

Correlation between the Solution Chemistry to Observed Properties of CdTe Thin Films Prepared by CBD Method

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ABSTRACT

The promising solar material Cadmium Telluride (CdTe) is successfully deposited on both plain glass and ITO coated glass substrates. Many variations in composition of the solution used for deposition of the film are made to optimize the deposition conditions. The bandgap calculated from optical transmission studies is found to be a function of the Cd/Te atomic ratio in the film. The atomic ratio in the film is a function of the Cd/Te concentration ratio in the solution used in deposition. Based on several experimental data points an equation involving the Cd/Te atomic ratio in the film and the chemistry of the solution is deduced.

Keywords: Photovoltaic Cell; Thin Film; CdTe; Atomic Ratio; Chemical Bath Deposition (CBD)

1. Introduction

Non-silicon based thin film solar cells are making a head way in the solar market. By 2010, the thin film solar cell production had grown to 13%. The largest share of production was the CdTe cells at 6% and at second place was silicon based thin film solar cells at 5% [1].

CdTe is a direct band gap semiconductor. The reported room temperature band gap of CdTe is in the range of 1.4 - 1.5 eV [2-4] ideally suited for solar radiation absorption. Energy conversion efficiency of CdTe/CdS cells has been increasing continuously over the past few decades. At present, the best in the class CdTe cell has solar power conversion efficiency of 17.3% and at the module level an efficiency of 14.4% is achieved [5]. The theoretical calculations by Fahrenbruch *et al.* [6] indicated highest achievable efficiency for these cells to be 17% which is close to what has been achieved experimentally.

Many fabrication techniques have been attempted for depositing CdTe thin films. Some are controlled atmosphere based techniques like sputtering [4,7,8], thermal evaporation [9-13], e-beam evaporation [14,15], Molecular-Beam Epitaxy (MBE) [16,17], Metalorganic Chemical Vapor Deposition (MOCVD) [18] etc. All these techniques are extremely expensive and require specialized equipment. In addition the size of the sample that can be coated is also limited because of the inherent inhomogeneous coating characteristics of the techniques. Many groups have attempted deposition of CdTe thin

film by three electrode electro-deposition technique [19-22]. Though larger samples can be prepared by this method by having larger electrolyte bath and adjusting the counter electrode surface area, only one sample can be deposited at a time. The process will not have a high throughput in terms of thin film preparation.

The Chemical Bath Deposition (CBD) technique has been widely used for depositing thin films [23,24]. The advantage of the CBD process for depositing thin films are, 1) The process is simple and cost effective, does not require expensive equipment; 2) Large samples and many samples can be coated at a time. In CdTe/CdS solar cells, CdS is deposited from chemical bath [11,25]. The CBD process for CdS is well established [26-28]. However the chemical bath process for CdTe is still in its infancy. There have been a few reports in the literature on the CBD process for preparing CdTe thin films. Padam and Malhotra [29] deposited CdTe on glass, ITO-coated glass, Si wafer and mica using solutions of CdCl₂ and TeO₂ in alkaline medium along with triethanolamine (TEA) and hydrazine hydrate. Klochko *et al.* [30,31] deposited CdTe thin film in acidic medium using CdSO₄ and TeO₂. Deivanayaki *et al.* [32] used cadmium acetate and TeO₂ to deposit CdTe thin films but there is no mention of the pH of the solution. Garadkar *et al.* [33] deposited CdTe thin films using Sodium Tellurosulphite as a source of tellurium and CdSO₄ for obtaining Cd²⁺ ions. Most of the depositions have been carried out at temperatures in the range of 60°C - 95°C. In some cases during deposition the solution was stirred. However, there is no systematic study of compositional variations of the starting solution

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and the actual deposition procedure. In this paper, results on the effect of some of the deposition parameters like the concentration of the complexing agent, deposition time, and the concentrations of cadmium and tellurium salts on the quality of the CdTe thin film obtained are presented.

2. Experimental Details

Cadmium acetate (CA), $\text{Cd}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$ was used as a source for Cadmium and TeO_2 for Tellurium in the solution. Triethanolamine (TEA) is the complexing agent for Cd and Hydrazine hydrate as the reducing agent to reduce Te^{4+} to Te^{2-} . TeO_2 was dissolved in hot dilute Sulphuric acid whereas cadmium acetate (CA) was dissolved in deionized water. To CA solution TEA and hydrazine hydrate were added. 25% ammonia solution was used to adjust the pH to 13. TeO_2 Solution was added in the end and the solution was heated to 92°C in less than 30 min. In the initial experiments the substrates were immersed after the solution attained the required temperature. But later it was found that the quality of the film was better when the substrate was introduced before starting to heat the solution. The slides were taken out of the solution after a stipulated times of 30, 45 and 60 mins. The slides were washed in boiling water and sonicated with acetone to remove the loosely adhering particles. Plain glass slides and ITO coated glass slides were used as substrates for the deposition of thin films. The compositions of the solution used for deposition of the film are given in **Table 1**. In addition, time was also a variant.

Table 1. Composition of the solution for different experiments. The Cadmium and Tellurium are in molarity where as TEA and Hydrazine Hydrate are expressed in milliliters.

Sample	Cd	Te	TEA	HH
1	1	0.07	30	7
2	1	0.07	28.5	7
3	1	0.07	25	7
4	1	0.07	24	7
5	1	0.07	23	7
6	1	0.07	20	7
7	1	0.07	18	7
8	0.7	0.05	20	5
9	0.5	0.05	12	5
10	0.7	0.07	25	7
11	0.5	0.07	25	7
12	0.3	0.07	25	7
13	0.5	0.05	32	7
14	0.7	0.07	30	7
15	0.5	0.07	30	7
16	0.3	0.07	30	7
17	0.5	0.07	32.6	7

The characterization of the films was carried out by recording the X-ray diffraction patterns using Philips XRD 'X'PERT PRO diffractometer using $\text{Cu-K}\alpha$ radiation ($\lambda = 1.5418 \text{ \AA}$). ULTRA 55, Field Emission Scanning Electron Microscope (Karl Zeiss) was used to study the particle size and film thickness. The films deposited on glass substrate were used for the annealing and grain growth studies. The films were heated in air or inert atmosphere to different temperatures. The chemical analysis of the films was carried out by using an Induction Coupled Plasma Spectrophotometer (ICPOES) Thermo Scientific iCAP 6500, ICP Spectrometer. The samples for ICP measurements were prepared by dissolving the film in minimum amount of dilute acids. Perkin-Elmer Lamda 35 UV visible spectrophotometer was used to record the absorption spectrum in the wavelength range of 400 - 1100 nm. The atomic force microscopic studies were carried out using Dimension ICON with ScanAsyst2 machine. The Seebeck coefficient and resistivity measurements were carried out using homemade probes and Keithley meters to measure the voltages.

3. Results and Discussion

The as deposited films when seen with naked eye had a shining finish. The color of film varied from dark ash color to deep brown. The film thickness was around 250 - 300 nm. The AFM pictures of as deposited films on ITO coated and plain glass are shown in **Figure 1** and the roughness profile of the film coated on glass is also shown. The surface roughness seems higher in the ITO coated substrate than on glass substrate. On glass substrate the roughness is about ± 5 nm.

3.1. Crystallographic Studies

The films deposited on glass substrates were thinner than the ones deposited on ITO coated glass substrates. The crystallinity of the films was also different in the two cases with films on ITO coated glass being more crystalline while that on plain glass substrate more amorphous. The ITO coating may be providing nucleating sites for the CdTe during deposition where as on a glass surface such nucleating sites would not be present. Substrate surface effects on nucleation and growth of thin films would be critical and such effects are seen in electrodeposition of CdTe thin films [34]. The as-deposited films on glass substrates did not show any pattern in the XRD. However, the sample #8 deposited on glass for 60 mins with subsequent annealing in inert atmosphere did show the XRD peaks corresponding to the cubic CdTe structure (JCPDS file No. 15-0770). **Figure 2** shows the XRD pattern of as-deposited and annealed sample.

The XRD pattern of as deposited sample #16 ($t = 45$ min) on ITO coated glass substrate is shown in **Figure 3**.

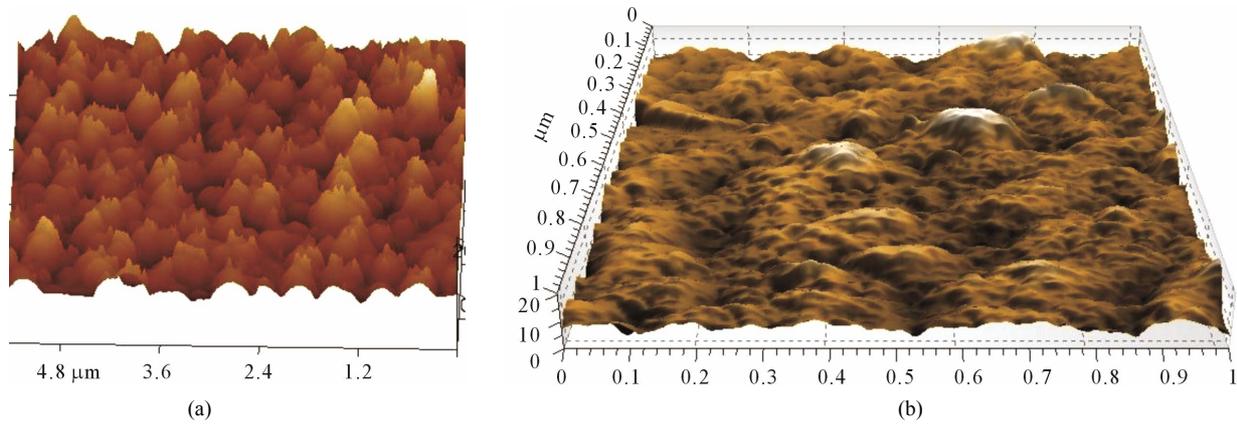


Figure 1. Atomic Force Micrographs of thin film deposited on (a) ITO glass and (b) Plain glass; (c) is the surface profile of the film deposited on glass.

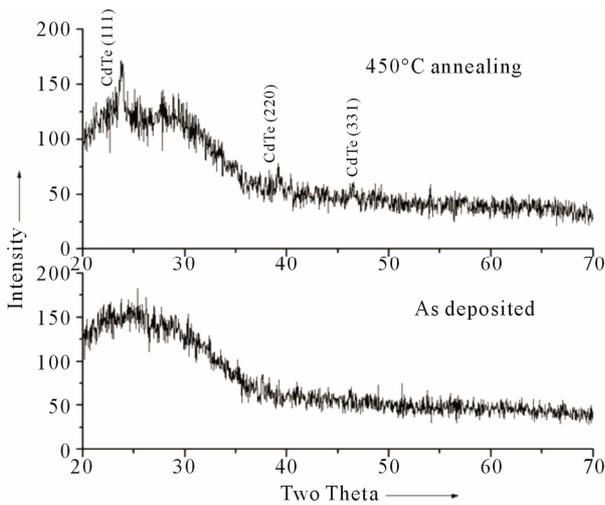


Figure 2. XRD pattern of as deposited and inert gas annealed sample #8.

The main diffraction peaks are at 2 theta of 23.45, 30.47, 35.2, 40.68, 46.3 and 50.84. The peaks at 23.45, 40.68 and 46.3 correspond to diffraction from (111), (220) and (311) planes of cubic CdTe (JCPDS file No. 15-0770). Inter planar spacings calculated for these two theta an-

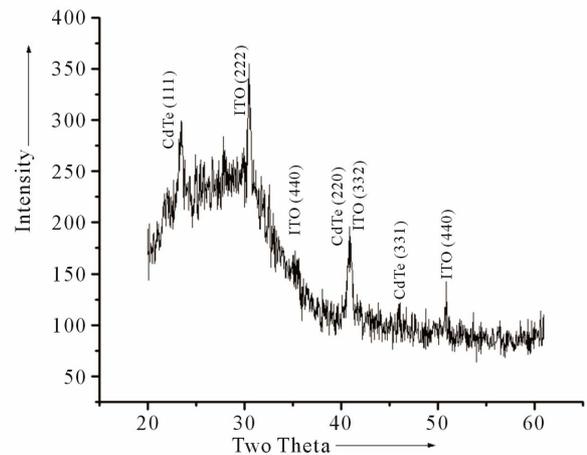


Figure 3. XRD pattern of sample #16 deposited for 45 mins on ITO coated glass substrate.

gles match well with the JCPDS file. The other peaks are due to ITO (JCPDS 6-416). The structure of CdTe deposited is predominantly cubic and reasonably crystalline. The unit cell parameters calculated using the peak positions from XRD is 6.4424 Å which is in good agreement with the reported value [31,34,35].

3.2. Effect of Annealing

The top down SEMs of the film surface are shown in **Figures 4** and **5**. The as deposited films on glass were amorphous in nature with small grain structure. The film surface looks clean, homogenous and dense with no pin holes. Annealing experiments were carried out in air and in inert atmosphere on sample #8 ($t = 60$ min). The grains grow to similar sizes in the two atmospheres. The grain boundaries are created and the grain growth is occurring through grain boundary movement. In the thin films annealed in air the grain growth is very clear whereas in the inert atmosphere annealed samples, up to about 350°C, there is slow grain growth and at higher temperatures, the grain disintegrate into finer particles.

The grain growth trends are shown in **Figure 6**. In both atmospheres up to 350°C, the grains grow at a rate ~ 0.3 nm/degree. Beyond that temperature, the rate of growth increases by an order of magnitude in the case of air annealed samples.

Using grain growth equation [34],

$$(d_t^2 - d_0^2)^{1/2} = Kt^n \quad (1)$$

Expressing K as [36],

$$K = K_0 \exp^{-(Q/RT)} \quad (2)$$

where d is the grain size at time t , K grain growth constant and n is the grain growth exponent. n is estimated to be 0.5 for shorter annealing times. Thus, by plotting $\ln(d_t^2 - d_0^2)$ versus inverse temperature, the activation energies for grain growth are calculated. For the air annealed sample, an activation energy was 0.08 eV for $T < 350^\circ\text{C}$ and 0.66 eV for $350^\circ\text{C} < T < 500^\circ\text{C}$ where as for the inert atmosphere annealed sample Q is found to be 0.16 eV up to 350 C. These values are much lower than the reported value of 0.99 and 1.17 eV for CdCl_2 treated and untreated CdTe films for a film thickness of 1.4 μm [34]. Activation energy of 2.5 eV is reported for a 2 μm thick electrodeposited CdTe film [37] that is close to the activation energy of 2.44 eV for Cd diffusion in CdTe at minimum Cd vapor pressure. The rate of recrystallization

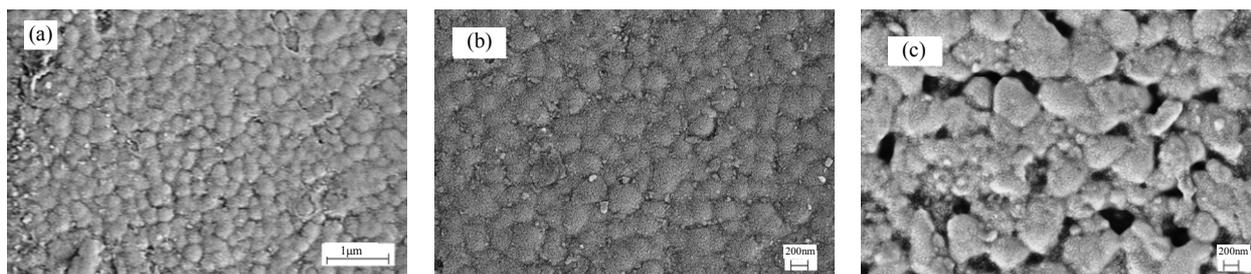


Figure 4. SEM of air annealed sample # 8 at different temperatures: (a) As-deposited; (b) 150°C; (c) 500°C.

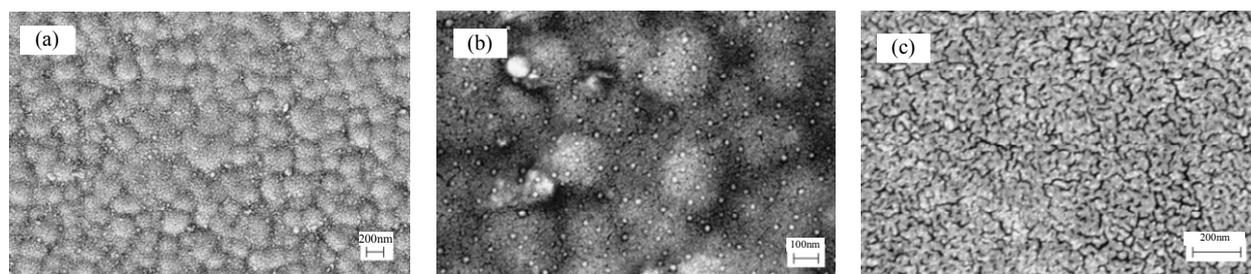


Figure 5. SEM of sample #8 annealed in inert atmosphere at different temperatures: (a) 150°C; (b) 250°C; (c) 450°C.

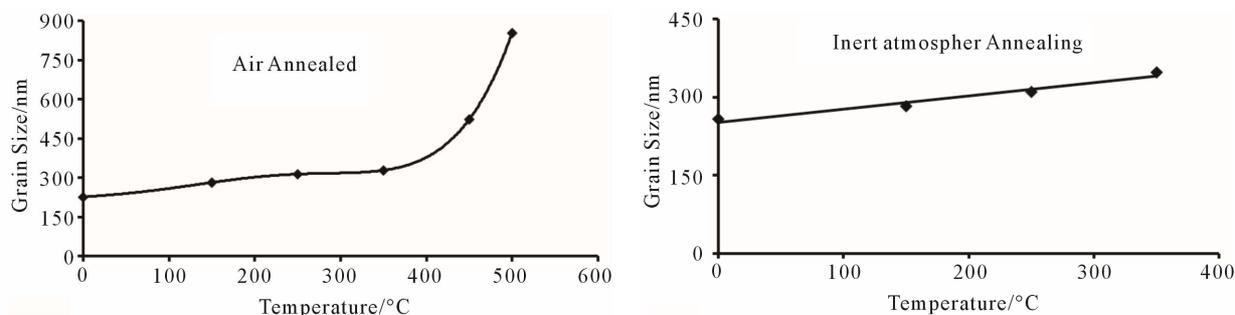


Figure 6. The average grain diameter of particles in the film as a function of annealing temperatures.

was higher with the presence of CdCl_2 compared to without CdCl_2 . It was concluded that the grain growth in these films should be Cd diffusion limited. The films deposited in the present study, from XRD pattern, does not show a $\langle 111 \rangle$ preferred orientation as in the electrodeposited films. The activation energy for grain growth is also dependent on the film thickness because the rate of grain growth is inversely proportional to the film thickness [38]. Thus, in these thin films (< 300 nm) grown from solution, the grain growth may be driven by the grain boundary diffusion rather than by Cd diffusion.

3.3. Optical Bandgaps and Film Compositions

Typical optical absorption spectrum of the CdTe film is shown in **Figure 7**. Optical absorbance was determined from the variation of the optical transmission with wavelength (λ). The absorbance α is related to the optical bandgap through the relation [39],

$$\alpha = A(h\nu - E_g)^n / h\nu \quad (3)$$

where A is a constant, $h\nu$ is the energy of incident radiation, E_g is the bandgap of the material and n is the exponent. The value of n depends on the nature of transition and in the case of CdTe, since the transition is known to be direct allowed ($\Gamma_{15} - \Gamma_1$) [40,41], n is taken to be 0.5. By simplifying the above equation,

$$(\alpha h\nu)^2 = A'(h\nu - E_g) \quad (4)$$

By plotting $(\alpha h\nu)^2$ as a function of $h\nu$ and extrapolating the straight line part of the plot to energy coordinate, it is possible to determine the optical bandgap of CdTe. Optical bandgap varied from about 1.25 to 1.78 eV for various samples and thus it is dependent on the deposition conditions as seen in **Figure 8**.

The effect of Cd/Te molar concentration ratio in the solution used for the deposition of thin films on the bandgap is studied. **Figure 9** shows the variation of bandgap as a function of the Cd/Te ratio used in the solution for depositing the films with 25 mL TEA and 30 mL TEA. Two different deposition times are considered, for

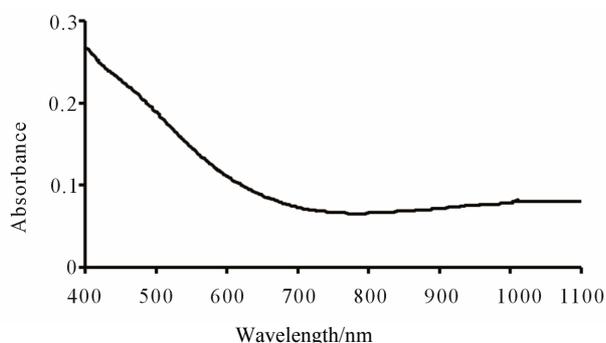


Figure 7. Absorbance spectrum of sample #5.

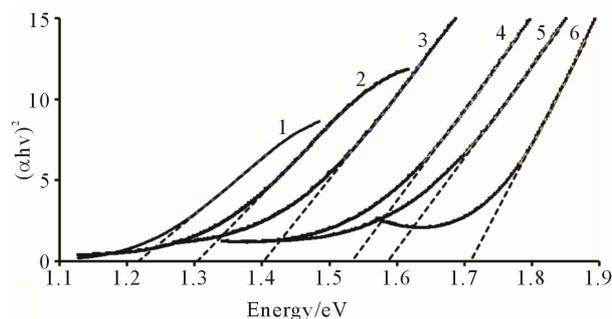


Figure 8. Optical absorption as a function of energy of radiation; samples (1) #6 (deposition time $t = 30$ min); (2) #3 ($t = 30$ min); (3) #5 ($t = 30$ min); (4) #17 ($t = 45$ min); (5) #15 ($t = 60$ min) and (6) #3 ($t = 60$ min).

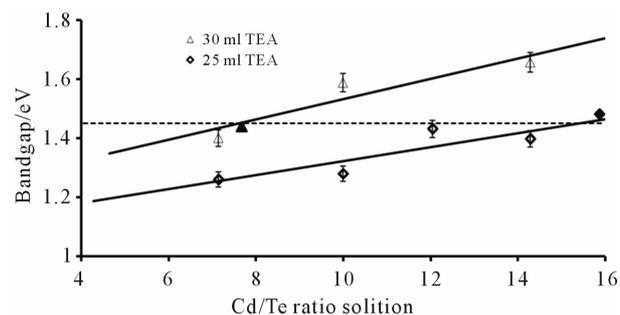
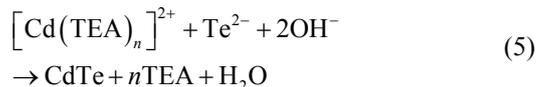


Figure 9. Variation of bandgap with ratio of Cd/Te ions in the deposition bath. The open triangles are for solutions with 30 mL TEA, open diamonds for solutions with 25 mL TEA. The solid lines are least square fit lines and the broken line is an indicator for bandgap of 1.45 eV. The solid triangle and diamond correspond to the experimental data carried out using calculated compositions from the fitted line.

25 mL TEA experiments 45 min data and for 30 mL TEA 60 min data are used. As the Cd/Te ratio decreases the bandgap also decreases. From these plots the Cd/Te ratio that is required to obtain a bandgap of 1.45 eV is calculated. The bandgaps of the CdTe films obtained with these calculated Cd/Te ratios are shown as solid triangles and diamonds in the figure and these data points agree well with calculated numbers from the fitted lines. Thus, the chemistry of the solution used for the deposition of the thin film plays a major role in controlling the optical properties of the deposited film.

The Cd/Te atomic ratio (AR) in the thin films is determined from ICP measurements. The AR of the film depends on the thin film deposition conditions. It is not just the Cd/Te ratio in the solution used for deposition but also on the amount of TEA. TEA is used as a complexing agent for Cd ions. The role of TEA is to prevent the precipitation of $\text{Cd}(\text{OH})_2$ under alkaline conditions. Cd-TEA complex on dissociation through the reverse reaction facilitates the controlled release of Cd^{2+} ions. These ions then combine with the Te^{2-} ions present in the

solution to form CdTe. The stoichiometry of the deposited films can thus be controlled by varying the concentration of the complexing agent [42]. The role of hydrazine hydride is to reduce Te^{4+} ions in TeO_2 to Te^{2-} . The reaction may be described as,



In **Figure 10**, the bandgap is plotted as a function of the Cd/Te atomic ratio in the film as determined by ICP. The straight line is fitted through all the points on the plot and the equation is,

$$\text{Bandgap}(\text{eV}) = -2.308 * \text{AR} + 3.687 \quad (6)$$

There is a relation between the solution chemistry and the atomic ratio in the film. There are many components involved in tuning the solution chemistry. In order to obtain a relationship between the various components of the solution and the AR of the film the quality control statistical tool, Minitab, was used. The parameters that were used to obtain the relation were the Cd, Te, TEA and HH content in the solution in addition to the time of deposition. The relationship that was obtained,

$$\begin{aligned} \text{AR} = & 1.07 - 0.00433\text{TEA} + 0.209\text{Cd} \\ & - 0.11\text{Te} - 0.00416\text{Time} \end{aligned} \quad (7)$$

Under the concentration ranges the experiments are carried out, the amount of hydrazine hydrate did not have a major contribution in determining the Cd/Te atomic ratio in the film.

In order to validate all the correlations that are obtained from the experimental data fitting, the straight line fits of **Figure 9** were used to calculate the required Cd/Te concentration ratio in solution to obtain a band gap of 1.45 eV. For example, with 25 mL TEA and 0.07 M Tellurium in the solution the cadmium that is required according to the data fit is 1.11 M. Experiment was carried out with these conditions and the bandgap of the resulting film was found to be 1.48 eV. From the fit of data plotted on **Figure 10**, the AR required to obtain a

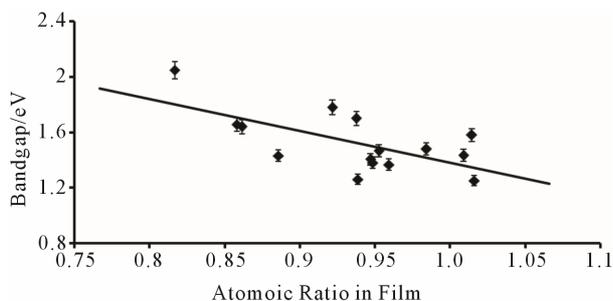


Figure 10. Band gap as a function of the Cd/Te atomic ratio in the thin film. The solid line is the trend line and the dotted broken line corresponds to the band gap of 1.45 eV.

bandgap of 1.45 eV is 0.97, AR predicted by equation 7 for the above experimental conditions is 0.94 and the AR measured on the above film is 0.99. Thus, the chemistry of the solution can be related to the bandgap of the film obtained through the Cd/Te atomic ratio in the thin film.

3.4. Electrical Properties

Electronically, CdTe exhibits amphoteric semiconducting behavior. By suitably doping CdTe it is possible to make it a n and p-type conductor. Intrinsically, Cd deficiency makes CdTe a p-type conductor while with Te vacancies it becomes a n-type conductor. The Cd deficiency state lies close to the upper edge of the valence band while Te vacancy state lies close to the lower edge of the conduction band in CdTe. When Cd/Te ratio approaches 1 the resistivity is maximum [13]. The electrical resistivity of some of the films was in the mega ohm range.

The sign of the Seebeck coefficient is an indicator of the nature of charge carriers in the material. The as deposited film had a positive Seebeck coefficient that increased in to higher value with air annealing at 350°C as reported in **Table 2**. This could be due to partial grain growth and grain boundary development. Also, there may be some amount of oxidation taking place at the grain boundaries. When annealed in inert atmosphere at 350°C, the Seebeck coefficient of the film increased to much smaller extent than when annealed in air.

4. Conclusions

Cadmium Telluride thin films have been successfully deposited from solution on both glass and ITO coated glass substrates. The grain morphology of the as deposited films on ITO glass was found to be more crystalline than on plain glass. XRD pattern corresponding to cubic CdTe is obtained only after annealing in inert atmosphere for the films deposited on plain glass. The grain growth studies of these films have been studied by annealing them in air as well as in inert atmosphere.

The optical studies in the UV-Visible radiation range show that the bandgap varies from about 1.26 eV to over 1.8 eV depending on the composition of the film. Cd/Te atomic ratio in the film is correlated with the bandgap and also with the Cd/Te component ratio in the starting solution that is used for deposition of the films. The ef-

Table 2. Seebeck coefficient of sample #2 after annealing in air and inert atmosphere at 350 C.

Sample	Seebeck Coefficient, μVK^{-1}
As deposited	7.3
Air annealed@350 C	348
Inert atmosphere annealed@350 C	15.2

fect of TEA is also studied in detail and it is found that optimum amount of TEA is essential for obtaining the right Cd/Te atomic ratio in the film. A relation correlating the chemical composition of the solution to the Cd/Te atomic ratio in the film is deduced. Thus, it is shown that the chemistry of the solution used for deposition of the film determines the optical properties of the film produced.

The Seebeck coefficient measurements confirmed the p-type conduction in the films.

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