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THE INFLUENCE OF REDUCED GRAVITY ON
THE CRYSTAL GROWTH OF ELECTRONIC MATERIALS

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The imperfections in the grown crystals of electronic materials, such as compositional nonuniformity, dopant segregation and crystalline structural defects, are detrimental to the performance of the opto-electronic devices. Some of these imperfections can be attributed to effects caused by Earth gravity during crystal growth process and four areas have been identified as the uniqueness of material processing in reduced gravity environment. The significant results of early flight experiments, i.e. prior to space shuttle era, are briefly reviewed followed by an elaborated review on the recent flight experiments conducted on shuttle missions. The results are presented for two major growth methods of electronic materials: melt and vapor growth. The use of an applied magnetic field in the melt growth of electrically conductive melts on Earth to simulate the conditions of reduced gravity has been investigated and it is believed that the superimposed effect of moderate magnetic fields and the reduced gravity environment of space can result in reduction of convective intensities to the extent unreachable by the exclusive use of magnet on Earth or space processing. In the Discussions section each of the significant results of the flight experiments is attributed to one of the four effects of reduced gravity and the unresolved problems on the measured mass fluxes in some of the vapor transport flight experiments are discussed.

INTRODUCTION

High quality single crystals of semiconductors are needed for various important applications in the electronic and optical devices. The imperfections in the grown crystals, such as compositional nonuniformity, dopant segregation and crystalline structural defects, are detrimental to the performance of the opto-electronic devices. Some of these imperfections can be attributed to effects caused by Earth gravity during crystal growth process. To date, four potential benefits have been identified for material processing in reduced gravity:

(1). Elimination of hydrostatic pressure and its influence on the defect formation.

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(2). Reduction of natural buoyancy-driven convective flows arising from thermally and/or solutally (compositionally) induced density gradients in fluids.

(3). Containerless process during solidification of liquid to reduce the stress exerted on the solid during growth and post-growth cooling as well as to suppress reaction or interaction between liquid and container.

(4). Elimination of sedimentation and Stokes migration in the fluids.

However, subtle effects, such as surface tension driven convection (Marangoni effect), are also often masked by gravitational buoyancy-related effects.

In next section, a brief review of the earlier US flight experiments on the growth of semiconductors will be presented. Then we will review the results of recent shuttle flight experiments on the crystal growth of electronic materials sponsored by the NASA Microgravity Science and Applications program. The effects of reduced gravity on the properties of the crystals which were grown by two major growth methods, growth from the melt and the vapor phases, will be discussed individually. Selected ground-based experiments which simulates the reduced gravity environment by applying magnetic fields during melt growth will also be discussed.

BRIEF REVIEW OF EARLY FLIGHT EXPERIMENTS

The significant results of the crystal growth of electronic materials on early missions, such as Skylab and Apollo-Soyez Test Project (ASTP), are reviewed briefly:

1. Melt Growth.

Six ingots of $Ga_{1-x}In_xSb$ with x = 0.1, 0.3 and 0.5 were solidified by a gradient freeze method on Skylab 3 and 4 [1]. The twinning in the flight ingots was 70 % less than in the corresponding ground-grown ingots and the distribution of voids was more uniform in the flight ingots. It is believed that foreign particles at the growth interface can cause nucleation of bubbles, twins and grain boundaries. On Earth the particles would tend to settle on the interface whereas in space the particles would remain dispersed rather uniformly in the melts due to the elimination of sedimentation and Stoke migration. Thus, during the growth process more twins would be formed on Earth while the bubbles would be generated more uniformly in space-grown ingots.

Seeded melts of Te doped InSb were gradiently solidified on Skylab 3 and 4 [2]. Because of the reduction of the buoyancy-driven convection diffusion-controlled segregation of the dopant as determined from Hall measurements and microscopically uniform dopant distribution evidenced from the chemical etching of the sample were achieved in the space-grown InSb ingots. Also, the melt, not wetting the ampoule wall, solidified in a containerless manner.

Also, six ingots of InSb, one heavily doped with Se, were processed on Skylab 3 and 4 by seeded containerless solidification [3,4]. Single crystals with extremely low density of defects were obtained and the maxima in the axial dislocation distributions coincided with the perturbation caused by a maneuver of the spacecraft.

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Single crystals of Ga doped Ge were partially remelted and grown by gradient freeze method on the Apollo-Soyuz mission [5]. Again, the surface features of the space-grown crystals indicated that the actual contact area between the sample and the confining ampoule wall was reduced to a fraction of 1%. Diffusion-controlled segregation as well as compositionally homogeneous materials were achieved in space.

2. Vapor Growth

Six vapor transport experiments of GeSe and GeTe were performed on the Skylab 3 and 4 missions using GeI_4 as a transport agent [6]. The space-grown crystals demonstrated a measurable improvement on the surface morphology. However, the mass fluxes in all cases were greater than predicted for a diffusion-controlled transport and it was postulated that other transport modes in a reactive solid-gas phase system exist.

On the later Apollo-Soyuz mission, three transport experiments on the systems $GeSe_{0.99}Te_{0.01}$- GeI_4, $GeS_{0.98}Se_{0.02}$- $GeCl_4$ and GeS - $GeCl_4$ - Ar were performed [7]. Findings similar to those on the GeSe and GeTe experiments [6], such as the improvement in the crystalline morphology and the higher mass fluxes than predicted for a diffusion-controlled transport, were reported. It was suggested that the existence of "thermochemically" induced convective flows would explain the unexpected transport rates observed in the flight experiments.

RECENT FLIGHT EXPERIMENTS

Most of the recent NASA Microgravity Science and Applications flight experiments on the growth of electronic materials were conducted on the space shuttle missions. The experimental hardware, i.e. the crystal growth facilities, were much more sophisticated than those employed in the early years. The onboard supporting equipment, such as the residual acceleration measuring system, was also greatly improved. These missions included Spacelab, the first International Microgravity Laboratory (IML-1), the first United States Microgravity Laboratory (USML-1) and the second United States Microgravity Payload (USMP-2). The significant findings of these experiments are reviewed below.

1. Melt growth

a. Crystal growth of CdZnTe. Two CdZnTe samples were processed in the Crystal Growth Furnace (CGF) using the seeded Bridgman-Stockbarger method [8]. Careful inspection of the flight samples showed that each sample solidified without wall contact throughout the shoulder region and that when wall contact was resumed, it was only on one side of the ampoule. It was suggested that partial wall contact was caused by an unexpected acceleration asymmetry which was confirmed by the on board acceleration measurements. The characterization technique of Fourier Transform Infrared (FTIR) transmission and infrared microscopy on both the ground and flight samples suggested that the materials were close to the stoichiometric composition and that the samples were experienced similar cooling rates for both the ground and flight samples.

However, some of the most interesting results were that (1) the surface morphology of the wall contact, partial wall contact and no wall contact regions of the flight samples were significantly different, (2) the structural perfection examined by x-ray rocking curves, synchrotron topography and dislocation etch pit analysis indicated that the flight samples, especially in the no wall contact section, contained much fewer defects than the ground samples produced in the otherwise same conditions. In fact, the etch pit density caused by crystal imperfections was reduced from 5 to 10×10^4 cm^{-2} to 500 to 2500 cm^{-2} and it was thought to have resulted from the near absence of the hydrostatic pressure in the microgravity environment which allowed the melt to solidify with minimum or no wall contact, resulting in very low stress being exerted on the crystal during growth and post-growth cooling.

b. Directional solidification of HgZnTe. A preprocessed $Hg_{0.84}Zn_{0.16}Te$ solid solution alloy ingot was back-melted and partially directionally resolidified in the CGF during the USML-1 mission [9,10]. The main objectives for the USML-1 phase of the investigation were: (1) to establish whether preprocessed alloy crystals can be successfully quenched, back-melted and regrown maintaining nearly steady-state compositions, (2) to freeze the diffusion boundary layer and from analysis of the boundary-layer composition to establish a value for the HgTe-ZnTe interdiffusion coefficient and (3) to perform detailed microstructural, electrical and optical characterization on both the grown and the rapidly frozen portions of the flight sample as well as the ground-grown samples to evaluate the effects of reduced gravity. The experiment was inadvertently terminated at about 30% of planned completion. About 5.7mm of sample had been grown at that point.

Detailed surface photomicrographs of the sample clearly showed significant topographical differences between the space- and ground-grown portions. Measurements of the ZnTe content of the sample indicated that the back-melting portion of the experiment was successfully accomplished and the melt back interface location was within 0.5mm of the desired value. The compositional measurements also indicated that the desired steady-state growth for the axial composition was reached at about 3mm into the growth. Structural characterization of the sample showed that both the ground- and flight-portions contained only a few grains and the crystallographic orientation was maintained following back-melting and space-growth. The size and the structural network of the dislocation pits were significantly different for the ground- and space-grown portions of the crystal. High ZnTe content areas were detected in the very top quenched-in portion of the ground-based samples but were not found in the top portion of the flight sample. It was believed that during the cooling process on Earth a significant portion of the solid particles of high ZnTe content that supercooled in the melt just in front of the freezing interface floated to the top of the sample.

The other significant finding was that the interface shape, radial compositional variations and the quenched-in dendritic structures of the flight sample all showed an asymmetric behavior and it was suggested that an

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unanticipated transverse residual acceleration of 4 to 8×10^{-7} g_0, as measured by the Orbital Acceleration Research Experiment (OARE), was the most likely cause.

c. Directional solidification of Se doped GaAs. Two Se-doped GaAs samples were seededly grown in CGF on the USML-1 mission [11]. After two translation periods, the first one at 2.5 μm/s and the second at 5.0 μm/s, the translation was stopped and the remaining sample melts were solidified using a gradient freeze technique. Contrary to the above results in section a and b, no free surface was observed and the diameter of the grown region was constant. The melt-back interfaces were concave (toward the solid), symmetric about the growth axis and have the same deflection for all the ground and flight samples reported and polycrystallinity started to occur in the later stage of the growth for all the samples. In both flight samples, voids in the centerline of the crystals, indicative of bubble entrapment, were found to correlate with the position in the crystal when the translation rates were doubled. The Se segregation data, both axial and radial, indicated that the growth was in the complete mixing regime for all the samples except the first 0.5cm crystal grown in flight sample #1 which was grown under a diffusion controlled condition. It was concluded, with the support from the onboard acceleration measurements, that a large acceleration event occurred at the one hour (about 1cm) point into the experiment which drove the segregation behavior of the sample from diffusion controlled growth to that of complete mixing.

d. Directional solidification of HgCdTe. A $Hg_{0.8}Cd_{0.2}Te$ alloy crystal was grown in the Advanced Automated Directional Solidification Furnace (AADSF) on the USMP-2 mission [12]. The major objective of the research was to establish the limitations imposed by gravity during growth on the quality of bulk, solid-solution, semiconducting crystals having a large separation between their liquidus and solidus curves. During the mission the orbiter was maneuvered into several different attitudes with the results that the residual acceleration vector was aligned differently with respect to the growth axis of the sample. Identifiable regions exist in which a transverse vector has pushed the melt against the wall and allowed it to readily contact away from the opposite wall. X-ray scattering showed that the regions pulled away from the wall tended to be less strained or of higher quality than the opposite surface and considerably better than the ground-based sample. Composition determination on the surface of the material demonstrated significant difference, dependent on the direction of the residual acceleration vector. Preliminary results of radial compositional distribution on two wafers grown during two different orbiter attitudes clearly showed the importance of the residual acceleration vectors. The radial compositional segregation of one wafer was asymmetrical whereas that of the other wafer was symmetrical. While the transverse component of the residual acceleration was about the same during the growth of these wafers the axial residual acceleration was opposite in direction with the stabilized axial acceleration (from the hot to the cold end) producing more symmetrical radial compositional distribution. These are clear indications of three-dimensional fluid flow. A significant portion of the ingot was

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grown with a component of the vector aligned in the direction from liquid to solid and synchrotron x-ray images showed it to be single crystal and of much lower defect density than found in ground-based growth. The characterization on the space-grown crystal is continuing and further microstructural, optical and electrical characterization promise to provide additional data on the potential benefits of growth in the reduced gravity environment.

2. Vapor growth

a. Crystal growth of HgI_2 by physical vapor transport. A single crystal of HgI_2 was grown by seeded physical vapor transport technique in a closed ampoule aboard the Spacelab 3 mission [13]. The experiment studied two of the four microgravity effects as stated in the INTRODUCTION section: (1) the HgI_2 crystals grown on Earth are prone to slip by the force of its own weight, thereby increase the density of defects and (2) to study the effect of convection on the growth rate and the homogeneity of the crystals. During the flight experiment, either due to the changes in the heat transfer mechanism or, as stated in Ref. [13], because of the higher stability of the crystal surface, the difference between the settings of the source and the seeded section had to be increased by 1.6 °C to initiate growth.

The characterization results showed significant improvements of the space-grown crystal over the ground-grown ones. Firstly, when comparing the measured electrical properties, the electron and hole mobility of the space-grown crystal were about twice of the best values ever achieved under ground-grown conditions and the mobility-lifetime product for holes, the most critical factor for detector performance, was improved by a factor of 7; although this performance of the space-grown crystal deteriorated somewhat with time. Secondly, the gamma ray rocking curves of the space-grown crystal showed one single peak as expected of a single crystal without grain boundaries while that of the ground-growth crystals consisted of three peaks, indicating three grains at slight angles with respect to each other.

The above two types of crystals as well as another type of HgI_2 grown in an identical manner but from higher purity starting materials were examined in a recent study using high resolution x-radiation diffraction [14]. Although the long range lattice crystallinity of the ground-grown crystals was slightly better than the other two materials, an additional phase was observed in the ground-grown crystals but not present in the space-grown nor in the terrestrial crystals grown from high purity materials which also displayed a device performance characteristics approaching that of the Spacelab crystal. While the improved performance of the Spacelab crystal was traceable to its higher carrier mobility, for the purified ground-grown HgI_2 the improvement was attributed to longer carrier lifetimes. Thus, it was concluded that the absence of additional phase precipitates appears to be much more important to device performance than the generally high level of lattice uniformity.

A similar experiment was performed later on the IML-1 mission. The gamma ray rocking curves confirmed the previously described superior crystalline quality of the space-grown crystal to the ground-based ones. The results of the high

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resolution monochromatic diffraction images demonstrated that the long range order in crystalline perfection of the space crystal has improved tremendously and was a factor of 15 higher than that of the ground-grown ones. The measured electrical properties of the space crystal, although compared favorably with the average properties of the ground-grown materials, were not as good as the results reported from the previous Spacelab mission.

b. Crystal growth Of GeSe by physical vapor transport. Two experiments on the crystal growth of GeSe in closed ampoules containing 4 and 8 atm pressures of Xe were conducted on the STS-7 shuttle flight. The effects of microgravity on the crystal growth and mass fluxes of the GeSe-Xe system were investigated [15]. The space-grown crystals were much larger and showed considerably improved surface and bulk morphologies comparing to the corresponding ground-grown crystals. In addition, the mass transport rates observed in the microgravity environment were in close agreement with the theoretically predicted values for diffusion-controlled mass transport and those values for the horizontally ground-based transport experiments were, respectively, 7.5 and 23 times larger than the flight results for ampoules containing 4 and 8 atm Xe partial pressures.

c. Epitaxial growth of HgCdTe by chemical vapor transport. Two epitaxial growth experiments of HgCdTe layers on (100) CdTe substrates by chemical vapor transport in closed ampoules using HgI_2 as transport agent were performed aboard the USML-1 mission. The preliminary results [16] showed the following improvements of the space-grown materials: (1) while the best ground-grown wafers have a wavy type step-terrace structure the space-grown layers appeared mirror-smooth under the same magnification (100-500 X), (2) the local compositional variations of the HgCdTe wafers were about 2 to 3 times smaller for the space than the ground-control samples and (3) the width of the x-ray diffraction rocking curves for the USML-1 samples were about one half of that of the ground-based samples.

GROUND SIMULATION OF FLIGHT EXPERIMENTS

Magnetic damping of convective flows in electrically conductive melts can be used to provide a higher degree of control on convection in the melt and thus provide an alternative method to simulate the environment of reduced gravity. Investigation of the effects of magnetic field on the crystal growth began in the early 1950s [17] and its application to improve melt growth has been studied for more than 25 years [18,19]. On Earth, the use of magnetic fields is now a standard technology for elimination of turbulent convective perturbation present during large diameter Czochralski growth of Si and GaAs. However, studies focused on elimination of convective mixing through application of magnetic fields have been much fewer because of the much larger field strengths required to eliminate or significantly reduce melt flows in the laminar regime. The following Bridgman-type growth experiments have been conducted. Matthiesen and co-workers [20] have grown Ga doped Ge in an axial field of 3 Tesla (T) and observed diffusion-controlled segregation of Ga in the first 3cm length of the

grown crystal. The recent investigation by Szofran et al. exhibited a diffusion-controlled segregation through the length of a Ga doped Ge ingot grown under an axial field of 5T. Becla et al. [21] applied a 3T field strength for HgMnTe and observed the elimination of radial compositional undulations . Su et al. used a transverse field up to 0.5T for HgCdTe and HgZnTe and observed compositional radial modifications suggesting a non-axisymmetric flow structure in the melts [22]. Recently, Watring et al. [23] have grown HgCdTe in the presence of an applied 5T axial field. The axial compositional profiles exhibited diffusion-controlled growth with the exception of an abrupt compositional rise at 10cm from first-to-freeze. The lateral solutal segregation effects were in agreement with calculations based on the nonplanar morphology of the growth interface and the assumption of only diffusion mass transfer.

DISCUSSIONS

Most of the significant results of the various flight experiments described above can be categorized into the four microgravity related effects on the crystal growth described in INTRODUCTION with some of the findings yet unresolved.
  1. Melt growth
In melt growth, the effects of reduced gravity from the first and third phenomena, i.e. elimination of hydrostatic pressure and containerless process of liquid, can not be easily separated. These effects were reported in most of the flight experiments and, usually, resulted in better quality on the grown crystals. Space-grown crystals with extremely low defect density were obtained during the containerless processing of InSb [3,4]. The etch pit density in the no wall-contact section of the space-grown CdZnTe crystals improved by a factor of 50 or more comparing to that in the ground-grown crystals [8]. In the flight experiment of directional solidification of HgCdTe [12] x-ray scattering also showed that the regions pulled away from the wall tended to be less strained or of higher quality than the opposite surface and considerably better than the ground-based sample.
The reduction of natural buoyancy-driven convection in the reduced gravity environment (second phenomena described in INTRODUCTION) was evidenced in the growth of Te doped InSb [2], Ga doped Ge [5] and for the first 0.5cm crystal grown in sample #1 of Se doped GaAs [11], where diffusion-controlled dopant segregation was obtained. This might also account for the difference in the size and structural network of the dislocation pits observed in the ground- and space-grown HgZnTe [10]. The reduction in twins and more uniform distribution of bubbles in the GaInSb crystals grown in space [1] could be the result of elimination of sedimentation and Stoke migration (the fourth phenomena).
The sudden doubling of the furnace translation rate during the growth of Se doped GaAs [11] is believed to be the cause for the formation of voids along the centerline of the space-grown crystals. However, It is still not clear what caused the Se segregation results which implied that the growth was in the complete mixing regime for the samples except for the first 0.5cm crystal in flight sample

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#1 and the conclusion [11] that a large acceleration event drove the growth condition from diffusion controlled to that of a complete mixing needs more rigorous substantiation.

Another very important result was the significant effects of residual acceleration experienced on board the spacecraft on the growing crystals, especially in the recent flight experiments where the supporting acceleration measuring systems were extensively utilized. This effect caused the partial wall contact of the space-grown CdZnTe [8] and HgCdTe [12] crystals. The symmetrical radial compositional distribution observed in the HgCdTe wafer grown in a more stabilizing axial residual acceleration, as opposed to the asymmetrical behavior for that grown under a destabilized acceleration condition, is additional evidence of the importance of the residual accelerations. The asymmetric behavior of the interface shape, the radial compositional distribution and the quenched-in dendritic structures of the HgZnTe flight sample [9,10] was also considered to be caused by an unexpected transverse residual acceleration of 4 to 8×10^{-7} g_o. At the same time, the damping effects of magnetic fields on the electrically conductive melts have been demonstrated extensively on Earth. However, the results of scaling laws indicate that at currently available field strength only small-diameter samples (less than 1cm) can be grown on Earth under diffusion-controlled conditions [24]. Extrapolation of these results to operation in space indicates that deployment of magnets with field strength in the order of 0.1T can result in the growth of large diameter crystals under diffusion-controlled conditions. Numerical modeling results indicate that the superimposed effect of moderate magnetic fields and the reduced gravity environment of space can result in reduction of convective intensities to the extent unreachable by the exclusive use of magnet on Earth or space processing. It is expected that magnetic fields on board the spacecraft can be used to control deleterious fluid flows which were caused not only by the residual acceleration but also by the Marangoni or thermocapillary effects during melt growth in reduced gravity.

2. Vapor growth

The main objective of the flight experiments on the vapor transport of GeSe, GeTe [6], GeS, GeSeTe and GeSSe [7] conducted by Wiedemeier and co-workers was to investigate the effects of microgravity on the vapor transport rates. However, the measured mass fluxes in space were much greater than those observed in the ground-based experiments as well as that predicted by a one-dimensional diffusion model [25]. A later study by Buchan and Rosenberger [26] reported on the observation of Ge-Se-I vapor species and on the presence of $GeSe_2(s)$ second phase in the $GeSe-GeI_4$ system. These observations were not taken into account in the analysis of Ref. [25]. After re-evaluating their previous analysis by including the presence of both GeSe and $GeSe_2$ in the source material Palosz and Wiedemeier [27] reported even greater discrepancies - the flight mass fluxes were 5 and 40 times higher than the values predicted by the diffusion model. It was concluded [7,27] that the discrepancy

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was caused by unknown effects of homogeneous gas phase reaction occurred in the chemically complex vapor transport system.

While the problem was not yet resolved there were some questions in the analyses of the transport rates in Ref. [25,27]. Firstly, in a Ge-chacolgenide system, e.g. Ge-Se, the solid-vapor equilibrium reaction: $GeSe(s) = Ge(g) + 1/2$ $Se_2(g)$ was never considered in these analyses and the material was assumed to congruently sublime into $GeSe(g)$ molecule, i.e. $GeSe(s) = GeSe(g)$. From the partial pressure measurements on the Ge-Te system [28,29] it was found that (1) the partial pressures of Te_2 along the Te-saturated $GeTe(s)$ are comparable to that of the GeTe molecular species at the temperature range of 550 to 700 °C, (2) the Te_2 partial pressures can decrease by a factor of 20 from the Te-saturated to the Ge-saturated $GeTe(s)$ and (3) the width of the homogeneity range for GeTe can be as high as 1.2 at. % - the solidus compositions for $GeTe(s)$ at 680 °C are 49.83 and 51.01 at. % Te [29]. Therefore, if the source material is assumed to be one single phase of $GeTe(s)$ the exact stoichiometry of the material need to be defined so that the equilibrium partial pressure of Te_2 can be determined. On the other hand, if it is assumed that the source material is a mixture of $GeTe(s)$ and $GeTe_2(s)$ the partial pressures of Te_2 over the Te-saturated $GeTe(s)$ should be used as the equilibrium pressures over the source. Secondly, Colin and Drowart have reported that they have detected another vapor species, $GeTe_2(g)$, at about one-fifth the concentration of $Te_2(g)$ in their mass spectroscopy-knudsen cell measurements on $GeTe(s)$ between 351° and 691 °C [30]. Therefore, all the above issues should be taken into account to re-evaluate the measured transport rates for the ground-based and microgravity conditions.

The effects of reduced gravity on the space-grown crystals were mainly in the following two areas: (1) the elimination of hydrostatic pressure and (2) the reduction of buoyancy-driven convection. The improved surface morphology in the early flight experiments on the grown GeSe, GeTe [6], GeSeTe, GeSSe and GeS [7] crystals as well as the recent space-grown GeSe [15] crystals and HgCdTe [16] layers were attributed to the reduction in convection intensities. The improvements in the electrical properties and the structural perfection in the space-grown HgI_2 [13] crystals could be attributed to either one or both effects although the absence of additional phase precipitates observed in the space-grown crystals and the terrestrial crystals grown from high purity materials appeared to be more important to device performance than the lattice uniformity.

ACKNOWLEDGMENTS

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