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**CONTROLLED CADMIUM TELLURIDE THIN FILMS FOR SOLAR
CELL APPLICATIONS**

Emerging Materials Systems for Solar Cell Applications

Quarterly Progress Report No. 2 for July 9–October 8, 1979

By
K. Vedam

November 1979

Work Performed Under Contract No. AC04-79ET23013

Materials Research Laboratory
The Pennsylvania State University
University Park, Pennsylvania

U.S. Department of Energy

Solar Energy

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QUARTERLY PROGRESS REPORT NO. 2

for the period

July 9, 1979 to October 8, 1979

Controlled Cadmium Telluride Thin Films for Solar Cell
Applications

(Emerging Materials Systems for Solar Cell Applications)

to

U.S. Department of Energy

Division of Solar Energy

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Under Contract No. DE-AC04-79ET23013

MATERIALS RESEARCH LABORATORY

THE PENNSYLVANIA STATE UNIVERSITY

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ABSTRACT

Numerous cadmium telluride films with excess Cd or Te have been prepared by controlled rf sputtering technique and characterized. It is found that slight excess of Cd or Te affects the structure, as well as the electrical resistivity of the film, markedly. Ohmic contacts with fairly low contact resistance have been achieved on Cd-rich specimens.

1. INTRODUCTION

Even though cadmium telluride has many of the characteristics of an ideal semiconductor for use as a highly efficient solar cell, very little work has been done on this material, mainly because of the following three reasons - (i) erratic and non-reproducible performance of thin films, (ii) surface problems including contacts and (iii) exceedingly short minority carrier life times. The present work was undertaken to tackle this problem in a systematic manner by (a) preparing the films by a controlled rf sputtering technique and (b) characterizing such films at every stage so that the cause for the erratic behavior reported in earlier studies can be identified and the conditions for the preparation of films with reproducible and desired properties can be established.

Obviously, the first step in such a program is to start with the highest purity CdTe commercially available. Due to the unusually long delivery time of the sputtering target made of such high purity material, research work utilizing such target could be started only during this reporting period. Further, some mechanical failure in the high vacuum line of the sputtering system caused a further delay in the actual starting of the project. However, the system is now in full operation and significant progress has been made both in the sample preparation and characterization aspects of the program. In what follows some of the results obtained are presented and discussed.

2. EXPERIMENTAL DETAILS

The self doped CdTe films were prepared by rf-sputtering using an MRC sputtering system. From our earlier studies we know that the nature of the sputter deposited CdTe film is found to be strongly dependant on the sputtering conditions. In fact, we can produce either crystalline (cubic and/or hexagonal) or amorphous

Fig. 1. Schematic diagram of R. F. sputtering system.

films of CdTe purely by controlling the Ar pressure, r.f. voltage and power and the physical geometry of the sputtering target, substrate, etc. In addition, the substrate temperature during sputtering, also influences the nature of the film. Further, the above sputtering parameters are also found to affect the adherence of the film to the substrate as revealed by scratch and abrasion tests developed in this laboratory.

2.1 Preparation:

All films were sputtered from a 5" CdTe target in an MRC rf-sputtering system. A schematic of the sputtering system is shown in Fig. 1. This system has the capability of sputtering up or down as well as changing the substrate-to-target distance. The CdTe target is of MARZ grade quality from Materials Research Corporation, with no impurity concentration greater than 1 ppm. The sputtering chamber is routinely pumped down to less than 1×10^{-6} torr before the Ar sputtering gas is admitted to the chamber. The Ar gas pressure was maintained at 20 mTorr in all the runs. Films were prepared at 3 different rf power levels, ranging from 50 to 150 W. Sputtering time in each case was varied to obtain nearly the same range of thicknesses for each run. The substrate-to-target distance was maintained at 35mm. Table 1 gives the details of various preparation conditions.

The substrates used included Fisher glass microscope slides (25 mm x 12mm x 1mm) and molybdenum. They were ultrasonically cleaned sequentially in detergent, distilled water, and alcohol and then stored in alcohol. The molybdenum slides were stored in acetone. The substrate set up used in most of the experimental runs is shown in Fig. 2. The figure also shows the position of the Cd pellet. While the glass substrates were used for thickness, x-ray diffraction and electrical properties measurements, the interspaced molybdenum substrates were used for compositional analysis.

Fig. 2. Experimental arrangement of the target and substrate in the sputtering chamber.

Table I

Relevant Sputtering Parameters Employed in this Study

Sample No.	Argon Pressure (in Torr)	rf Power (in watts)	rf Voltage (volts)	Sputtering Time (minutes)	Substrate to Target Distance (mm)	Dopant	Substrate
PSCD-001	20	50	500	80	35	Cd	Glass
2	20	100	550	45	35	Cd	Glass
3	20	150	750	32	35	Cd	Glass
4	20	50	525	80	35	Te	Glass
5	20	100	575	45	35	Te	Glass
6	20	150	750	32	35	Te	Glass
7-1 to 16	20	50	480	120	35	Te	Glass & Mo
8-1 to 16	20	100	575	70	35	Te	Glass & Mo
9-1 to 16	20	150	625	45	35	Te	Glass & Mo
10-1 to 16	20	50	475	123	35	Cd	Glass & Mo
11-1 to 16	20	100	550	70	35	Cd	Glass & Mo
12-1 to 16	20	150	610	45	35	Cd	Glass & Mo

Fig. 3. Thickness of film as a function of radial distance measured from the Cd pellet end of substrate holder.(Sample No. PSCD-10).

2.2 Characterization:

Film thicknesses were measured mechanically using a Talysurf-10 Profilometer. The thickness of the film deposited in a typical run is shown in Fig. 3, as a function of radial distance r , measured from the Cd-pellet end of the substrate holder. X-ray diffraction pattern of CdTe films on glass were obtained with a GE diffractometer using $\text{CuK}\alpha$ radiation operating from $2\theta = 20^\circ$ to 50° . The results are presented in the next section. The optical absorption data were obtained for a few selected samples using Cary 14 spectrophotometer.

An ion scattering (ISS) and an Auger electron spectrometer (AES) combined in the same ultra-high vacuum system, designed at the Materials Research Laboratory, was used for the compositional analysis of CdTe films on molybdenum substrates. Since this facility is rather unique, a brief description of the set-up is given below. A schematic diagram of the instrument is shown in Fig. 4. The samples mounted in a carousel type target holder are inclined 45° relative to the ion beam for ISS. A 180° rotation of the target holder about the Z-axis positions the sample (previously analyzed with ISS) normal to the electron beam for AES. A total of eight samples can be examined using either (or both) spectrometer(s) without breaking the vacuum.

The ion scattering spectrometer is a first generation 3M Model 520 unit. Four probe ions (He-3 , He-4 , Ne-20 and Ar-40) can be used. The electronic system has been augmented to include (1) rastering of the ion beam to eliminate the "edge effect" from the bombarded crater, (2) signal gating, and (3) automated control of scanning and data acquisition by a PDP 11/20 data processor.

Computer control of the ion scattering spectrometer is accomplished through an interface module designed by and constructed in our laboratory. In the present set-up, the number of primary ions scattered from the surface at an angle of 90° is measured at energies from 0 to 99.9% of the incoming primary ion energy in incremental steps of 0.1%. Initial information, such as the number of ele-

Figure 4. ION SCATTERING-AUGER ELECTRON SPECTROMETER

ments to be scanned, the energy windows and data acquisition time, and the time between sets of scans are programmed into the PDP-11/20 data processor. In each scan, the data is stored in core and processed before starting the next cycle of data acquisition. In this manner, the depth profiles of as many as ten (or more) different elements can be obtained in a completely automated analysis.

The Auger electron spectrometer is based on a Physical Electronics Industries Model 15-25G high resolution, double-pass, cylindrical mirror analyzer (CMA). Primary excitation is accomplished by means of an internal, co-axial electron gun. Depth profiling can be done utilizing the sputtering capability of the ion beam in the ion scattering spectrometer to remove successive layers of a surface for Auger electron analysis. Acquisition and processing of the Auger spectra using the PDP 11/20 computer is currently being developed in order to extract Auger line shapes from the experimental data.

The ISS measurements were made using a 1-mm-diam, 1500 eV, $^{20}\text{Ne}^+$ ion beam at a beam current density of $\sim 28 \mu\text{A}/\text{cm}^2$. The data were collected over preselected energy ranges of the Ne-20 ions scattered from Cd and Te to enable the computer (1) to search for the peaks in the appropriate Cd and Te energy "windows" and (2) to subtract the background and store the information for further data processing. Collection and processing of data took less than 1 minute between sets of scans spaced at intervals of ~ 2 minutes. Each film was scanned until reasonably stable Cd and Te signals were noted.

AES analyses were made using a 0.25mm, 1.5KeV, 20 μA primary electron beam. A modulation voltage of 2 volts peak-to-peak was chosen in recording the derivative spectra at a rate of 2eV/sec.

2.3 Electrical Characterization

2.3.1 Multi-Functional Circuit: A variety of measurements for systematic scanning of a large number of CdTe samples is accomplished efficiently and accurately using a multi-functional circuit designed and built in our laboratory.

FIG. 5. SCHEMATIC DIAGRAM OF THE MULTIFUNCTIONAL CIRCUIT

1 - CURRENT REGULATOR	5 - START CIRCUIT CURRENT	S3 (EITHER V OR I_{sc})
2 - SAMPLE VOLTAGE (WITH LOAD)	S1 (OPEN - V_{oc} ; V WITH LOAD
CLOSED - CURRENT SOURCE MODE)	S4 (I_{sc} - MAGNITUDE)
3 - CURRENT MEASUREMENT	S2 (OPEN - V_{oc}/I_{sc}
CLOSED - V WITH LOAD)	S5 (CURRENT-COURSE MAGNITUDE)
4 - GROUND REFERENCE FOR OFFSET		

The circuit is capable of functioning in the following manner:

- (1) Solar Cell Current-Voltage indicator
 - (a) Short Circuit Current ($.1\mu\text{A}$ resolution at 1% accuracy)
 - (b) Open Circuit Voltage (load of $10^{12}\Omega$)
 - (c) Current/Voltage under load condition from 0 to $5\text{K}\Omega$
(continuous)
- (2) Constant Current Source: capable of driving $10\text{M}\Omega$ load
- (3) 3-point probe: For contact resistance measurements using Shockley ladder test pattern.
- (4) 4-point probe: For film bulk resistivity measurements.

Voltage differences between various points of internal and/or external to the circuit are measured using high input impedance voltage follower IC chips ($R_{in} = 10^{12}\Omega$). Maximum voltage difference is obtained by means of a voltage-controlled current source and voltage regulator IC chips (maximum current $\approx 5\text{mA}$, minimum current $\approx 0.1\mu\text{A}$). The short circuit current, with minimum leakage current, is measured using a current-to-voltage convertor IC ship.

The complete circuit schematic is shown in Fig. 5. Multiple position switches conveniently change the functional mode and enable the measurements involving different magnitudes of currents and voltages. Two terminals are provided for measurement on two terminal device. One additional external probe is provided for 3 or 4 pt. probe measurements. The circuit requires supply voltages of $\pm 15\text{V}$ with ground.

2.3.2. Resistance Measurement: Electrical measurements were performed on a Shockley Ladder test pattern to obtain resistivity ρ , sheet resistance $\rho_{\square}$, and contact resistance R_c as functions of the sputtering parameters. The electrical parameters are obtained by passing a constant current through the end contacts of the ladder and measuring the voltages at various "rungs" of the ladder structure with high input impedance op-amps (see multifunctional circuit: Fig. 5).

Fig. 6. (a) Shockley Ladder Test Pattern,
(b) Solar Cell Structure.

Fig. 6a shows the ladder test pattern used in the experiment. The separation between "rungs" of the ladder is 2.5mm. The separation between end pads is 20mm and the width of the pattern can be varied by carefully scribing the sample. Fig. 6b shows the structure of the solar cell. The Ni metal contacts were deposited by shadow masking within the sputtering chamber (for both sputter etched and non-etched samples). Perimeter scribing of each ladder is often necessary to ensure electrical isolation from possible nickel layer shorts to the glass substrate at the edges. Resistance variation with temperature up to 300°C was observed for some earlier samples with annealing times up to about 10 minutes. However, all data presented in this report were obtained from non-annealed samples. Also, preliminary photoconductivity measurements were made on some earlier samples by monitoring the change in resistance at maximum light intensity of the solar simulator.

3. RESULTS

The results of our compositional analysis structure and electrical measurements are described in the following subsections.

3.1 Chemical Composition Analysis:

A typical variation of the cadmium and tellurium ISS signals with analysis time is shown in Fig. 7 for sample 010-7. Both Cd and Te signals were low in the beginning, primarily due to impurities on the outermost layer. That is, the number of Cd and Te scattering centers in approximately 250 μ m x 1000 μ m analysis area in ISS are smaller initially. As the impurities are sputtered away by the ion beam, the scattered signals assume stable values.

There are two commonly used methods for measuring the surface composition. The first involves comparison of the signal from the sample (I_x) to that obtained from a pure elemental standard (I_{xs}), according to Eq. (1):

Fig. 7. Variation of the Cd (●) and Te (+) ISS signals and the atomic concentration of Cd (○) vs. time for sample No. 010-7.

$$C_x = I_x / I_{x,s} \quad (1)$$

A second method makes use of the following relation:

$$C_x = (I_x / K_x) / \sum_j (I_j / K_j) \quad (2)$$

where C_x is the concentration of X, I_x is the signal from X, K_x is the sensitivity relative to an arbitrary standard, and the summation is over all the elements present.

The inherent advantage of the second method [Eq. (2)] is that after initial calibration for the instrumental sensitivities, concentrations can be determined from the experimental data without the need of inserting standards together with the test samples. We should stress, however, that only relative concentrations can be obtained by this method, inasmuch as hydrogen is not detected by either ISS or AES. Nevertheless, use of Eq. (2) is a convenient method of comparing the Cd and Te concentrations among these films.

Using Eq. (2), the relative concentration of Cd for sample 010-7 is also plotted on the right ordinate scale of Fig. 7. The ISS sensitivities for Cd and Te were determined from previous sputter-deposited and ion-plated CdTe films whose compositions were measured by electron microprobe techniques. The plot shows that although the Cd and Te signals change with analysis time, the Cd concentration attained a stable value fairly quickly. Calibration and random errors contributed to a total error of ~2% in the measured values.

The chemical compositions of the Cd-doped films are plotted in Fig. 8, while the results for the Te-doped samples are shown in Fig. 9. In each case, the resulting compositions were either Cd or Te rich depending upon the nature of the dopant and the distance of each sample from it. That is, for the Cd-doped films, the Cd concentrations of samples 010-5 and 010-7 were the same to within experimental error. These results were not unexpected inasmuch as both samples were underneath the Cd island. For samples situated more than ~80mm radially from the Cd island (i.e. 010-12 to 010-16) the film compositions ap-

Fig. 8. Variation of Cd concentration (at %) vs. sample number for Cd-doped CdTe films sputter-deposited at 50 watts (O), 100 watts (+), and 150 watts (●) rf power levels.

proached the 50% level.

The variation of composition as a function of radial distance from the Te dopant, Fig. 9, showed similar type behavior. As in the case of the Cd-doped films, Fig. 8, the experimental data suggest one further important observation, namely, that as the rf power level increases from 50 watts to 150 watts, the gradient in composition between samples located directly below the Cd or Te dopant and those situated greater than 84mm from it decreases. This shows that in addition to the effect of radial distance of the substrate from the dopant, the rf power levels provide additional control in the tailor-making of these films with the desired doping levels. Reproducibility studies are currently being made and will be reported shortly.

3.2 Structure:

For each run (50W, 100W and 150W) X-ray diffraction patterns of selected films were obtained. Fig. 10 shows a composite of X-ray patterns of the samples prepared at 50W power at four different radial distances (see Fig. 2) measured from the Cd-rich end of the substrate holder. Similar composites for films at 100W and 150W are shown in Figs. 11 and 12. As can be seen from the figures, the CdTe films exhibit both cubic and hexagonal phases. This is in agreement with the results of our earlier studies carried out with an older undoped CdTe (and not high purity) target⁽¹⁾. A closer inspection of Figs. 10 a, b, c and d and also Figs. 11 and 12 reveals that while the hexagonal phase is predominant at small values of r , the cubic phase increases with increasing r . In conjunction with the data shown in Fig. 8, the above results means that the hexagonal phase is dominant for Cd rich films, and that the amount of cubic phase increases as stoichiometry is approached. Also, the (002) peak at 23.7° , for Cd-rich films, is dominant while for the more stoichiometric films the (100) peak at 22.7°

⁽¹⁾ See Appendix 1 of the First Quarterly Progress Report (Contract No. DE-ACOE-79ET23013) dated August 1979.

Fig. 9. Te concentration (at %) vs. sample number for Te-doped CdTe films sputter-deposited at 50 watts (O), 100 watts (+), and 150 watts (●) rf power levels.

Fig. 10. X-ray diffraction patterns obtained on CdTe samples #PSCD 010, as a function of distance from the cadmium pellet end of the substrate holder.

Fig. 11. X-ray diffraction patterns obtained on CdTe samples #PSCD 011, as a function of distance from the cadmium pellet end of the substrate holder.

Fig. 12. X-ray diffraction patterns obtained on CdTe samples #PSCD 012, as a function of distance from the cadmium pellet end of the substrate holder.

Fig. 13. Typical optical absorbance of sputter deposited CdTe film.

predominates. Thus, the preferential orientation of the hexagonal phase of the films changes with composition at the three different power levels. Furthermore, at the near-stoichiometric compositions where the cubic phase becomes a more significant portion of the hexagonal/cubic diphasic mixture, the cubic phase is preferentially oriented with (220) planes parallel to the film surface. It is interesting to note that the (220) cubic and (002) hexagonal are related crystal planes indicating a polytypism-controlled growth behavior. More tests, especially at Te-rich compositions, are being done to get a better understanding of the structural phase relations and their control.

Fig. 13, is a typical example of the results of optical absorption studies on two samples. It is seen that the optical absorption edge is near 850nm. It may be recalled that our earlier studies⁽²⁾ had indicated that the absorption edge for the hexagonal 2H phase is at 808nm, whereas for the pure cubic 3C phase it is at 919nm. Hence, the presently observed value of 850nm for the absorption edge indicates that the film is diphasic, again in agreement with the results of X-ray diffraction results discussed above, as well as our earlier results⁽²⁾ obtained on not so pure CdTe film.

3.3 Resistivity

3.3.1. Bulk Resistivities: Film bulk resistivity has been found to be a strong function of the radial distance, r , from the source pellet in both the cadmium and Tellurium rich films. Resistivity values were calculated with a knowledge of the sample geometry and the results of resistance measurement. The bulk resistivity is given by

$$\rho = \rho_{\square} t$$

and the sheet resistance, $\rho_{\square}$, is given by

$$\rho_{\square} = R \frac{W}{L},$$

(2) B. E. Knox and K. Vedam, "Coating Science & Technology," Final Report No. AFCL-TR-76 dated January 31, 1976; Contract No. F19628-75-C0110.

Fig. 14. Resistivity of sputter deposited CdTe films as a function of distance from Cd pellet end of the substrate holder.

where W = width of the ladder

L = length between consecutive rungs of ladder

R = measured voltage/current ($\frac{V}{I}$) ratio

and t = Thickness of film.

A wide range of resistivity values ($0.03\Omega\text{cm}$ to $5M\Omega\text{cm}$) were measured for numerous Cd rich samples. Limited number of Te rich samples were tested and they indicate resistivity values ranging from $10\Omega\text{cm}$ to $10M\Omega\text{cm}$. Typical results for Cd rich samples produced by two different sputtering conditions are presented in Fig. 14. The rapid rise of ρ with the increase of radial distance from the pellet is to be noted. Indeed ρ was found to be slightly varying even on the same ladder sample from one end to other, as may be seen in Figs. 15(a) and 15(b) in which the potential drops across the resistive ladder pattern are plotted. The ohmic nature of the contacts were determined by I/V plots on a curve tracer using potential reversal across the sample as shown in Fig. 16 for a Cd rich sample.

From Figs. 8 & 9, and the above data we see a definitive relationship between film composition and resistivity indicating that control of resistivity is possible. It is interesting to note that very small changes in stoichiometry produce large changes in resistance. The results are in complete agreement with the earlier results⁽³⁾ obtained on films prepared from not so pure CdTe target. Analysis of all these results indicate that it is possible to reproducibly prepare self-doped CdTe films of low resistivity by controlled R.F. sputtering technique.

3.3.2. Contact Resistance: Contact resistance values were determined using the potential distribution plots obtained from the ladder test pattern shown in Figures 15(a) and 15(b). Due to finite contact resistances at both ends of the sample the potential distribution show discontinuities. However, by extra-polating the straight line plots a transfer length L_T can be identified at

(3) Work performed under a contract with National Patent Development Corporation (NPDC) prior to the start of the present contract. Vide page 3 of last Quarterly Report for details.

Fig. 15. (a) Potential distribution measured along the rungs of the ladder test device. Sample #PSCD 001.

Fig. 15. (b) Potential distribution plot on the ladder test device. Samples #PSCD 010 and PSCD 012.

Fig. 16. Curve Tracer display showing I-V characteristic of Cd rich CdTe film. Sample #PSCD 012-6. Vertical axis I-0.05 MA/div.; Horizontal axis V = 0.2 V/div.

both ends which when combined with the sample sheet resistance value will yield the contact resistance according to the relationship,

$$R_c = \rho \frac{L^2}{T}$$

Contact resistance and sheet resistance values are presented in Fig. 17, as functions of radial distance from the source pellet which correspond to the bulk resistivity data of Fig. 14. These samples were metallized with Ni after sputter etching and this resulted in low value of contact resistance. On the other hand we have observed that the samples which are not sputter etched before metallizing, exhibit large contact resistance.

4. SUMMARY AND FUTURE PLANS:

Numerous CdTe films with varying excess cadmium concentration were prepared by controlled R. F. sputtering technique and these films were examined for their chemical composition, structural and electrical characteristics. It is found that crystal structure (in other words the degree of hexagonality) of the film can be varied by varying the Cd/Te ratio in the film. Earlier studies had revealed that similar structural variation can be achieved by varying the sputtering parameters. Furthermore, such compositional variation is found to affect the resistivity of the sample markedly. Ohmic nature of the contacts was established for Cd-rich samples, fairly low values of contact resistance can be achieved if the specimens are sputter etched before metallization.

Similar studies will be conducted on Te-rich samples which have been and are being prepared by controlled rf-sputtering technique. When once the optimum sputtering conditions are established, then p-n, MIS and Schottky barrier type solar cells will be constructed and evaluated.

Fig. 17. Sheet resistance $\rho_{\square}$ and contact resistance R_c as a function of radial distance from Cd pellet end of the substrate holder. CdTe samples PSCD 001 and PSCD 002 (both n-types).

5. PERSONNEL

The following personnel are involved in this project for varying amounts of time:

Dr. M. B. Das

Dr. S. Krishnaswamy

Dr. L. Tongson

Mr. P. Swab

Mr. R. Petkie

The project has benefitted from frequent discussions with Drs. R. Messier and S. Fonash.