

Characterisation of Electrodeposited Cadmium sulphide Thin Films for Application in CdTe Solar Cells

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Abstract

Cadmium sulphide thin films have been grown from acidic electrolyte prepared from analytical grade reagent using electroplating technique. The films were grown on glass/FTO substrate in an acidic medium of pH 2.50 and temperature of $\sim 85^\circ\text{C}$. The layers were intended for use as window layers in CdTe thin film solar cells. The structural study show that the films were crystalline with the preferred orientation along the (100)H phase of cadmium sulphide (CdS). The optical study shows that the bandgaps matched well with the bulk bandgap of CdS (2.42 eV) after the cadmium chloride (CdCl₂) treatment. The films also show improved morphology after CdCl₂ with increased roughness than in the case of the as-deposited films. Photoelectrochemical (PEC) cell study indicate that all the layers in both the as-deposited and after the CdCl₂ were n-type in electrical conduction.

Keywords: Electrodeposition, CdS, Thin films, n-type, CdCl₂ treatment

1.0 Introduction

Cadmium sulphide (CdS) is a wide bandgap II-VI semiconductor material with a direct optical bandgap of 2.42 eV ($\lambda=514.5$ nm) (Nishino *et al.*, 1999) at room temperature. CdS is an n-type material with absorption coefficient of 4×10^4 cm⁻¹ (Zarębska and Skompska, 2011). These optoelectronic properties make CdS suitable especially as a window layer in solar cells when paired with absorber materials such as CdTe (Basol, 1984; Echendu *et al.*, 2014; Ojo and Dharmadasa, 2016) and CIGS (Kannan Ramanathan, Keane and Noufi, 2005; Bhattacharya, 2013). CdS has also been utilised in areas such as sensors (Brands, 2016), transistors (Walker *et al.*, 2014) and radiation detectors (Fowler and Grant, 1963; Johnson and Laboratories, 1970). The deposition of CdS thin films has been demonstrated using both high temperature complex systems such as closed space sublimation (CSS) (Cruz, Ferreira and Pinheiro, 2014; Paudel, Xiao and Yan, 2014) and low-cost methods such as electrodeposition (Abdul-Manaf *et al.*, 2015; Salim *et al.*, 2016). The main focus of the PV community is to reduce the Watt per hour ($\text{\$W}^{-1}$) cost using low-cost materials and processing methods. Electrodeposition, a low-cost method of semiconductor growth has shown the flexibility of semiconductor growth from both aqueous and non-aqueous solutions (Sasikala,

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Dhanasekaran and Subramanian, 1997) and has been proven to be manufacturable in the production of solar panels (Cunningham, Rubcich and Skinner, 2002). It also provides advantage for the control of deposition parameters, and hence material properties.

Electrodeposition of CdS thin films has been carried out using both 3-electrode (Lade, Uplane and Lokhande, 1998) and 2-electrode (Salim *et al.*, 2016) systems. Another simple method used to produce electronic quality CdS is by the use of chemical bath deposition (CBD) method. However, this method is not suitable due to its batch process of chemical waste which is a great environmental concern due to the Cd element contained therein. On the other hand electrodeposition is a continuous process with less chemical waste, is considered a better option.

Presented in this paper is the cathodic electrodeposition of CdS thin films using 2-electrode system for application as window layers in CdS/CdTe solar cell devices. Prior to incorporation into devices, the films were characterised using various analytical techniques for their structural, optical, morphological and electrical properties so that high electronic quality CdS can be used for the fabrication of high-efficiency solar cells.

2.0 Preparation of CdS electroplating electrolyte

The CdS bath was prepared using 300 ml of deionised water containing 0.075 M CdCl₂ (purity 99.995%) and 0.15 M (NH₄)₂S₂O₃ (purity 98%); which are the sources of Cd and S ions, respectively. The electrolyte was contained in a 500 ml Teflon beaker which was in turn contained in a 1000 ml glass beaker filled with deionised water (i.e. a water bath) to maintain uniform heating of the electrolyte. The pH of the electrolyte was adjusted to 2.50±0.02 using HCl or NH₄OH. The temperature of the bath was maintained at ~85°C throughout the deposition process. All chemicals and the substrates were purchased from Sigma-Aldrich, UK. All the experiments were conducted in Materials and Engineering Research institute (MERI) Laboratories of Sheffield Hallam University, UK.

The cleaning of the substrate was carried out using soap solution then rinsed in deionised water. The substrate was further degreased in methanol and then rinsed in deionised water and dried in air afterwards. The glass/FTO cathode was attached to a carbon electrode using polytetrafluoroethylene (PTFE) tape while the anode is a high purity carbon rod. The electrodeposition process was carried out using computerised Gill AC potentiostat (ACM instrument). Glass/FTO substrate was used for the deposition of all CdS layers in this work.

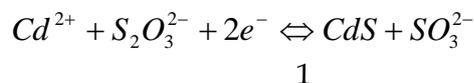
3.0 Result and discussion

3.1 Cyclic voltammetry

The approximate growth voltage for the deposition of CdS thin films was determined using cyclic voltammetry. Using this approximate voltage range, several CdS layers were deposited and characterised to obtain the best growth voltage (V_g) for the deposition of near stoichiometric CdS layers. Figure 1 shows the cyclic voltammogram for the deposition of CdS from aqueous acidic solution. The pH of the electrolyte was adjusted to 2.50±0.02 at room temperature using NH₄OH or HCl and the deposition was carried out at ~85°C with a scan rate of 3 mVs⁻¹. The cyclic voltammetry was taken in the voltage range (0-1500) mV. Based on the reduction potential of sulphur ($E^0=+0.144$ V) (Nishino *et al.*, 1999) and Cd ($E^0=-0.403$ V) (Vanysek, 2000), sulphur with more positive redox potential, will start to deposit first on the cathode (Nishino *et al.*, 1999) at voltages around 200 mV where the deposition current begins to rise (as observed in the inset diagram of Figure 1). It is expected that Cd

with more negative redox potential will start to deposit at a voltage around 850 mV where a sharp increase in deposition current is noticed.

The electrochemical equation for the deposition of CdS from Cd^{2+} and $S_2O_3^{2-}$ is given by equation 1 (Zarębska and Skompska, 2011).



The co-deposition of S and Cd to form near stoichiometric CdS is expected to occur at voltages around 1100 mV to 1300 mV. As shown in the voltammogram (Figure 1), the shaded portion indicates (not to scale) the region for the deposition of near stoichiometric CdS thin films. Thin films of electronic quality CdS can be deposited within a wide voltage range as experimentally confirmed.

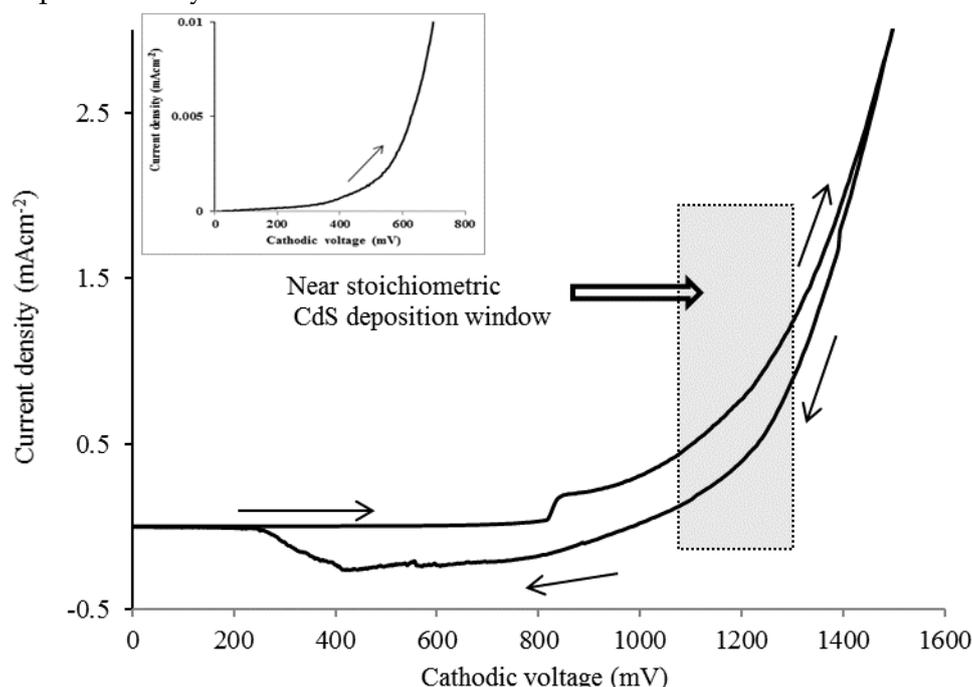


Figure 1: Cyclic voltammogram for an electrolyte consisting of 0.075 M $CdCl_2$ and 0.15 M $(NH_4)_2S_2O_3$. The pH and temperature of the electrolyte were 2.50 ± 0.02 and $\sim 85^\circ C$ respectively.

At higher voltages around 1400 mV, deposition of dendrites on the cathode was observed. Therefore, this region was avoided due to their poor electronic quality.

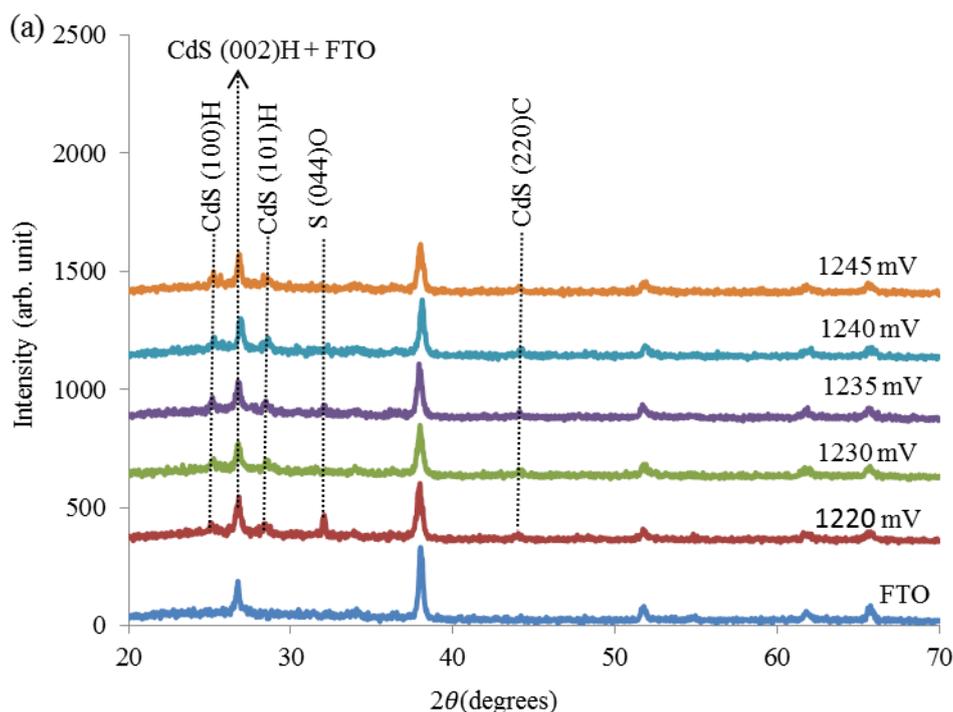
In the reverse cycle, elemental Cd and Cd from CdS start to dissolve into the bath around 980 mV and the negative peak around 400 mV could be due to dissolution of S from the cathode.

3.2 X-ray diffraction

Figure 2 shows the XRD patterns of (a) as-deposited and (b) $CdCl_2$ treated thin films of CdS grown at different growth voltages. The XRD patterns were studied in the 2θ range ($20-70^\circ$). The growth voltages were selected based on the approximate deposition voltages obtained from the voltammogram scan for the growth of near stoichiometric CdS thin films. The growth voltages were changed in steps of 5 mV within the estimated voltage range and each CdS layer was grown for a duration of one hour. After growth, the samples were rinsed with deionised water and dried in air. The as-deposited samples grown at each voltage were cut into two. One of the samples set was left as-deposited and the other annealed in the presence of $CdCl_2$ at $400^\circ C$ for 20 minutes in air. After characterisation, the growth voltages were

further narrowed down close to the near stoichiometric region in the range (1220-1245) mV for the purpose of analysis. The best material was obtained by monitoring the most intense peak among the entire XRD spectra.

The XRD spectra of both (a) as-deposited and (b) CdCl₂ treated CdS layers shown in Figure 2 revealed that the films were polycrystalline in nature and consists of both hexagonal and cubic crystal structures in both as-deposited and after CdCl₂ treatment. The three diffraction peaks observed at approximate 2θ values of 24.9, 28.3 and 26.5° corresponds to reflections from (100), (002) and (101) hexagonal phase of CdS respectively. These peaks match well with JCPDS file number: 01-080-0006 for hexagonal CdS crystal phase. The peak at 2θ=~26.5 corresponding to (002) was however not considered for further analysis because of its overlap with the peak of the underlying FTO substrate ((022)H+FTO). The small peak at 2θ=~43.5° corresponds to reflection from (220) cubic phase of CdS. The observation of (100), (002) and (101) reflections in CdS films is well documented in the literature (Morris and Vanderveen, 1993; Sasikala, Dhanasekaran and Subramanian, 1997; Abdul-Manaf *et al.*, 2015; Salim *et al.*, 2016). The peak at 2θ=~32.1° for sample grown at 1220 mV can be observed and is related to the reflection from the (044) orthorhombic elemental sulphur phase. The presence of this peak in CdS films has been reported for both CBD (Barote, Yadav and Masumdar, 2011) and electrodeposited CdS layers (Fauzi, 2015). The appearance of this peak in CBD grown CdS was attributed to the high concentration of S used in the deposition solution (Barote, Yadav and Masumdar, 2011) which correlates to the higher concentration of sulphur than cadmium used in this experiment.



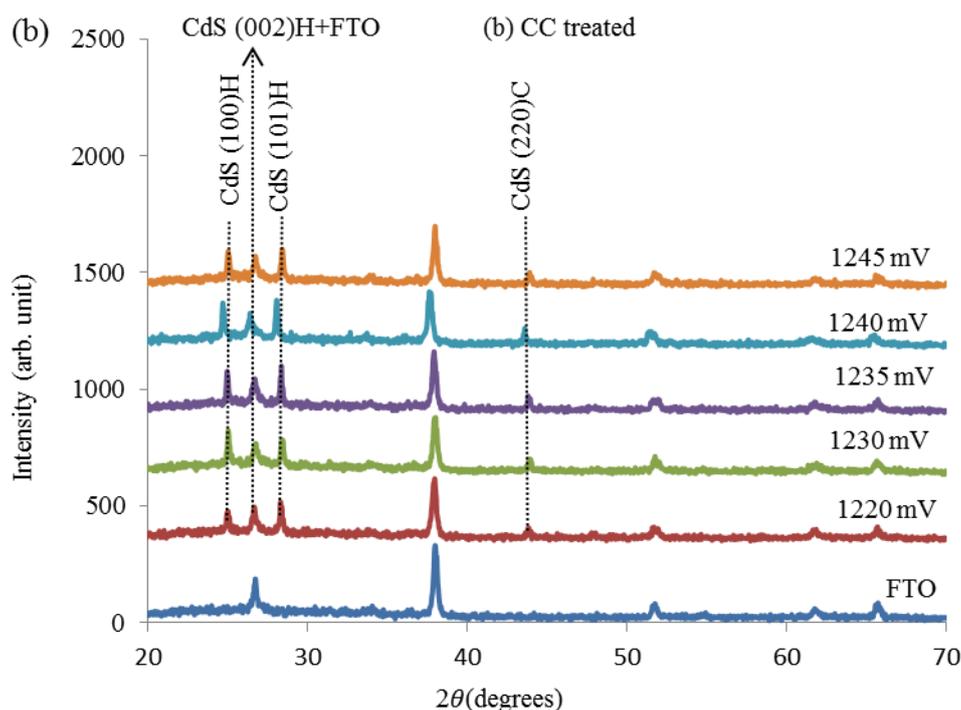


Figure 2: X-ray diffraction pattern for (a) as-deposited and (b) CdCl₂ treated layers at 400°C for 20 minutes in air.

Based on the peak intensities in these (as-deposited) films, the preferred orientation is along the (100) hexagonal phase of CdS at $2\theta \approx 24.9^\circ$ grown at voltage of 1235 mV. This peak shows the highest intensity and was therefore considered the best CdS growth voltage for the as-deposited films. Figure 2(b) shows the XRD patterns of the films after annealing the samples in the presence of CdCl₂ at 400°C for 20 minutes in air. After CdCl₂ annealing, the previously weak and poorly crystalline peaks of (100) and (101) corresponding to hexagonal CdS has shown significant improvement in their peak intensities while reverse is the case for the previously intense peak corresponding to (002) H+FTO peak. The increase in intensities of the two peaks indicates recrystallisation due to the incorporation of the CdCl₂ fluxing agent during annealing of the films. The increase in the intensities of the hexagonal (100) H and (101) H phases of CdS after CdCl₂ treatment was also reported by Fathy *et al.* (Fathy *et al.*, 2014).

The increase in the peak intensities of the CdS peaks such that they dominate the (002)H peak after the CdCl₂ annealing is very interesting to see. Observed also is the disappearance of the (044) orthorhombic peak related to S at $2\theta \approx 32.1^\circ$ in these films. Fauzi also observed the disappearance of the S peak after CdCl₂ treatment (Fauzi, 2015). The highest crystallinity in these samples has shifted from the 1235 mV in the as-deposited films to sample grown at 1240 mV after the CC treatment. Therefore, the preferred orientation in the CdCl₂ annealed films is along the (100) hexagonal phase of CdS at $2\theta \approx 24.9^\circ$ for sample grown at voltage of 1240 mV. Though, most of the samples show comparable peak intensities for both the as-deposited and CdCl₂ treated states.

Figure 3 shows the plot of the XRD peak intensities vs. growth voltage for the (a) as-deposited and (b) CdCl₂ annealed layers of CdS. It is clearly seen that the intensity of (100)H peak in the as-deposited samples settled at ~ 100 counts for all samples. After annealing in the presence of CdCl₂, the samples showed an increase in their peak intensities indicating improved crystallinity in the layers. The highest peak intensity shifted from the growth

voltage of 1235 in the as-deposited to 1240 after the CdCl_2 treatment. This shows that CdS layers with better crystallinity can be grown over a wide voltage range.

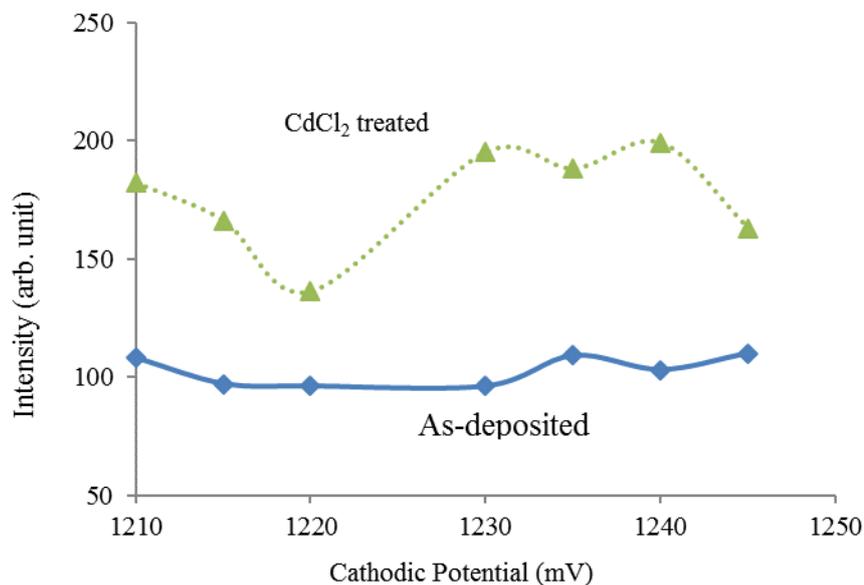


Figure 3: XRD peak intensity vs. growth voltage for as-deposited and CdCl_2 treated thin films of CdS.

3.3 Optical studies

Figure 4 shows the plots of absorption coefficient, α versus photon energy ($h\nu$) for the (a) as-deposited and (b) CdCl_2 treated CdS layers grown at their respective optimised growth voltages. All the layers were grown on glass/FTO substrates for one hour. After growth, the as-deposited sample at each growth voltage was cut into two. The first set of samples was left as-deposited and the other set was annealed in the presence of CdCl_2 at 400°C for 20 minutes in air. The energy bandgap (E_g) in each case were estimated by extrapolating the straight line portion of the absorption coefficient edge to photon energy (at $\alpha=0$) (Tauc, 1966).

The bandgaps were obtained for as-deposited samples grown in the range (1210-1245) mV with the bandgaps scattered in the range (2.37-2.45) eV. However, only the optimised growth voltages of 1235 mV and 1240 mV for as-deposited (Fig.4(a)) and CdCl_2 treated (Fig.4(b)) were selected and plotted in Figure 4 for simplicity. Their E_g bandgaps were found to be 2.40 and 2.42 eV for the as-deposited and the CdCl_2 treated respectively.

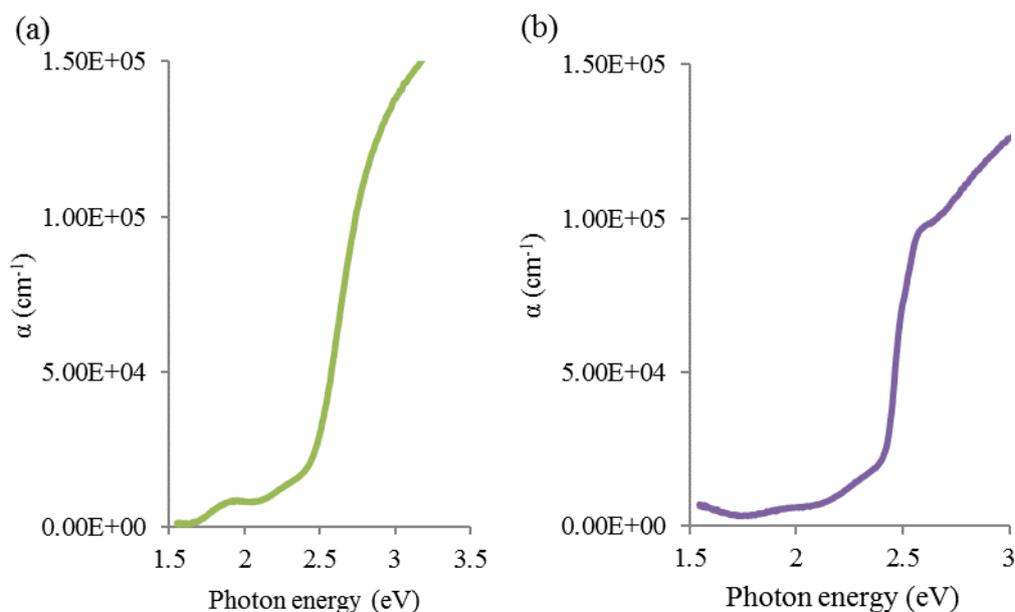


Figure 4: plots of α vs $h\nu$ (a) as-deposited and (b) CdCl_2 treated CdS layers grown at their respective optimised growth voltages.

After annealing in the presence of CdCl_2 (Figure 4(b)), very interesting results in all the layers were observed irrespective of the growth voltage of the films. The estimated bandgaps for all layers show 2.42 eV equal to the bulk energy bandgap of CdS ($E_g=2.42$ eV) as shown in Figure 5. The decrease in the bandgaps of the as-deposited layers to lower photon energy corresponding to energy bandgap of 2.42 eV after CdCl_2 annealing is interesting to see. This indicates the importance of annealing thin films in the presence of CdCl_2 in order to activate their optoelectronic photovoltaics for high performance solar cell devices. The advantages of the CdCl_2 treatment include grain growth, recrystallisation and defect passivation (Dharmadasa, 2014). During CdCl_2 heat treatment, the small grains in the as-deposited layer will coalesce to form bigger grains which reduce the concentration of grain boundaries in the films. As a result, most of the grain boundaries are consumed thereby reducing light transmittance through these gaps and the measured bandgaps will give values close to the bulk bandgap of the CdS material.

This experiment has also shown that CdS thin films can be grown over a wide voltage range without compromising its essential optoelectronic properties suitable for application as a window material in the fabrication of relevant device structures.

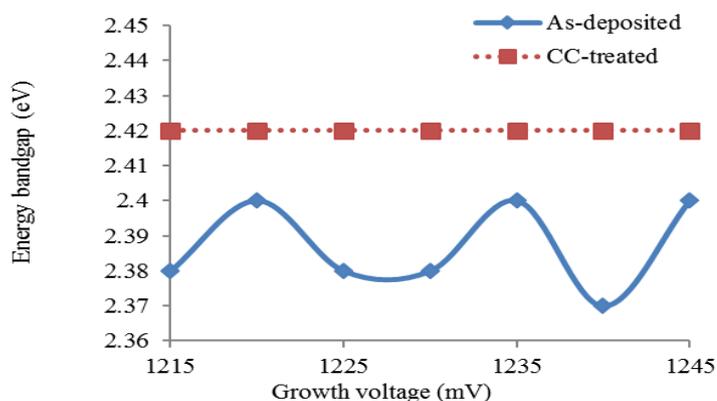


Figure 5: Energy bandgaps for as-deposited and CdCl_2 treated CdS layers grown on glass/FTO substrates at different growth voltage range (1215-1245) mV for one hour.

Further to the absorption coefficient measurement analysis, the transmittance of the layers was estimated in the wavelength range (530-700) nm. Figure 6(a) shows that the typical transmittance of the as-deposited samples measured within the wavelength range show transmittance in the range (70-95)%.

For the annealed samples in the presence of CdCl_2 (Figure 6(b)), the typical transmittance of the films is seen to slightly improve in the range (60-96)% with smooth and sharper absorption edges when compared with the as-deposited layers. These plots show only the optimised voltages in the as-deposited and the CdCl_2 treated layers.

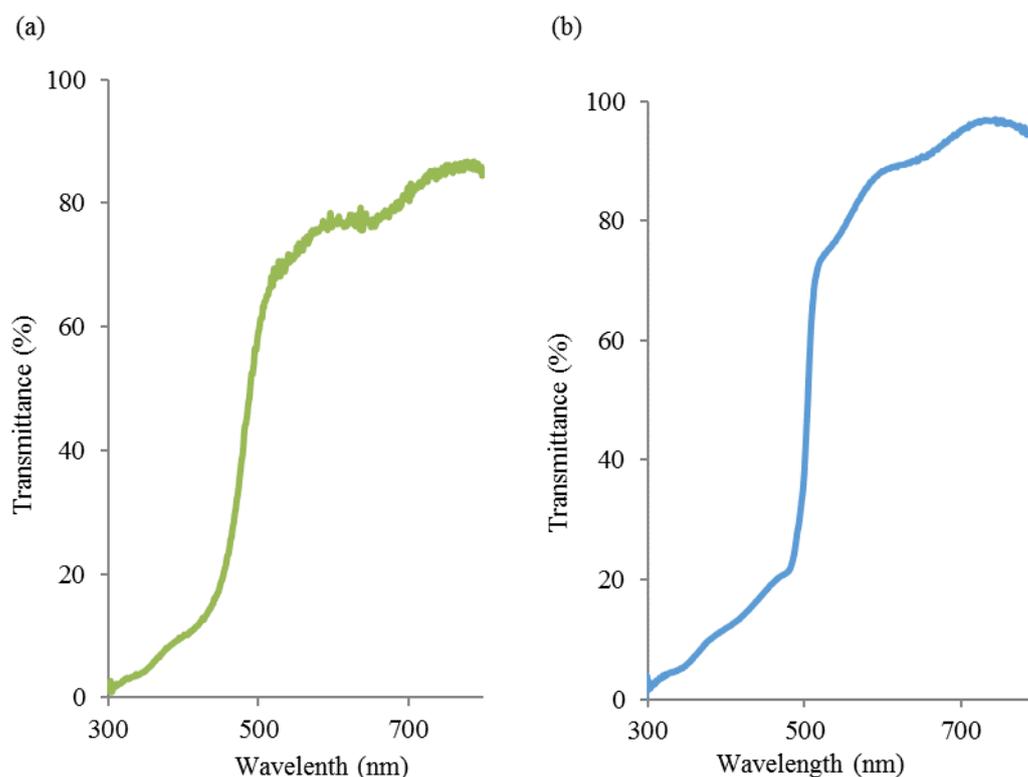


Figure 6: Transmittance spectra for (a) as-deposited and (b) CdCl_2 treated CdS layers grown for one hour at the optimised growth voltages (annealing was carried out at 400°C for 20 minutes in air).

3.4 Scanning electron microscopy

To study the effect of CdCl_2 surface treatment of the CdS layers, a layer was grown on glass/FTO substrate for one hour at the optimised voltage of 1240 mV. Thereafter, the layer was divided into two samples. One piece was left as-deposited and the other annealed in the presence of CdCl_2 at 400°C for 20 minutes in air for comparison.

Figure 7 shows the SEM images of (a) as-deposited and (b) CdCl_2 treated CdS layer. It is seen that the as-deposited sample is covered with agglomerations of small grains showing good surface morphology with virtually no exposure of pinholes and fairly smooth morphology. After annealing in the presence of CdCl_2 (Figure 7(b)), it is observed that the films show a very dense and compact morphology.

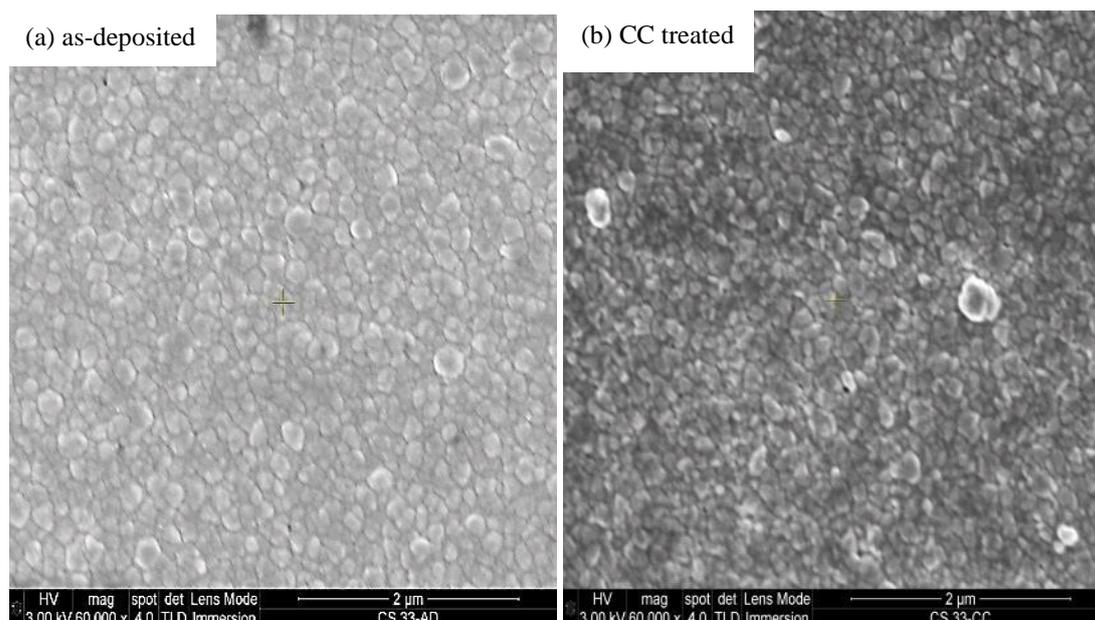


Figure 7: SEM images of (a) as-deposited and (b) CdCl₂ treated CdS layers annealed at 400°C for 20 minutes in air.

3.5 Photoelectrochemical cell measurements

Photoelectrochemical (PEC) cell measurements of electrodeposited CdS grown at different growth voltages were carried out on both as-deposited and CdCl₂ treated samples to confirm the electrical conductivity type of the layers and to also study how the CdCl₂ treatment affected the electrical properties of these layers. All samples were grown for one hour using a voltage range (1210-1245) mV. The as-deposited CdS layer grown at each voltage was divided into 2. The first set of eight samples was left as-deposited while the other set of eight samples was annealed in the presence of CdCl₂ at 400°C for 20 minutes in air. All samples in both as-deposited and CdCl₂ treated condition showed n-type behaviour as expected. The magnitude of the PEC signal is seen to increase in the CdCl₂ treated layers which could be due to improvement in both structural and electrical properties of the layers. The measured PEC signal for as-deposited and CdCl₂ treated CdS layers grown at different growth voltages were plotted in Figure 8.

All samples show n-type electrical conductivity in both as-deposited and CdCl₂ treated conditions as expected. The naturally n-type electrical conduction type of CdS is attributed to S vacancies and Cd interstitials which are intrinsic donor defects in CdS semiconductor material (Wu *et al.*, 2010).

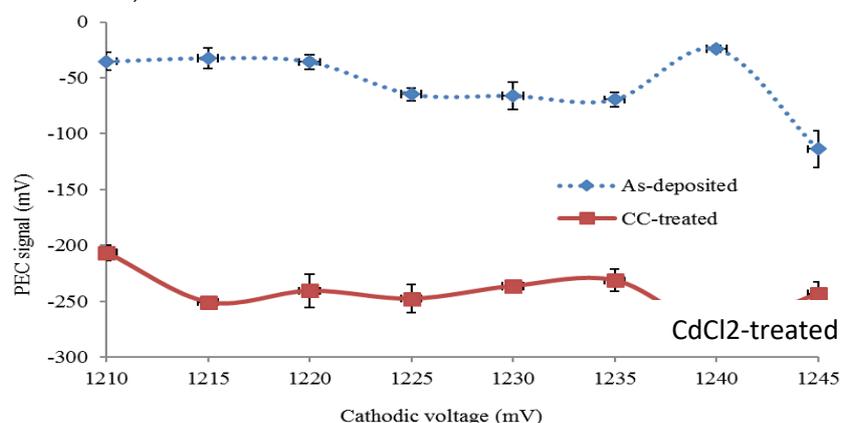


Figure 8: PEC signal measurements as a function of cathodic voltages for as-deposited and CdCl₂ treated CdS layers grown at different growth voltages.

4. Conclusions

CdS thin films were successfully grown using electroplating technique from simple 2-electrode system in a cathodic mode. The growth temperature and pH of the deposition electrolyte were $\sim 85^{\circ}\text{C}$ and 2.50 ± 0.02 respectively. The suitable deposition voltage was established after employing relevant characterisation techniques and the best growth voltage range was found to be between 1220 and 1250 mV. Structural properties using XRD show that CdS thin films were polycrystalline in nature in both the as-deposited and CdCl₂ treated condition. The as-deposited films, consist of sulphur rich layers with the elemental sulphur peak observed in layers grown at deposition voltage of 1220 mV. The XRD spectra show the presence of hexagonal and cubic crystal phases in both as-deposited and CdCl₂ annealed layers. After annealing at 400°C for 20 minutes in the presence of CdCl₂, the peak related to sulphur disappeared. The annealing process is usually carried out to improve the optoelectronic properties of the semiconducting layers. In this experiment, all bandgaps were observed to shift to the bulk bandgap of CdS (2.42 eV).

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