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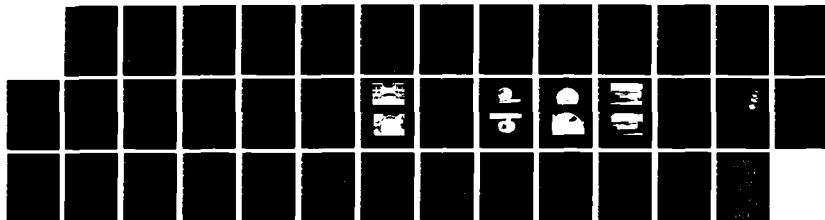
LARGE PLATE COTE SYNTHESIS BY SEALED VESSEL TRANSPORT
(U) AMORPHOUS MATERIALS INC GARLAND TX
G R CRONIN ET AL 31 DEC 85 DAAG29-85-C-0004

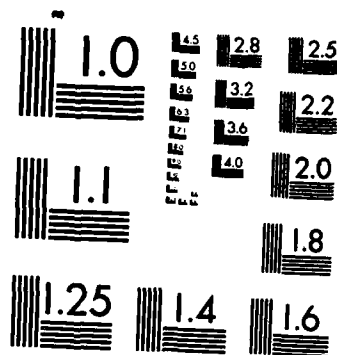
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LARGE PLATE CdTe SYNTHESIS BY SEALED
VESSEL TRANSPORTFINAL TECHNICAL REPORT
DECEMBER 31, 1985G. R. CRONIN AND A. R. HILTON
PRINCIPAL INVESTIGATORS

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VESSEL TRANSPORT

FINAL TECHNICAL REPORT
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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) A compounding process originally developed for the preparation of large plates of IR transmitting chalcogenide glass has been applied to cadmium telluride. A three zone furnace system is used to vaporize, condense and compound near stoichiometric quantities of 5-nines grade cadmium and tellurium. A controlled directional freeze results in circular plates up to 25 cm in diameter which are free of voids or gross inclusions and contain large areas of single crystal materials.		

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20. ABSTRACT (Continued)

A heat treating process under cadmium and tellurium vapor has been developed which results in a 100 fold improvement in infrared transmission over non heat treated material. Absorption coefficients less than 0.002 cm^{-1} have been observed from 2.5 to $25\mu\text{m}$ wavelength on samples ranging up to 2 cm in thickness.

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SUMMARY

Compound
A compounding process originally developed for the preparation of large plates of IR transmitting chalcogenide glass has been applied to cadmium telluride.

A three zone furnace system is used to vaporize, condense and compound near stoichiometric quantities of 5-nines grade cadmium and tellurium. A controlled directional freeze results in circular plates up to 25 cm in diameter which are free of voids or gross inclusions and contain large areas of single crystal material.

micron meter
A heat treating process under cadmium and tellurium vapor has been developed which results in a 100 fold improvement in infrared transmission over non heat treated material. Absorption coefficients less than $0.002/\text{cm}$ have been observed from 2.5 to $25\mu\text{m}$ wavelength on samples ranging up to 2 cm in thickness. The attainment of near theoretical transmission, as expected, is dependent upon the sample thickness, anneal temperature, pressure of cadmium or tellurium used in the anneal, and length of time of treatment. Details of the compounding process as well as the annealing process are included in the text.

Start
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 too 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.

CONT'D Pg. 1
Large plates of high purity large grain cadmium telluride were grown in a sealed system. Cadmium telluride was compounded from the elements, which are sublimed through a filter into a separate growth chamber 6", 8" or 10" in diameter. Grain size was increased by using a controlled freezing technique. Material thus produced was evaluated regarding purity, optical homogeneity and suitability as substrates for HgCdTe growth.

A. Program Goals

Specifically, the goals of this program can be stated as follows:

1. Apply the compounding techniques developed for the production of IR transmitting chalcogenide glass to the preparation of large area cadmium telluride plates.
2. Prepare 6, 8, and 10 inch diameter plates with thickness of 1 inch or greater without voids, gross inclusions or excessive dendritic growth.
3. Obtain large single crystal regions (one inch square or greater) from which oriented substrates can be cut.
4. Develop a heat treating technique resulting in low absorption optical grade material.

II. APPLICATION OF THE AMTIR METHOD TO THE GROWTH OF CdTe

A. Preparation of AMTIR-I glass

A simplified diagram depicting the glass compounding and casting process developed by Amorphous Materials, Inc. to produce AMTIR-I, is shown in Figure 1. A high purity 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 purification, compounding the glass and casting the plate) into a single continuous process.

In the first step, both chambers are evacuated and heated to remove moisture. A 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 chamber 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 wall, 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. 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.

IR GLASS COMPOUNDING - CASTING SYSTEM

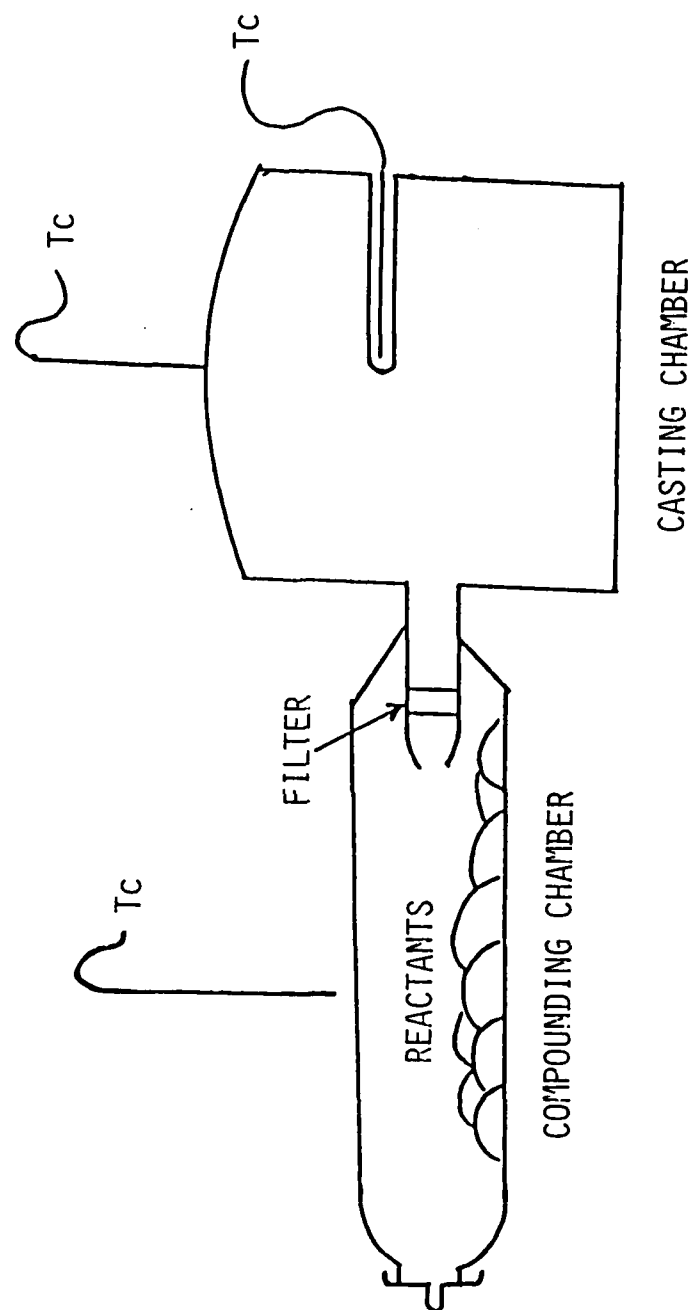


FIGURE 1

AMTIR-I glass is compounded in very unique 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 and resealed for reuse. The compounding tubes are not saved. The impurities within the high purity 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.

II-B MODIFICATION OF THE GLASS PROCESS FOR THE GROWTH OF CdTe

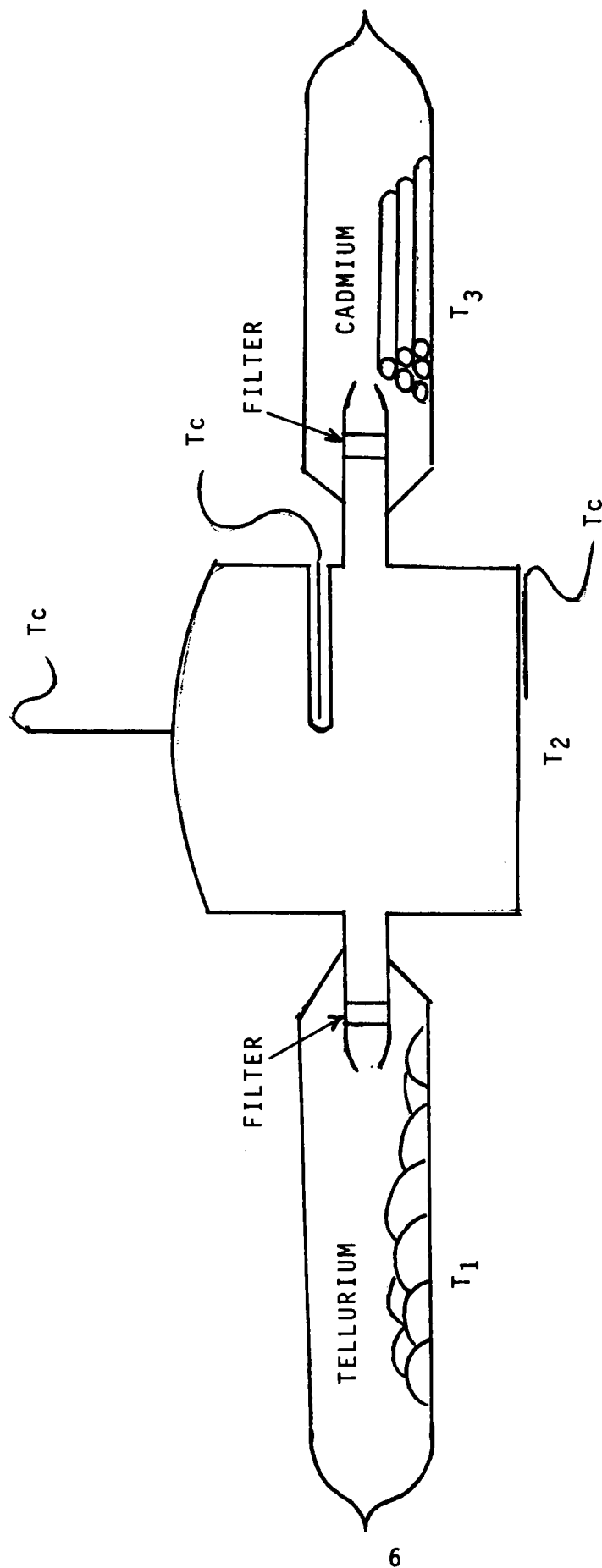
The process used for the growth, from a near stoichiometric melt, of large plates of CdTe is essentially the same as that previously described for the preparation of AMTIR-I glass. The major modification is the addition of a second side arm onto the central plate chamber to contain the second elemental component (Figure 2). Early in this program it was determined that better results were obtained if the cadmium and tellurium were vaporized and condensed separately into the central chamber rather than as the pre compounded Cd Te. The compounding process was carried out by the slow distillation of first cadmium, then tellurium into a central evacuated chamber. The distilled vapors in both cases were filtered by means of a quartz fiber plug placed near the entrance to the central chamber. The cadmium is slowly transferred to the central chamber over a time period of about four hours at 750°C (vapor pressure approximately one atmosphere). Then the tellurium is transferred at 800°C (vapor pressure approximately 150mm) over a time period of 8-10 hours.

After material transfer, the central chamber is placed on an automatic temperature program which is designed to bring the CdTe to just above the melting point (1092°C). This is followed by a slow cooling cycle to promote the growth of large area crystals, a quench cycle, and finally a slow anneal cycle. The complete temperature cycle is shown in Figure 3.

During the slow cool from above the melting point cycle, a low volume (1-2 lit./min.) air flow is directed on the bottom center surface of the central quartz chamber. The purpose of this cooling air is to ensure that the freezing is initiated at the center of the plate and crystal growth occurs outward towards the periphery.

The cooling air is channelled from the center of the plate outward through spaces cut in the quartz fiber insulation on which the casting chamber is resting (see Figure 4). If the cooling air flow is sufficient, freezing

CADMIUM TELLURIDE COMPOUNDING ARRANGEMENT



TRANSFER

T ₁	800°C
T ₂	400°C
T ₃	750°C

COMPOUND

T ₁	800°C
T ₂	1100°C
T ₃	800°C

FIGURE 2

CENTER CHAMBER (T₂) TEMPERATURE PROGRAM

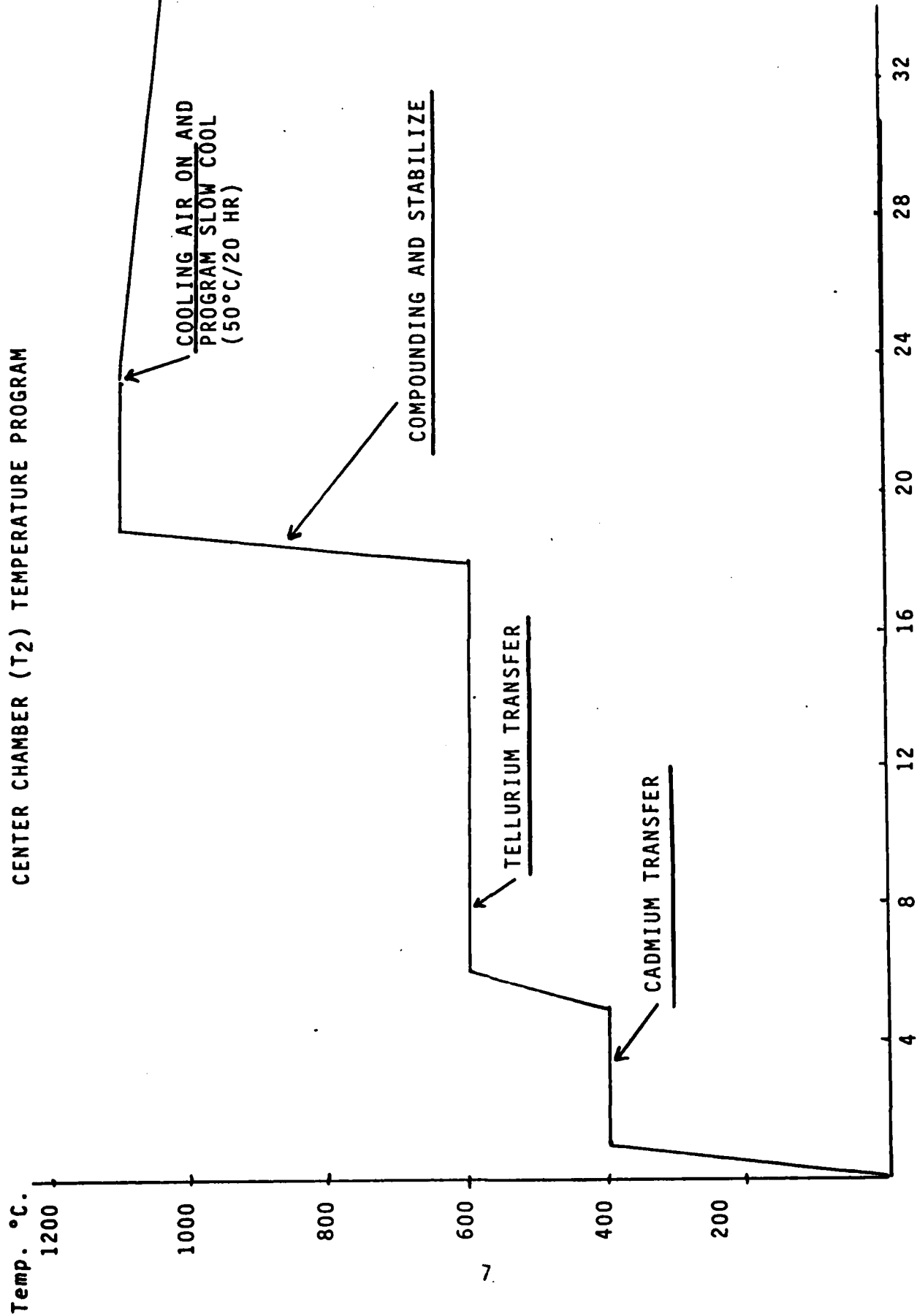


FIGURE 3

COOLING AIR FLOW PATTERN ON BOTTOM OF GROWTH CHAMBER

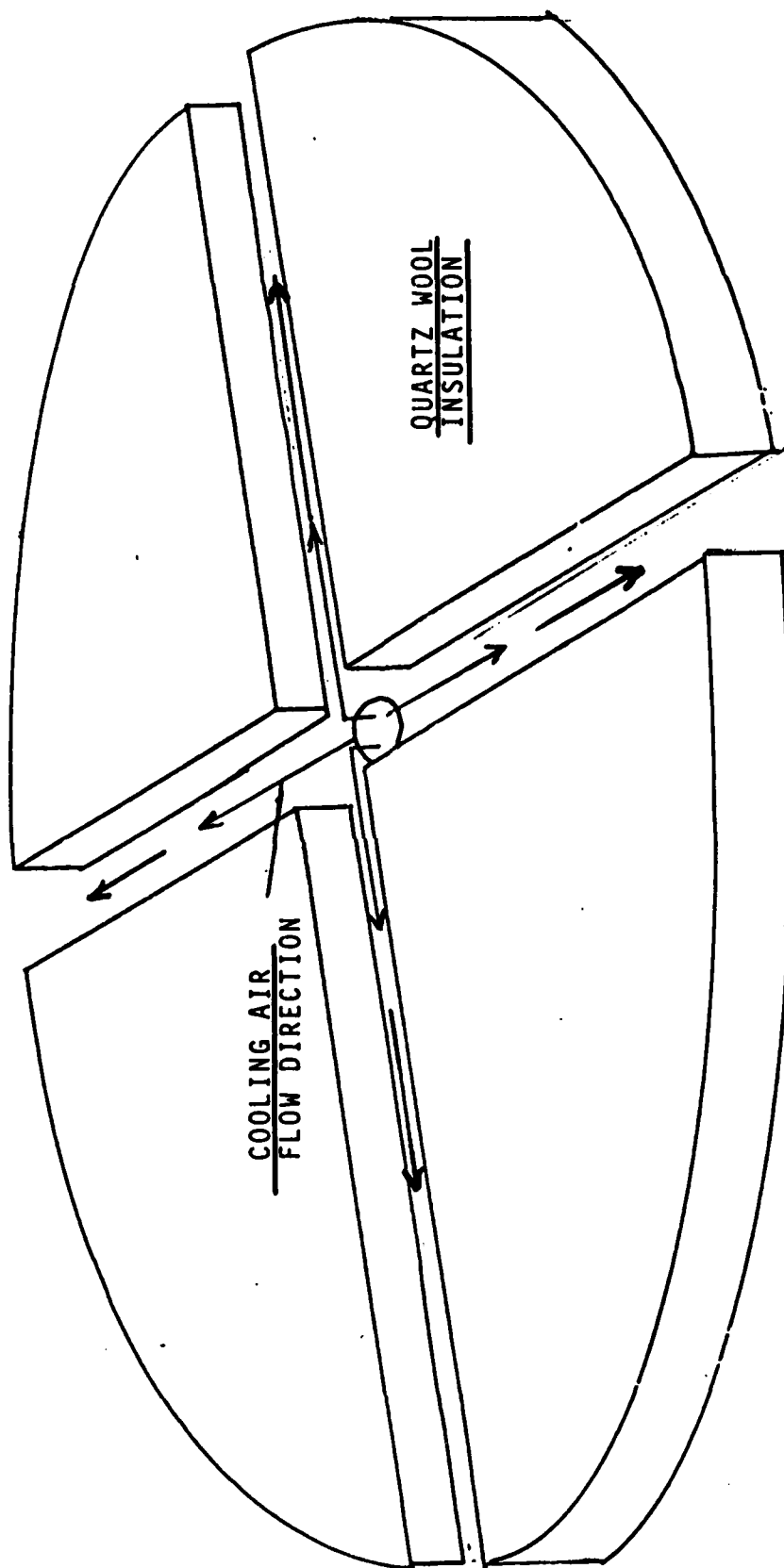


FIGURE 4

of the plate is initiated at the center of the bottom of the melt and proceeds simultaneously in an outward and upward direction as shown in Figure 5. Evidence for this type of freezing pattern is provided by the appearance of the upper surface of the cooled plate. Due to the fact that a 100% stoichiometric melt is difficult to achieve under these conditions, a small amount of residual dendritic material is usually observed on the outer periphery of the upper surface of the plate indicating that this was, in fact, the last portion of melt to freeze.

A photograph of the furnace system prior to removal of the quartz chamber containing an 8 inch diameter plate is shown in Figures 6 & 7.

III. RESULTS AND ACCOMPLISHMENTS

The work for the present reporting period has concentrated on two aspects of the program:

1. Confirm the consistency of the process by repeating the preparation of CdTe plates of thickness of one inch or greater which are void free, high purity and with large individual single crystal regions.

2. Develop a subsequent heat treatment process which can be used for preparing optical grade material of any size shape, or thickness.

A. Plate Preparation, No. 61

A total of six 6-inch diameter plates were compounded under the present contract. Plate #61 was compounded from 1.499Kg cadmium (5-nines purity) and 1.843Kg tellurium, (also 5-nines grade). The cadmium was lightly etched in dilute HCl before being sealed in the side chamber. The tellurium, in chunk form, was loaded in the as received condition. A vacuum of less than 3 microns was maintained on the compounding chamber for approximately 2 hours prior to sealing the chamber closed. Transport of the cadmium and tellurium occurred without incident in the usual manner and the plate chamber was slowly brought up to 1112°C. A 12-14 C temperature gradient was maintained between the center of the chamber and its lower surface. Unfortunately the automatic temperature control program allowed the central chamber temperature to overheat (1163°C) and as a result, cooling between this temperature and the freezing temperature of 1095°C occurred too rapidly. Although the resulting plate which weighed 2.441Kg, was normal in appearance, i.e. free of cracks, free of dendritic growth etc., the interior of the plate contained numerous spherical voids approximately 1-2mm in diameter. In addition, the

PREFERRED FREEZING DIRECTION OF LARGE AREA PLATES

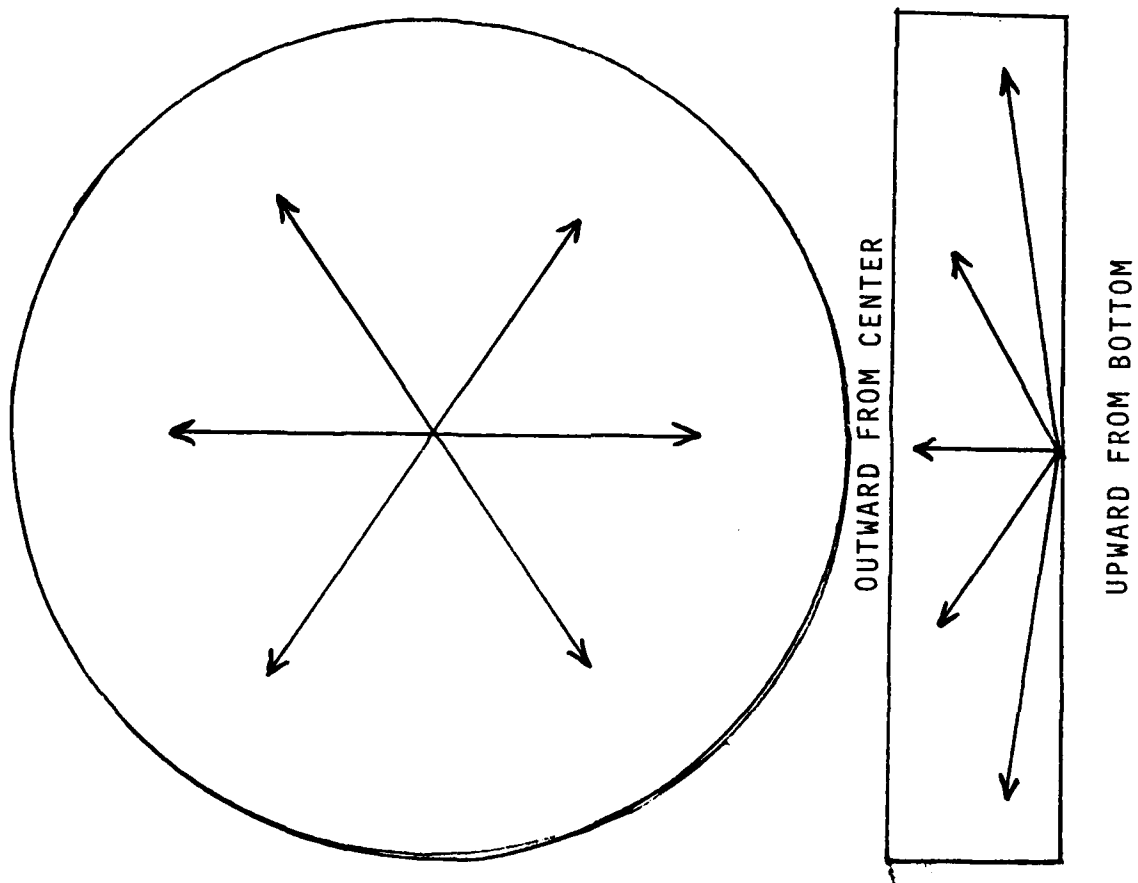


FIGURE 5

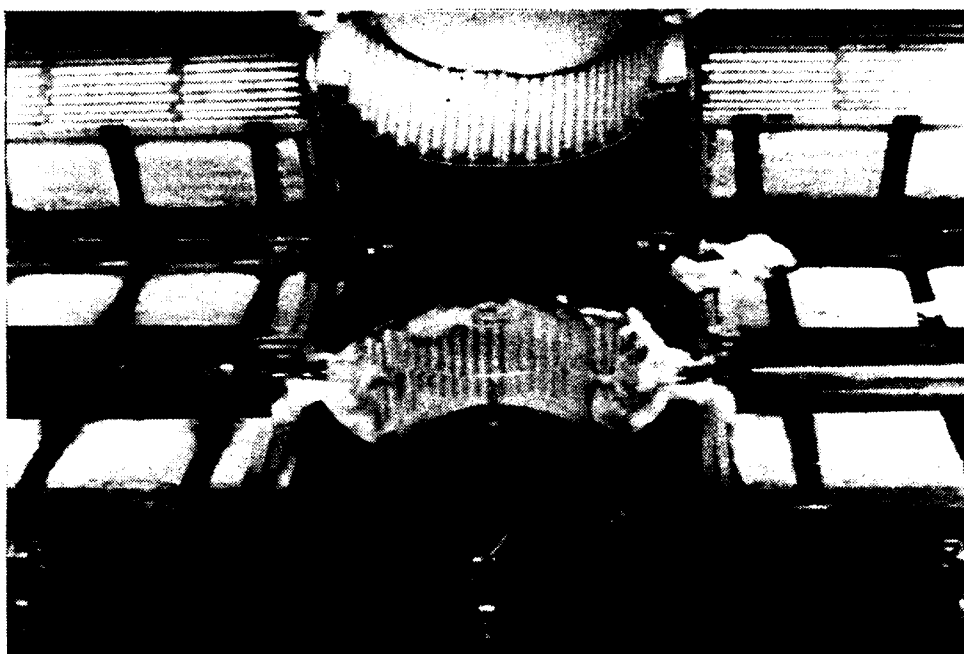


FIGURE 6
3 - ZONE FURNACE WITH 8 INCH DIAMETER CHAMBER
AFTER COMPOUNDING



FIGURE 7
CENTER CHAMBER OF 3 - ZONE FURNACE AFTER COMPOUNDING
8 INCH DIAMETER PLATE

The compounding and cooling cycles proceeded normally, the quartz chamber remained intact and the finished plate was removed within 72 hours. The surface appearance of this plate was somewhat rough and uneven, not as smooth and shiny as normally observed, but there was no indication of voids or inclusions and the plate was free of cracks. The single crystal regions of this plate averaged approximately 2.25 cm² in area. The plate was used in later heat treatment experiments to increase the IR transmission.

E. Plates #65 & 66

The final two plates prepared under this contract were probably the best plates in every respect we have seen. Both plates weighed 3.4Kg upon removal from the quartz chamber (Figure 8). Plate #65 was compounded from cadmium and tellurium alone (Cd 1890g and Te 2146g) while plate #66 contained an additional 40g of zinc, or approximately 3.5 mole %. Large single crystal regions were present in both plates (see Figure 9) and there appeared to be no significant difference in the size of the single crystal regions between the pure CdTe plate and the one containing zinc. Both of these runs were noteworthy in that a minimum of residue was observed in the cadmium and tellurium chambers after the compounding process had been completed (see Figure 10). This was not true of the preceding runs where a considerable amount of residue, probably carbon rich, was observed in both chambers. Although the starting material is reported to be of 99.999% purity, the carbon content is never reported in the chemical analysis and would appear to be the major impurity in both cadmium and tellurium.

IV. HEAT TREATMENT STUDIES

The primary purpose of this work was to develop a time-temperature-sample thickness model which could be used to prepare optical quality CdTe of any size, shape or thickness from melt grown plates. For guidelines in all the heat treatment studies we used previous technical reports by Shiozawa, Jost & Roberts.⁽¹⁾ In their work a variety of time/temperature experiments were made on vapor grown samples of millimeter thickness. In most cases however, samples received cadmium vapor treatment first, followed by a separate tellurium vapor treatment.

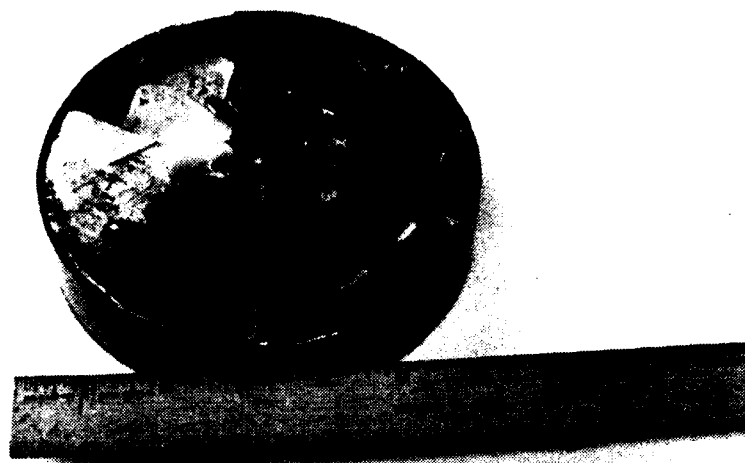


PLATE 65

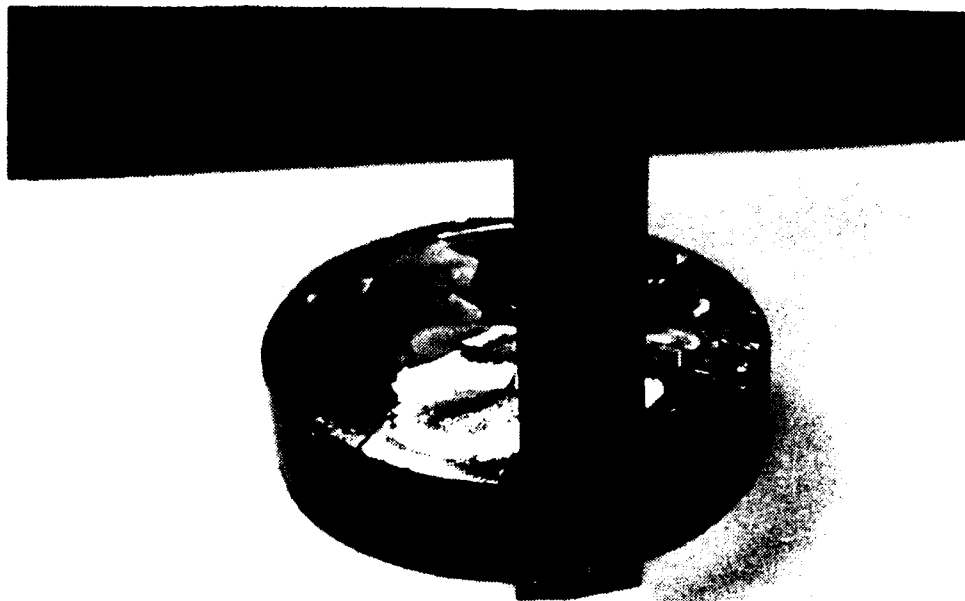


FIGURE 8
PLATE 65 AND 66 AFTER REMOVAL FROM THE COM-
POUNDING CHAMBER IN THE "AS GROWN" CONDITION

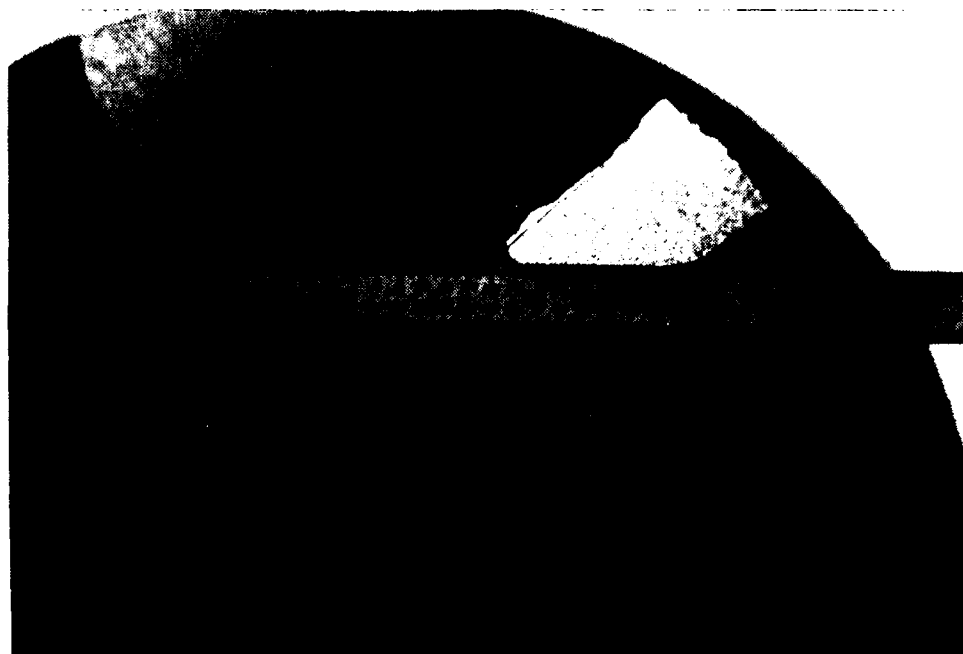
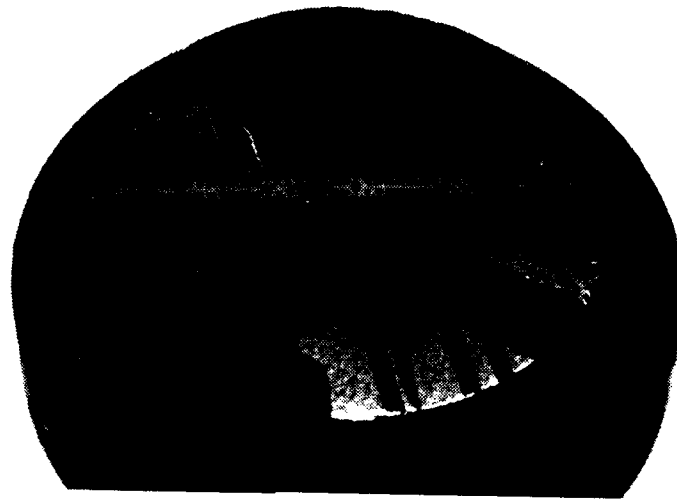
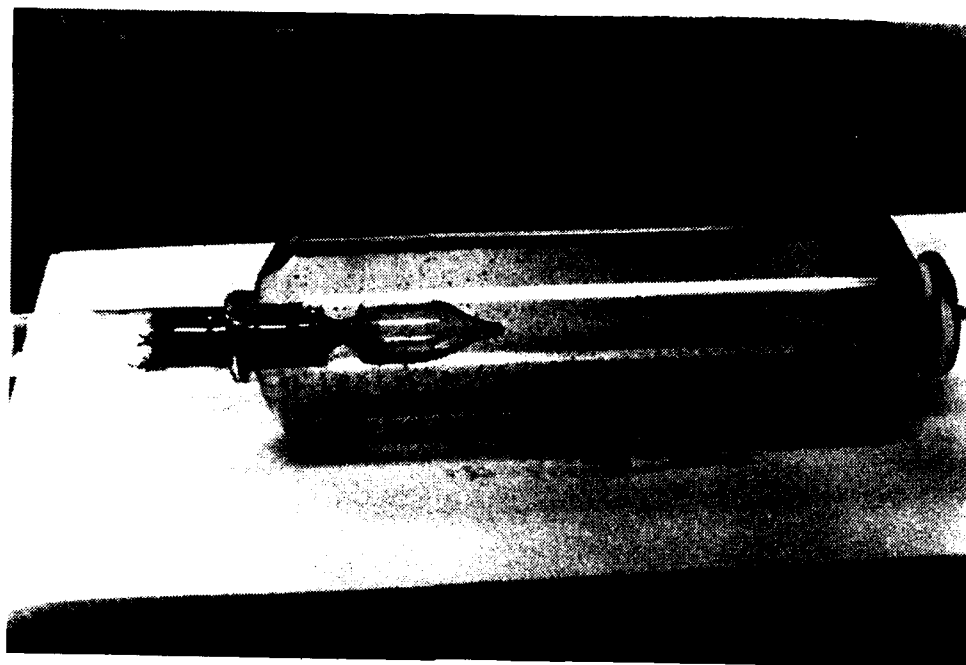


FIGURE 9
LAPPED SURFACE OF PLATE NO. 65 SHOWING
RELATIVELY LARGE AREA SINGLE CRYSTAL
REGIONS



CADMIUM CHAMBER

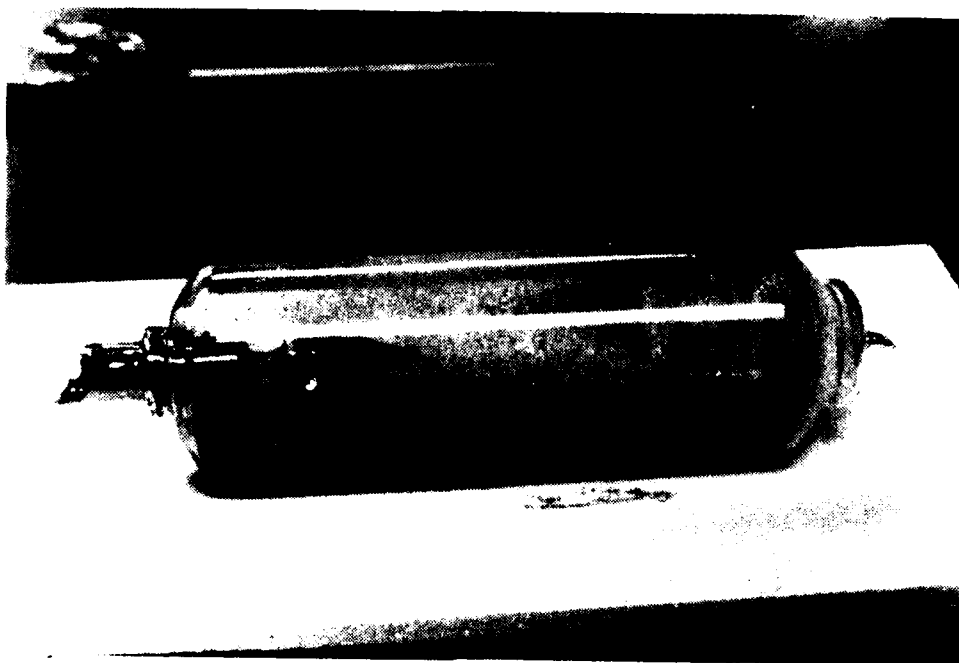


FIGURE 10
CADMIUM AND TELLURIUM CHAMBERS AFTER DISTIL-
LATION AND COMPOUNDING. NOTE RESIDUE, PROBABLY
CARBON RICH

A. Cadmium vapor treatment

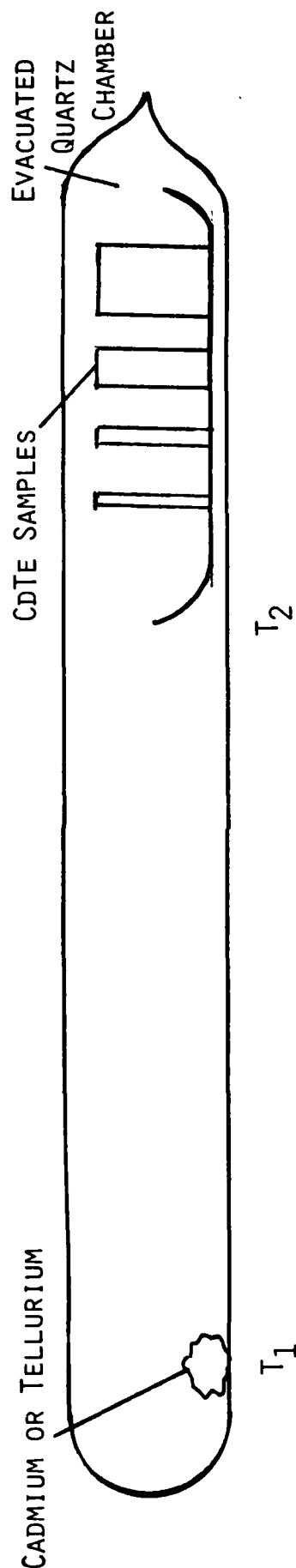
For this work we prepared a total of twenty samples all cut from the same plate and, for the sake of uniformity, from the same quadrant. Five groups of samples were prepared in four thickness ranges: 2mm, 5mm, 10mm and 20mm. Each sample measured approximately 2 cm X 2 cm. The plates, from which these samples were cut, were well compounded, free of voids, cracks or dendritic growth and contained large area single crystal regions. The first group of four samples (different thickness) was used as a control and received no heat treatment but underwent lapping and polishing with the heat treated samples. Each of the remaining four groups were placed in evacuated quartz tubes along with a piece of freshly etched cadmium (see Figure 11). The samples were placed upright in a small quartz boat inside the tube so that both surfaces were equally exposed to the vapor. All four evacuated quartz tubes containing the samples were placed in the heat treating furnace simultaneously and then withdrawn, one at a time, after one day, two days, four days and eight days. The results of the cadmium annealing are shown in Figures 12, 13 and 14. The IR transmission results of the control samples are shown in Figure 15. For the sake of simplicity only the wavelength region between 5 microns and 8 microns are shown. For the untreated samples, as expected, there is no wavelength dependence of transmission indicating that the absorption is due to tellurium precipitates. After 2 days of Cd vapor heat treatment all of the samples show a strong wavelength dependency of IR transmission indicative of free carrier absorption. Figure 16 shows a plot of absorption coefficient vs treatment time for all four thicknesses and the results show that after only 2 days of treatment, all the samples except the 20mm thickness were saturated. Table 1 summarizes the calculated absorption coefficients for all the samples at three selected wavelengths, 2.5, 5.0 and 10 microns.

B. Tellurium vapor treatment

Following the Cd vapor heat treatment and subsequent IR transmission measurements, the samples were reloaded into clean quartz tubes, maintaining group identity, for the Te vapor heat treatment. In this manner the group of sample thickness which had received one day, two day, four day and eight day heat treatment under cadmium also received the same length of treatment under tellurium.

The results of the total Cd and Te heat treatments are shown in Figures 17, 18 and 19. The IR transmission of all samples increased markedly following the tellurium treatment and as Figure 19 shows, nearly 8 days were required to obtain maximum transmission on the 20mm thick sample.

HEAT TREATING CdTe



18

T°C	- 700°C	CD AND TE
V. P.	- 380MM	CD, 30MM TE
TIME	- 1, 2, 4, 8	DAYS
SAMPLE		
THICKNESS	- 2, 5, 10, 20MM	

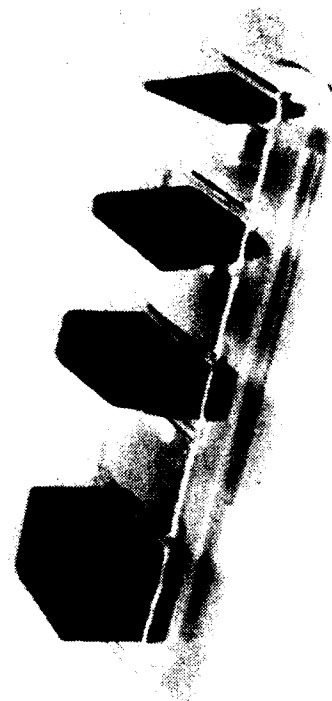
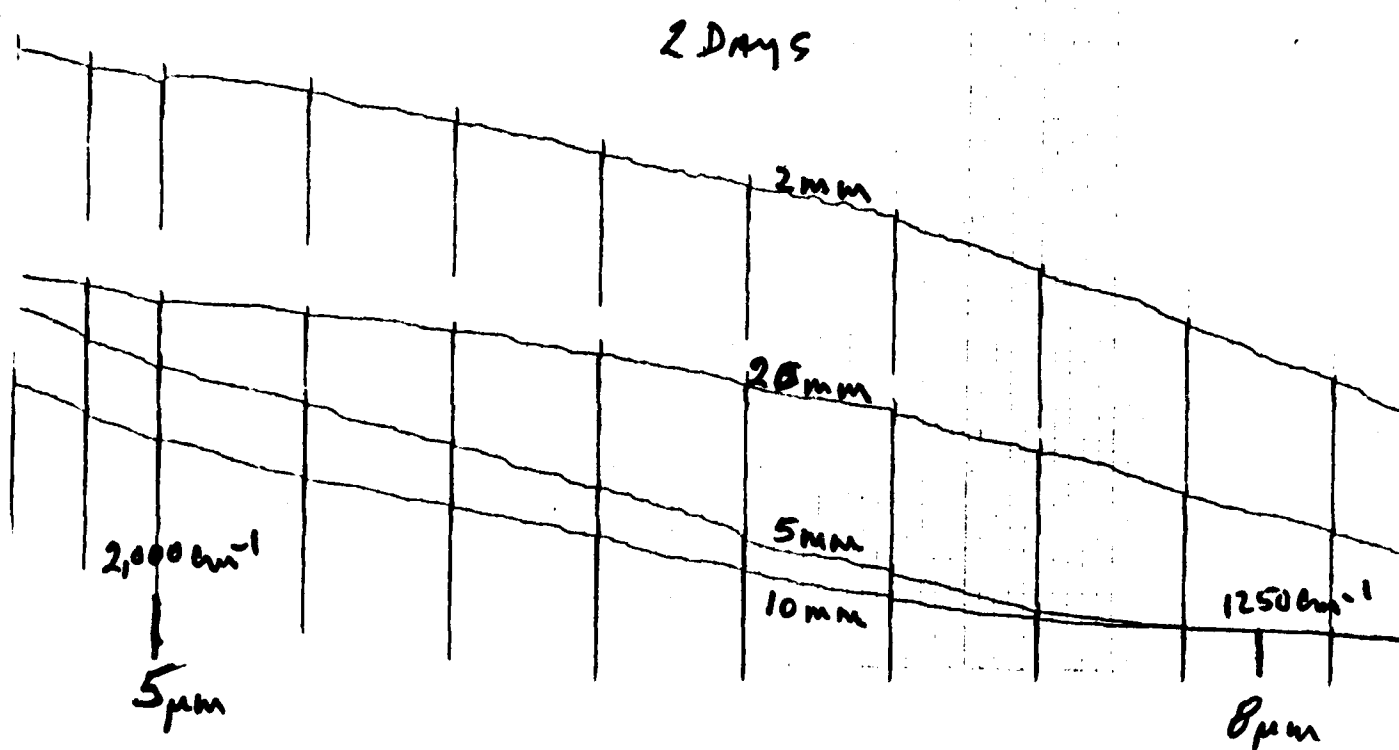


FIGURE 11

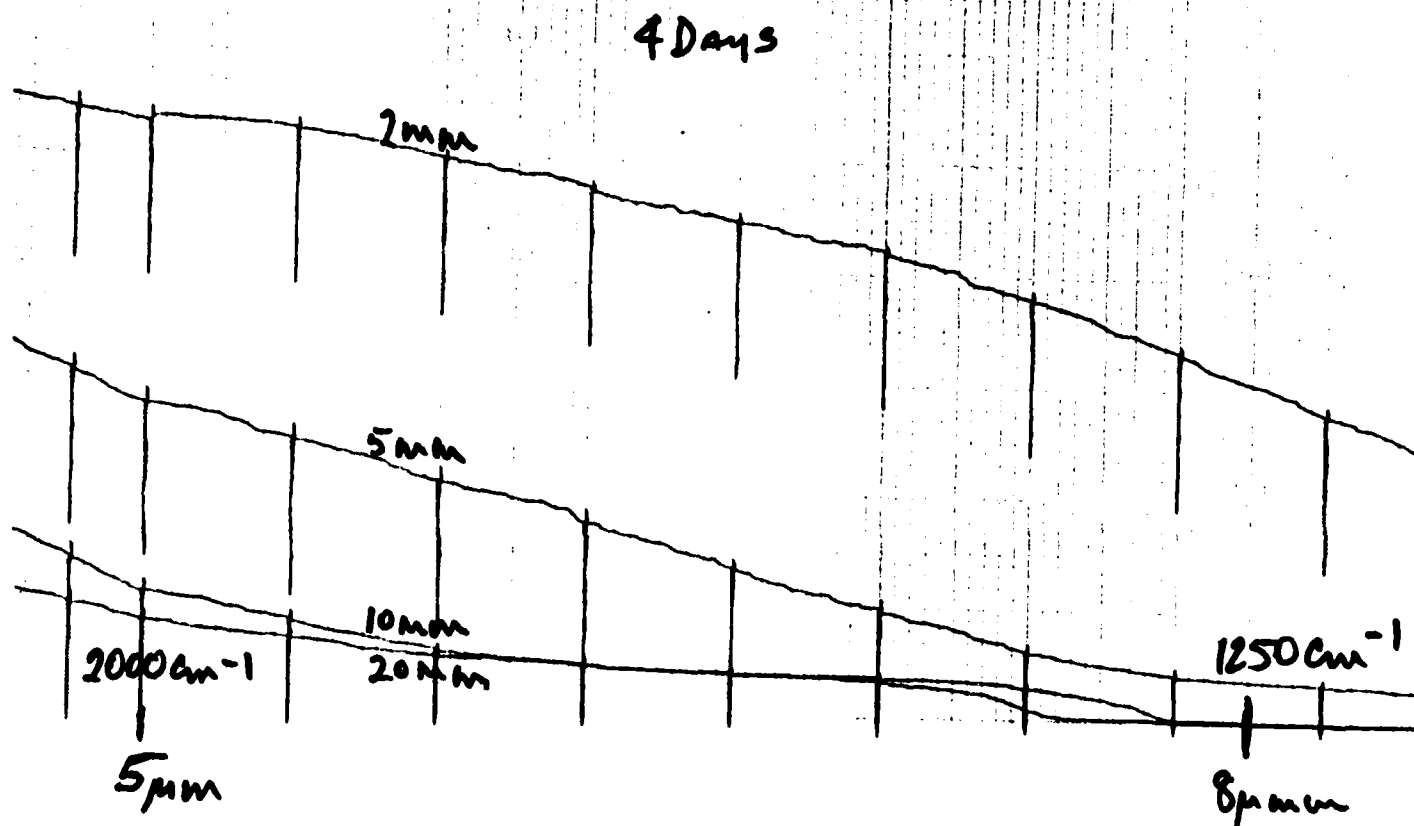
VAPOR ANNEAL OF CADMIUM TELLURIDE PLATES
2 DAYS OF TREATMENT WITH
0.5 ATM. CD VAPOR AT 700°C



ABSORPTION IS DUE TO FREE CARRIERS
P-TYPE
WAVELENGTH DEPENDENCE

FIGURE 12

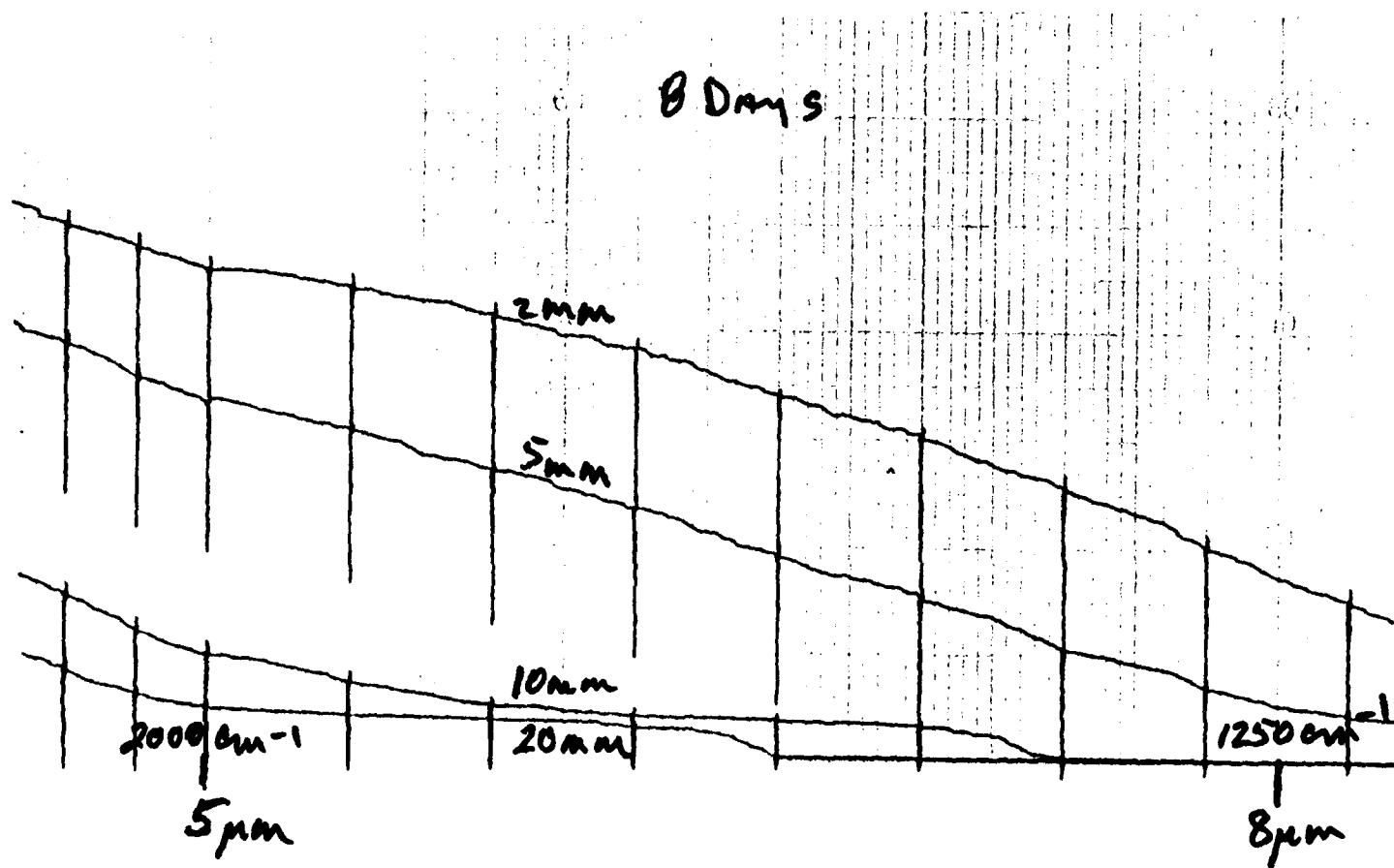
VAPOR ANNEAL OF CADMIUM TELLURIDE PLATES
4 DAYS OF TREATMENT WITH
0.5 ATM CD VAPOR AT 700°C



ABSORPTION IS DUE TO FREE CARRIERS
P-TYPE
WAVELENGTH DEPENDENCE

FIGURE 13

VAPOR ANNEAL OF CADMIUM TELLURIDE PLATES
8 DAYS TREATMENT WITH
0.5 ATM. CD VAPOR AT 700°C



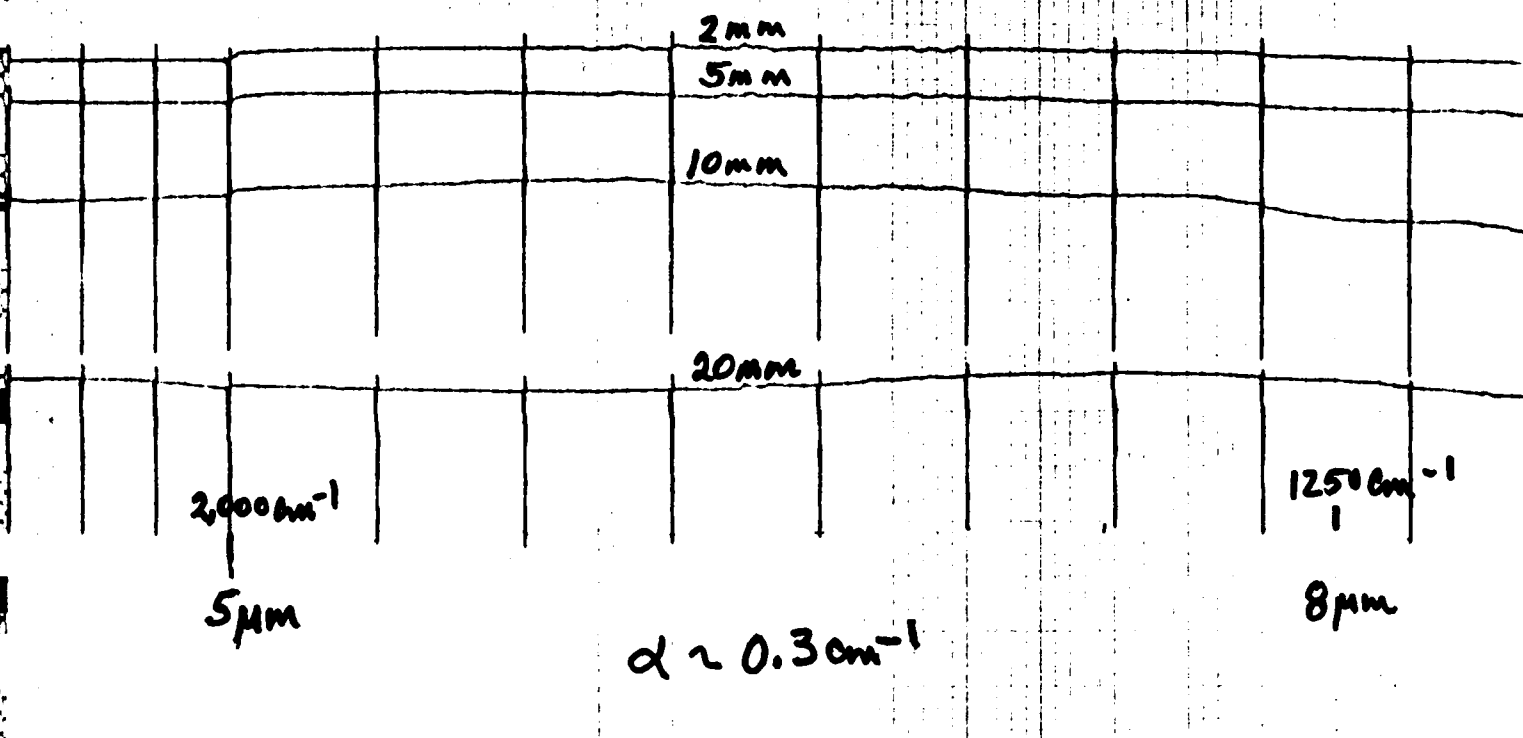
ABSORPTION IS DUE TO FREE CARRIERS
P-TYPE
WAVELENGTH DEPENDENCE

FIGURE 14

VAPOR ANNEAL OF CADMIUM TELLURIDE PLATES

NO TREATMENT

0 DAYS



ABSORPTION IS DUE TO TELLURIUM PRECIPITATES.
NO WAVELENGTH DEPENDENCE.

FIGURE 15

IR ABSORPTION OF HEAT TREATED
CADMIUM TELLURIDE CRYSTALS AS
A FUNCTION OF THICKNESS
Cd @ 700°C

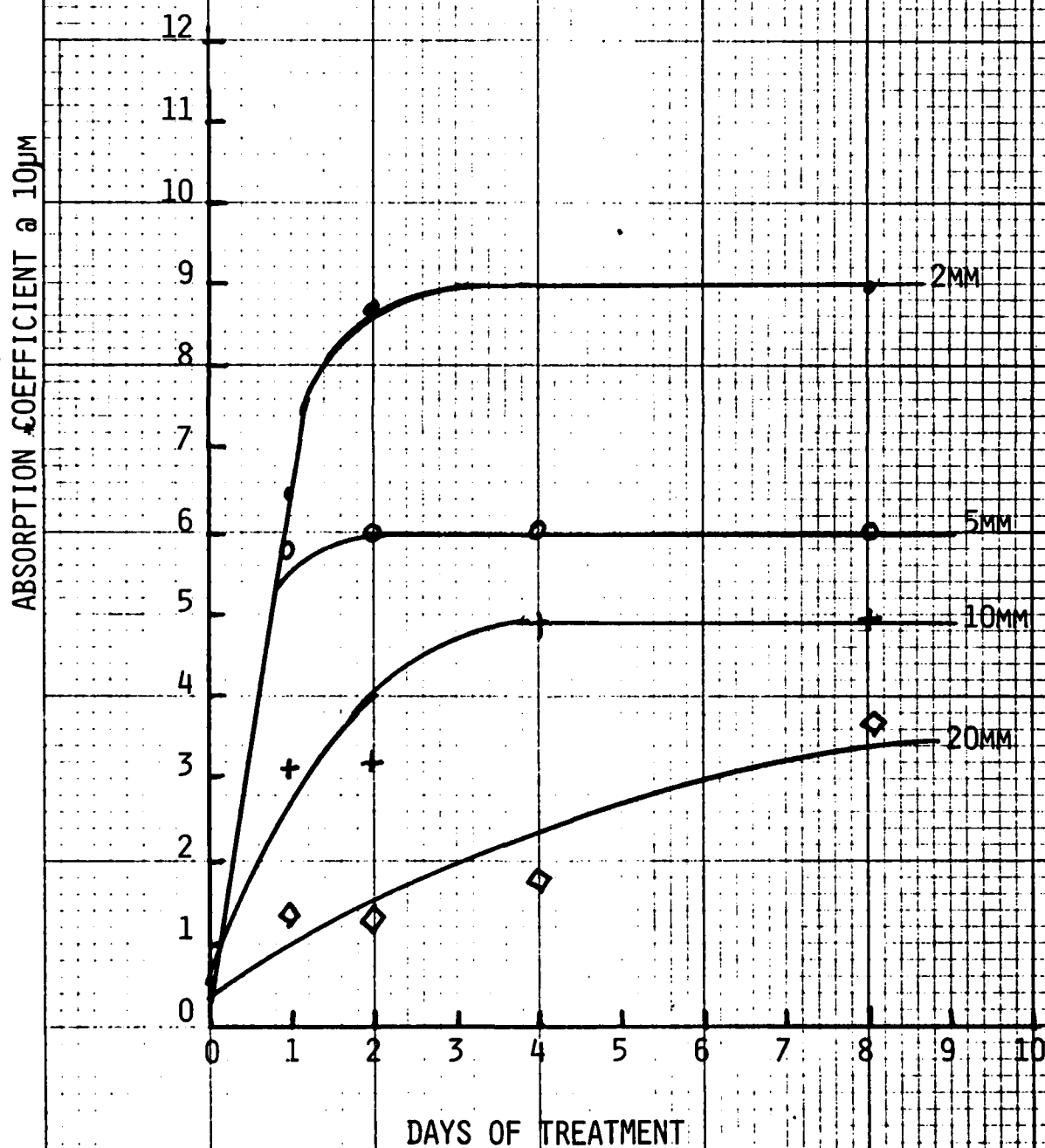


FIGURE 16
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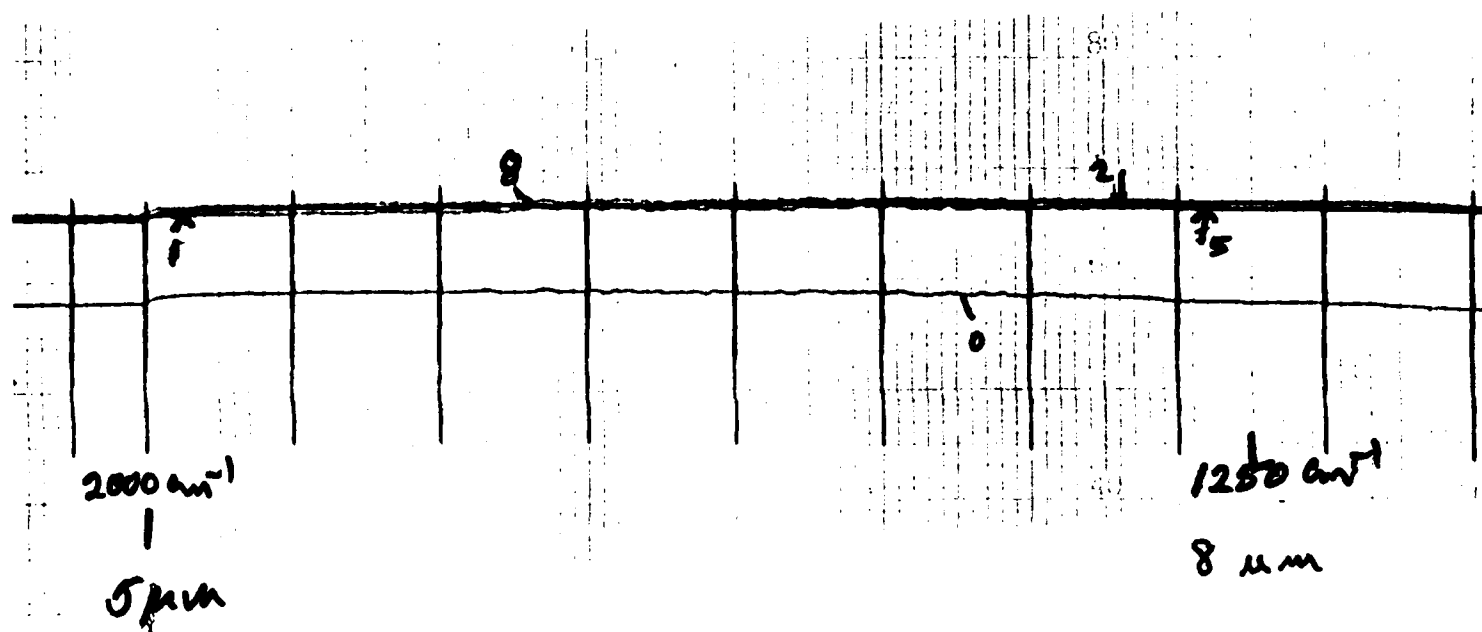
TABLE 1

IR ABSORPTION OF CADMIUM TELLURIDE CRYSTALS
AFTER HEAT TREATING WITH CADMIUM VAPOR @ 700°C

THICK. MM	WAVELENGTH μm	0 DAYS CM-1	1 DAY CM-1	2 DAYS CM-1	4 DAYS CM-1	8 DAYS CM-1
2	=2.5	0.24	0.4	0.24	0.24	0.48
	=5.0	0.24	1.8	1.3	1.0	1.8
	=10	0.31	6.4	8.5	4.6	8.9
5	=2.5	0.23	0.25	0.25	0.25	0.25
	=5.0	0.25	1.5	1.5	1.4	1.4
	=10	0.23	5.9	5.9	5.9	5.9
10	=2.5	0.21	0.56	0.21	0.28	0.28
	=5.0	0.21	1.9	1.2	1.9	1.9
	=10	0.21	3.1	3.1	4.9*	4.9*
20	=2.5	0.28	0.56	0.31	0.30	0.24
	=5.0	0.32	0.61	0.42	0.59	1.3
	=10	0.35	1.4	1.2	1.7*	3.7*

*ESTIMATED

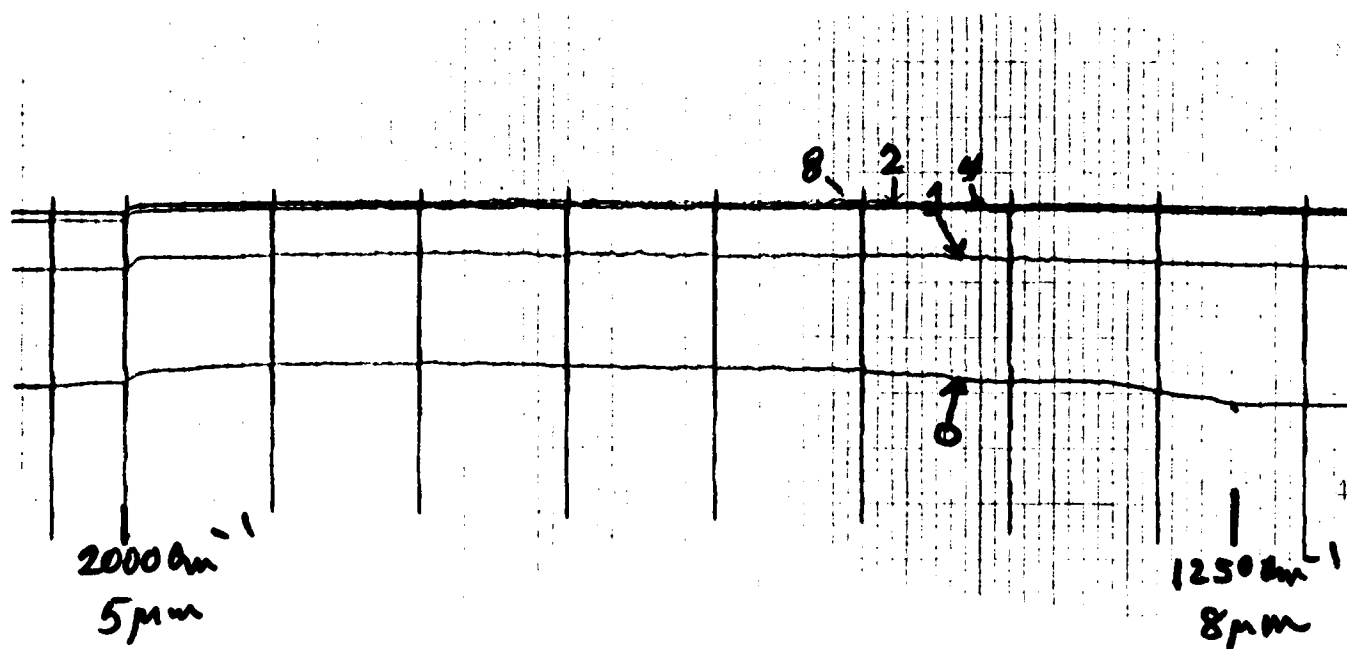
VAPOR ANNEAL OF CADMIUM TELLURIDE PLATES
 EQUAL TREATMENT DAYS Cd @ 700°C/DAYS TE @ 700°C
 5MM THICKNESS



1/1 ABSORPTION $\alpha < 0.002 \text{ cm}^{-1}$
 NO WAVELENGTH DEPENDENCE

FIGURE 17

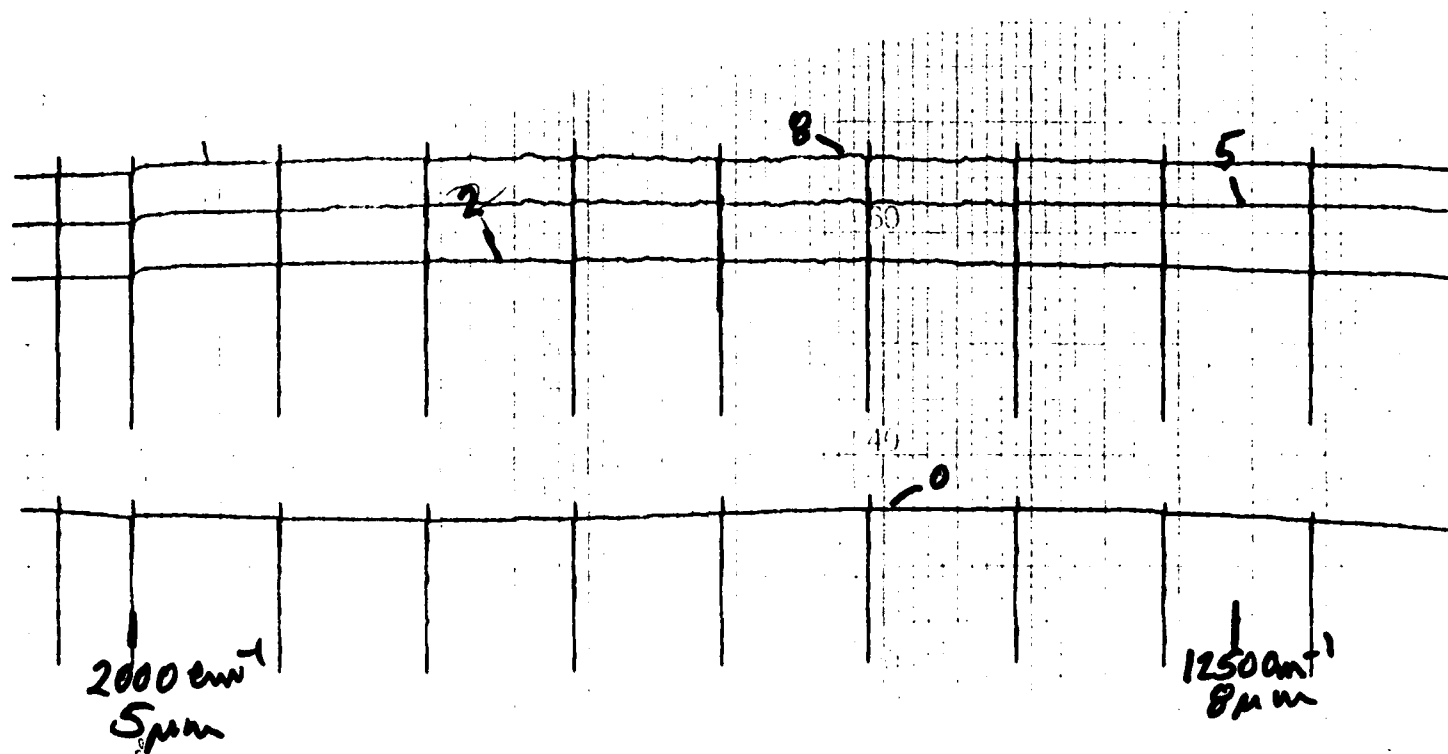
VAPOR ANNEAL OF CADMIUM TELLURIDE PLATES
 EQUAL TREATMENT DAYS Cd @ 700°C/DAYS Te @ 700°C
 10MM THICKNESS



4/4 ABSORPTION $\alpha < 0.002 \text{ cm}^{-1}$
 NO WAVELENGTH DEPENDENCE

FIGURE 18

VAPOR ANNEAL OF CADMIUM TELLURIDE PLATES
 EQUAL TREATMENT DAYS Cd @ 700°C/DAYS TE @ 700°C
 20MM THICKNESS



8/8 ABSORPTION $\alpha < 0.002 \text{ cm}^{-1}$
 NO WAVELENGTH DEPENDENCE

FIGURE 19

The complete results of both heat treatments are shown in Table 2. Absorption coefficients as low as 0.002 cm^{-1} were estimated, which represents about a 100 X improvement in transmission over the untreated material.

One of the heat treated samples was sent to Laser Power Corporation of San Diego, California for further evaluation and they also reported an absorption coefficient in the range of $.002 \text{ cm}^{-1}$ by laser calorimeter measurement.

V. CONCLUSIONS

The chalcogenide glass compounding process has been modified to produce, reproducibly, large area plates of cadmium telluride. The slow freezing rate and the controlled directional freeze appears to be a reliable method of producing relatively large volumes of randomly oriented single crystal regions. No single preferred growth orientation was observed and in general, the plates were well compounded, free of voids or inclusions, with very little, if any, dendritic growth. The effect of heat treatment on optical absorption was repeated a number of times in addition to the experiments described in this report. In every case the sample temperature as well as the source temperature were maintained at 700°C . The length of time was adjusted according to the sample thickness as dictated by the results of Table 2. In every case a definite increase in IR transmission was observed providing that both cadmium and tellurium heat treatments were applied. These results are consistent with a model which involves the initial presence of tellurium precipitates which are removed or neutralized by the cadmium vapor treatment. The excess cadmium diffused into the material is then neutralized by the subsequent low pressure tellurium vapor treatment.

TABLE 2
 ANNEAL TREATMENT OF CADMIUM TELLURIDE PLATES
 ANNEAL TEMPERATURE - 700°C
 CADMIUM TEMPERATURE - 700°C (0.5 ATM.)
 TELLURIUM TEMPERATURE - 700°C (30MM Hg)

TIME REQUIRED FOR MAXIMUM EFFECTS		
<u>PLATE THICKNESS</u>	<u>CADMIUM</u>	<u>TELLURIUM</u>
2MM	< 2 DAYS	< 1 DAY
5MM	< 2 DAYS	< 1 DAY
10MM	< 4 DAYS	< 2 > 1 DAY
20MM	8 DAYS	< 8 > 5 DAYS

NO TREATMENT α 10UM 0.2-0.3 CM-1

8 DAYS Cd @ 700 + 8 DAYS Te @ 700 α < 0.002 CM-1*

*(< 0.5% ABSORPTION 0.4-2 CM SAMPLES)

REFERENCES

1. "Single Crystal Cadmium Telluride High Energy IR Laser Windows, L. R. Shiozawa, J. M. Jost, and D. A. Roberts, Contract F3361-71-C-1777, AFML, WPAFB, December 1971.

PAPERS AND PRESENTATIONS

1. "Infrared Focal Plane Array Materials, Processing and Devices", DARPA/DSO Research Program, BDM Bldg., Tyson's Corner, VA April 2-4, 1985.
2. To be presented: SPIE - The International Society for Optical Engineering, First Annual Symposium, Los Angeles, CA, January 19-24, 1986.

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