. This InSb alloy was loaded into the growth ampoule and sealed using the procedure described in Ref. 21.

Ground Experiments

NASA's Marshall Space Flight Center (MSFC) had a Transparent Video Furnace fabricated that enabled direct observation of the solid-melt interface during directional solidification. This apparatus consisted of a hot zone and a cold zone separated by an air gap. A video camera was placed such that it could record whatever happened in this air gap. An accelerometer mounted at the base enabled recording of the acceleration along X, Y, and Z axes. The furnace control unit, data-acquisition system (IBM PC AT), video monitor, and video recorder were all mounted on a rack adjacent to the furnace as shown in Ref. 21.

The ampoules were taken to MSFC for experiments under normal gravity on the ground. An ampoule was placed in the furnace such that a part of the ingot was in the gap between the hot and cold zones. The video camera was focused in the gap on the lowest part of the ingot. The temperature of the furnace was increased to melt the ingot. The temperature of the hot zone was adjusted until the solid-liquid interface was visible in the gap so that the video camera could be focused on the interface. The video camera was moved back and forth and the lighting was adjusted until a clear image of the interface could be seen on the video monitor. The temperature of the hot zone was recorded and the same temperature was used later in the flight experiments. The next step was to determine the appropriate translation rate. The choice of ampoule translation rate depended on the fact that the solid-liquid interface had to be in the field of view of the camera until all or most of the ingot was directionally solidified. The optimum translation rate was determined by observing the solid-liquid interface at various translation rates. Once the proper hot zone temperature and translation rate were determined, the ground experiments were performed.

For an experiment the ampoule was positioned in the furnace as desired. The temperature of the hot zone was set and the furnace was switched on. Once thermal equilibrium was reached, the translation unit was switched on and the ampoule was translated at the rate determined earlier. Simultaneously, the data-acquisition unit was switched on. The data collected included the hot zone temperature, translation rate of the ampoule, position of the ampoule in the furnace, XYZ acceleration data, and the temperature of the ampoule at four different positions along its length. Simultaneously, the video recorder was switched on to record the solidification phenomena. Once the ingot was completely into the cold zone, the translation unit, data-acquisition system, and the video camera were switched off. The furnace was cooled to room temperature and the ampoule was taken out.

Flight Experiments

For flight experiments, the Transparent Video Furnace was taken to Ellington Air Base near NASA's Johnson Space Center in Houston, TX. This apparatus was flown on NASA's KC-135 Microgravity Research Aircraft Facility, which can produce repeated periods of low gravity for ~20 s intervals using parabolic flight patterns. The furnace was loaded and

mounted tight onto the floor of the KC-135. Two hours before the flight, the furnace was switched on and the temperature of the hot zone was increased. Thermal equilibrium was attained before flight. Just before takeoff, the furnace was covered with a hood as per KC-135 safety requirements. After takeoff, the aircraft flew to a predetermined altitude of 28,000 ft and flew level (straight) for a few minutes. Just as the aircraft executed its first parabola, the data-acquisition system was switched on. Simultaneously, translation was started along with the video recorder. All units were shut off at the end of the last parabola. The aircraft then returned to base. During this time the furnace was shut off, allowed to cool, and the ampoule taken out. One ampoule was run on each flight, with one flight per day. Table 1 lists the parameters for the two sets of experiments performed in the Transparent Video Furnace. The taped videos were viewed later and the observations were correlated with the properties of the resulting ingots.

Centrifuge Experiments

Two different centrifuges were used for the experiments. The first centrifuge was located at the Gagarin Cosmonaut Training Center outside Moscow and had an arm length of 18 m. The second centrifuge was located at the Department of Bridges and Roads facility at Nantes in France and had an arm length of 5.5 m. The centrifuges and the furnaces used for crystal growth are described in detail in Ref. 19. The furnaces used in the two different centrifuges were very similar. Each furnace had two zones consisting of a ceramic tube wound with platinum-rhodium wire as the heating elements. Directional solidification of the melt was performed by the gradient freeze technique, i.e., the temperature of each zone was programmed down at a constant rate. The furnace was mounted at the end of the arm of the centrifuge and oriented such that the resultant acceleration was along the axis of the furnace. Each ampoule was wrapped with ceramic fiber insulation, enclosed in a stainless-steel cartridge, and loaded into the furnace. The ingots in the centrifuge ampoules were directionally solidified under acceleration levels ranging from 1 to 10 times earth's gravity g. The InSb ingot was melted and the temperature was allowed to stabilize before the centrifuge was put into rotation. After 2.5 hs, the furnace temperature was programmed to decrease slowly at 20°C/h. Once the centrifuge rotation was stopped, the last part of the melt was quenched by turning off the power to the furnace. The temperature gradient in the furnace was estimated to be 20°C/cm and the freezing rate approximately 10 mm/h. Table 2 lists the parameters for the ingots grown in these two centrifuges.

Characterization Methods

The ingots were removed from the quartz ampoules by soaking the ampoules in 48% hydrofluoric acid for 6 to 8 h to reduce the ampoule wall thickness. Each ingot was sliced longitudinally down the center using a Lastec Model 2006 wire saw. One-half of each ingot was cut into 18-mm-long sections for characterization. The samples were mounted in epoxy and mechanically polished. The mounted samples were etched for 8 to 12 s with CP4 etch (2 parts HNO_3, 1 part HF, and 1 part CH_3COOH). The samples were rinsed immediately in deionized water followed by methanol. This procedure reveals grain and twin boundaries in InSb.[20] To quantify the microstructure, grain and twin boundaries were counted across the width of the ingot at 2-mm intervals along the longitudinal axis using a Nikon Optiphot-2 optical microscope. Statistical analysis was performed on counts of twin boundaries and grain boundaries. A one-sided Student's t-test with paired comparisons versus length for grain boundaries and twin boundaries was performed for the ingots.[22] The t-test allowed us to determine the probability of the number of boundaries in one ingot being greater than that in another ingot. The t-test was performed by using the Number Cruncher Statistical System (NCSS).

Table 1 InSb ingots solidified in the Transparent Video Furnace

Experiment	InSb	Growth location	Inner diam., mm	Cross section	Length, cm
First set	Undoped	Ground	5	Cylindrical	6.4
	Undoped	KC-135	5	Cylindrical	4.7
	Undoped	KC-135	5	Cylindrical	8.0
Second set	Te-doped	Ground	9	Cylindrical	6.3
	Te-doped	KC-135	9	Cylindrical	6.8
	Te-doped	Ground	7.9	Triangular	4.8
	Te-doped	KC-135	7.9	Triangular	13.0

Table 2 InSb ingots grown in a centrifuge at high gravity

Experiment	InSb	Gravity level	Centrifuge	Length, cm
First set	Undoped	1 g	18.0-m arm	5.3
	Undoped	3 g	18.0-m arm	6.8
	Undoped	5.2 g	18.0-m arm	6.6
	Undoped	7 g	18.0-m arm	6.9
	Undoped	9 g	18.0-m arm	5.4
Second set	Te-doped	5.2 g	18.0-m arm	7.9
	Te-doped	9 g	18.0-m arm	5.3
	Se-doped	9 g	18.0-m arm	8.4
	Se-doped	2.2 g	5.5-m arm	5.8
Third set	Se-doped	1 g	18.0-m arm	6.0
	Se-doped	5 g	18.0-m arm	7.0
	Se-doped	10 g	18.0-m arm	8.2
	Se-doped	5.2 g	18.0-m arm	7.0
	Te-doped	1 g	18.0-m arm	6.7

The ingots were then repolished. The polished samples were etched with two solutions to reveal any Te or Se impurity striations in InSb:

Etchant 1—50 ml HNO_3, 37 ml HF, 30 ml CH_3COOH, and 1 ml Br_2 for 4–10 s.

Etchant 2—1 part HF, 1 part CH_3COOH, and 1 part $KMnO_4$ (0.05 M) for 4–10 min.

Results and Discussion

Surface Appearance and Behavior

The main aim of performing the KC-135 experiments was to determine if a high contact angle of the melt would cause the melt to pull away from the ampoule wall under low gravity. We had speculated that the separation of the melt from the ampoule would occur during the low-gravity period of the KC-135 aircraft flight. Also, we had expected the melt to freeze after it had pulled away from the ampoule wall and thereby produce smaller diameter crystals. However, we could not observe any separation of the melt from the ampoule wall in any flight experiment, using cylindrical ampoules of two different inside diameters and triangular ampoules. The total time for which low gravity could be obtained on the KC-135 aircraft might have been too small for separation of the melt from the ampoule wall. Hence, experiments need to be performed in environments that will enable longer times of low gravity to be available before we can come to a firm conclusion regarding the cause for smaller diameter crystals.

Matthiesen[23] performed experiments on the KC-135 aircraft to study the behavior of InSb and Ge melts. He also did not observe any separation of the melt from the ampoule wall for InSb samples, but postsolidification analysis of Ge samples indicated that separation of the melt from the ampoule wall did take place under low gravity. Matthiesen speculated that InSb samples formed oxides on their surface due to leaks in the end seals of the ampoules and that this oxide adhered to the quartz ampoule and prevented separation of the melt from the ampoule wall. It is likely that in our KC-135 experiments the InSb samples also had a thin layer of oxide on their surface. The other important fact that one should remember is that in the KC-135 aircraft experiments, the ampoules were subjected to alternating periods of low and high gravity. Each 20 s period of low gravity was separated by 40 s of high gravity (~ 1.8 g).

An unexpected phenomenon was observed in our KC-135 experiments. Bubbles present in the melt on the surface of the ampoule wall moved toward the solid-liquid interface when the interface contacted the bubbles. We would expect a floating bubble to move from a region of higher surface tension to a region of lower surface tension. Since InSb is a normal melt, the surface tension increases with decreasing temperature.[24] Thus, we would expect on this basis that if the bubble moved, it would move away from the interface rather than toward it. Therefore a surface tension gradient is unlikely to have caused the bubble motion. It is more likely that the minimization of total surface energy was responsible. Figure 1 is a schematic representing the bubble motion. The solid line represents the solid-liquid interface before it came into contact with the bubble, while the dashed line represents the solid-liquid interface after it had moved past the bubble. "A" represents the bubble before it contacted the solid-liquid interface, while "B" represents the bubble after it was contacted by the solid-liquid interface. When the solid-liquid interface comes into contact with the end of the bubble marked "c," the contact angle changes at this end, compared to the opposite end of the bubble marked "d." The end marked "c" in the melt starts freezing with a particular contact angle. At the same time the other end of the bubble marked "d" is still in the melt and hence free to move. Because of the different contact angles between ends "c" and "d," the end "d" moves towards the solid-liquid interface to minimize the total surface energy. The important point to remember here is that the solid-liquid interface was nearly perpendicular to the ampoule wall.

Matthiesen[25] had observed in his KC-135 experiments with InSb that the solid separated from the ampoule wall as the solid was cooling down. The same behavior was observed in our KC-135 and ground experiments performed in the Transparent Video Furnace. The solid pulled loose from the ampoule wall close to the solid-liquid interface at irregular intervals. The solid would be expected to separate from the ampoule wall when the stress due to differential thermal expansion exceeds the sticking stress.

Lagowski et al.[10] proposed solidification of nonwetting melts in ampoules of triangular cross sections in space. They predicted that this would yield cylindrical crystals contacting the ampoules along only thin lines in the middle of each triangular face. Experiments by Sen and Wilcox[9] however showed that a nonwetting melt did not form a liquid column in triangular ampoules in the KC-135. The liquid pulled away from the ampoule only in the corners. This was in agreement with the theoretical work done by Sen and Wilcox.[11] But in all our KC-135 flight experiments, the InSb grown in the triangular ampoules was always triangular in cross-section.

The InSb ingots used for ground-based, KC-135, and centrifuge growth experiments had a number of large and small bubbles on their outer surface. We believe these bubbles were formed due to the dissolved gases that were entrapped between the ingot and the ampoule wall during ampoule preparation. These bubbles remained on the surface of the ingots even after performing ground based and KC-135 solidification experiments. However, the surfaces of all of the ingots grown in the centrifuge were extremely shiny and clean. The ingots grown in the centrifuge had very few tiny bubbles, or in some cases no bubbles, on their outer surface. Therefore, it seems that processing InSb in a centrifuge reduced or eliminated bubble incorporation at the ampoule wall. It is likely that the higher g levels caused the bubbles to break free of the ampoule wall and rise up to the vapor space at the top of the ampoule, thereby resulting in their elimination.

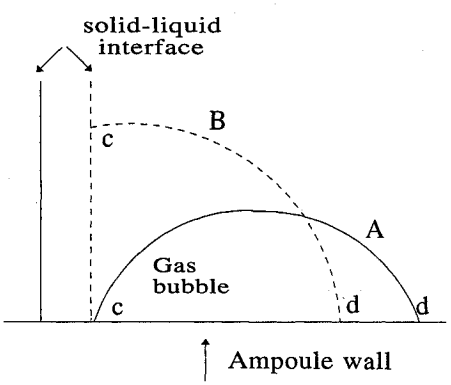

Fig. 1 Schematic represents the motion of a bubble present on the ampoule wall when the solid-liquid interface comes into contact with the bubble: a) before contact, and b) after contact.

Striations

No impurity striations were observed in any of the doped InSb ingots grown on the ground, on the KC-135, or in the centrifuge. Any rapid change in the freezing rate causes a change in the impurity composition in the growing crystal. These changes can be revealed in the form of striations by using etchants whose etching rates are sensitive to impurity concentration. Since no impurity striations were observed, it can be concluded that the freezing rate did not fluctuate during solidification of the doped InSb ingots grown under any condition.

Microstructure

The results of the statistical analysis of the grain and twin boundary counts are presented here. The undoped ingot grown at 9 g had one very large grain; it was a single crystal through most of its length. But this crystal was heavily twinned. An undoped InSb ingot grown at 7 g had the fewest number of twins compared to the other ingots grown in the first set of centrifuge experiments. Similarly, the Te-doped InSb ingot grown at 9 g and the Se-doped ingot grown at 10 g had the fewest number of the twins compared to the rest of the ingots grown in the second and third set of centrifuge experiments, respectively. The Te-doped ingot grown at 9 g and Se-doped ingot grown at 5.2 g had fewer grain boundaries than the other ingots grown during the second and third set of centrifuge experiments, respectively. The Te-doped ingot grown at 9 g had two large grains, while the Se-doped ingot grown at 9 g had a number of small grains. The presence of Se in InSb resulted in small grains, while Te in InSb resulted in much larger grain size. Therefore, the microstructure depended on the type of dopant present in the InSb ingot.

To illustrate the magnitude of the effect of acceleration/g level on the microstructure of InSb ingots, Figs. 2–7 were plotted. Figures 2–7 indicate the average number of grain boundaries and twin boundaries per millimeter vs the g level for different ingots. Figures 2 and 3 include error bars representing the standard error of the means at different g levels for grain and twin boundaries, respectively, for ingots obtained from the first set of centrifuge experiments. Figures 4 and 5 and Figs. 6 and 7 are similar plots for the second and third set of centrifuge experiments, respectively.

To check the reproducibility of the grain and twin boundary counts, ground-based control experiments were performed. Five ingots of Se-doped InSb were grown at 1 g in the laboratory. These ingots were analyzed for grain and twin boundaries. The standard error for the mean number of boundaries for these five experiments is 0.0115 mm^{-1} for grains and 0.05 $^{-1}$ for twins. The standard error is a measure of the random variations that can be expected in the grain and twin boundary counts. Comparison of the error bars for the ground-based control experiments with the centrifuge experiments indicates the acceleration and doping influenced the microstructure more than random variations due to other factors. In contrast to the centrifuge results, t-tests revealed that the microstructure of the ingots grown on the KC-135 aircraft was not significantly different from that of those grown on the ground.

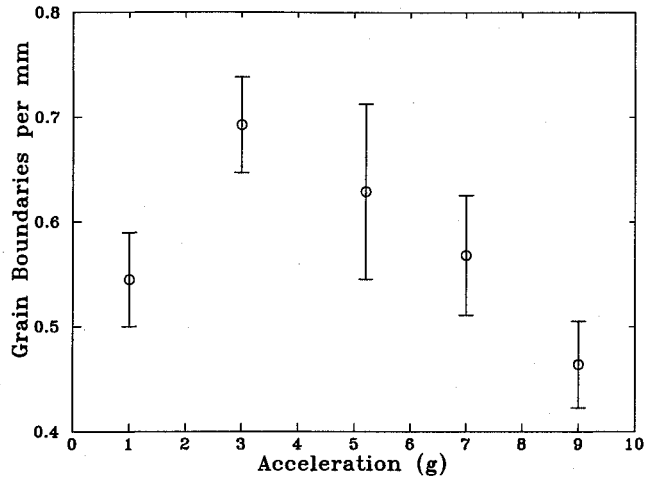

Fig. 2 Mean number of grain boundaries per millimeter of the ingot width vs *g* level for undoped ingots grown in the first set of centrifuge experiments.

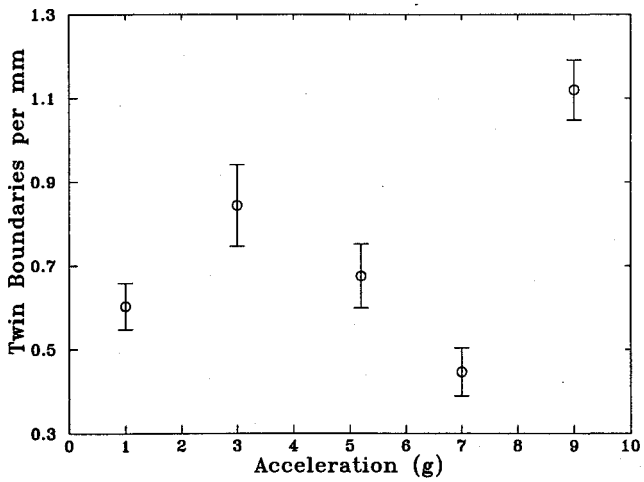

Fig. 3 Mean number of twin boundaries per millimeter of the ingot width vs *g* level for undoped ingots grown in the first set of centrifuge experiments.

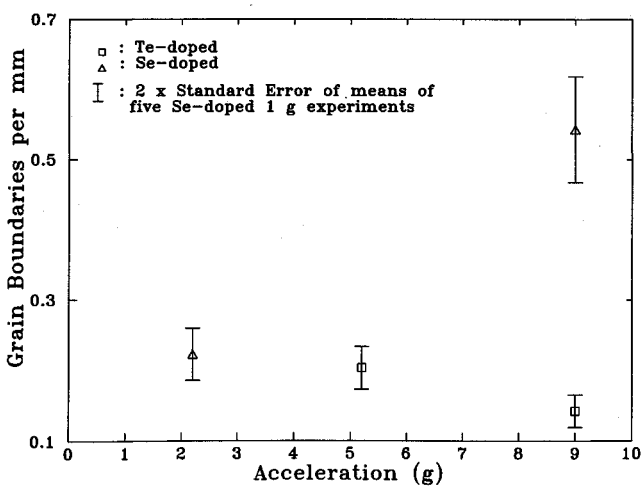

Fig. 4 Mean number of grain boundaries per millimeter of the ingot width vs *g* level for doped ingots grown in the second set of centrifuge experiments. Standard error from mean of five Se-doped ingots solidified at 1 *g* is 0.0115 mm^{-1}.

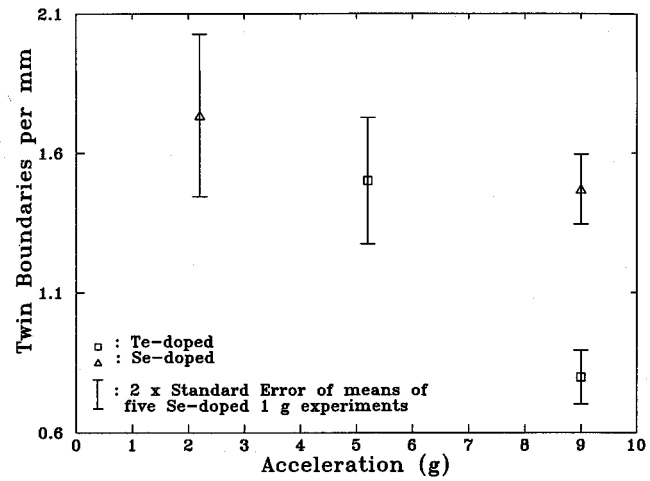

Fig. 5 Mean number of twin boundaries per millimeter of the ingot width vs *g* level for doped ingots grown in the second set of centrifuge experiments. Standard error from mean of five Se-doped ingots solidified at 1 *g* is 0.05 mm^{-1}.

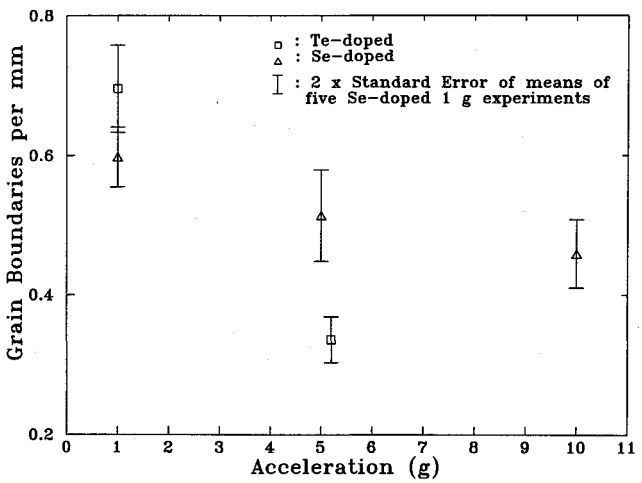

Fig. 6 Mean number of grain boundaries per millimeter of the ingot width vs *g* level for doped ingots grown in the third set of centrifuge experiments. Standard error from mean of five Se-doped ingots solidified at 1 *g* is 0.0115 mm^{-1}.

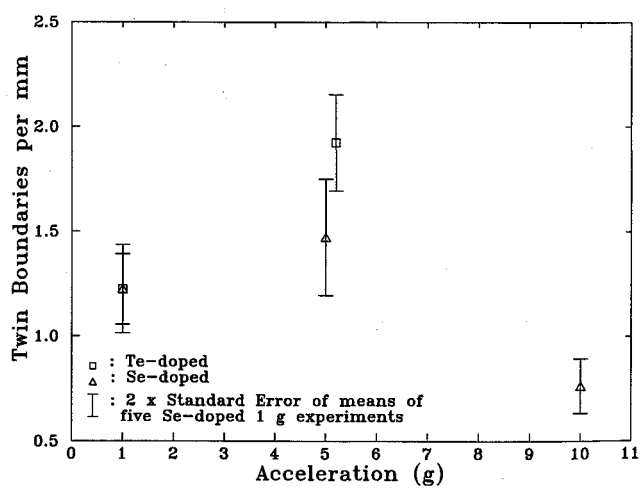

Fig. 7 Mean number of twin boundaries per millimeter of the ingot width vs *g* level for doped ingots grown in the third set of centrifuge experiments. Standard error from mean of five Se-doped ingots solidified at 1 *g* is 0.05 mm^{-1}.

Fig. 8 Microcracks in a Te-doped ingot grown at 9 *g*.

Etching of Te-doped InSb ingots grown in the centrifuge above 7 *g* revealed microcracks as seen in Fig. 8. Microcracks were never observed in undoped InSb ingots, Se-doped ingots, or in ingots grown on the Earth or in the KC-135. The Te concentration in the InSb ingots was approximately 10^{19} atoms/cm^3. It is likely that the Te concentration varied significantly in ingots grown in the centrifuge. Variations in dopant concentration in the ingot would give rise to compositional stresses and microcracks.

Conclusions

Directional solidification experiments on the KC-135 aircraft did not yield smaller diameter crystals. The motion of gas bubbles on the ampoule wall during the low gravity cycle of the KC-135 aircraft was explained based on the minimization of surface energy when the freezing interface contacted the bubbles. Growth of InSb in a centrifuge changed the microstructure and characteristics of the ingots to a considerable extent, that was a function of doping. Microcracks appeared in Te-doped ingots grown above 7 *g*.

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