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"Crystal Growth of CdTe in Space and Thermal Field Effects on Mass Flux and Morphology"

H. Wiedemeier

Summary

The primary, long-range goals of this project are the development of vapor phase crystal growth experiments, and the growth of technologically useful crystals in space. The necessary ground-based studies include measurements of the effects of temperature variations on the mass flux and crystal morphology in vapor-solid growth processes. For in-situ mass flux measurements dynamic microbalance techniques will be employed. Crystal growth procedures and equipment will be developed to be compatible with microgravity conditions and flight requirements.

During the recent period of effort, emphasis was placed on the further development of crystal growth and the investigation of relevant transport properties of CdTe. Significant progress has been accomplished concerning the better understanding and stoichiometry control of the physical vapor transport properties of CdTe. The dependence of the mass flux on source temperature has been experimentally established. Most importantly, CdTe synthesis and pretreatment procedures are being developed that yielded considerable improvements in mass transport rates, and mass fluxes which are independent of the amount of source material. Most recent experimental transport rates are in very close agreement with theoretically predicted data for different deviations of the source material from stoichiometry. The combined results to date demonstrate that a higher degree of stoichiometry control of CdTe than before has been achieved during this period of investigation.

Based on the above, a CdTe crystal growth experiment, employing physical vapor transport, yielded very promising results. A crystal boule of about 15 mm length, 12 mm maximum diameter, and a weight of about 6 g was grown. Optical microscopy and X-ray diffraction studies revealed that the boule contained several large sized crystal grains of a high degree of single crystallinity. Further characterization studies of the CdTe crystals are in progress. In view of the reported difficulties in the growth of CdTe single crystals, the crystal growth experiment performed as part of this effort may be considered as rather successful.

The reaction chamber, furnace dimensions, and ampoule location of the dynamic microbalance system were modified in order to minimize radiation effects on the balance performance. Operational testing procedures have been resumed and sensitivity measurements have been initiated.

Introduction

The primary objectives of this project are the development of vapor phase crystal growth experiments of electronic materials, and the growth of single crystals of such materials in space. The microgravity environment provides a significant reduction in the average magnitude of the gravitational force, but most other experimental parameters are the same as on earth. It is, thus, of critical importance to distinguish gravity-related from gravity-unrelated effects on crystal growth, in

order for commercial crystal growth in space to become a reality. In addition, all ground-based processing parameters must be optimized in order to observe genuine microgravity effects on crystal morphology and properties. The major parameters affecting crystal growth and quality in a normal gravity environment are the mass flow and temperature conditions. Very little experimental information is available concerning the effects of temperature perturbations on mass flux and crystal growth. It is, therefore, difficult to determine "a priori" the relative impact of the above parameters on crystal morphology under normal and reduced gravity conditions. For closed-tube vapor transport systems, we have demonstrated a relationship between transport mode and boundary layer thickness [1]. In addition, we have shown previously that even under vertical, stabilizing conditions the vapor phase of transport systems is thermally not stable [2]. The interrelation between temperature perturbations, fluid flow, and crystal morphology indicates the complexity of reactive solid-vapor transport systems. For the design of meaningful crystal growth experiments in space it is of great importance to understand as much as possible the interrelation between crystallographic, thermodynamic, and fluid dynamic parameters and their relative impact on crystal morphology and properties. Under these conditions, it is possible to fully exploit the benefits of a microgravity environment for crystal growth.

Scientific and Technical Basis

The development of crystal growth experiments in space and the growth of electronic materials of improved properties in microgravity environment are the dominant aspects of this program. Based on an extensive literature available of electronic materials, cadmium telluride apears to be one of the most valuable semiconducting materials presently available. Its useful properties for nuclear radiation and infrared detection, for high energy laser windows, for solar cells, and as substrates for HgCdTe crystals are well-known and have been exhaustively discussed in the literature. Frequently, CdTe single crystals are grown from the condensed phase (melt or solution) [3]. Although these techniques produce crystals with high growth rates, the main problems encountered include poor grain size, lack of control or orientation of grains, and a variety of other crystalline defects [3,4]. The development of crystal growth experiments in space and the growth of electronic materials of improved properties in microgravity environment are the dominant aspects of this program. Based on an extensive literature evaluation of electronic materials, cadmium telluride appears to be one of the most valuable semiconducting materials presently available. Its useful properties for nuclear radiation and infrared detection, for high energy laser windows, for solar cells, and as substrates for HgCdTe crystals are well-known and have been exhaustively discussed in the literature. Frequently, CdTe single crystals are grown from the condensed phase (melt or solution) [3]. Although these techniques produce crystals with high growth rates, the main problems encountered include poor grain size, lack of control of orientation of grains, and a variety of other crystalline defects [3,4].

The growth of CdTe from the vapor phase offers a possibility of producing crystals of high compositional uniformity, high resistivity, and low density of imperfections [5,6]. A closer analysis of these and other recent literature concerning the vapor transport of CdTe reveals inconsistencies and discrepancies in transport and growth rates of CdTe. Experimental evidence produced in our laboratory for this sytem strongly suggests that the above discrepancies are caused by stoichiometry problems of the source material. Practically no systematic studies of stoichiometry effects on

mass transport and crystal growth of CdTe in closed ampoules are available in the literature. A similar situation exists for the correlation of crystalline quality and growth parameters for closed ampoule CdTe growth. To achieve better control of the transport and growth process of CdTe, a thorough understanding of the mass transport - growth mechanism and its dependence on the overall stoichiometry of the system is required. This is also of importance because of the relation between stoichiometry, mass flux, growth rate and properties of the grown crystal. These intrinsic, gravity-independent parameters of a system must be controlled in order to unambiguously determine the effects of gravity-dependent parameters on crystal growth.

For the analysis of the average and momentary mass flux of the vapor growth system, dynamic microbalance techniques will be employed. For this purpose, a vacuum microbalance facility has been assembled. From the observed mass flux and thermochemical data of the system, the dominant transport mode can be deduced. In addition, the effects of induced and inherent temperature oscillations on the mass transport rates are of particular interest with respect to variations in mass flux and flow pattern. The dynamic microbalance technique offers the potential to observe changes of transport phenomena in-situ under actual transport conditions. Independent measurements of the vacancy formation in $Hg_{0.8}Cd_{0.2}Te$ [7] have demonstrated the suitability of this technique for transport studies in closed ampoules.

In-situ measurements of transport phenomena are of basic scientific and technological value. In the context of this program, their primary function is to support the crystal growth effort. In order to distinguish between the effects of the above discussed different parameters on crystal growth, the stability of experimental parameters must be well established. We have demonstrated that even minute fluid dynamic disturbances in the vapor phase have a significant effect on the uniformity and crystallographic perfection of $Hg_{0.8}Cd_{0.2}Te$ single crystals [8]. To minimize such disturbances on ground is important for the interpretation and optimal use of microgravity effects in space. In addition, reduced convectional instabilities in space offer the possibility to grow CdTe crystals of larger diameters than available on gorund for comparable levels of perfection. However, the interrelations of the above indicated stoichiometry and of convectional effects on mass transport and growth of CdTe must be experimentally analyzed and understood in order to develop flight conditions. The systematic investigation of this problem is the immediate goal of our CdTe effort.

<u>Progress Accomplished</u>

<u>Development of Theoretical Transport Model for CdTe</u>

In order to compare experimental data with expected mass fluxes for "ideal" conditions, a transport model for the physical vapor transport of CdTe was developed as part of this effort. This requires the computation of partial pressures from thermochemical data, and of diffusion coefficients based on physical constants of the vapor species. Individual mass fluxes were computed based on the assumption of 1-dimensional diffusive-advective flow. The presence of residual gas in the ampoule was considered. The computations were performed for increasing deviations from stoichiometry of the overall solid-vapor phase composition. The program developed applies iterative procedures for a given set of experimental parameters (temperatures of the source and deposition product, partial pressures of vapor species, and source to deposit distance).

A basic theoretical model for the physical vapor transport of CdTe and a temperature difference of 850° -> 830° was developed and reported earlier [9] by us. A comparison of predicted and experimental mass flux data [9] revealed good agreement with respect to the overall trend of the mass fluxes as a function of stoichiometry of the system. However, there were considerable differences in the magnitude of the predicted and observed mass fluxes, the latter being lower than the former, particularly in the vicinity of the stoichiometric composition of the solid-vapor transport system [9]. These differences could be related to the actual homogeneity range of CdTe.

In connection with on-going mass transport rate experiments of CdTe, to be discussed below, it appears desirable to employ higher source temperatures than used previously [9] for the crystal growth of CdTe. Higher source temperatures would not only increase the mass flux, but a corresponding increase of the growth temperature would also increase the surface mobility of Cd and Te atoms on the growing surface of the crystal. Both changes are expected to increase the growth rate and crystalline quality of CdTe. Therefore, in order to compare experimental transport rates with predicted data, the theoretical transport model for CdTe was modified and adjusted to a temperature difference of 900° -> 880^oC. In addition, two different residual pressures of CO have been considered, namely 0.03 and 0.006 atm. A source to deposit distance of Δl = 10 cm was assumed for these computations. The theoretical flux curves are graphically represented in Fig. 1. The abszissa shows the excess Cd and Te in mg per cm^3 free ampoule volume relative to the stoichiometric (Cd:Te =1:1) composition. The overall shapes of the theoretical flux curves are very similar to that reported earlier [0], but the magnitudes of the predicted data are different from those of the earlier computations. The theoretical curves predict a significant decrease in mass flux for even minute deviations of the solid-vapor system from stoichiometry.

Mass Transport Experiments on CdTe

CdTe source materials employed for mass flux experiments were synthesized from stoichiometric (1:1) ratios of elemental Cd (99.9999%) and Te (99.9999%), which were further purified by sublimation prior to the synthesis of CdTe. The elemental mixture was annealed in evacuated, sealed fused silica ampoules at about 850°C for several days. Debye-Scherrer X-ray diffraction patterns of the annealing product confirmed the chemical and structural identity of CdTe. The fused silica ampoules employed for the synthesis of CdTe and for the transport experiments were cleaned and pretreated as discussed before [8]. Several different types of mass flux experiments were performed with the above source material. Any additional treatment of this source material prior to use is discussed in connection with the particular experiments.

We have observed and reported previously [9] that annealing of the source material under dynamic vacuum conditions and at elevated temperatures prior to sealing of the transport ampoule has a significant effect on the mass flux. With increasing annealing temperature the mass flux increases [9]. In order to standardize the pretreatment procedures employed for the present experiments, the synthesized souce material was transferred from the synthesis ampoule to the transport ampoule and attached to the vacuum system. Prior to sealing, the ampoules containing the source material were continuously evacuated and annealed for 5 hours at about 500°C. After sealing, the ampoule was used for the particular experiment.

In order to determine the dependence of the mass flux on the source temperature, a series of experiments was performed in which amounts of 1g of source material, synthesized and pretreated as discussed above, were transported at different source temperatures. For this purpose, the temperature difference between the souce and condensation region was fixed at $\Delta T = 20°C$ and the source to deposit distance was $\Delta l = 10$ cm. The transport experiments were performed under vertical, stabilizing conditions. The results to date in terms of mass flux as a function of source temperature are graphically represented in Fig. 2. The data indicate a relatively steeper increase in mass flux in the temperature range of 825°C to 900°C than at source temperatures above about 900°C. With temperatures greater than 900°C, the mass flux appears to level off. The latter observation (above 900°C) is not consistent with theoretical considerations which suggest that the mass flux should continuously increase with source temperature, at least within the temperature range investigated. The slight deviation of experimental data from theoretical expectations indicates that residual deviations from "stoichiometry" of the preannealed source material become more effective at higher temperatures and reduce the mass flux. This is also indicated by the reproducibility of the mass fluxes at a source temperature of 900°C. The source materials used for the experiments at 900°C were pretreated as discussed above, but come from different batches of elements and/or synthesized CdTe. The combined observations suggest that at least part of the residual deviations from stoichiometry could be trace impurities of the source material.

In order to further investigate the stoichiometry problems, special purification procedures of CdTe are being developed in our laboratory. Emphasis in these procedures is on the minimization of exposure of the source material to regular and even controlled atmospheres. The procedures include repeated sublimation of the synthesized source material in a multi-compartment reaction tube at elevated temperatures followed by annealing at 500oC for about 5 hours under dynamic vacuum conditions. Because the above development is still in progress and consists of several individual steps, a standardized procedure and detailed results will be reported at a later time. Some results to date for the latter material are included in Fig. 2. The mass fluxes observed are consistently greater than those obtained with the other source materials, and the dependence of the mass flux on source temperature is similar to that of the other source materials. A further interpretation of the limited results obtained with the specially treated source material is presently not justified.

For crystal growth in space to be commercially viable, large crystals of high quality have to be produced. That requires the use of large amounts of source material. We have observed and reported previously [9] that the mass flux of synthesized source materials, annealed at 400oC for 3.5 hours, decreases considerably with increasing amount of source material. These earlier data, obtained for a temperature difference of 863o -> 848oC, are included for comparison in Fig. 3. Based on the results of Fig. 2, a source temperature of 900oC was chosen for analogous measurements of the dependence of mass flux on the amount of source material. For this purpose, a fixed temperature difference of 900o -> 880oC and a source to deposit distance of $\Delta l = 10$ ... were employed. The synthesized source materials were annealed at 500oC for a period of 5 hours prior to sealing. The results of these experiments in terms of mass flux as a function of amount of source materials are shown in Fig. 3. The magnitudes of the mass fluxes are greater than those reported earlier [9], because of the higher source temperature of the present experiments. The overall trend of the mass fluxes with source material amount is very similar to that observed earlier (Fig. 3). The source materials employed for the experiments using 1g amounts were pretreated the same way, but come from different batches of elements of CdTe. The combined observations strongly support the above discussed effects of small deviations from stoichiometry on the mass flux. Even for minute deviations, such effects are magnified for large amounts of source material, as demonstrated by the data in Fig. 3. In order to further support the critical importance of stoichiometry control of the source material, some preliminary mass flux data of specially treated source materials are included in Fig. 3. For the same temperature difference (900o -> 880oC), the mass fluxes of the specially treated sources are about five to six times greater than those of the other sources for large amounts of source material. In addition, the mass fluxes of the "high purity" sources are apparently independent of the amount of source material. The latter results indicate that a higher degree of stoichiometry control than ever before has been achieved for these experiments. Because of the technological implications with respect to growth rate and crystal quality, the development effort and investigation of the stoichiometry effects are being continued.

The systematic investigation of the effects of deliberate deviations from stoichiometry of CdTe source material on mass flux has been continued. Earlier experiments for the temperature difference 850° -> 830°C havd been reported previously {9] and are included for camparison in Fig. 1. The present experiments are performed for a temperature difference of 900°C -> 880°C and a source to deposit distance of $\Delta l = 10$ cm. The first set of transport experiments is based on synthesized CdTe, as discussed above, but without annealing at 500°C prior to sealing. For the second series of experiments, CdTe source materials synthesized (as discussed above) and annealed at 500°C for about 5 hours prior to sealing were used for the transport experiments. For both series, 1g amounts of source materials were employed. It should be noted that, except for the stoichiometric composition (zero excess of Cd or Te), the loaded ampoule after annealing at 500°C and prior to sealing had to be briefly removed from the vacuum system to add the desired excess of elemental Cd or Te. After this addition, the ampoule was evacuated again and sealed. The results to date of these experiments in terms of mass flux versus excess Cd or Te (relative to the stoichiometric composition) in the source material are shown in Fig. 1. Both sets of data for the temperature differences 900° -> 880°C are in very close agreement with the functional trend of mass flux versus stoichiometry predicted by the theoretical curves. The differences in magnitude between the two theoretical curves is a result of different residual CO pressures employed for the theoretical computations. The magnitude differences between the two series of experiments clearly reflect the influence of the source treatment, namely annealing at 500°C prior to sealing, which was employed for one series of experiments. This annealing procedure apparently improved the stoichiometry of the source material to such an extent, that the mass fluxes increased by about a factor of five to six relative to the untreated source material and for same excess amounts of Cd or Te. Higher transport rates yield increased growth rates and shorter growth times which are of immediate practical importance for space experiments. The magnitude differences in mass fluxes between the present data (900° -> 880°C) and earlier results (850° -> 830°C, [9]) reflect the different transport temperatures for the same pretreatment procedures.

The combined experimental results of Figs. 1-3 are internally consistent and demonstrate considerable progress in the better understanding of the mass transport behavior of CdTe. The effect of source temperature on the mass flux has been established. The previosly observed dependence of the mass flux on the amount of source material [9] has been further investigated, and preliminary results to date indicate that the mass fluxes of "properly" treated source materials are independent of the amount of souce material. The magnitude of the mass fluxes has been considerably improved, and the agreement of experimental results (for souce materials annealed at 500°C) with theoretical predictions is rather close. The data in Fig. 1 also indicate that the homogeneity range of CdTe is very narrow. The quantitative determination of the homogeneity range of CdTe, the effects of stoichiometric deviations of the source material on the growth rate and on crystal properties are of primary importance for this program. The interrelations of these effects with those of convective contributions to the mass transport and growth processes are rather complex.

Growth of CdTe Crystal

In order to investigate the correlation of present results on the mass transport properties with the actual growth of CdTe single crystals, growth experiments have been initiated during this period of effort. As mentioned earlier, the primary emphasis of crystal growth experiments is on the development of crystal growth parameters for microgravity conditions. This, in turn, requires a thorough understanding of gravity related and unrelated effects on the growth properties. For the design of meaningful space experiments it is necessary to differentiate the effects of intrinsic parameters, such as stoichiometry and purity, from extrinsic variables, such as convective disturbances or ampoule growth, on crystal growth and quality. In accordance with this approach, growth experiments are planned in which controllable variables are changed systematically in order to observe and measure corresponding changes in the crystal growth properties of CdTe.

The source material employed for the present growth experiment consisted of CdTe synthesized, as discussed above, from elemental Cd and Te, which were purified by sublimation prior to synthesis. No additional treatment was applied to the starting material. The growth ampoule of fused silica was of 15 mm inner diameter and 10 cm in length, rounded at the source region end and conically shaped in the growth region. The ampoule was pretreated as described earlier [8]. After loading the ampoule was evacuated and sealed at a pressure of 10^{-6} torr or less.

The furnace used had a nearly isothermal temperature profile of several cm lengths with a maximum temperature of about 900°C. The temperature difference between the source and condensation end was less than 10°C. The ampoule was positioned in the vertical furnace with the source material at about 900°C, and the ampoule was pulled at a slow rate to maintain the growth surface of the crystal at about the same temperature.

The crystal boule of CdTe obtained by this procedure is shown in Fig. 4a. The boule has the external shape of the conical part of the ampoule, and has a very shiny, metallic black appearance. The boule is about 15 mm long, has a maximum width of about 12 mm, and weighs about 6g. About one-third from the tip of the boule, the crystal shows a macroscopic irregular boundary suggesting an interference with the growth process at this time leading to the formation of grains. The crystal surface is nearly flat, of high reflectivity, and displays several facets around the edge. These macroscopic observations indicate that the boule is at least partly single crystalline and contains several grains. The optical photomicrograph using Nomarski differential interference contrast microscopy, of the native surface of the crystal boule in Fig. 4b confirms the mirror smooth surface morphology and the presence of grains.

The natural cleavage plane of the zinc blende type structure of CdTe is parallel to the (110) set of planes. Two typical large sized grains, obtained by cleaving the crystal boule, are shown in the optical photograph of Fig. 5a. The smooth (110) cleavage planes of metallic black luster are clearly visible in the photograph. It should be noted that the presence of smooth cleavage planes nearly always indicate a high degree of single crystallinity of the corresponding grains. This is confirmed by the Laue X-ray back reflection pattern in Fig. 5b of the cleavage plane of the CdTe grain (from Fig. 5a). The Laue pattern clearly reveals the two-fold symmetry of the

(110) plane. The Laue spots are well-defined and indicate a considerable degree of single crystallinity of the large grains.

Further characterization studies of the CdTe crystal boule, employing chemical etching and other techniques, are in progress. Preliminary results indicate the occurrence of etch patterns which may be related to the type and density of dislocations.

In view of the well-known difficulties encountered in the growth of CdTe single crystals, it is justified to consider the present growth experiment rather successful. It should also be noted that this experiment was performed on the basis of systematic investigations of the mass transport properties of CdTe performed in our laboratory under the present contract.

Mass Flux Phenomena Measurements

After the dynamic microbalance facility had been assembled, operational testing procedures of the individual and of interfaced units were initiated during the previous period of effort [9]. These procedures have been continued as part of the present effort. Occasional vacuum problems as a result of heat conduction from the reaction chamber of the system to the vacuum connections required a modification of the furnace dimensions, heated zone lengths, and transport ampoule location relative to the balance system. Because of the complexity of the system with respect to heat losses from the furnace and sensitivity of the balance, the optimum relationship between these parameters cannot be accurately predetermined, but must be tested experimentally. The reaction chamber and furnace dimensions have been modified during the recent period of effort, and operational testing of the facility has been resumed.

As apparent from this report, primary emphasis during this period of effort has been placed on the further development of the CdTe crystal growth experiment and on the investigation of relevant mass transport properties of cadmium telluride. This is in line with the overall priorities of the program. Because of the rather limited man-power available for this particular project, the investigation of mass flux phenomena by microbalance techniques could only be continued at a minor level of effort.

Preliminary Conclusions

During the present period of investigation, the experimental and theoretical effort was primarily devoted to the further investigation of the mass transport and crystal growth properties of CdTe. Significant progress has been achieved in the better understanding of the interrelation between the stoichiometry of the source material and the transport behavior of CdTe. The computational model for the physical vapor transport of CdTe has been extended to higher temperature ranges. The dependence of the mass flux on the source temperature has been experimentally established. Most importantly, synthesis and source material treatment procedures are being developed to clarify the effects of stoichiometry on mass flux. Results to date show that source materials subjected to these special procedures yielded significantly greater mass transport rates than observed previosly, and mass fluxes that are independent of the amount of source material. In addition, the most recently observed mass fluxes are in very close agreement with theoretical predictios of the mass transport rates of CdTe as a function of deviation from stoichiometry of the

overall system. The combined results demonstrate that a considerably improved stoichiometry control of the source material has been accomplished.

Based on the above investigatios, a CdTe crystal growth experiment yielded a large crystal boule. Preliminary characterization of the boule by optical microscopy and Laue X-ray diffraction studies revealed a very smooth, native growth surface, the presence of crystal facets at the edge of the surface, and the existence of large crystal grains within the boule. The rather smooth cleavage plane and Laue patterns demonstrate the high degree of single crystallinity of the CdTe grains. These observations will be further supported by additional crystal characterization studies. In view of reported difficulties encountered with CdTe crystal growth, the results of this experiment are very promising.

Because of the limited man-power available for this project, a reduced effort was devoted to the modification and continued testing of the dynamic microbalance facility.

Future Plans

In accordance with the priorities of the Center and of this project, future activities will be directed towards the further development of CdTe crystal growth in microgravity environment. This involves two major aspects for the immediate future. The further quantitative investigation and identification of the effects of stoichiometry on the mass flux and crystal growth properties of CdTe represent one important part of our approach. The second aspect is concerned with the differentiation of gravity unrelated effects from those of convective disturbances on crystal growth properties. The experimental approach will be supported by theoretical considerations. The results to date and to be obtained provide the scientific and technological basis for the definition of optimum experimental parameters for CdTe crystal growth in microgravity.

Within the limitations of available man-power, the above mass transport and crystal growth experiments will be supported by in-situ measurements of transport phenomena in closed ampoules. For this purpose, the dynamic microbalance facility will be employed. Emphasis will be on the observation of temperature oscillation effects on the transient behavior of vapor transport systems. The task for the immediate future is concerned with the determination of sensitivity limits of the balance with respect to temperature variation effects on the vapor phase.

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(SEE FIGURES ATTACHED)