



Synthesis and Characterisation of CdTe Thin Films

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Abstract

The present global energy, largely from the conventional sources can be depleted in the near future, hence the need for a sustainable alternative sources energy for the growing world population. Photovoltaic (PV), a renewable energy source has been identified as the best alternative source for a sustainable energy due to its cheap, simplicity and environment friendly nature. The PV solar cell devices were usually made from materials with specific properties called semiconductors. One of such semiconductor material is cadmium telluride (CdTe), which is used as an absorber layer in CdS/CdTe solar cell. Cadmium telluride (CdTe) thin films have been synthesised using a simple and cost-effective 3 – electrode electroplating technique. The deposition was carried out from aqueous acidic electrolyte composed of laboratory grade reagent CdSO₄ and TeO₂. The films were deposited on a cathode made from glass/fluorine doped tin oxide (glass/FTO) substrate. A saturated calomel electrode (SCE) was used as the reference with respect to platinum (Pt) anode. The pH and temperature of the bath were 2.00±0.02 and 80°C respectively. This indicates that the films were deposited in an acidic medium. After deposition, each sample was divided into two, one left as-deposited and the other annealed in the presence of CdCl₂. Thereafter, the films were characterised for their structural, morphological and optical properties. The structural properties studied using x-ray diffraction (XRD) showed that the material is crystalline in nature with preferred orientation along the (111) and (211) cubic phases of CdTe. The surface morphology of the films showed that the CdTe material is crystalline with visible grains agglomeration. Increase in grain size with increase in annealing temperature was observed from the films morphology. Optical studies revealed that the bandgap for both as-deposited and the annealed materials show a similar bandgaps of 1.50 eV. Photoelectrochemical (PEC) cell studies were conducted to find the electrical conduction type of the layers in both as-deposited and annealed conditions. The study showed that the material is p-type at the lower voltages ($V < 828$ mV) and n-type at higher voltages ($V > 828$ mV). After CdCl₂ treatment, all layers transformed to p-type. The successful deposition and characterisation of CdTe thin films have been carried out and the results from the various characterisation techniques show that the materials will be suitable for solar cell development.

Keywords: CdTe, electroplating, CdCl₂-treatment, PEC cell

1. Introduction

Cadmium telluride (CdTe) thin films are very important due to their various applications in electronic devices such as solar cells (Bulent M. Basol, 1984). CdTe, a II-VI binary semiconductor materials have been found suitable in complementing material scientist effort to find a material that can directly convert most of the sunlight falling on it into electricity. It is for this reason that CdTe stands out to be one of the most researched and promising semiconductor materials in the production of both laboratory scale and large area optoelectronic devices such as the solar panels. CdTe has a direct and near ideal bandgap (E_g) of 1.45 eV for one bandgap and single p-n junction solar cells with absorption coefficient $>10^4$ cm⁻¹ (Bube, n.d.). CdTe can be n- or p-type in electrical conduction (Chaure et al., 2003; Takahashi et al.,



1985) depending on the molar concentration or added dopants. These are among the properties that make it a suitable material for application in solar energy conversion. CdTe can be grown using low-cost techniques and the material can absorb more than 90% of photons with energy greater than $E_g=1.45$ eV using only about 2000 nm thick CdTe layer.

CdTe thin films have been grown using a number of deposition methods (Dharmadasa et al., 2014). These include close-spaced sublimation (CSS) (Gnatenko et al., 2012), electrodeposition (ED) (Bulent M. Basol, 1984), molecular beam epitaxy (MBE) (Sugiyama, 1984), metalorganic chemical vapour deposition (MOCVD) (Mora-Seró et al., 2004), pulsed laser deposition (PLD) (Ghosh et al., 2012) etc. Electroplating is a low-cost technique, and offers an advantage of growing materials with both n-type and p-type electrical conductivity using intrinsic doping by simply changing the deposition potential.

In electroplating, CdTe can be synthesised using either aqueous [1,11] or non-aqueous (Pandey et al., 1996) electrolytes. In acidic or alkaline (Abdul-Manaf et al., 2015; Arai et al., 2006). In 1928 (Pandey et al., 1996), Mathers and Turner demonstrated for the first time that CdTe can be grown from aqueous solution. The successful deposition and characterisation of CdTe using $CdCl_2$ (Abdul-Manaf et al., 2015) and $Cd(NO_3)_2$ (Salim et al., 2015) precursors have been recently carried out.

This communication give the results of CdTe thin films grown on glass/FTO substrates using 3-electrode system from electrolyte containing 1 M $CdSO_4$ and TeO_2 . The layers were further studied for their various properties using analytical techniques such as XRD, morphology, PEC cell measurement, optical absorption. The effects of $CdCl_2$ (CC) treatment on the structural, optical, and electronic properties of the layers have also been investigated. This is to optimised the material parameters using analytical characterisation so that quality and high efficiency Cds/CdTe solar cell will be fabricated (The fabricated solar cells were not reported in this work). Therefore, only the the material characterisation results were published in this paper.

2. Materials and Methods

The materials/reagents required for this work includes $CdSO_4$ powder (> 99%) purity and high purity TeO_2 (99.999%) powder which provided the Cd and Te sources respectively. The cathode was glass/FTO substrates while the anode is a Pt strip. The reference electrode was a SCE and the deposition electrolyte was prepared from deionised water. Ammonium hydroxide (NH_4OH) and dilute sulphuric (H_2SO_4) acid were used to adjust the pH of the bath. Gill AC potentiostat was used as power source while the heating of the bath was provided by hot-plate with magnetic stirrer. The substrates were clean using organic solvent (acetone & methanol) and rinsed in deionised water. All materials/chemicals were purchased from Sigma-Aldrich, UK.

The electrolyte was prepared from 1 M $CdSO_4$ and TeO_2 solution in 800 ml of deionised water. The pH of the bath was adjusted to 2.00 ± 0.02 using either H_2SO_4 and/or NH_4OH dilute solution. The temperature of the bath was $\sim 80^\circ$ C. The substrates (cathode) were clean in methanol and rinsed in deionised water before insertion into the bath. After deposition, the substrates were again clean in deionised water and then allowed to dry in open space air. Thereafter, the substrates were cut into two, one left as-deposited and the other annealed in $CdCl_2$ for onward characterisation of the materials.

3. Results and discussion

3.1 Cyclic voltammetry

In the electrodeposition (ED) method of semiconductor growth, cyclic voltammetry is usually employed to determine the approximate deposition voltage for materials. This technique measures the current-voltage (I-V) through a conducting aqueous solution containing the ionic species of the semiconductor materials. Figure 1 shows the voltammogram scan for an aqueous solution containing 1M CdSO₄ and a small amount of TeO₂. The use of small amount of TeO₂ is due to the high difference in the reduction potential (E⁰) values of the constituent species of Cd (E⁰=-0.403 V) and Te (E⁰=+0.593 V) (Vanysek, 2000) materials. The growth temperatures (T_g) was ~70°C. The pH of the bath was adjusted to 2.00±0.02 using dilute aqueous solution of H₂SO₄ or NH₄OH.

The process for the deposition of CdTe on the cathode from acidic aqueous electrolyte as proposed by Panicker *et al.* (Panicker, 1978). The first mechanism is a diffusion control which involves the reduction of $HTeO_2^+$ to Te which reacts with Cd²⁺ to form CdTe on the surface of the cathode. The expression for the formation of CdTe on the cathode are given in equations 1 and 2.

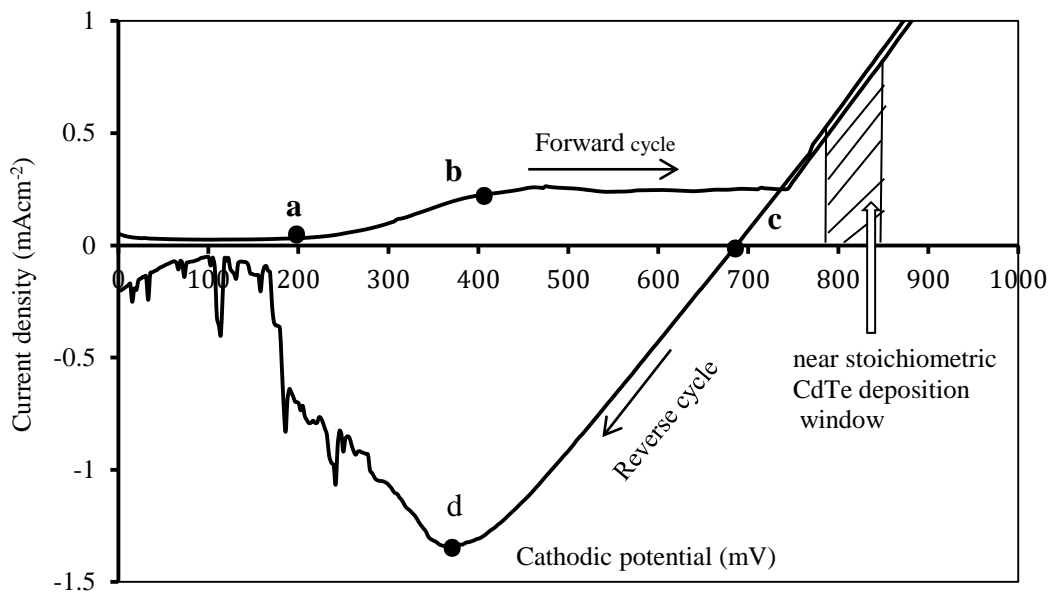
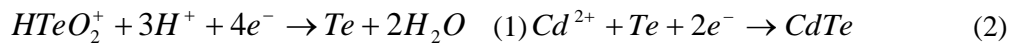


Figure 1: Cyclic voltammogram of aqueous solution consisting of 1M CdSO₄ and TeO₂ with glass/FTO cathode. The reference electrode used for the system is saturated calomel electrode (SCE).

The formation of CdTe thin films on the cathode is highly influenced by diffusion process. Looking at the voltammogram (Figure 1) and with reference to the reduction potential (E⁰) values of the elements involve in the electrochemical process, Te with redox potential of +0.593 V, will deposit first on the cathode and this starts at cathodic voltage around 200 mV (point *a*) and cadmium with redox potential of -0.403 V starts to deposit at cathodic voltage around 450 mV (point *b*). The deposition of near stoichiometric CdTe window was narrowed down and is indicated by the shaded portion of the voltammogram in the cathodic voltage range (780-840) mV. In the reverse cycle, it is observed that the

dissolution of elemental Cd and Cd from CdTe starts to occur at growth voltage of 695 mV (point *c*). A large negative peak at cathodic voltage around 370 mV (point *d*) could be due to dissolution of Te from the cathode.

4. Results and Discussion

4.1 X-ray diffraction

The X-ray diffraction (XRD) was carried out to study the structural properties of the CdTe thin films. Figure 2 shows a typical XRD spectra for the (a) as-deposited (AD) and (b) after CdCl₂ (CC) treatment. In this experiment, the growth voltages were changed in steps of 2 mV in order to obtain the approximate deposition range for the growth of near stoichiometric CdTe material with the highest crystallinity. To achieve this, the growth voltage (Figure 2) was narrowed down in the range of (830-838) mV within the vicinity of the inversion voltage (V_i) (see figure 4) and it shows that the material grown at the cathodic voltage of 834 mV shows better crystallinity with the highest (111) peak intensity for the as-deposited films. Therefore, this growth voltage is considered the best for the as-deposited materials. Therefore, only the material grown at the best growth voltage (834 mV) is plotted as shown in Figure 2.

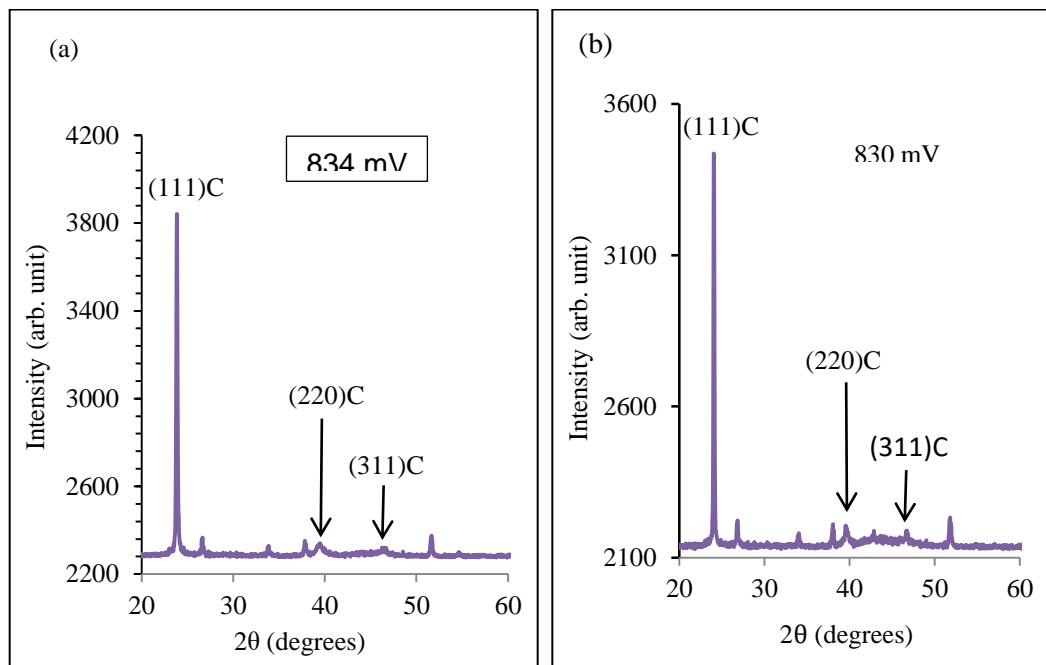


Figure 2: XRD spectra of (a) as-deposited and (b) CC-Treated CdTe thin films.

The XRD pattern, irrespective of growth voltage the, CdTe thin films revealed that the material are polycrystalline in nature with strong preferred orientation along the (111) cubic phase of CdTe at $2\theta = \sim 24.0^\circ$. CdTe usually grows with a preferred orientation along the (111) peak for low temperature grown materials irrespective of the growth method (Jaegermann et al., 2009). Other two weak peaks at approximately 2θ values of $\sim 39.8^\circ$ and $\sim 46.7^\circ$ correspond to reflections from (220) and (311) cubic phases of CdTe respectively. These peaks data matches well with Joint Committee on Powder Diffraction Standards (JCPDS) reference file number 01-075-2086. The strict requirements in the quality of materials for application in solid-state electronics especially solar cells are not only important but necessary for optimum device performance. The as-deposited films are usually not suitable for application in solar cell due to their poor electronic properties as a result of defects and high density of grain boundaries.

Therefore, the as-deposited materials must undergo a post growth heat treatment in the presence of CdCl₂ (CC or any other suitable Cl containing salt. After CC heat treatment (Figure 2(b)), the highest crystallinity in the sample grown at 834 mV in the as-deposited films shifted to a layer grown at 830 mV. This is attributed to recrystallisation and annealing out defects in these materials. Therefore, 830 mV is taken as the best material growth voltage with respect to SCE under the experimental conditions used. It is important to get materials that show highest possible XRD peaks which mean high crystallinity suitable for applications in solar cells. Table 1 shows the summary of the XRD parameters obtained for the best materials in the as-deposited and the CC treated CdTe thin films with respect to the (111) peak. The parameters obtained from the XRD machine include; the angle position (2θ), d-spacing (Å) and crystallite size. The crystallite size (D) of the films was calculated using the full width at half maximum (FWHM), β, using the Scherer's formula (Monshi et al., 2012) given in equation 3.

$$D = \frac{0.94\lambda}{\beta \cos\theta} \tag{1.0}$$

Where D is the crystallite size in nm, β is the FWHM in radian, λ = 1.54 Å is the wavelength and θ is the position of the angle in degrees (Bragg's angle). The obtained parameters in the table show that the surface treatments have modified their structural properties which indicate recrystallisation, defects passivation and the release of compressive stress incorporated into the material during growth. The crystallite sizes were calculated for the best growth voltages.

Table 1: Summary of XRD data for the best growth voltages for as-deposited (AD) and CC treated CdTe layers.

Condition of material	V _g (mV)	2θ (deg.)	d-spacing (Å)	FWHM (deg.)	Crystallite size, D (nm)	Plane of orientation	Assignment
AD	834	23.9	3.73	0.162	52.3	(111)	CdTe/Cubic
CC	830	24.1	3.70	0.129	65.2	(111)	CdTe/Cubic

In XRD, the magnitude of intensity signifies how crystalline a material is. While it is desirable to fabricate solar cells with highly crystalline materials, the photovoltaic output of the devices does not solely depend on the crystallinity of the material but equally on other important factors such as the quality of the heterojunction, optimum doping concentration etc. Another observation is the decrease in the full width at half maximum (FWHM) from 0.162 degree in the as-deposited to 0.129 degree for layers annealed in the presence of CC. This indicates grain growth and recrystallisation in these films (Lalitha et al., 2004). In other words, crystallite size increases with decrease in FWHM as shown in Table 1.

4.2. Morphology studies

Figure 3 shows two CdTe layers grown for 1 hour at three different cathodic voltages of 826, 828 and 830 mV within the vicinity of the V_i (see figure 4). After growth, each sample was cut into two parts for as-deposited (AD) and CdCl₂ (CC) treatment. Figure 3 (a & b) shows AD and CC treated CdTe layers grown at 826 mV and Figure 3 (d & e) shows AD and CC annealed CdTe layers grown at 828 mV while Figure 3 (g & h) shows AD and CC treated CdTe layers grown at 830 mV. The AD films in each case were covered with large clusters or agglomerations of small grains consisting of nano-crystallites. The clusters have varying sizes up to sub-micron sizes for largest ones. The small cluster size is in the range (200-300)

nm. The morphology of this layer shows fairly compacted clusters with scattered pinholes. After annealing in the presence of CC at 400°C for 15 minutes in air, the films show grain growth and recrystallisation becoming more compact with an increase in the average cluster size in the range (200-500) nm for layers annealed in the presence of CC.

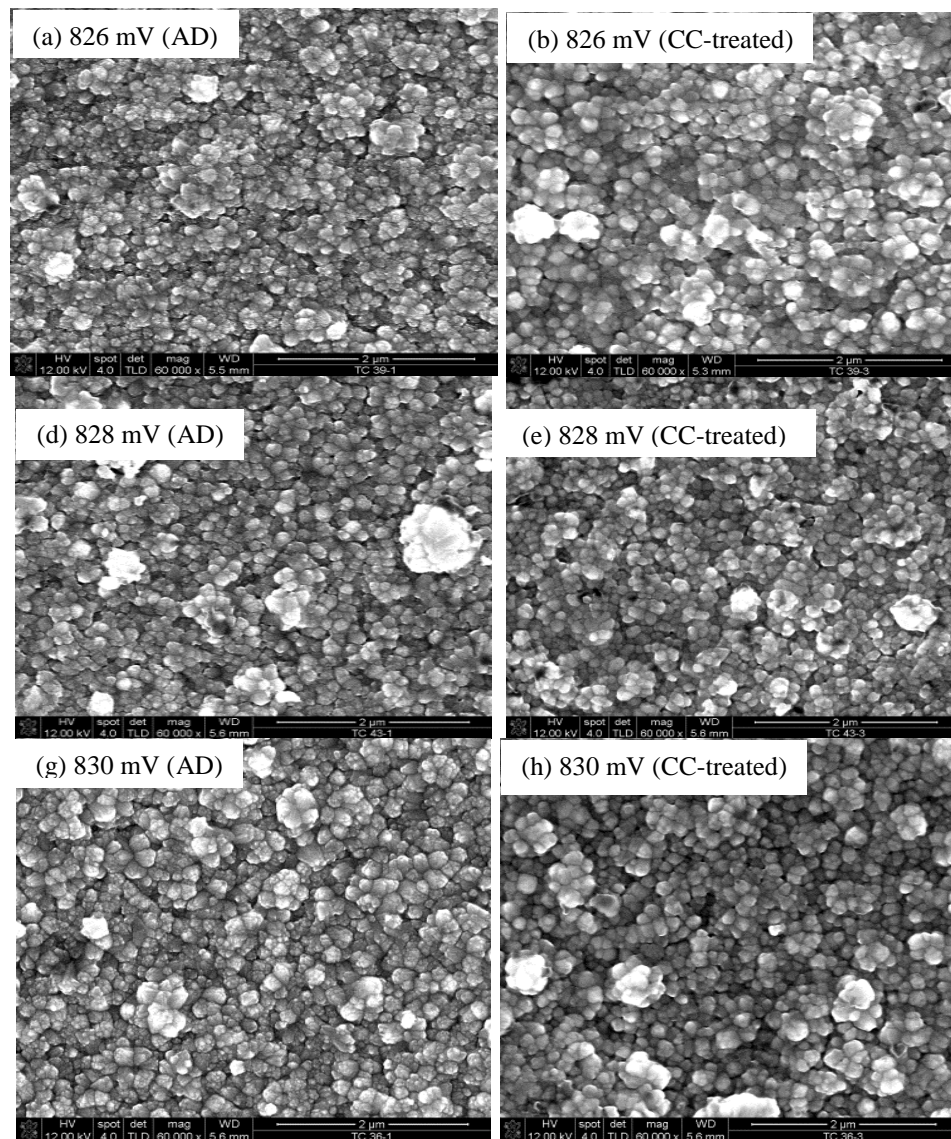


Figure 3: Typical SEM images for CdTe thin films grown at different growth voltages. All annealing was done at 400°C for 15 min in air.

4.3. Photoelectrochemical cell measurements

Photoelectrochemical (PEC) cell measurements were carried out to confirm the electrical conductivity type of the CdTe thin films. Many samples were grown at different growth voltages. The samples were grown at voltages between (820-836) mV.

Figure 4 show the PEC signals observed for as-deposited and CC treated CdTe layers. The CdTe layers grown at low cathodic voltages are Te-rich and p-type in electrical conduction, while at higher cathodic

voltages; the layers are Cd-rich and n-type in electrical conduction. In between these two regions, there exists an inversion voltage (V_i) which produces near stoichiometric material with highest crystallinity and suitable electronic properties for solar cell fabrication. Materials grown at this point are near stoichiometric with intrinsic semiconducting properties and the Fermi level should be right in the mid-gap. It can be observed from Figure 4 that moving to higher voltages from the V_i increases n-type doping while moving to lower voltages increases the p-type doping. Materials grown at voltages higher than the V_i are Cd-rich and the possible defects at this region are Te-vacancies (V_{Te}), Cd in Te sites (Cd_{Te}) and Cd interstitials (Cd_i). Whereas the films grown at voltages lower than the V_i are Te-rich with possible defects of Cd-vacancies (V_{Cd}), Te in Cd sites (Te_{Cd}) and Te interstitials (Te_i). The value of V_i may shift, depending on the factors such as Te concentration, stirring rate, growth temperature and pH value.

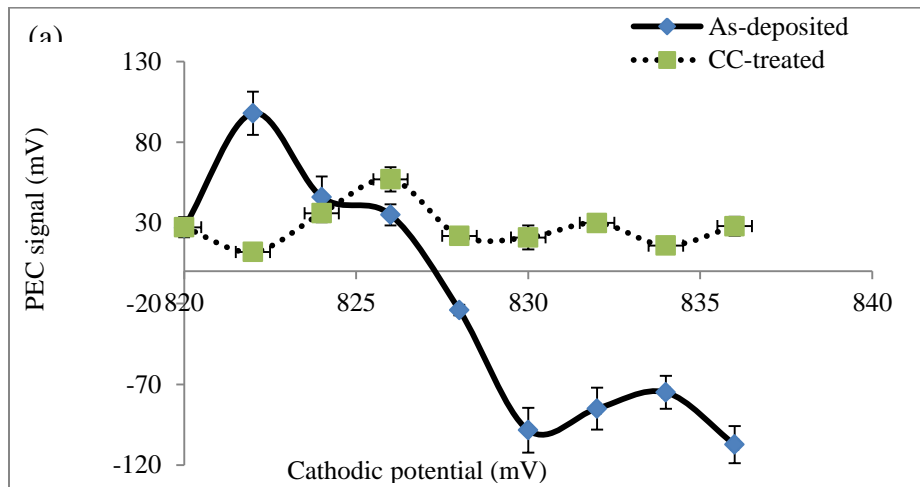


Figure 4: PEC signal as a function of growth voltage for as-deposited and $CdCl_2$ -treated CdTe thin films grown at different cathodic voltages. $CdCl_2$ annealing process was carried out at $400^\circ C$ for 15 minutes in air.

After CC treatment, materials show that p-type materials move towards n and n-type materials move to p-type conduction. It should be noted that this is due to Fermi level (FL) movement within the CdTe bandgap. There is a possibility for a material to move from p-type to n-type (Meyers, 1988; Yang et al., 2010) and from n-type to p-type (B. M. Basol et al., 1985; Duffy et al., 2000) after annealing. This shows that the direction of movement of the FL in the bandgap depends on the initial condition of the layer, doping effects and heat-treatment conditions.

4.4. Optical absorption studies

Optical absorption studies were carried out to estimate the optical energy band gap (E_g) of the materials. Figure 5 shows the plot of the square of the absorbance (A^2) vs photon energy ($h\nu$) for a typical CdTe material. This is to study the absorption edge and the optical energy bandgap of the material. Energy band gaps are usually determined or estimated from the plots of the $(ah\nu)^2$ vs photon energy ($h\nu$) popularly known as Tauc plot. This is estimated by extrapolating the straight line portion of the square of the absorption (A)² edge to the photon energy ($h\nu$) axis ($A^2 = 0$) (Tauc, 1966).

In this experiment, as-deposited CdTe layers grown at different potentials were cut into two pieces. The first set of the samples was left as-deposited and the other set annealed in the presence of CC at $400^\circ C$ for 15 minutes in air. This is to check the effects of the CC treatment on the initial material properties and

how it affects the bandgap of these layers. It can be observed that from the Figure 5a and 5b, the bandgaps of the CdTe layers in both as-deposited and CdCl₂ material show similar bandgap values of 1.50 eV similar to the bulk bandgap of CdTe (1.45 eV). It is important to note that only the best material in each of as-deposited and CdCl₂-annealed samples were plotted as shown in Figure 5.

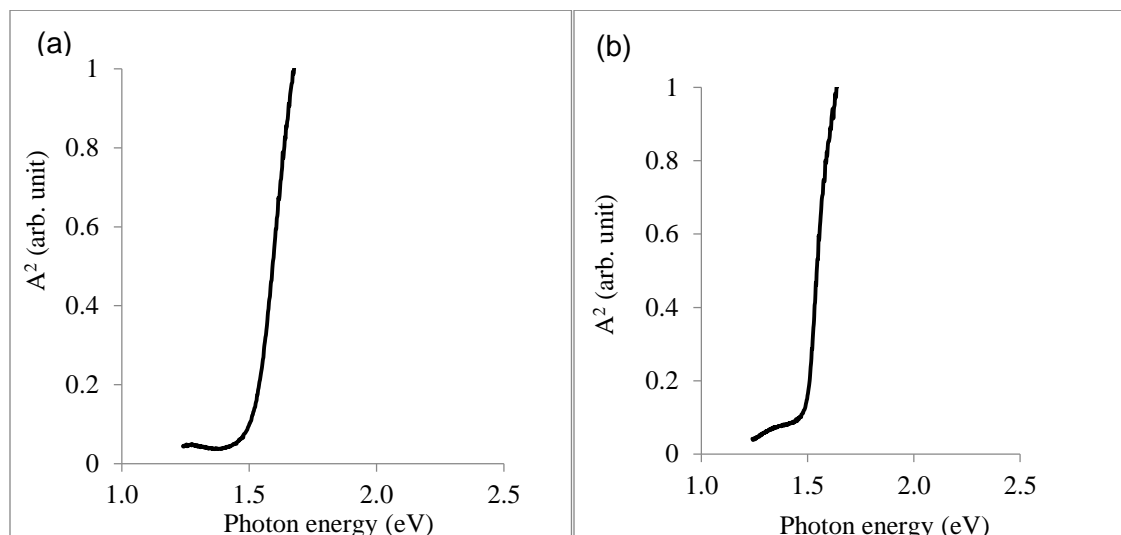


Figure 5: Absorption coefficient for (a) as-deposited and (b) CdCl₂-treated CdTe layers. The CdCl₂ annealing was carried at 400°C for 15 minutes in air.

5. Conclusions

Thin films of CdTe have been successfully electroplated from an aqueous electrolyte on glass/FTO substrate from aqueous acidic electrolyte using 3E systems. In this method, saturated calomel electrode (SCE) was used as the reference electrode. The working electrode (cathode) and the anode were glass/FTO substrate and platinum sheet respectively. The CdTe semiconductor was electroplated from aqueous electrolyte consisting Cd and Te ions. PEC results of the as-deposited layers indicate that both n-type and p-type electrical conduction types can be achieved by simply changing the deposition voltage.

Structural studies using XRD show that the CdTe films are polycrystalline in nature with a preferred orientation along the (111) cubic phase. The surface morphology of the AD films show clusters consisting of small grains in the range (200-300) nm but after CC treatment, the grains coalesced to form few microns size grains. It also shows that increase in grain size is dependent on the annealing temperature. Optical absorption studies show that, both as-deposited and CdCl₂ annealed layers show similar bandgap values of 1.50 eV which is similar to the bulk bandgap of CdTe (1.45 eV).

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