LARGE PLATE CdTe SYNTHESIS BY SEALED VESSEL TRANSPORT

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(214) 494-5624

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REPORT PERIOD: JANUARY 27-APRIL 30, 1982

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The first quarterly technical report was delayed one month in order to have some results to report. The program did not actually begin until the first week in February. Early efforts were spent in ordering materials and equipment with very little experimental results by the end of March. The original schedule presented in the contract will be resumed for the second quarter.
SUMMARY

The goal of this program is to use vapor growth to form polycrystalline plates of cadmium telluride (CdTe) 6", 8" and 10" in diameter. The resultant material will be evaluated as substrates for mercury-cadmium-telluride (HgCdTe) growth and as an infrared optical material. Late in the program, mercury vapor will be used in an attempt to grow HgCdTe layers on the large area CdTe plates.

Previous workers have grown large plates of CdTe 6" in diameter transporting the vapor using an inert gas. The starting material was pre-compounded. The method to be used in this program is to compound the material and transport the vapor through a filter into the casting chamber for growth in one operation under vacuum. Solid state recrystallization carried out at high temperatures over a long period of time will produce the desired large grain structure. Preliminary experiments were carried out using standard 8" diameter chambers while awaiting materials to construct 6" units. High purity material was compounded but failed to transfer into the growth chamber. The filter separating the chamber apparently was plugged due to attack by the cadmium vapor. The decision was made to suspend the use of filters until other problems could be worked out. Several attempts were completely lost due to failure of the quartz chambers.

Two 6" diameter plates were grown using material compounded during the preliminary runs. The first plugged partially illustrating the need for insulation on the chamber. Also, CdTe stuck to the quartz walls indicating the need to coat with gra-
phite. The second run was much more successful. A plate about 0.5" thick was formed. Almost half the plate had large grains. Some portions showed good infrared transmission. One piece grown in a hot zone showed near theoretical transmission of 66%. Attempts to transfer material and grow to higher temperatures in order to improve quality were not successful.

Transfer of the material at the lower temperatures has been too fast. Next quarter methods will be developed to transfer more slowly and then raise the temperature for recrystallization. Better coating of the quartz will be needed in order to compound and grow in one operation. The results are encouraging and it appears that a practical process may be worked out. Scale up to larger diameters does not appear as a severe problem at this time.
I INTRODUCTION

A crucial problem in the volume production of U. S. Government FLIR systems is the availability of mercury-cadmium-telluride (HgCdTe) detector arrays. Certainly fabrication of the arrays is difficult but the major problem is the availability of high quality detector material. The production of bulk HgCdTe alloy is a slow, difficult low yield process. The preferred method to produce detector materials is to grow HgCdTe layers epitaxially on a high quality cadmium telluride (CdTe) substrate. However, the availability of high quality CdTe is also limited. Part of the reason for the shortage is that the conventional method for preparing CdTe crystals is to slowly (0.1"/hour) zone refine small diameter (1") crystals using the Stockbarger technique. The purpose of this program will be to develop an alternative approach.

Large plates of high purity large grain cadmium telluride will be grown from the vapor in a sealed system. Cadmium telluride will be compounded from the elements and sublimed through a filter into a separate growth chamber 6", 8" or 10" in diameter. Grain size will be increased by solid state recrystallization. Material thus produced will be evaluated regarding purity, optical homogeneity and suitability as substrates for HgCdTe growth. Attempts will be made to grow HgCdTe layers by mercury vapor deposited on large area plates insitu.

II. VAPOR GROWTH OF CADMIUM TELLURIDE

A. General

Cadmium telluride is formed from the group II element
cadmium and the group VI element tellurium. All II-VI compounds, as they are called, are difficult to grow because of their high melting points and the fact that both constituent elements are volatile. An appreciable vapor pressure exists for each element at the solid melting point which leads to complications for melt growth. For the same reason, vapor growth of the solid becomes attractive.

Solid CdTe may be grown from the vapor at temperatures considerably below its melting point, 1097°C, according to the following equation:

\[
\text{Cd (g)} + \frac{1}{2} \text{Te}_2(g) \rightleftharpoons \text{CdTe (s)}
\]

Where:  
\( g \) signifies gas or vapor  
\( s \) signifies solid

The reaction as written is reversible which indicates that solid CdTe will sublime or vaporize into its constituent elements. Thus, the source of Cd or \( \text{Te}_2 \) gas may be either the pure heated elements or a heated source of pre-compounded CdTe solid.

The quality of the CdTe grown from the vapor (or melt) depends upon maintaining the exact ratio of 1:1 between the cadmium and tellurium atoms. The pressure of cadmium vapor (\( p_{\text{Cd}} \)) and the pressure of tellurium vapor (\( p_{\text{Te}_2} \)) in equilibrium over solid CdTe at the growth temperature is very critical and a function of temperature. Deviations from stoichiometry will occur if the ratios are not closely controlled. Supplying the vapors from pure elemental sources heated separately and mixed together at the reaction sight is not too accurate. Even the use of inert carrier gasses does not improve the situation to any great extent. Sublimation of pre-compounded CdTe followed by removal of the vapor to the reaction sight is far more accurate. The compounded material may
be weighed to an accuracy of 0.1% or better. The accuracy of the ratios may be maintained through direct sublimation in a closed system or through use of an inert carrier gas in a flowing system.

One quality criteria not mentioned thus far is crystal-linity. In melt growth, crystals of one single grain may be grown provided that a seed is used and growth rates are very slow. Such restrictions are also the prime limitation relative to diameter of the crystal. In vapor growth, crystals are grown on other crystal surfaces of the same or similar structure, or they grow on an amorphous substrate with spontaneous nucleation and growth occurring over the entire surface. The first case may produce single crystals provided that growth conditions are carefully controlled. The latter method may produce large grain material provided high growth temperatures are maintained over long periods of time.

B. Previous Work

The first extensive investigation of the preparation of CdTe was carried out by D. de Nobel (1) of Phillips. The results published in 1959 details the thermodynamic properties, optical, electrical and semiconducting properties of the materials. The liquidus-solidus curve and related pressure-temperature studies have served as the basis for melt growth of CdTe over the years. Discussions related to melt growth will not be repeated here only as they are relative to vapor growth.

L. R. Shiozawa and co workers at Gould (now Cleveland Crystal Laboratories) carried out an extensive investigation
beginning in the late 1960's concerning the vapor growth of II-VI compounds generally (2) and cadmium telluride (3) specifically. Diagrams from their papers will be used to discuss the problems involved in vapor growth of cadmium telluride.

Figure 1 shows the simple binary phase diagram of the Cd-Te system (2). The diagram indicates a single compound is formed, CdTe which has a congruent melting point of 1097°C. The term "congruent melting point" indicates that except at perfect stoichiometry, below the 1097°C melting point, both Cd rich and Te rich liquids exist in equilibrium with pure CdTe. The point is better illustrated in the diagram (4) shown in figure 2. Only in a very narrow range of stoichiometric ratios does the pure CdTe and its equilibrium vapor pressure exist. The range is less than one part per hundred thousand or 0.001 percent. Minimum vapor pressure at all temperature exist along this line. Equilibrium vapor pressure would be exactly in the right ratio. However, considering the accuracy required, it is more likely that the solid cadmium telluride would be either Cd rich or Te rich. Vapors above the solid would reflect this fact and be larger than those above exactly stoichiometric material.

The pressure of cadmium at the cadmium rich boundary stability field as a function of temperature (2) is shown in figure 3. The tellurium rich (2) is shown in figure 4. In both diagrams, the data is compared to the pressure for the pure element, P Cd (1) and P Te2 (1). For the low temperature, the measured values are very close to those for the pure liquid or close to those corrected according to RAOUlt's Law. ROULT's law states the vapor pressure of a component is lowered by its atomic fraction in a liquid mixture:
FIG. 1. PHASE DIAGRAM OF THE Cd-Te SYSTEM.
FIG. 2. SOLID STABILITY FIELD OF CdTe.
FIG. 3. Cd-RICH BOUNDARY OF THE SOLID CdTe STABILITY FIELD. (PRESSURE-TEMPERATURE PROJECTION.)
FIG. 4. Te-RICH BOUNDARY OF THE SOLID CdTe STABILITY FIELD. (PRESSURE-TEMPERATURE PROJECTION.)
\[ P_{Cd} = x \cdot P_{Cd}(1) \]

Where

- \( P_{Cd} \) is the pressure of cadmium above the melt
- \( P_{Cd}(1) \) is the pressure of pure cadmium at the temperature of measurement
- \( x \) is the atomic fraction of cadmium in the melt.

A similar statement may be made for the pressure of tellurium. The calculated curves using \( x \) are a simple application of Roult's law. Those using the \( Z \) subscript indicates that association between the atoms in the liquid are taken into account. The curves labeled with the \( M \) subscript correspond to the minimum pressure conditions which exist with only pure Cd Te solid present.

The diagrams indicate that the pressure of cadmium may become very large, 4-6 atmospheres, around 1000°C when compounding cadmium telluride. The pressure at the congruent melting point, 1097°C, is about 1.5 atmospheres. The vapor pressure of tellurium is quite low throughout the compounding range. The trick to compounding without an explosion is to stay close to the perfect stoichiometry point and allow time for the liquid phases to become small in quantity. The trick in vapor growth is to be sure that the subliming material is near the stoichiometric ratio so that the resulting vapors are close to the proper ratio. Maintenance of the proper ratio during vapor growth is complicated by the fact that tellurium exists as a diatomic gas, Te2. The effusion rate for a gas is inversely proportional to the square root of its molecular weight and directly proportional to its pressure. Therefore, for CdTe sublimation, we find the effusion ratios to be (2)
$$R \text{Cd/R Te}_2 = (\text{PCd/PTe}_2) \left( \frac{\text{MW Te}_2}{\text{MW Cd}} \right)^{\frac{1}{2}}$$

Where
- R is effusion rate
- P is pressure
- MW is molecular weight

Even though the vapor starts out at the perfect 2/1 ratio, the cadmium atoms move faster changing the effusing gas mixture ratio. For Cd Te, the ratio becomes 1.33 instead of 2. Build up of the excess component may limit the deposition rate in a sealed system where equilibrium type conditions may be established. The equilibrium constant is given by:

$$K_{\text{Cd Te(s)}} = (\text{PCd}) (\text{PTe}_2)^{\frac{1}{2}}$$

The build up of one component over the other shifts the equilibrium and suppresses further sublimation. Some have used capillary tubes to allow the excess component build up in the vicinity of the source to be pumped away or eliminated. Others have used a carrier gas to carry the subliming vapors, with perfect ratios, to the deposition surface.

C. Vapor Growth of CdTe Plates

Shiozawa (3) and co-workers at Gould used physical vapor deposition (PVD) to grow CdTe plates up to 6" in diameter. Figure 5 illustrates the type of systems used. Pre-compounded CdTe was sublimed from the hot zone of a furnace to a slightly cooler zone. Best results were obtained when growth temperatures of 1000-1050°C were used. Recrystallization was allowed to occur over periods up to 400 hours (17 days).

Figure 6 illustrates the type of grain sizes which were obtained. Growth and recrystallization at 950°C produced small grains. Fast deposition and short growth periods even at the high temperatures produced plates with small grains and voids.
FIG. 5. HORIZONTAL SYSTEM FOR CRYSTAL GROWTH BY GRAIN GROWTH
FIG. 6. Lapped Surfaces of CdTe Window Blanks Highlighting Grain and Twin Patterns.
The systems were operated semi-sealed. Argon gas was used as a carrier. Pressures up to 1 atmosphere was used but 180 mm, the vapor pressure of stoichiometric Cd Te at 1050°C, was the preferred pressure. Later versions of the system used graphite parts to minimize the effects of air leaks. Also, inert gas was flowed through the furnace to protect the graphite components.

Plates thus produced were heat treated with both Te$_2$ and Cd vapors at temperatures of 700-800°C. Treatments were alternated and lasted 1-5 days. Measured absorption at 10.6 μm was 0.001-0.002 cm$^{-1}$. The effect of heat treating on the infrared transmission of one of their CdTe plates is shown in figure 7. From the transmission for Cd treatment alone, one can see that dual treatment is necessary. After treating with Te$_2$ vapor, almost theoretical transmission was obtained.

III APPLICATION OF THE AMTIR METHOD TO THE GROWTH OF CdTe

A. Preparation of AMTIR-1 glass

A simplified diagram depicting the glass compounding and casting process developed by Amorphous Materials, Inc. to produce AMTIR-1, is shown in figure 8. A high purity quartz container is placed in a dual zone resistive heated furnace. Zone 1 contains the round empty chamber which will serve later in the process as the casting mold. Zone 2 contains the glass compounding chamber. The process employed at Amorphous Materials combines all three glass processes (element purifications, compounding the glass and casting the plate) into a single continuous process.
FIG. 7. OPTICAL TRANSMISSION OF Ga-DOPED CdTe
FIGURE 8. GLASS PROCESS DIAGRAM

PURIFICATION

COMPOUNDING

DISTILLATION

CASTING
In the first step, both chambers are evacuated and heated to remove moisture. Small amount of aluminum wire is added to the elements to act as a getter of oxides in the molten glass. After several hours, the chambers is sealed off while still under vacuum by heating and collapsing the pumping tube. The furnace is closed and need not be opened again until the glass is quenched.

The temperature is raised in both chambers while rocking the furnace. The casting chamber is heated to prevent premature transfer of the elements from the compounding chamber. The furnace is rocked for several hours to insure that all the elements have reacted properly and are uniformly mixed.

Rocking is stopped and the glass casting chamber cooled to allow transfer of the compounded glass into the casting chamber. Distillation occurs through a porous quartz filter which removes all particulate matter. The distillation generally takes 12-15 hours. The amount of material passed through the frit varies depending upon diameter and thickness. A 6" diameter plate 2" thick requires about 4Kgms of glass while a 8" diameter plate 2" thick requires 7Kgms of material. It is interesting to note that the purity of the finished material improves with quantity prepared. The total amount of impurities from quartz walls, vacuum system, etc. remains constant while the ending concentration decreases with increase in mass. The distilled glass is mixed by rocking. The glass is then allowed to cool while rocking down to the casting temperature range. Rocking is stopped and the furnace leveled up. The glass is rapidly cooled using compressed air to the anneal range 370°C. The glass is annealed 6-8 hours before being allowed to cool to room temperature.
AMTIR-1 glass is compounded in very unique and valuable high purity quartz containers. Casting chambers 6", 8" and 10" in diameter are routinely prepared. Methods have been worked out to open the chambers with a diamond saw so that the cast plate may be removed without breaking. The chambers are cleaned up and resealed for reuse. The compounding tubes are not saved. The impurities within the high purity beginning reactants are left behind during the distillation. The residues are sometimes quite large indicating the effectiveness of the method in preparing high purity solid materials.

B. Modification for Vapor Growth of Cd Te

The temperature range involved in cadmium telluride compounding and solid state recrystallization is 1050-1100°C, well above the compounding temperature used for AMTIR-1. However, the furnaces are of sufficient caliber to reach and maintain these temperatures continuously. Times involved are much longer. From start to finish the AMTIR-1 process requires 48 hours to produce a plate. High quality cadmium telluride grown from the vapor with solid state recrystallization (grain growth method) requires 300-400 hours (12-17 days) to produce good quality material 1" thick. For a 6" diameter plate, 2700gms of cadmium telluride would be grown. Considering all these factors, the procedure carried out would be as follows:

1. The correct amounts of high purity cadmium and tellurium will be weighed out and placed in the compounding chamber as before. For a 6" plate, about 2700 grams will be required. High purity aluminum wire will be added (10-20ppm) to serve as an oxide getter. The casting chamber may need to be carbon coated using graphite formed from the pyrolysis of acetone.
2. The compounding and casting chambers will be heated to 200°C to remove moisture and the chamber will be tipped off.

3. The casting chamber will be heated to 1100°C. The compounding side will be heated to the boiling point of cadmium (765°C) and rocking begun. Over a period of hours, the temperature will be increased to 1100°C while rocking to insure complete compounding of the cadmium telluride.

4. Rocking will cease and material transfer begun. The casting chamber side will be reduced to a temperature of 1000-1050°C to bring about transfer of material to the plate side. The high temperature will be maintained so that grain growth will be a continuous process. Particulate matter and oxide impurities will remain behind in the compounding chamber. The time involved may be as long as 10-15 days.

5. After completion of the run, the cadmium telluride plate will be removed from the chamber using a horizontal glass saw as is done for AMTIR plates.

C. Program Objectives

A large diameter, thick, high purity, large grain plate of cadmium telluride will be formed. Individual large grains 1-4cm³ in volume will be visible in the surface of the plate after lapping and light etching. Evaluation and treatment of the plate depends upon the desired use.

First emphasis of the program will be to establish the growth parameters using 6" diameter chambers. Purity of resultant material will be verified by chemical analysis. Electrical, mechanical and optical evaluation techniques will be
used where appropriate. After growth parameters are established, scale up for 8" diameter and 10" diameter plates will occur.

Substrates will be cut out of 6" 8" and 10" diameter plates for evaluation for HgCdTe epitaxial growth. Disks 2" in diameter and 0.1" thick will be prepared for evaluation by groups designated by DARPA.

Disks 2"D x 0.25" thick will be prepared for optical homogeneity verification. The disks will be polished flat and paralleled in order to carry out MTF based image spoiling tests. Additionally, lenses will be fabricated for evaluation in the U. S. Army small imager FLIR common Module.

The last goal of the program will be to grow large area HgCdTe layers insitu. Mercury vapor will be admitted into the 6", 8" and 10" diameter CdTe growth chambers in an attempt to produce layer growth of the right composition.

IV. RESULTS FOR THE QUARTER

A. Equipment Changes

The compounding and casting unit designed and built during the previous facility development program (1979) was equipped with Barber Colman 524 controllers which functioned to only 1000°C. The present program requires the capability to exceed the CdTe melting point of 1097°C. Therefore, new units had to be purchased. We selected Barber Colman 5643 units which may reach 1200°C and control to ± 2°C at that temperature. The units have digital display and may be set up in a digital manner using a keyboard and the display. Variables such as pro-
portional band, reset, rate, etc. may be set digitally which leads to excellent reproducibility between individual units and run to run.

The overtemperature control units on the panels were also rated for only 1,000°C. The two units were removed and sent to the local service shop for modification to extend the temperature range to 1200°C. Meanwhile, we will operate without overtemperature protection.

B. Materials

Five nines cadmium bars and five nines tellurium chunks were ordered from ASARCO. Zone refined five nines bars of tellurium were also ordered from KBI. Delivery occurred in 4-6wks.

Amorphous Materials had discontinued preparing 6" plates of AMTIR-1. The 8" plates were considered much more efficient from a labor standpoint. Supplies required to prepare 6" chambers were no longer available from our local quartz supplier and had to be special ordered. Some delay was experienced awaiting delivery.

C. Temperature Measurements

Control of temperature and thermal gradients is of paramount importance in vapor growth. In order to ascertain the magnitude of the temperature errors, an 8" diameter casting chamber and connecting compounding tube were constructed with temperature wells placed inside at strategic points. The chambers were evacuated and sealed off while containing enough
tellurium (10gms) to provide ½ atmosphere of pressure at 1100°C.

Measurements inside the reaction (R) chamber showed a variation of 1010-1048°C while the controller was set at 1060°C. Serious problems existed which were corrected by the use of additional insulation.

Measurements within the casting (C) chamber showed a variation of 1018-1022°C while the controller was set to control at 1040°C. The gradients were much smaller. The temperature is monitored as a matter of standard procedure and used in the process control rather than controller settings.

D. Preliminary Attempts.

The time required to receive 6" diameter quartz materials was uncertain. The decision was made to begin using the standard 8" diameter compounding and casting quartz containers. Such units are available everyday if needed. The compounding and casting chambers are separated by a quartz wool filter of our own design and construction.

The first two attempts succeeded in compounding 5Kgms. and 3Kgms. of high purity material but none was transferred into the casting chamber. Material from run #2 showed 45% transmission beginning at 2.5μm but dropping off rapidly to about 5% at 12μm. Free carrier absorption appears to be the cause. The next three attempts using 8" chambers were completely unsuccessful due to failure of the quartz. Failures occurred above 1000°C. Examination of the failed systems also revealed that the quartz wool filter was being attacked and plugging up. The attack was probably due to cadmium vapor and
could not be avoided. The decision was made to abandon the use of a filter for the present.

E. First 6" Diameter Plates

The first run with a 6" chamber concentrated on just being able to transfer the material from one chamber to another. A chamber was constructed with no filter and the charge placed in the R side was 2Kgms of pre-compounded material from an earlier run. After evacuating heating and sealing the unit, the controllers were set with R at 1060\(^\circ\)C and C at 1020\(^\circ\)C. A slight flow of air was blown on the bottom of the 6" casting chamber. No transfer occurred. The C side was turned down to 975\(^\circ\)C. All the material transferred in less than 12 hours. The material was left to grow for 30 hours at that temperature and slowly cooled down using the CAM control unit.

A plate was formed but it was very thin. About 500gms of the beginning 2000 was found in the transfer tube between the two chambers. Also, some of the CdTe deposited and grew on the walls of the round casting chamber. Obviously, there was a variance in temperature for the walls of the chamber. Insulation was needed. The thin plate was stuck to the quartz indicating a need for carbon coating of the chamber surfaces.

Precompounded material was used for the second attempt with a 6" chamber. Quartz wool was placed around the transfer tube and the outside of the casting chamber. The charge was 2Kgms. of CdTe. Both chambers were evacuated and heated to 500\(^\circ\)C. Acetone vapor was admitted to the chambers and a carbon film (graphite) allowed to form. At the completion of
this operation, the unit was again evacuated and sealed off.

Transfer occurred with the R ride set of 1060°C and C at 980°C. A slight flow of air was used on the bottom of the casting chamber. All the material was transferred in 17 hours, overnight. The C side was then raised to 1050°C to facilitate recrystallization. The monitored temperature inside the chamber was only 1035°C indicating a growth temperature close to 1030°C. The material was allowed to grow 100 hours and then slow cooled.

Examination revealed that most all the material had been transferred and most all was in the bottom in the plate. The only exception was a small amount of material deposited opposite the entry of the transfer tube and above the insulation wrapping. The plate on the bottom had a wedge indicating a thermal gradient. The minimum thickness was about 0.5". A small amount of material grew up the inside wall of the container. A photograph of the plate as it appeared upon removal from the chamber is shown in figure 9A. Note the taper in thickness and the ridge around the outside. The thinner area corresponds to a slightly higher temperature. The diagram in figure 9B. shows the general appearance of the plate. The large area of large grains corresponds to the thinner, hotter portion of the chamber.

The plate was ground flat and parallel. Both sides were polished. Some fracture lines appeared. Examination with an IR microscope revealed extensive precipitation at grain boundaries. In the small (mm's) grain area, scattering was extensive. In the large (cm's) grain area, general precipitation occurred
Figure 9 A. 6" Diameter Plate After Removal From Chamber.

Figure 9 B. Diagram of Plate Structure
but was not nearly as extensive. The measured infrared transmission of the plate in the large grain area rose from 20% at 2.5μm to 30% at 25μm. The thickness was 0.4". The transmission increase indicates scattering due to particles and or poor polish.

The grains of the piece of material found above the insulation on the chamber wall were large. A piece cut and polished for evaluation 0.175" thick showed IR transmission of 57% at 2.5μm rising to 66% at 25μm. The results indicate the need to recrystallize at a higher temperature.

The final 6" diameter run for the period was an attempt to transfer and grow the CdTe at a higher temperature. A growth temperature of 1050°C was chosen. The temperatures were set with R at 1100°C and C at 1060°C. The monitored temperature inside the chamber was 1048°C. The flow of air under the chamber was not used. The furnace was opened the next day and it appeared the CdTe had transferred. However, it is very difficult to see when the furnace is 1000°C or more. The material was left to grow 76 hours and then cooled. When examined, it was discovered that no transfer had occurred.

F. Discussion of Results

The goal of this program is to compound CdTe, transfer the material into a second chamber through a filter and re-grow the material in a large grain plate. We have not yet at this time achieved all four goals. Two goals have been partially achieved: Compounding CdTe and growing a large grain plate.
Transfer from one chamber to the other can be achieved thus far at a low temperature, around 950-975°C. The temperature is below the boiling point of tellurium which may help to alleviate the problem of Te₂ vapor build up. The rapid sublimation at low temperatures leads to voids in the material. We will have to learn to control the rate of sublimation.

Use of the filter has been suspended to promote progress. The filter failure is due to cadmium vapor attack. Carbon coating of the filter may help to protect the wool. Another possibility is to use a fritted disk in place of the wool.

Thus far, we have not succeeded in compounding CdTe and then transferring the material. Quartz failure has occurred in almost every cast of compounding. We are not sure if the failure is the result of cadmium attack or if the failure has been random. Both plates thus far have been formed from pre-compounded material. We will need to do a better job of graphite coating the quartz.

Increasing the growth temperature after transfer is a simple matter. The trick is to transfer the material at a slow rate so that regrowth will produce a good quality plate.

The results are encouraging. Proper conditions may be selected which will allow achievement of all our goals. We may be able to apply our methods to other materials provided enough vapor pressure and phase information is available.
V. PLANS FOR NEXT QUARTER

1. Transfer CdTe into the casting chamber at a slower rate perhaps by lowering the temperature of the R side as well as the C side.

2. Improve the carbon coating of the Quartz chambers both C and R sides.

3. Raise the regrowth temperature to 1050-1075°C.

4. Fabricate a large area filter which may be precoated with carbon for protection.
REFERENCES

1. "Phase Equilibria and Semiconducting Properties of Cadmium Telluride" By: D. de Nobel
   Phillips Research Reports 14 361-399 (1959)

2. "Research on Improved II-VI Crystals" By: L.R. Shiozawa and J. M. Jost. Clevite Corporation, Cleveland, Ohio

3. "The Application of Physical Vapor Deposition to Semiconductor Materials for Use as High Power Infrared Windows"
   Gould Laboratories, Cleveland, Ohio