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CONTROLLED CADMIUM TELLURIDE THIN FILMS FOR SOLAR CELL APPLICATIONS

Quarterly Progress Report No. 3 for October 9, 1979—January 8, 1980

MASTER

By
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February 1980

Work Performed Under Contract No. AC04-79ET23013

The Pennsylvania State University
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U.S. Department of Energy

Solar Energy

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QUARTERLY PROGRESS REPORT NO. 3

for the period

October 9, 1979 to January 8, 1980

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

Under Contract No. DE-AC04-79ET23013

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and
S. V. Krishnaswamy

THE MATERIALS RESEARCH LABORATORY

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ABSTRACT

The main emphasis during the third quarter of the program was laid on the improvement of the quality of sputtered films, their characterization and use in the fabrication of Schottky barrier type diodes and solar cell structures. Films prepared under different conditions and on different substrates were examined by SEM showing nodular growths under certain conditions.

I-V, C-V and photovoltaic characteristics were measured on numerous samples based on n- and p-type films on Ni substrates having top metallization of either evaporated Au and Al. The n-type samples showed up to 200mV V_{oc} and small short-circuit currents. The characteristics observed are indicative of the presence of interfacial layer and surface states. Surface state's capacitance were measured on p-type samples metallized with Au.

During the forthcoming period, we plan to investigate Hall mobility in p- and n-type films, and pn homojunction structures for electrical and photovoltaic characteristics. Efforts will also be extended in overcoming the problems in the neutralization of workable Schottky barrier devices and field-effect resistors for doping and mobility profiling.

1. Introduction

A research program is being pursued to carry out a systematic study on the preparation and characterization of rf-sputtered CdTe thin films with a view to understand their properties and evaluate their usefulness in photovoltaic applications and finally optimize the efficiency of the resulting solar cell structures. Our extensive past experience in rf-sputtering in general and some experience of sputtering CdTe, in particular, provide the background for this research. From our earlier studies, we find that the nature and properties of the sputtered CdTe are strongly dependant on the sputtering conditions, such as rf power, Ar pressure and physical geometry and arrangement of the target substrate temperature etc.

During the first two quarters, substantial progress were made in the preparation and characterization of CdTe films. We can now prepare films over a wide range of sputtering conditions with reproducible physical properties. By employing self-doping technique, we have been able to prepare both Cd as well as Te rich films. We can also vary the crystal structure (specifically, the degree of hexagonality) by varying the Cd/Te ratio in the film. Furthermore, such compositional variation is found to affect the resistivity of the sample. A wide range of resistivity values ($0.03 \Omega\text{cm}$ to $5 \text{ M}\Omega\text{cm}$) were measured for the Cd-rich samples. Tellurium-rich samples were also tested and they indicate resistivity values ranging from $10 \Omega\text{cm}$ to $10 \text{ M}\Omega\text{cm}$. In the next section, we describe the progress made during this reporting period.

2. Progress During the Past Quarter

During this reporting period of the program, primary emphasis has been directed towards fabricating MS and MIS structures using n-type as well as p-type films so that electrical characterization could be carried out on these

materials. These characterization should yield carrier concentration, mobility values. Although the primary emphasis has been devoted to electrical characterization, we have experimented varying preparation parameters such as rf power (and hence rf voltage), substrates, etc. to improve the quality and perfection of the films. While the preparation conditions are described in Section 2.1, materials characterization of the resulting film are described in section 2.2. Finally, section 2.3 describes our efforts in fabricating the various device structures and their electrical properties.

2.1 Film Preparation:

In the last reporting period, films were sputter deposited at 50 to 150W rf power levels. The films showed a mixture of cubic and hexagonal phases. However, we are interested in films that are primarily cubic. From our past experience with sputtering CdTe, we know that cubic films can be obtained by depositing at 10-30mTorr but at high rf powers ($> 300W$). This necessitated the use of another sputtering system, as the one used earlier had some limitations in reaching these higher powers (with very little reflected power and rf tuning problem). The new sputtering system (MRC 8520) (at present totally dedicated to CdTe work), is similar to the earlier one and has such capabilities as sputtering up, sputtering down, substrate etching etc. Preliminary runs were carried out at lower power levels to see that properties of films prepared are indeed system independent. This in fact, established one of our objectives of obtaining films with reproducible physical properties.

In all the runs carried out during this quarter Ar gas pressure was maintained at 20mTorr and the substrate to target distance was also kept constant at $\sim 35mm$. These parameters thus reflect no change from previous runs. Films were prepared at three different power levels ranging from 200 to 400W with the rf voltage ranging from 100V to 1650 volts. Table 1 gives details of various preparation

TABLE I

Relevant Sputtering Parameters Employed in This Study

Sample No.	Ar Pressure (mTorr)	rf Power (watts)	rf voltage (volts)	Sputtering Time (mins)	Substrates(s)	Dopant	Remarks
PSCD 020	25	150	550	50	Ni foil (chem. etch).	Cd	Sys. A; n/n^+ obtained by relative motion of the Substrate
021	25	200	610	40	Ni foil (chem. etch)	Cd	
022	25	200	650	45	Ni foil (chem. etch)	Te	
023-027	23	200	900	53	Ni*	Cd	Sys. E n/n^+ (new Sputtering System)
028	23	200	900	50	evaporated In*	Cd	
031	23	200	550	30	Ni*	Cd	Too Thin Film
032-035	23	200	800	60			
036	25	200	750	60	Ni (no sputter etch)	Cd	
038	23	200	1050	90	Ni, Ni/glass, glass	Cd	System overhauled, Plasma easy to tune and extremely stable
039-041	23	200	1050	90	Mo glass*	Cd	
042	23	200	1050	90	ITO (nesaglass)*	Cd	
044-045	23	400	1650	55-90	Ni, Mo & glass*	Cd	
048	23	300	1425	75	Ni, glass*	Cd	
049	23	200	1025	95	Mo and glass*	Cd	For X-ray and ISS and SEM work
050	23	300	1400	75	Mo and glass*	Cd	For X-ray and ISS and SEM work
051	23	400	1650	55	Mo and glass*	Cd	For x-ray and ISS and SEM work
052	23	200	1025	95	Mo and glass*	Te	For x-ray and ISS and SEM work
053	23	300	1400	75	Mo and glass*	Te	For X-ray and ISS and SEM work
054	23	400	1650	55	Mo and glass*	Te	For X-ray and ISS and SEM work

*Substrate Sputter-etching carried out

conditions.

2.1.a Substrates: As the primary emphasis has been on electrical characterization using MS or MIS structures most of the films were deposited onto Ni substrates. Glass substrates were also employed to obtain x-ray diffraction, thickness evaluation and to obtain SEM photographs. Films on Mo substrates have been used mostly for compositional analysis. However, in a few selected cases, films on Mo were used for electrical characterization also. A couple of films were prepared at one rf power (150W) on Metglass (Metallic-glass) substrates. Prior to deposition, the Ni substrates were usually sputter-etched. However, in selected cases, we have deposited films on chemically etched Ni substrates and Ni substrates that had no chemical etching but usual ultrasonic cleaning procedure. About 1 μ m thick In evaporated on glass slides were also tried as substrates.

2.2 Film Characterization

2.2.a Chemical Composition: The chemical compositions of the CdTe films were measured by ion scattering spectrometry (ISS). The experimental set-up for this were described in detail in the Second quarterly report⁽¹⁾. Briefly, the relative Cd and Te concentrations in these films were evaluated from the observed intensities by using previously determined ISS sensitivities for these elements. The calibration standards were sputter-deposited and ion-plated CdTe films whose compositions were measured by electron microprobe techniques.

Figure 1 shows the data obtained from Cd-doped CdTe films which were deposited at 200W, 300W, and 400W rf power levels. The Cd concentrations of samples situated 10mm from the Cd pellet (samples 049-1, 050-1 and 051-1) were ~55 at %. For samples deposited at 200W rf power, the Cd concentrations gradually decrease as the radial distance of each sample increases from the Cd dopant. This behavior is similar to the results obtained at lower power levels of 50 -

Fig. 1 Variation of Cd concentration (at %) vs. sample number for Cd-doped CdTe films sputter-deposited at 200 watts (O), 300 watts (X), and 400 watts (□) rf power levels.

150W which we reported in the Quarterly Report No. 2.

As the power level was increased to 300 and 400W, however, more abrupt changes in the doping levels as a function of radial distance from the Cd pellet were noted. For example, a Cd concentration of ~52 at % was measured for the 300W sample 050-3 situated 30mm from the dopant, while the Cd concentration of sample 051-3 which was deposited at 400W approached the 50% level. This sharper decrease in the composition may arise from a higher CdTe:Cd sputter-deposition-rate ratio at the higher rf-power levels as compared to the lower powers so that the films at large r are nearly stoichiometric. Such differential sputtering rates may arise due to intrinsic differences in the sputtering mechanisms between different types of materials (eg. metals and semiconductors) or thermal effects at both the sputtering target and the growing film. Bombardment effects at the substrate which are directly related to the substrate temperature, can be mapped out in terms of a spatial distribution by placing a phosphor screen (such as found in TV sets) in place of the substrate. We will be investigating this aspect in the next reporting period.

Figure 2 shows the measured concentrations of Te-doped CdTe films that were prepared at 400W rf power level. A 36%, 31%, and 9% excess Te were measured in samples 054-1, 054-3, and 054-5 which were located 10, 30, and 50mm, respectively, from the Te pellet.

2.2.b Structure: In the last quarterly report, we presented composites of x-ray patterns of Cd-doped CdTe samples prepared at 50, 100, and 150W. These composites were obtained from samples placed radially with the Cd pellet being on one end of the target. Here we are reporting our results on Te-doped CdTe (Te pellet on CdTe target with substrates placed radially) at various rf-power level preparation conditions. Fig. 3 shows a composite of x-ray

Fig. 2 Te concentration (at %) vs. sample number for Te-doped CdTe films sputter-deposited at 400 watts power level.

50W

Fig. 3 X-ray diffraction patterns obtained on CdTe sample #PSCD007, as a function of distance from Te pellet end of the substrate holder.

patterns of samples performed at 50W power at four different radial distances measured from the Te-rich samples. Similar composites at 100W and 150W are shown in Figs. 4 and 5.

As can be seen from these figures, the $\text{Cd}_{1-x}\text{Te}_x$ films exhibit both cubic and hexagonal CdTe phases in agreement with our earlier results. A closer inspection of Figs. 3, 4, and 5 reveals that the cubic phase increases (and hexagonal phase decreases) at large r (as stoichiometry is approached), particularly seen by the appearance of the (311) cubic peak. However, the Te-rich films exhibit non-crystalline solid (NCS) formation with increasing Te concentration. It is seen from Fig. 5 that the hexagonal phase is starting to increase as nonstoichiometry is increased. However, at high Te concentrations where the NCS formation becomes dominant, the interrelationship between the hexagonal and NCS phase is unclear at present. Absorption edge data are needed to find if indeed the NCS phase is hexagonal-like. If so, then the trend would be the same as that for the Cd-doped samples-stoichiometry promotes the cubic phase while off-stoichiometry (both Cd- and Te-rich) favors the hexagonal phase. Again, at the near-stoichiometric Te-rich composition where the cubic phase becomes a more significant portion of the diphasic mixture, the cubic phase is preferentially oriented with (220) planes parallel to the film surfaces. However, unlike Cd-rich films, the relative shift of the (100) peak to the (002) peak for Te-doped films cannot be established because of the above mentioned NCS formation. From Figs. 3, 4, and 5, we see that the extent of NCS formation increases as the power decreases and as the Te concentration increases. Both of these trends are consistent with other metal-chalcogenide systems in which low substrate temperature and high Te concentrations favor NCS formation. The peak at $2\theta = 40.5^\circ$ in those figures is still unresolved at this point. This peak lines up with Te peak. However, a similar peak occurs at about 40° for

100W

Fig. 4. X-ray diffraction patterns obtained on CdTe samples #PSCD008, as a function of distance from the Te pellet end of the substrate holder.

Fig. 5 X-ray diffraction patterns obtained on CdTe sample #PSCD009, as a function of distance from the Te pellet end of the substrate holder.

the Cd doped films which cannot be assigned to any Cd peak. Therefore, a possibility of non-stoichiometric of solid solution compound has to be considered. Also, the continuous change in the peak position from 40.5° to 39.3° as stoichiometry is approached, for all the figures presented here and in the last report, is another indication that a solid solution phase exists.

Figure 6 illustrates the morphology of a typical Cd-doped CdTe film deposited on Ni substrates at ~ 20 mTorr Ar and 200W rf power. Figure 6a depicts the top surface of the film (sample #036) deposited on 0.125mm thick Ni foil while Figure 6b (sample #024) is that for CdTe film on sputtered Ni film ($1\mu\text{m}$ thick) on a glass slide. The surface morphology of both the films are similar with grain size ranging anywhere from 0.1 to $0.3\mu\text{m}$. It is clear from the figure that while the films in general are smooth, substrate surface microstructure seems to have strong influence on the growth. Figure 7 is a composite of the SEM micrographs showing top surface of Cd-rich CdTe films obtained under similar preparation conditions and deposited on various substrates.

Some nodular growths appear in these films. Apart from these nodules and other macro-defect induced growths, the microstructure at the 2 micron level is very similar. Such nodular growths have been reported in other thin film preparation methods such as electro and electroless plating, thermal evaporation, etc.⁽²⁻⁴⁾. For instance, J. C. Knights et al.⁽⁵⁾ report these modules for a-Si:H films, prepared by glow-discharge decomposition thin film technique, which have good electronic characteristics for photovoltaic applications. We have also seen them in sputtered a-Si and a-Ge films⁽⁶⁻⁷⁾. Recently, Spalvins⁽⁸⁾ has observed this in ion plated Cu and Au polycrystalline films. Nodules are coating defects which generally result from spontaneous multi-nucleation at surfaces defects such as microscratches, sliplines, inclusions or impurities. They can grow individually or they can grow together forming aggregates. The size

Fig. 6 Scanning Electron Micrographs of Cd-doped CdTe films.

Substrate: (a) Ni foil, (b) sputtered Ni film.

Fig. 7 SEM micrographs revealing morphology of the top surface of Cd-rich CdTe films prepared under similar conditions and deposited onto (a) Ni foil, (b) Mo foil and (c) ITO (nesaqglass).

Fig. 8 SEM micrographs of CdTe film (Cd doped) on glass substrates (a) substrate not sputter etched, (b) substrate sputter etched, before deposition.

of the nodules $\sim 1\mu\text{m}$, whereas the aggregate structures can be as much as ten microns in diameter. The defect nucleation sites of the substrate are points of high energy concentration which provide the driving force for nucleation and growth of the nodules. These defect growths strongly influence the mechanical and electrical properties of the films. They can produce inhomogeneous stresses in the films, in turn creating pinholes and introducing microcracks in the films. We therefore believe such structural characterization is most desirable in order to understand and control film microstructure and device properties. For instance, Serreze et al.⁽⁹⁾ observed a large number of pinholes hampering their I-V. and C-V. evaluation. Perhaps these may be related to large numbers of such nodules being present.

Figure 8 shows the top surface morphology for films deposited on glass substrates. Figure 8a represents a film deposited on glass with no sputter etch treatment while Figure 8b represents a film deposited after sputter-etching the glass substrate. As can be seen from the figure, the general features are very similar; the nodules appear to be smaller in number for the sputter-etched sample.

2.3 Device Fabrication and Characterization

From the point of view of fabrication simplicity we decided to explore the possibility of developing Schottky barrier diode test devices and photovoltaic cell structures by evaporating Au and Al on both n- and p-type CdTe films sputtered on Ni substrates. We also used Mo and ITO substrates and a few samples were fabricated with a thin SiO_2 layer prior to metallization in an attempt to form MIS structures.

2.3.a I-V Characteristics: Typical I-V characteristics of diodes with Au on n-type, Al on p-type and Au on p-type CdTe sputtered films on Ni substrates

are given in Figures 9, 10 and 11, respectively. These characteristics indicate considerable departure from expected Schottky barrier diode characteristics. The n-type devices with Au contact appear to show nearly square-law and symmetrical characteristics, having low currents at high voltages resembling space-charge limited current. The Al metallization on p-type devices indicate somewhat expected exponential forward I/V characteristics but rather high current in the reverse direction. The Au metallization on p-type devices indicate symmetrical I/V characteristics similar to the n-type devices but the current levels are high even at low voltages. These characteristics strongly suggest that there are interfacial layers and/or presence of high density of surface states.

2.3.b Solar Cell Performance: As anticipated from the observed I-V characteristics, the majority of the solar cells indicated poor open-circuit voltage and short-circuit current performance. The highest V_{oc} of 200mV was observed in n-type devices with I-V characteristics similar to that given in Figure 9; they indicated negligible short-circuit current. A few samples showed short-circuit current in the region of $50\mu A/cm^2$ but negligible V_{oc} . P-type cells with Au and Al metallizations indicated negligible open-circuit voltage and short-circuit currents.

2.3.c C-V Characteristics: Small-signal capacitance of test diodes were measured using an r.f. bridge (B 601, Wayne Kerr) varying the frequency from 50KHz to 1MHz. We were interested in these measurements to find whether the capacitance was dependent on the bias voltage and frequency. The same measurements also directly indicated the value of the shunt conductance across the diode capacitance. Typical capacitance values measured on Au on n-type and Al on p-type devices were 40-100pF for a device area of 0.01 cm^2 and film

Fig. 9 Typical I-V characteristic of the metallized diodes based on n-type films ($0.2\Omega\text{-cm}$) on N^+ substrate.

Fig. 10 Typical I-V characteristic of Al metallized diodes based on p-type films ($10\Omega\text{-cm}$) on Ni substrate.

Fig. 11 Typical I-V characteristic of Au metallized diodes based on p-type films on Ni substrate.

thickness of about $2\mu\text{m}$. These are indicative of the fact that the CdTe film was entirely depleted even under zero bias conditions. Whether this behavior is due to the presence of deep level bulk traps or surface states or a combination of both is difficult to say. The capacitance behavior is very different in the case of Au metallization on p-type films as shown in Figure 12. The value of the capacitance is very large compared to the other two types of devices and its voltage dependence is also unusual. The capacitance showed negligible dependence on frequency. This capacitance cannot be interpreted directly in terms of the surface states without knowledge of the capacitance due to every interfacial layer. However, if the same can be regarded to be very large compared to that due to surface states it is possible to calculate a surface state density value of approximately $10^{12} \text{ cm}^{-2} \text{ eV}^{-1}$ for a capacitance of 1600pF when the device area is 0.01cm^2 .

3. Summary and Future Plans

We have established reproducibility of sputtered CdTe films and achieved a wide range of self-doped films of both p- and n-types. The range of resistivities for Cd-rich samples lies between $0.3\Omega\text{-cm}$ to $5\text{M}\Omega\text{-cm}$ and that for Te-rich samples lies between $10\Omega\text{-cm}$ to $10\text{M}\Omega\text{-cm}$. Structural characterization for Cd-rich films indicates mixed phases under present sputtering conditions. In Cd-rich films the hexagonal phase dominates and the amount of cubic phase increases as stoichiometry is approached. Preferential orientation of the hexagonal phase of the film changes with composition under present operating conditions. We have fabricated schottky barrier type test diodes and solar cell structures using both p- and n-type films on Ni substrates and they have been investigated for electrical characteristics and photovoltaic effects. The present technique of metallization appears to be unsatisfactory due to the possible presence of

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Fig. 12 Typical C-V characteristics of Au metallized diodes based on p-type films on Ni substrate.

interfacial layers and high density of surface states.

We plan to continue with the work in materials growth and characterization and would like to develop an "island" technique to incorporate new dopants (In, Au, Pb and I). It is also our plan to investigate alloying with hydrogen and nitrogen. For improvement of ohmic contacts we would investigate new metals and metallic oxides. We would continue with the fabrication of schottky barrier test devices with different surface treatments prior to metallization for both n-type and p-type films. For the direct measurement of mobility in different resistivity samples, Hall-effect test samples will be fabricated on glass substrates. An experimental set-up for such measurements has already been made and results of preliminary tests are encouraging. Once the Schottky barrier formation becomes a reality we would like to form Schottky barriers onto the Hall effect type test pattern and thus modulate the sample conductivity by the depletion mechanism. This would enable us to determine both mobility and dopant concentration variation within the thin film. Work in the formation of sputtered p-n junctions will also be undertaken in several steps. The first step would be to form p-n junctions on single-crystal materials of one conductivity type by sputtered desposition of the opposite conductivity type films.

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5. PERSONNEL

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

Dr. K. Vedam

Dr. M. B. Das

Dr. S. Krishnaswamy

Dr. L. Tongson

Mr. P. Swab

Mr. R. Petkie

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

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