Deposition and characterization of ho-type cadmium telluride films

T. L. Chu, Shirley S. Chu, F. Firszt, H. A. Naseem, and R. Stawski Southern Methodist University, Dallas, Texas 75275

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The deposition of CdTe films on foreign substrates by the direct combination of the elements in a gasflow system has the flexibility that the conductivity type and electrical resistivity of the film can be controlled by adjusting the composition of the reaction mixture. The deposition and properties of p-type CdTe films are emphasized in this paper because of its importance in thin-film solar cells. Graphite, W/graphite, mullite, and Corning 7059 glass were used as substrates for the deposition process. While CdTe films deposited on W/graphite and mullite substrates could be n or p type, depending on the composition of the reaction mixture, all films deposited on graphite substrates were p type, irrespective of the reactant composition, substrate temperature, or the purification of graphite, suggesting that carbon is electrically active in CdTe. The resistivity of p-type CdTe films on W/graphite and mullite substrates has been controlled for the first time by (1) using a Cd-deficient reaction mixture, and (2) adding dopants (AsH₃ or PH₃) to the reaction mixture. The resistivity versus composition relation was studied in detail. The optical properties of nearly stoichiometric, Cd-deficient and Te-deficient CdTe films were also investigated.

INTRODUCTION

Cadmium telluride has been under investigation for many years, and its preparation, crystal growth, properties, and device applications have been extensively reviewed. Being a direct-gap semiconductor with a room-temperature energy gap of 1.5 eV, CdTe is a promising photovoltaic material; single-crystalline homojunction and heterojunction solar cells have been prepared and characterized. CdTe is particularly suited for thin-film devices, since relatively short minority carrier diffusion length $(1-2\mu m)$ can be tolerated due to its short optical absorption length. The preparation and characteristics of single-crystalline and thin-film CdTe solar cells have been reviewed recently.

Most CdTe solar cells are of the heterojunction type due to the difficulties associated with the preparation of thin, high-conductivity surface layer. Available data on heterojunction devices indicate no serious loss of photovoltaic performance in transferring from a single crystal to thin films. The heterojunction devices require another semiconductor with considerably larger band gap, appropriate electron affinity, and the ease of preparing high-conductivity films usually referred to as the "window" material. The known window materials, such as cadmium sulfide, zinc oxide, tindoped indium oxide, etc., make use of anion vacancies and are all of n-type conductivity. Thus, p-type CdTe films are required for the fabrication of thin-film solar cells. In spite of the extensive work on thin-film CdTe heterojunction solar cells, very little information on the control of the conductivity type and carrier concentration in CdTe films is available.

In single-crystalline CdTe, the homogeneity region is only a fraction of one atomic percent on both the Cd-rich and Te-rich side of the stoichiometric composition. Although the cadmium and tellurium vacancies behave as acceptors and donors, respectively, the concentration of electrically active species is significantly less due presumably to the formation of interstitials or complexing defects. In CdTe doped with shallow dopants, the concentration of electrically active species is often less than that of the dopant, particu-

larly at high concentrations, due to the formation of interstitials or change complexes. The interstitials, charge complexes, or other defects could degrade the minority carrier diffusion length. These problems are further enhanced in thin-film CdTe.

The objective of this work is to prepare CdTe films with controlled properties in a reproducible manner. The reaction of Cd and Te vapor on the surface of heated substrates in a hydrogen (or helium) atmosphere in a gas-flow system has been used. This technique has the flexibility that the deviation of the CdTe film from stoichiometry can be controlled and the incorporation of electrically active dopant into CdTe films can be accomplished. p-type CdTe films of controlled resistivity have been deposited reproducibly by controlling the Cd/Te molar ratio in the reaction mixture or by incorporating dopants into the reaction mixture. The experimental procedures and results are discussed in this paper.

DEPOSITION OF CADMIUM TELLURIDE FILMS

The deposition of cadmium telluride films by the direct combination of Cd and Te vapor on the surface of mullite, graphite, carbon-coated graphite, and tungsten-coated graphite substrates at 500-650 °C in a hydrogen-flow system has been reported.³ The apparatus for the deposition of CdTe films and the temperature profile of the furnace are shown schematically in Fig. 1. A Cd container, a Te container, and the substrate were placed in a fused silica tube of 55mm i.d. in a multizone furnace with each zone separately heated and controlled. Hydrogen was used to carry Cd and Te vapors to the substrate surface. The composition of the reaction mixture was then determined by the temperatures of Cd and Te containers and the flow rates of the carrier gas through them and the reaction tube. In a few experiments, helium instead of hydrogen was used. Cd and Te₂ are the dominant species in the gas phase and combine to form CdTe surface of the substrate $(p_{\text{Cd}}^0) (p_{\text{Te}_2}^0)^{1/2} > K_{\text{CdTe}}$ at the substrate temperature. Although the partial pressures of Cd and Te in the reaction

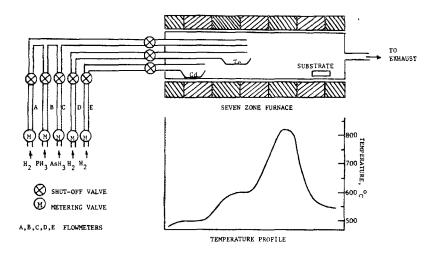


FIG. 1. Schematic diagram of the apparatus for the deposition of cadmium telluride films.

mixture are considerably lower than the equilibrium vapor pressures of the elements at the substrate temperature, any excess of cadmium or tellurium has been shown to deposit, at least partially, on the substrate surface. Thus, the Cd/Te molar ratio in the reaction mixture must be closely controlled in order to obtain nearly stoichiometric CdTe films. The nearly stoichiometric films can be p type (slightly Cd deficient) or n type (slightly Te deficient). When tungstencoated graphite, prepared by the thermal reduction of tungsten hexafluoride with hydrogen on the surface of one side of a graphite plate at 500 °C, was used as the substrate, the change in conductivity type of nearly stoichiometric films has been found to take place over a very narrow range of the composition of the reaction mixture. For example, a reaction mixture containing 0.264-Torr Cd and 0.117-Torr Te₂ produced n-type CdTe films at 575 °C, and the lowering of the cadmium partial pressure by 2% produced p-type films. The conductivity type of CdTe films was determined from the photovoltage of silver-Schottky barriers measured using a quartz halogen lamp and curve tracer. The photovoltage is positive for an n-type film and is negative for p-type material.

RESISTIVITY MEASUREMENTS OF p-TYPE CdTe FILMS

Since the current flows through the thickness of CdTe films in the operation of devices, the measurement of resistivity of CdTe films in the thickness direction is more useful. Ohmic contacts to p-type CdTe films were formed by electroless Au plating at about 100 °C to form Au/p-CdTe/subtrate structure. Due to the large work function of p-type CdTe, the p-CdTe/substrate such as W/graphite) interface is always rectifying. An example of the current-voltage characteristics of a Au/p-CdTe/W graphite structure is shown in Fig. 2. For positive bias on Au, the CdTe/substrate junction is forward biased, and the dynamic resistance consists of the resistance of CdTe and the Au/CdTe contact resistance. At bias voltages higher than about 0.3 V, the current-voltage characteristics deviate from the exponential relation due to contributions of series resistances. The Au/CdTe contact resistance depends on the resistivity of CdTe films and is relatively small for films of several hundred Ω cm resistivity as discussed below. Thus, the series resistance is associated mainly with the resistance of cadmium telluride. At higher voltages, the current-voltage relation is linear, and its slope may be used to calculate the resistivity of the CdTe films. For negative bias on Au, the CdTe/substrate junction is reverse biased, and the dynamic resistance consists of the resistance of CdTe, the Au/CdTe interface resistance, and the CdTe/ substrate interface resistance. Hence the CdTe/substrate interface resistance may be deduced from a comparison of the forward and reverse characteristics.

The resistivity of p-type CdTe films on mullite substrates was measured by the van der Pauw technique; the ohmic contacts to the films were also made by electroless Au plating. This measurement provides the lateral resistivity of the film which may be different from the resistivity in the thickness direction due to grain-boundary effects. This is particularly true in high-resistivity films, since the potential

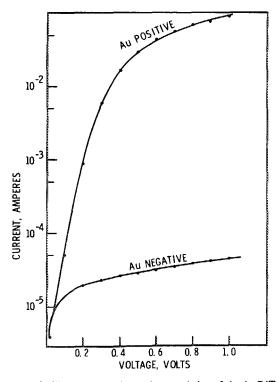


FIG. 2. The current-voltage characteristics of Au/p-CdTe/W/graphite structure.

barrier across the grain boundaries contributes to the resistivity measured by the van der Pauw technique, and this contribution decreases with increasing carrier concentration in the film. At sufficiently high carrier concentrations, the potential barrier at grain boundaries becomes negligible, and the van der Pauw measurements provide the average resistivity of the grains. On the other hand, the resistivity measured in the thickness direction of the film consists of contributions from the columnar grains and grain boundaries. Their relative importance depends on the carrier concentration in the grains and the chemical and structural properties of the grain boundaries. If the grain boundaries have a low impedance due to such factors as dopant segregation, the measured resistance is contributed mainly by the grain boundaries. Similarly, the grains contribute mainly to measured resistance when the grain boundaries are of high impedance.

CdTe FILMS ON GRAPHITE SUBSTRATES

High-purity graphite plates (Grade DFP-2 manufactured by POCO Graphite Incorporated) of 1-1.5 mm thickness were used as substrates for the deposition of CdTe films. The plates were purified by the manufacturer using halogen treatment at high temperatures. The purified graphite was claimed by the manufacturer to have a total ash content of 5 ppm or less⁴: major impurities being iron, silicon, aluminum, and magnesium of up to 5 ppm each.

CdTe films deposited on graphite substrates under a wide range of conditions are polycrystalline and are tightly adherent to the substrates. The average grain size in the film depends on the substrate temperature and the thickness of the film. At similar thicknesses, the films deposited at higher temperatures consist of larger grains than those deposited at lower temperatures. At a given temperature and a fixed partial pressure of tellurium, the growth rate increases rapidly as the Cd/Te molar ratio is first increased and gradually levels off at higher CdTe ratios. One problem associated with the use of graphite substrates is the porosity of graphite; the density of DFP-2 graphite is 1.84 g/cm³, corresponding to a porosity of about 18%. Consequently, the surface of graphite always exhibits a high concentration of pits which sometimes are tens of micrometers in size. The presence of pits on the substrate surface may prevent the uniform nucleation of CdTe during the initial stage of deposition. After the initial nuclei are formed, CdTe tends to deposit preferentially on these nuclei to form irregularly shaped crystallites. The subsequent film growth proceeds by the enlargement of these crystallites rather than by the generation of fresh nuclei. Because of the nonuniform nucleation, the coalescence of all adjacent crystallites cannot produce a completely continuous structure at finite thicknesses, $20 \,\mu\mathrm{m}$ for example. The formation of pinholes is more pronounced at higher substrate temperatures due to the lower rate of nucleation. Figure 3 reproduces the scanning electron micrograph of a cadmium telluride film of about 15-µm thickness deposited on a purified DFP-2 graphite substrate at 575 °C showing the pinholes in the film.

Attempts were made to determine the composition of the reaction mixture where the *n-p* transition in the CdTe

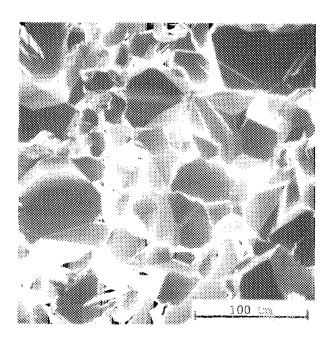


FIG. 3. Scanning electron micrograph of a CdTe film deposited on a graphite substrate at 575 °C.

film takes place. The deposition of CdTe was carried out at 575 °C; the partial pressure of Te₂ in the reaction mixture was fixed at 0.117 Torr and that of Cd was varied between 0.18 and 0.36 Torr. While one would expect that on the basis of the data obtained for CdTe films on W/graphite substrates, the films produced at 575 °C with Cd partial pressure higher than 0.264 Torr to be of *n*-type conductivity, all films were found to be p type. The deposition of CdTe films was also carried out at 530 °C using the partial pressures of cadmium and tellurium over a wide range, and all films were again p type. To determine if the p-type conductivity is related to contamination from the graphite substrates, the substrates were purified by treatment with hydrogen chloride at high temperatures in the following manner. Silicon-carbide (about 0.2-mm thickness) -coated graphite susceptor of $5 \times 1.3 \times 15$ cm³ in dimensions was placed in a fused silica reaction tube of 5.5-cm i.d. and heated at 1400 °C in hydrogen chloride at a flow rate of 1 1/min for 30 min. No impurities were liberated as is evidenced by the absence of any deposits in the cooler section of the reaction tube. Purified DFP-2 graphite plates of $3\times0.1\times14$ cm³ in dimensions were then placed on the silicon-carbide-coated graphite susceptor and heated in hydrogen chloride at 1400 °C for 30 min, a thin layer of white deposit was found on the wall of the reaction tube. However, the amount of condensed material was not sufficient for chemical analysis. The heating of the DFP-2 graphite in hydrogen chloride was repeated until no white deposit appeared. Many cadmium telluride films were deposited on DFP-2 graphite substrates purified by hydrogen chloride using Cd/Te molar ratios over a wide range. Since the CdTe films were deposited at temperatures considerably lower than the temperature used for hydrogen chloride treatment, the diffusion of impurities from the substrate into CdTe film during the deposition process is unlikely. The graphite substrates were also purified by refluxing in an aqua regia solution for six weeks followed by thorough rinsing

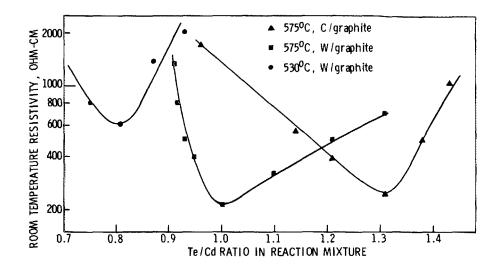


FIG. 4. Electrical resistivity of p-type CdTe films on W/graphite and C/graphite substrates as a function of the Te/Cd molar ratio in the reaction mixture.

with deionized water. Cadmium telluride films deposited on these substrates were also of p-type conductivity irrespective of the Cd/Te molar ratio in the reaction mixture. These results suggest that carbon is incorporated into the cadmium telluride films and is electrically active. Hydrogen is known to react with carbon at temperatures above 500 °C; however, the hydrogen transport of carbon is not the major cause of carbon incorporation into CdTe since the use of helium as the carrier gas produced the same results. The reaction between carbon and tellurium is most likely to be responsible for the incorporation of carbon into CdTe films (the formation of CSe₂ is well known). Carbon presumably occupies interstitial positions in CdTe because of its small size (the tetrahedral covalent radii of C, Cd, and Te are 0.77, 1.48, and 1.32 Å, respectively).

CdTe FILMS ON W/GRAPHITE SUBSTRATES

Graphite is not suitable as a substrate for the deposition of CdTe films because of the incorporation of carbon into the films during the deposition process. The reaction between carbon and tellurium may be minimized or eliminated by depositing $2-3\mu m$ of tungsten on the surface of graphite. The tungsten coating makes the graphite surface more homogeneous to yield more uniform and pinhole-free CdTe films. The electrical resistivity of CdTe films on W/graphite substrates may be controlled by (1) the Cd/Te molar ratio in the reaction mixture, and (2) the addition of dopant, such as arsine, to the reaction mixture. Both techniques have been investigated.

The electrical resistivity of p-type CdTe films on W/graphite substrates may be controlled within a limited range by varying the Te/Cd molar ratio in the reaction mixture. Figure 4 shows the resistivity of p-type CdTe films deposited on W/graphite substrates at 530 and 575 °C and on C/graphite (the carbon coating was used to provide a more homogeneous surface) substrates at 575 °C as a function of the Te/Cd molar ratio in the reaction mixture. Using W/graphite as substrates, the transition from n to p type in cadmium telluride films deposited at 575 °C occurs at a higher Te/Cd molar ratio than that deposited at 530 °C. The room-temperature resistivity of nearly stoichiometric p-type CdTe

films deposited at 530 and 575 °C is about $10^4 \Omega$ cm. As the Cd partial pressure in the reaction mixture is decreased, the resistivity of the film first decreases due to increasing Cd vacancy concentration and reaches a minimum, about 600 Ω cm for CdTe films deposited at 530 °C using a Te/Cd molar ratio of 0.8 and 200 Ω cm for cadmium telluride films deposited at 575 °C using a Te/Cd molar ratio of unity. The lower resistivity obtainable at higher substrate temperatures is presumably related to the higher defect concentration. As the cadmium partial pressure in the reaction mixture is further reduced, the resistivity increases due presumably to the formation of defect complexes. When carbon-coated graphite is used as substrates for the deposition of cadmium telluride films at 575 °C, the minimum resistivity occurs at a higher Te/Cd ratio as compared with W/graphite. This difference must be related to the electrical behavior of carbon in CdTe. The fact that n-type CdTe films cannot be deposited on graphite substrates indicates that carbon behaves as an acceptor in CdTe. However, this simple concept cannot account for the observed difference in the resistivity versus Te/ Cd ratio relation for CdTe films deposited on C/graphite and W/graphite substrates. It is likely that the interactions of native defects (such as Te interstitials) and carbon result in the formation of donor-type complexes, thus shifting the resistivity minimum in CdTe films on C/graphite substrates toward higher Te/Cd molar ratios. However, the magnitude of the resistivity minimum is similar for W/graphite and C/ graphite substrates. It should be pointed out that due to the use of a gasflow system, a large change in the Te/Cd molar ratio in the reaction mixture results only in a small change in the composition of deposited CdTe films, less than the accuracy of the electron-microprobe analysis. Also, no compositional inhomogeneities were detected across the films by the electron-microprobe analysis.

The effects of carbon on the resistivity of CdTe films become more pronounced at higher substrate temperatures. Figure 5 shows the resistivity of p-type CdTe films deposited on C/graphite and C/graphite/W substrates at 630 °C as a function of the Te/Cd molar ratio in the reaction mixture. The C/graphite/W substrate has no exposed graphite once a CdTe thin film is deposited, and the minimum resistivity, about 100Ω cm, occurred at a Te/Cd molar ratio of about

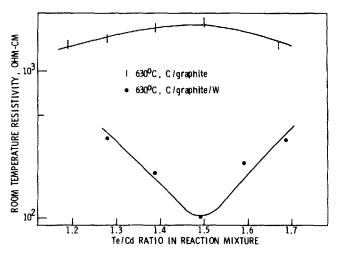


FIG. 5. Electrical resistivity of p-type cadmium telluride films on C/graphite and C/graphite/W substrates as a function of the Te/Cd molar ratio in the reaction mixture.

1.5. The resistivity of CdTe films on C/graphite substrates is essentially constant, about 2000 Ω cm, for Te/Cd molar ratios of 1.2-1.7 due to the contribution of carbon from the backside of the substrate.

It has been reported that oxygen is essential for the fabrication of efficient thin-film CdS/CdTe solar cells. The effect of oxygen on the electrical resistivity of p-type CdTe films was determined by introducing varied amounts of oxygen into the reaction mixture during the deposition process. The results are shown in Fig. 6; the resistivity of p-type CdTe films was lowered appreciably by the addition of up to about 0.05% oxygen and then leveled off at higher oxygen concentrations. This result suggests that oxygen acts as an acceptor at low concentrations, and the formation of complexes may dominate at high oxygen concentrations.

The electrical resistivity of p-type CdTe films may also be controlled by the addition of dopants (AsH₃ or PH₃) to the reaction mixture during the deposition process. In one series of experiments, for example, CdTe films were deposited on W/graphite substrates at 575 °C using a reaction mixture with a Te/Cd molar ratio of 0.91 (this ratio produces nearly stoichiometric p-type CdTe films) containing various con-

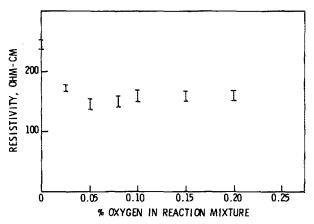


FIG. 6. Effect of addition of oxygen to the reaction mixture on the resistivity of p-type CdTe films.

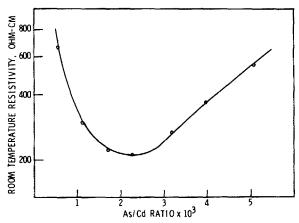


FIG. 7. Electrical resistivity of p-type cadmium telluride films as a function of the AsH_{χ}/Cd molar ratio in the reaction mixture.

centrations of arsine (85-ppm AsH₃ in H₂). The room-temperature electrical resistivity of arsenic-doped CdTe films was measured, and one set of typical results is shown in Fig. 7. If the arsine concentration in the reaction mixture is gradually increased, the resistivity of the film decreases, and a minimum, about 200 Ω cm, occurs at a AsH₃/Cd molar ratio of about 2×10^{-3} . The resistivity increases at higher arsine concentrations due presumably to the interaction of the dopant with the native defects. When the reaction mixture has a lower Te/Cd molar ratio, the incorporation of arsenic is less efficient.

The effective intragrain minority carrier diffusion length in several Cd-deficient and arsenic-doped CdTe films on W/graphite substrates have been measured by the scanned electron-beam-induced current method in an ETEC Autoscan scanning electron microscope. Large-grain specimens with flat surfaces were selected, and a Schottky barrier was formed to cover only a small portion of each of the grains under investigation. An electron beam was focused on the surface of a grain, and as the beam was moved from the edge of the Schottky barrier, the induced current was measured as a function of the distance of the electron beam from the edge of the Schottky barrier. The effective intragrain diffusion length deduced from the logarithm current versus distance relation is $2-4 \mu m$, considerably higher than that in n-type CdTe films, about $1 \mu m$.

CdTe FILMS ON MULLITE SUBSTRATES

Nearly stoichiometric CdTe films were deposited on mullite substrates at 500-600 °C by adjusting the CdTe molar ratio in the reaction mixture. These films may be n or p and exhibit high lateral resistivity, $10^4-10^5~\Omega$ cm, as measured by the van der Pauw technique. p-type CdTe films were also deposited by using PH₃ as a dopant. Figure 8 shows the room temperature lateral resistivity of phosphorous-doped CdTe films as a function of the PH₃/Cd molar ratio in the reaction mixture. Similar to the As-doped CdTe films on W/graphite substrates, the resistivity-PH₃/Cd ratio relation shows a minimum, 300-400 Ω cm, at a PH₃/Cd ratio of about 10^{-3} , and the resistivity increases at higher concentrations of PH₃. The room-temperature Hall mobility

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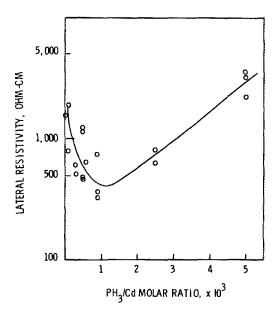


FIG. 8. Room-temperature lateral resistivity of p-type CdTe films as a function of the PH₃/Cd molar ratio in the reaction mixture.

in most samples is 5–10 cm²/V sec, thus the carrier concentration in the minimum resistivity film is about 10¹⁶ cm⁻³. Figure 9 shows the electrical resistivity of a phosphorous-doped CdTe film as a function of temperature in the range of 25–150 °C. The activation energy, about 0.65 eV, is associated predominately with the conduction process across the grain boundaries. The higher minimum lateral resistivity of CdTe films as compared with the minimum resistivity of CdTe films in the thickness direction is due presumably to the potential barrier at grain boundaries.

Attempts were made to measure the Au/p-CdTe contact resistance using CdTe films on mullite substrates. Several contact strips were applied to the surface of a sample of about 0.5×3 cm² in area and $20\,\mu\mathrm{m}$ in thickness by electro-

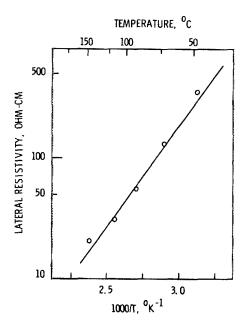


FIG. 9. Lateral resistivity of a phosphorus-doped CdTe film as a function of temperature.

less Au plating. A known current was passed through the end contacts of the specimen, and the voltage drop across the various contacts were measured. This voltage drop is a linear function of the distance between contacts, and in principle, the contact resistance can be deduced from an extrapolation of the voltage-distance relation to zero bias. However, the intercept at the voltage axis was too small to be determined accurately. It is estimated that for p-CdTe films of 200–500 Ω cm resistivity, the Au/p-CdTe contact resistance is less than 5% of the resistance of the film. The previous assumption of neglecting the contact resistance in the measurement of resistivity of p-CdTe films in the thickness direction is thus justified.

CdTe FILMS ON GLASS SUBSTRATES

CdTe films were deposited on Corning 7059 glass substrates at 530-540 °C. Nearly stoichiometric, Te-deficient (n-type of minimum resistivity), and Cd-deficient (p-type of minimum resistivity) films were obtained by adjusting the Te/Cd molar ratio in the reaction mixture. The deposited films were mechanically polished with alumina, and their optical absorption at room temperature measured using a Varian Model Cary 17 spectrophotometer. Figure 10 shows a plot of α^2 (α is the absorption coefficient) of a nearly stoichiometric CdTe film versus photon energy. The extrapolation of the linear portion of the plot to the energy axis yields an optical band gap of 1.50 eV for the fundamental absorption, essentially the same as the band gap of single-crystalline CdTe. The α^2 versus photon energy plots for Te- and Cd-deficient CdTe films also yield the same band gap. In electrochemically deposited CdTe films, the direct band gap of as-deposited n-type films (1.51 eV) was found to be higher than that of the converted p-type films through heat treat-

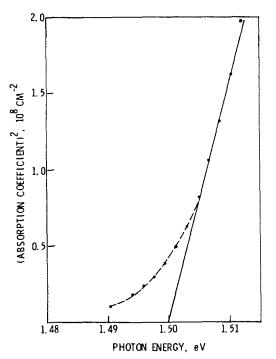


FIG. 10. Optical absorption data of a nearly stoichiometric CdTe film.

ment (1.46 eV). This difference was attributed to defect absorption in the heat-treated material.

SUMMARY

The deposition of CdTe films on graphite, W/graphite, mullite, and Corning 7059 glass substrates has been carried out under a wide range of conditions. Carbon has been shown to be electrically active in CdTe due presumably to the reaction of carbon and tellurium, and no n-type CdTe films can be deposited on graphite substrates. The effects of carbon on the resistivity of CdTe films are more pronounced at higher substrate temperatures. Using W/graphite as substrates, the change of conductivity type of nearly stoichiometric CdTe films has been found to take place over a narrow composition range of the reaction mixture. The resistivity of p-type CdTe films on W/graphite and mullite substrates has been controlled for the first time by (1) using a Cd-deficient reaction mixture, where the minimum resistivity obtainable decreases with increasing substrate temperature, and (2) adding dopants (AsH₃ or PH₃) to the reaction mixture, where the minimum resistivity is related to the dopant concentration. The minimum resistivity (lateral) of p-type CdTe films on mullite is higher than that on W/graphite substrates due to the effects of grain boundaries. The effective intragrain minority carrier diffusion length in p-type CdTe films on W/graphite substrates is appreciably higher than that in n-type films. The room temperature optical band gap of nearly stoichiometric, Cd-deficient, and Te-deficient CdTe films is 1.50 eV, same as the band gap of a single-crystalline CdTe.

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