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An investigation into the effect of rate of stirring of bath electrolyte on the properties of electrodeposited CdTe thin film semiconductors

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Abstract

Electrodeposition (ED) has been recognized as a low cost and scalable technique available for fabrication of CdS/CdTe solar cells. Photovoltaic activity of these electrodeposited semiconductor materials drastically depends on the ED growth parameters namely; electrodeposition potential, concentrations and ratios of concentrations of precursors used to prepare the bath electrolyte, pH of the electrolyte, deposition temperature and rate of stirring of the electrolyte. In order to grow thin films with good photovoltaic properties, it is essential to maintain these variables at their optimum ranges of values during electrodepositions. Hence, this study was conducted to investigate the dependence of the properties of electrodeposited CdTe thin film material on the rate of stirring of the bath electrolyte. The CdTe material was grown on glass/FTO (2×3 cm^2) and glass/FTO/CdS (2×3 cm^2) surfaces in bath electrolytes containing 1.0 mol/L $CdSO_4$ and 1.0 mmol/L TeO_2 solutions at different rates of stirring within the range of 0-350 rpm while keeping the values of pH of the electrolyte, deposition temperature and cathodic deposition potential with respect to the saturated calomel electrode at 2.3, 65 °C and 650 mV respectively. After the heat treatment at 400 °C in air atmosphere, the deposited samples with a good visual appearance were selected and evaluated based on their morphological, elemental, structural, optical and electrical properties in order to identify the optimum range of rate of stirring for electrodeposition of CdTe thin film semiconductors. Results revealed that, rates of stirring in the range of 60-85 rpm in a 100 mL volume of electrolyte containing the substrate and the counter electrodes in the center of the bath with a separation of 2.0 cm between them can electrodeposit CdTe layers exhibiting required levels of morphological, structural, optical and electrical properties on both glass/FTO and glass/FTO/CdS surfaces.

Keywords: Electrodeposition; Cadmium telluride; Stirring rate; Solar energy materials

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1. Introduction

At present, the photovoltaic industry is stepping forward rapidly due to the emerging energy crisis all over the world. Amid the photovoltaic (PV) devices at intense investigations, the CdS/CdTe based solar cells have an imperious position owing to their high power conversion efficiencies [1-4]. In these devices, the CdTe material plays the most important role as the absorbing material of the device [1] having the near ideal energy band gap in the range of 1.45-1.50 eV [5-7] for a single band gap p-n junction. There are many technologies available for manufacturing of PV devices and among them the technique of electrodeposition has gained a considerable attention among the PV community due to its low cost of production, scalability and ease of adaptability for large scale manufacturing of CdS/CdTe solar cells [8-9]. To produce good quality CdTe materials using the technique of electrodeposition it is essential to maintain the ED growth parameters namely; electrodeposition potential, concentrations and ratios of concentrations of precursors used to prepare the bath electrolyte, pH of the electrolyte, deposition temperature and rate of stirring of the electrolyte at their optimum values [10-11]. In this study, an attempt was made to investigate the effect of rate of stirring of the electrolyte on the properties of CdTe material grown on glass/FTO and glass/FTO/CdS surfaces due to the lack of investigations carried out on this aspect in literature [2]. Further, this study is in concurrent with our previous work on "Effect of stirring rate of electrolyte on properties of electrodeposited CdS layers" in 2016 [3] and the principles related to transport of ions towards working electrode during electrodeposition is used for interpreting the results and understanding the effects of solution stirring on the properties of electrodeposited CdTe layers.

2. Experimental method

CdTe layers were potentiostatically electrodeposited on fluorine doped tin oxide (FTO) glass (2×3 cm^2) and glass/FTO/CdS (2×3 cm^2) substrate working electrode surfaces in a three electrode electrolytic cell consisted of a saturated calomel reference electrode (DirectIon, UK) and a high purity graphite sheet counter electrode (99 %, Sigma Aldrich, USA). Potential controls over the electrochemical cell were achieved using a potentiostat/galvanostat (Metrohm Autolab PGSTAT128N, Netherlands). The bath electrolyte containing 1.0 mol/L $CdSO_4$ (Sigma Aldrich, 99 %, USA) and 1.0 mmol/L TeO_2 (Sigma Aldrich, 99.995 %, USA) placed in a 100 mL beaker (diameter: 5 cm and height: 6 cm) having the working and counter electrodes at the center of the solution parallel to each other with a separation of 2.0 cm was used to deposit CdTe layers on glass/FTO and glass/FTO/CdS surfaces separately. Six different rates of stirring (0, 60, 85, 125, 240 and 350 rpm) generated through uniform revolutions of a 2.0 cm long magnetic bar of a magnetic stirrer (Thermo Scientific, USA) were used for these electrodepositions. During each and every electrodeposition, the cathodic deposition potential, pH of the electrolyte, deposition temperature and deposition time were maintained constant at 650 mV, 2.3, 65 °C and 3 hrs. respectively. As electrodepositions were carried out in electrolytes at temperatures above 25 °C in an open cell, electrolytic bath continued to be evaporated. In order to maintain the solution volume at 100 mL, required volumes of ~1.0 mmol/L TeO_2 solution having the same pH and the same temperature were added into the bath at regular time intervals to bring the solution volume to 100 mL. ~1.0 mmol/L TeO_2 solutions were used in place of water for this purpose as the concentration of TeO_2 used in the electrolyte was very low owing to its very low solubility in water and the concentration of it diminishes very rapidly with the progress of the deposition.

At the end of the deposition process, all the samples were rinsed in de-ionized water and dried under a high purity nitrogen gas flow. Subsequently, the films that exhibited well adherent and uniform appearances were annealed at 400 ºC for 10 min. in air atmosphere. In order to determine the effect of rate of stirring of the electrolyte on the quality of CdTe layers deposited on glass/FTO and glass/FTO/CdS surfaces, their morphological, elemental, structural, optical and electrical properties were evaluated. The surface morphology and elemental properties were studied using scanning electron microscopy (EVO LS15, Germany) and energy dispersive X-ray spectroscopy (EVO LS15, Germany) respectively. The structural properties were studied using the technique of X-ray diffraction (Siemens D 5000, Germany) while optical properties were studied using the UV-Visible absorption spectroscopy (Cintra 202 UV-Vis, Australia). Further, the electrical properties namely; electrical conductivity type, V_{oc} and J_{sc} associated with the semiconductor/electrolyte interface were studied using a three electrode photo-electrochemical cell containing of 0.10 mol/L $Na_2S_2O_3$ (99 %, Sigma Aldrich, USA) electrolyte that consisting of a CdTe/electrolyte junction, graphite counter electrode and the saturated calomel reference electrode. The experiments were repeated three times in order to confirm the results.

3. Results and discussion

3.1 Visual appearance of CdTe layers electrodeposited on glass/FTO and glass/FTO/CdS surfaces at different rates of stirring

Fig. 1 shows the visual appearance of heat treated CdTe samples grown on glass/FTO and glass/FTO/CdS surfaces under different rates of stirring.

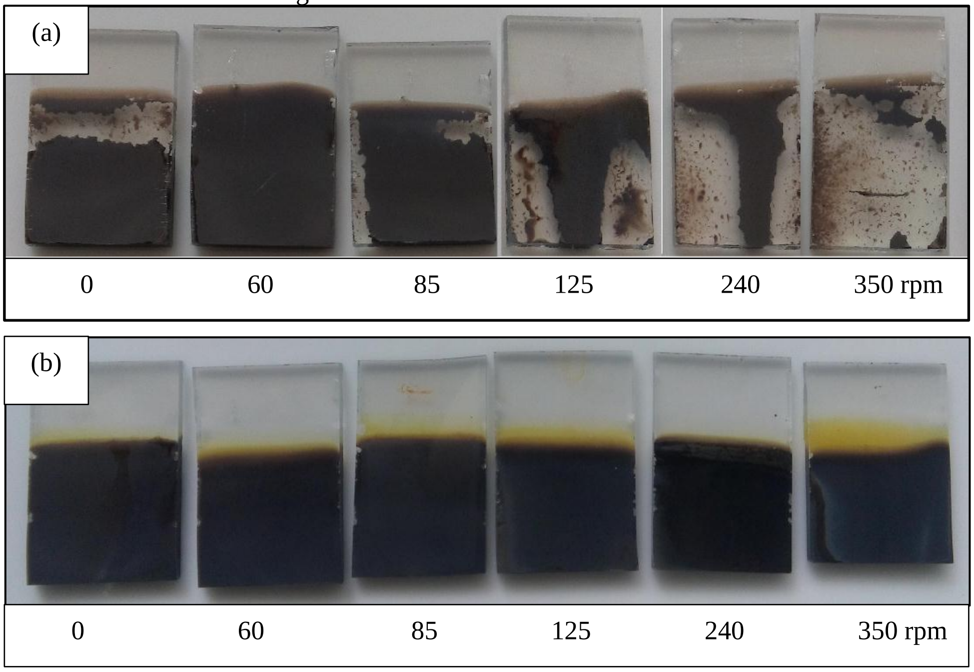

Fig. 1: Visual appearance of heat treated CdTe samples electrodeposited at stirring rates of 0, 60, 85, 125, 240 and 350 rpm on (a) glass/FTO and (b) glass/FTO/CdS surfaces.

As Fig.1a indicates, the CdTe layers deposited are not well-adhered at several rates of stirring indicating that the rate of stirring has a pronounced effect on the strength of sticking of CdTe layers deposited onto the surface of the substrate. When stirring rates ranging from 0 to 350 rpm are considered, the latter rates produced loosely bound CdTe layers. As discussed in our previous work on "Effect of stirring rate of electrolyte on properties of electrodeposited CdS layers" in 2016 [3], this could be due to the high convection rates produced upon the high rates of stirring. As a result, the pressure variations formed can produce collisions between electro-active species and the glass/FTO surface peeling the Cd, Te or CdTe species out of the deposited surface [11-12].

Further, the CdTe layers deposited at non-stirring conditions also disclose slight peeling off effect in the upper part of the CdTe layer. This might have occurred due to the low rates of supply of ions and poor rates of ion exchanges at the electrode/electrolyte interface. Following reactions are expected to occur in the bath electrolyte of CdTe. Accordingly, $\mathrm{Cd^{2+}(aq)}$ and $\mathrm{HTeO_2^+(aq)}$ ions are reduced to $\mathrm{Cd(s)}$ and $\mathrm{Te(s)}$ forms respectively to produce $\mathrm{CdTe(s)}$ at the surface of working electrode.

$$\mathrm{Cd^{2+}(aq) + 2e \rightarrow Cd(s);\ E_o = -642\ mV\ vs.\ SCE} \qquad (1)$$

$$\mathrm{HTeO_2^+(aq) + 3H^+(aq) + 4e \rightarrow Te(s) + 2H_2O(l);\ E_o = -551\ mV\ vs.\ SCE} \qquad (2)$$

$$\mathrm{Cd^{2+}(aq) + HTeO_2^+(aq) + 3H^+(aq) + 6e \rightarrow CdTe(s) + 2H_2O(l)} \qquad (3)$$

As explained in our previous work on "Effect of stirring rate of electrolyte on properties of electrodeposited CdS layers" in 2016 [3], ion transportation towards the electrode at non-stirring condition occurs mainly due to the processes of migration and diffusion. Convection under no stirring condition that can occur due to small thermal gradients possible to present in solution can be neglected in comparison to the magnitudes of migration and diffusion. Hence, Cd^{2+} and $HTeO_2^+$ ion concentrations near and around the electrode continue to decrease as a function of time while building a concentration gradient in the nearby solution layer and it may produce non-uniform and non-stoichiometric CdTe material having poor adhesive qualities [3]. Within the entire range of stirring rate, the samples deposited on glass/FTO surfaces at 60 and 85 rpm show comparably good physical appearance. However, the sample grown at 85 rpm has a slightly disordered CdTe layer.

As Fig. 1b reveals, the visual appearance of the CdTe layers grown on glass/FTO/CdS surfaces slightly varies with the variations in the rate of stirring of the electrolytic bath analogous to that of CdTe samples grown on glass/FTO surfaces. Further, it is clear that, glass/FTO/CdS surface is a better substrate than that of glass/FTO for electrodepositing of CdTe thin layers. The sample grown at non-stirring state has a non-uniform CdTe layer. As explained previously, this may due to low rates of ion supply and poor rates of ion exchanges at the electrode/electrolyte interface and hence the deficiency of Cd^{2+} or $HTeO_2^+$ ions near and around the working electrode [3]. Hence the resulting non-stoichiometric CdTe materials may exhibit non-uniform surface topology. The samples grown at the stirring rates of 60 and 85 rpm exhibit good physical appearances. This may be due to the supplying of Cd^{2+} and $HTeO_2^+$ ions to the substrate surface at a rate sufficient enough for formation of stoichiometric layers of CdTe over this range of stirring rates.

The CdTe layers grown on glass/FTO/CdS surface at the stirring rates in the range of 125-350 rpm were not uniform. A slight peeling off nature was observed over the edges of the samples grown at the rates of 125 and 350 rpm. Further, the sample grown at 240 rpm has slightly different colour (black) than that of others [4]. As discussed before, high impact collisions resulting upon the higher stirring rates may be the reason behind [11-12]. Further, the colour variation observed in the sample grown at 240 rpm could be due the formation of a slightly Te rich layer at higher ion transportation rates.

3.2 Surface morphology of CdTe layers electrodeposited on glass/FTO and glass/FTO/CdS surfaces at different rates of stirring

Since there were significant differences in the visual appearances of the CdTe layers grown on glass/FTO and glass/FTO/CdS surfaces at different rates of stirring, surface morphology of these samples were studied. Fig. 2 shows the SEM images of the heat treated CdTe layers grown on glass/FTO at different rates of stirring.

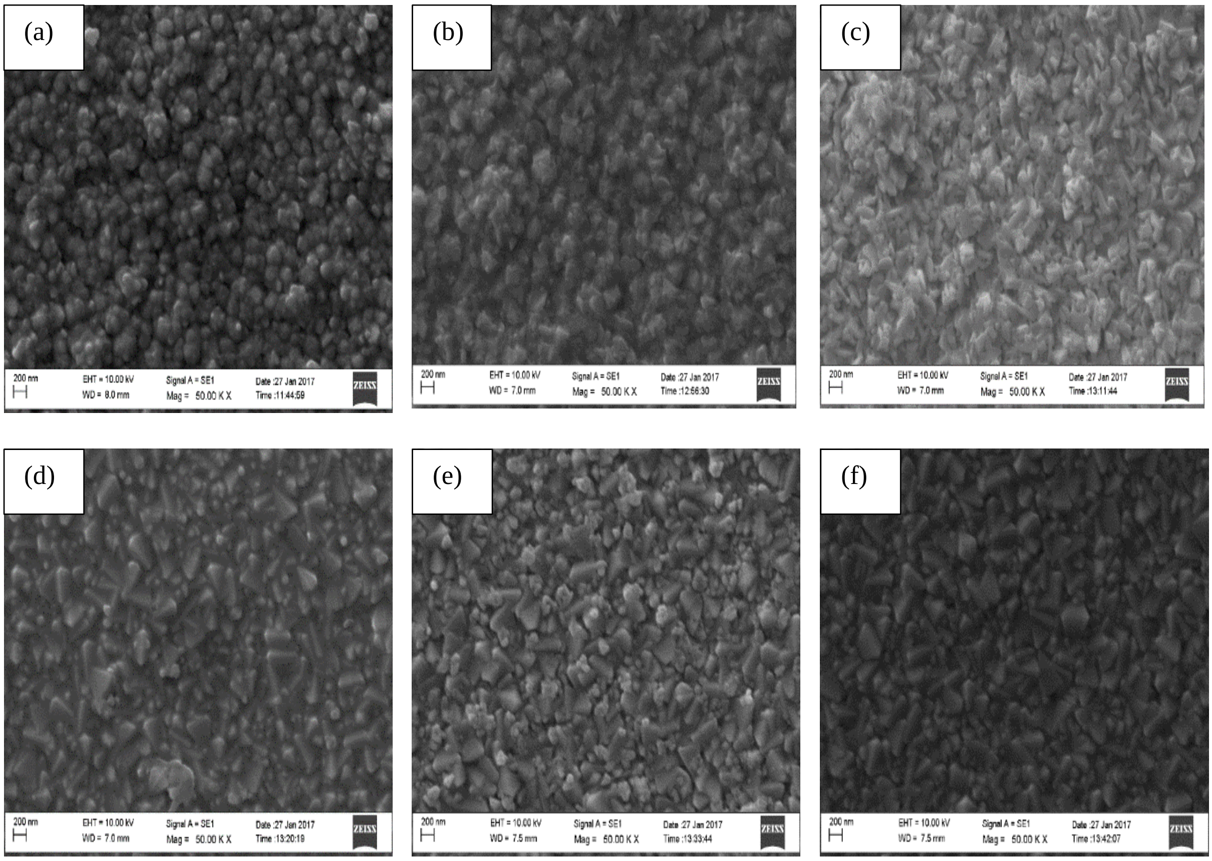

Fig. 2: 50, 000 times magnified SEM images of CdTe layers electrodeposited on glass/FTO surfaces at (a) non-stirring state, (b) 60 rpm, (c) 85 rpm, (d) 125 rpm, (e) 240 rpm and (f) 350 rpm, after the heat treatment.

The SEM results shown in Fig. 2 are totally in line with the visual appearance of CdTe samples shown in Fig. 1a. The surface topology of the CdTe samples grown at non-stirring state, at stirring rates of 60 and 85 rpm are presented in Fig. 2a, b and c respectively reveal the presence of a nano scale grain distribution with an average particle size of ~80 nm. As noted in previous section 3.1, these CdTe layers appeared to be agglomerated clusters that are formed by the assortment of nano size grains [2, 13-16]. Among these three samples, the CdTe layers grown at non-stirring state and 60 rpm have ordered grain distribution with a regular grain shape. The CdTe layer grown at 85 rpm has an irregular shaped grain spreading with a considerable upsurge in surface roughness. The surface morphology of the CdTe layers grown at 125, 240 and 350 rpm are presented in Fig. 2d, e and f respectively. As elucidated by the figures, a poor surface coverage can be noticed in them and the top surface of the glass/FTO substrate is clearly visible as observed from Fig. 1a.

At higher stirring rates, non-uniform ion transportations to substrate surface can result in poor adherence of the material onto the substrate. This could result highly porous layers and short-circuited current paths within the material leading to poor electronic properties. Also, the reduced optical properties can be expected due to these disordered layers ultimately resulting poor device parameters.

Fig. 3 shows the SEM images of the heat treated CdTe layers grown on glass/FTO/CdS at different rates of stirring.

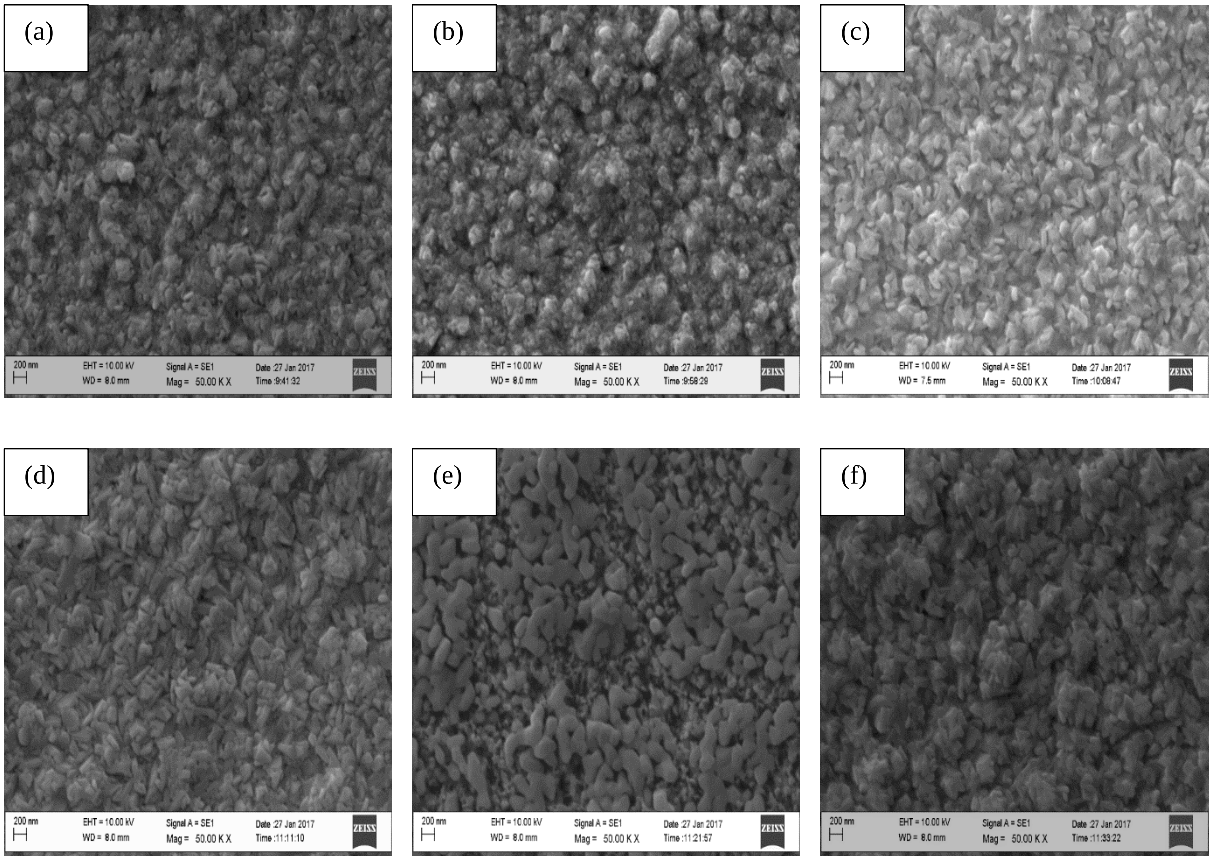

Fig. 3: 50, 000 times magnified SEM images of CdTe layers deposited on glass/FTO/CdS surfaces at (a) non-stirring state, (b) 60 rpm, (c) 85 rpm, (d) 125 rpm, (e) 240 rpm and (f) 350 rpm, after the heat treatment.

The surface topology of the CdTe samples grown on glass/FTO/CdS surfaces at non-stirring state, at stirring rates of 60 and 85 rpm presented in Fig. 3a, b and c respectively reveal the presence of an agglomerated nano scale grain distribution and it is similar to the CdTe samples grown on glass/FTO surface. However, the shape of the grains of the sample grown at 85 rpm slightly differs from the samples grown at non-stirring state and 60 rpm. The surface morphology of the CdTe layers grown at 125, 240 and 350 rpm are presented in Fig. 3d, e and f respectively. As illustrated by the figures, non-uniform surface coverage can be noticed. The sample grown at 240 rpm has a totally different surface morphology and, the visual appearance observed in section 3.1 could be due to these nano-scale changes in the grain distribution. Further, the surface roughness of the sample grown at 350 rpm is higher than that in the other samples and it could be a result of increased rates of ion supply upon the increased rate of stirring.

Vigorous ion transportations at higher stirring rates can disturb the equilibrium of the ions on the substrate and hence result in non-uniform CdTe layers. This can result in highly porous layers and short-circuited current paths within the material leading to poor electronic properties. Also, structural and optical properties could reduce due to the damaged layers ultimately leading to poor performances in devices.

3.3 Elemental properties of CdTe layers electrodeposited on glass/FTO and glass/FTO/CdS surfaces at different rates of stirring

The elemental composition of CdTe layers grown on glass/FTO and glass/FTO/CdS surfaces were studied using the technique of energy dispersive X-ray (EDX). The summary of the ratios of Cd/Te is presented in Table 1. Accordingly, the CdTe layers grown at non-stirring state on both glass/FTO and glass/FTO/CdS exhibited slightly higher amount of Te than the samples grown at other rates of stirring and the latter exhibited very close ratios of Cd/Te. Further, the vigorous supply of ions towards the working electrode upon the higher rates of stirring may diminish the formation of uniform and well adhered layers of CdTe on both glass/FTO and glass/FTO/CdS substrates.

Table 1: Cd/Te ratio of the CdTe layers deposited on glass/FTO and glass/FTO/CdS surfaces at different rates of stirring after the heat treatment.

Stirring rate/rpm	glass/FTO/CdTe	glass/FTO/CdS/CdTe
	Cd/Te ratio	Cd/Te ratio
0	1.04	1.05
60	1.08	1.08
85	1.06	1.08
125	1.07	1.08
240	1.07	1.07
350	1.07	1.07

3.4 Structural properties of CdTe layers electrodeposited on glass/FTO and glass/FTO/CdS surfaces at different rates of stirring

Fig. 4 shows the X-ray diffractrograms of the CdTe layers deposited on glass/FTO and glass/FTO/CdS at the stirring rates of 0, 85 and 240 rpm.

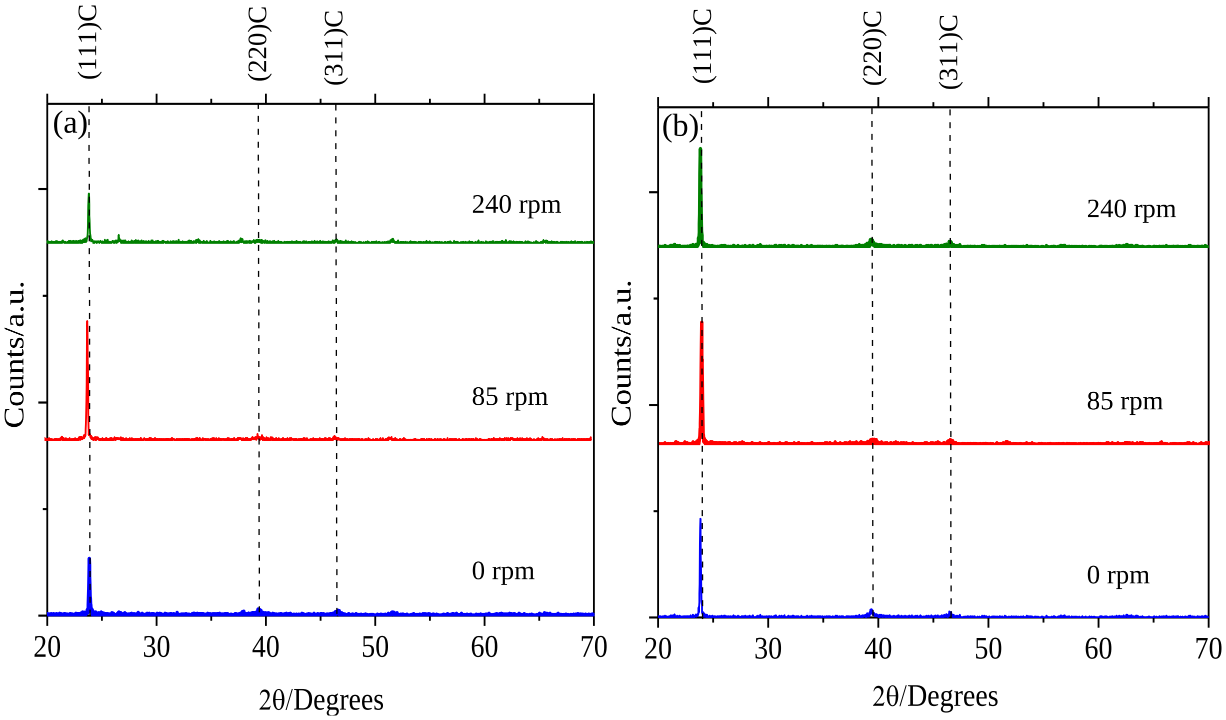

Fig. 4: X-ray diffractograms of CdTe layers deposited on (a) glass/FTO and (b) glass/FTO/CdS surfaces at 0, 85 and 240 rpm after the heat treatment.

In Fig. 4, the diffraction peaks can be observed along the planes of (111), (220) and (311) at 2θ of 24°, 39° and 46° respectively [2-3] evidencing the cubic crystalline structure of all the samples that have been analyzed. When CdTe layers were grown, the peak intensities of the CdTe layers deposited on glass/FTO/CdS were higher than the CdTe layers deposited on glass/FTO. The surface structure of glass/FTO/CdS may favour the deposition of CdTe material on it and due to well adherent nature of the CdTe layers on glass/FTO/CdS, it may enhance further deposition of CdTe layers showing intensified peaks in respective diffractograms. Further, CdTe layers deposited on both glass/FTO and glass/FTO/CdS at 85 rpm exhibited the highest peak intensity corresponding to the crystallographic plane of (111) and it reflects the stability of the material observed under section 3.1 and 3.2.

3.5 Optical absorption spectroscopy

Tauc plots obtained for CdTe layers grown on glass/FTO and glass/FTO/CdS surfaces at different rates of stirring are presented in Fig. 5.

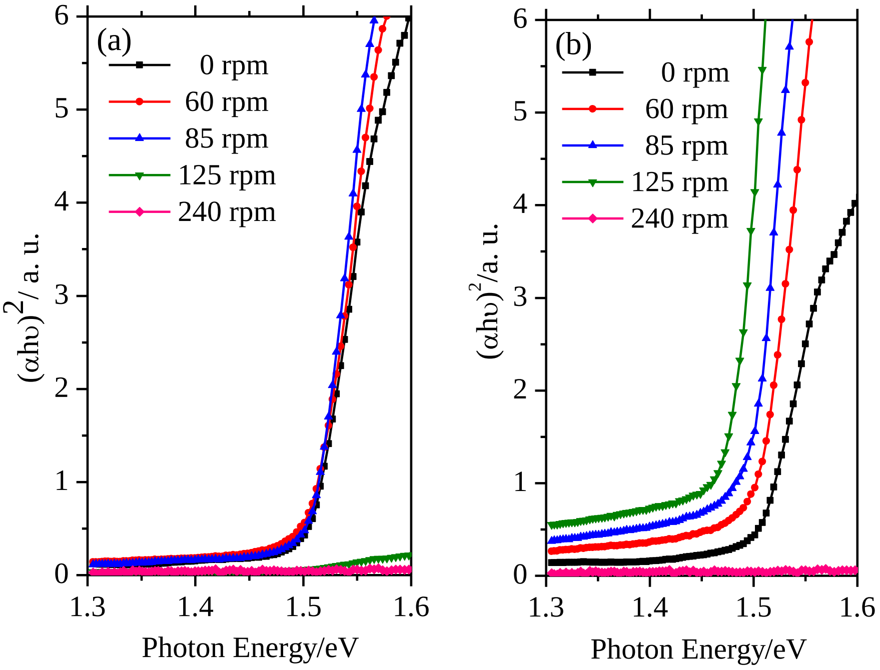

Fig.5: Tauc plots obtained for CdTe layers grown on (a) glass/FTO and (b) glass/FTO/CdS surfaces at different rates of stirring within the range of 0-350 rpm.

As can be seen from Fig. 5a, the CdTe layers that grown on glass/FTO surfaces at 0, 60 and 85 rpm exhibit good optical absorption properties than that of the samples grown at 125, 240 and 350 rpm which are in line with the observed visual appearances and detected surface morphologies. As can be estimated by the Tauc plots shown in Fig. 5a, the band gap energies of the CdTe layers electrodeposited at 0, 60 and 85 rpm were found to be in the range of 1.49-1.50 eV which is within the expected theoretical E_g range [1,5,17-18]. However, the samples deposited at 125 and 240 rpm exhibited considerably low absorbances which made them disqualified as an absorber material of the CdS/CdTe solar cell device. As the CdTe layers grown at 350 rpm were peeled off from the glass/FTO substrate, a significant deviation from absorbances of the other samples was noted and it did not show the corresponding Tauc plot within the absorbance range considered and a result similar to that was observed for the CdTe sample grown on glass/FTO/CdS. The non-uniform and highly rough nature of surface with irregular shaped grains observed in the samples may contain non-stoichiometric CdTe or other material formed due to the incorporation of O atoms on CdTe that having different optical absorption properties.

As Fig. 5b reveals, the CdTe layers grown on glass/FTO/CdS surfaces at 0, 60, 85 and 125 rpm exhibit good optical absorption properties than that of the samples grown at 240 and 350 rpm. The estimated band gap energies of the CdTe layers electrodeposited at 0, 60, 85 and 125 rpm were found to be in the range of 1.48-1.50 eV. However, the samples deposited at 240 rpm exhibit a considerably low absorbance and hence, it is not suitable for using as an absorber material of the CdS/CdTe solar cell devices.

3.6 Solid/liquid junction J-V characteristics in a photo-electrochemical cell

In a photo-electrochemical (PEC) cell, the Schottky barrier formation at the interface of CdTe/electrolyte (solid/liquid) takes place and band alignments alter accordingly and hence can be considered as a simple cell [5]. Assuming this nature, J-V characteristics were obtained and all the ED-CdTe samples were identified to be p-type in electrical conductivity. The open circuit voltage (V_{oc}) and short circuit current density (J_{sc}) values obtained for CdTe layers grown on glass/FTO surfaces at different rates of stirring are presented in Table 2.

Table 2: PEC cell V_{oc} and J_{sc} values for CdTe layers grown on glass/FTO surfaces at different rates of stirring; 0, 60, 85, 125, 240 and 350 rpm.

Stirring rate/rpm	Average V_{oc}/mV	Average J_{sc}/μA cm^{-2}
0	140	31
60	240	42
85	203	39
125	183	35
240	170	28
350	105	23

Fig. 6a and b respectively show the variation of V_{oc} and J_{sc} of CdTe samples grown on glass/FTO surfaces at the CdTe/electrolyte interface as a function of stirring rate.

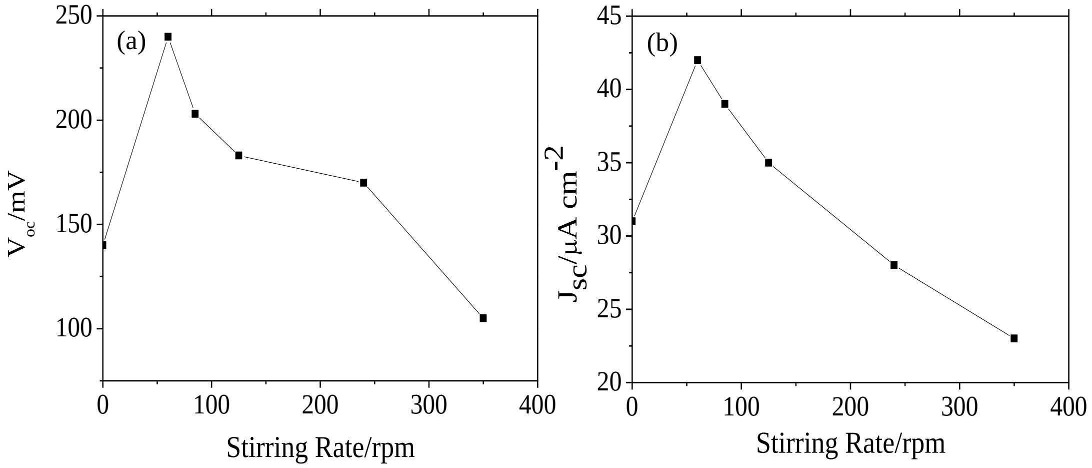

Fig. 6: The variation of (a) V_{oc} and (b) J_{sc} of CdTe samples grown on glass/FTO surfaces at the CdTe/electrolyte interface as a function of stirring rate within the range of 0-350 rpm.

As can be seen from Table 2 and Fig. 6, the sample grown at 60 rpm shows the best electrical properties. The higher stirring rates (125-350 rpm) exhibit lower electrical properties (V_{oc} and J_{sc}). This result highly agrees with the morphological and optical properties discussed above and consequently it reveals that, the rate of ion/mass transportation towards the working electrode is drastically important in producing well-adhered and PV active CdTe layers [3].

Though the sample grown at non-stirred state has a reasonable visual appearance, its electrical properties are poor to that of the sample grown at 125 rpm. Further, the CdTe layers electrodeposited at 85 rpm exhibit considerably high V_{oc} and J_{sc} values which is second only to the sample deposited at 60 rpm. Observed electrical properties are in good agreement with the visual appearances, surface morphologies and the optical properties of the CdTe layers grown at different rates of stirring. These observations indicate that, the layers grown at moderate stirring rates in the range of 60-85 rpm can produce CdTe materials with considerably good electronic qualities.

The open circuit voltage (V_{oc}) and short circuit current density (J_{sc}) values obtained for CdTe layers grown on glass/FTO/CdS at different rates of stirring are presented in Table 3.

Table 3: PEC cell V_{oc} and J_{sc} values for CdTe layers grown on glass/FTO/CdS at different rates of stirring; 0, 60, 85, 125, 240 and 350 rpm.

Stirring rate/rpm	Average V_{oc}/mV	Average J_{sc}/μA cm^{-2}
0	345	285
60	369	350
85	418	360
125	480	340
240	83	0
350	100	250

Fig. 7a and b respectively show the variation of PEC cell V_{oc} and J_{sc} of CdTe samples grown on glass/FTO/CdS surfaces at the CdTe/electrolyte interface as a function of rate of stirring.

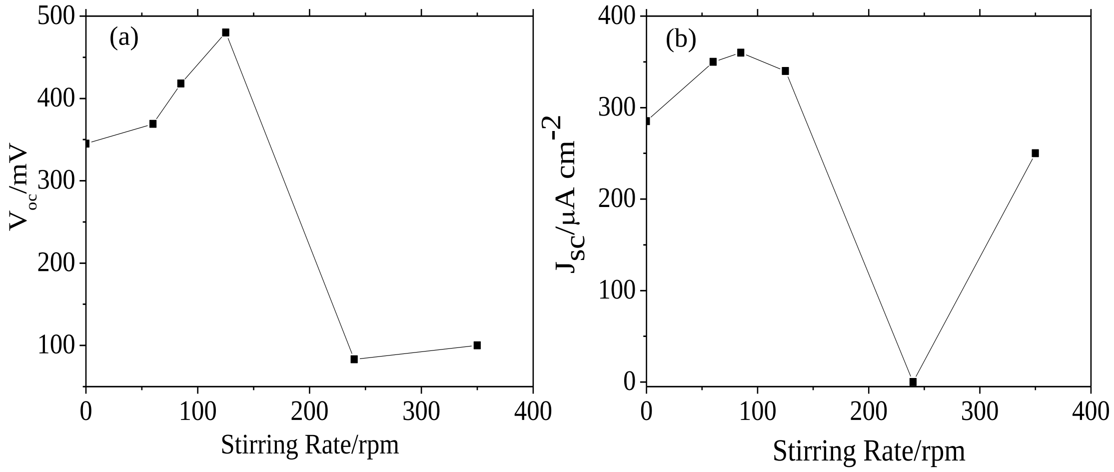

Fig. 7: The variation of (a) V_{oc} and (b) J_{sc} of CdTe samples grown on glass/FTO/CdS surfaces at the CdTe/electrolyte interface as a function of rate of stirring within the range of 0-350 rpm.

As can be seen in Table 3 and Fig. 7, the sample grown at 125 rpm shows the best V_{oc} while the sample grown at 85 rpm exhibiting the best J_{sc}. The higher stirring rates in the range of 240-350 rpm exhibit lower electrical properties (V_{oc} and J_{sc}). These observations highly agrees with the morphological, structural and optical properties discussed above and accordingly it reveals that, the rate of the ion/mass transportation towards the working electrode is significantly important in producing uniform and PV active CdTe layers.

Although the sample grown at non-stirring state has a reasonable surface morphology and optical properties, its electrical properties are lower than that of the samples grown at the stirring rates in the range of 60-125 rpm. The observed visual appearance, surface morphology and the optical properties of all the CdTe layers grown on glass/FTO/CdS surfaces are compatible with the measured electrical properties of the samples. Hence, the layers grown at moderate stirring rates in the range of 60-125 rpm can be used to produce CdTe materials having considerably good electronic qualities.

4. Conclusion

The influence of the rate of stirring on ED-CdTe layers has been successfully studied. The visual appearance, surface morphology, elemental, structural, optical and electrical properties obtained indicate that, the CdTe layers with good optoelectronic properties can be grown on glass/FTO surface from a solution volume of 100 mL at a stirring rate around 60-85 rpm and on glass/FTO/CdS surface it is 60-125 rpm producing orderly flows of electrolyte during stirring. Furthermore, stirring rates higher than 125 rpm are not suitable due to high turbulences and hence CdTe layers produced are low in electrical, optical and structural properties and poor in surface morphology. Conjointly, non-stirring condition is also not suitable as it causes low rates of ion transportation causing low electrical properties. When, both glass/FTO and glass/FTO/CdS surfaces are considered, the rate of stirring in the range of 60-85 rpm is suitable to grow quality CdTe layers having good optoelectronic properties.

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