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# Electrodeposition of CdTe from BmimCl: Influence of substrate and electrolytic bath



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#### ABSTRACT

Potentiostatic electrodeposition of Cadmium Telluride (CdTe) films from the alkaline ionic liquid (IL), butyl methyl imidazolium chloride (BmimCl) at 80 °C has been carried out at an applied potential of  $-1.45\,\mathrm{V}$  vs. platinum (Pt) wire. The influence of the substrate and electrolytic IL bath content on the morphology and composition of electrodeposited CdTe thin films has been presented. The sheet resistivity of the modified FTO substrate increases to  $\sim 17.7\,\Omega/\mathrm{sq}$ . While treatment of the substrate prior deposition changes the individual CdTe morphology, the composition as well as the film compactness has been found to vary with the treatment of the IL based electrolytic bath. SEM micrograph shows inter-connected needle-like and herring bone morphology of electrodeposited CdTe along with agglomerated lumps of Te and flecks of Cd. Interdependency of surface morphology and elemental composition of electrodeposited CdTe films were observed by SEM & EDX analysis. It has been shown that hydroxylated FTO surface facilitates three dimensional (3D) nucleation and growth in the presence of  $O_2$  in the electrolytic bath. 3D nucleation and growth mechanism leads to Cd-rich CdTe films. Electrodeposition that does not proceed through 3D nucleation and growth shows only Te-rich layer. Also, the net charge transfer was found more in Cd-rich CdTe layers as compared with Te-rich CdTe deposits.

#### 1. Introduction

Cadmium Telluride (CdTe) is an II-VI stable compound semiconductor formed from cadmium (Cd) and tellurium (Te) with an optimum direct band gap ( $E_g = 1.45 \, eV$ ) [1,2] and higher absorption coefficient (10  $^{5}\,\mbox{cm}^{-1}$ ) [3]. Hence, it has been mainly used in CdTe photovoltaics [4,5]. Apart from its use as a photovoltaic material, CdTe has also been used in sensors, rectifiers, and detectors [6]. The preparation of CdTe thin films by various non-vacuum processes like electrodeposition [7], spray pyrolysis [8], and chemical bath deposition [9] as well as vacuum techniques like close space sublimation, physical vapour deposition and sputtering [10] has been well investigated by many research groups. However, in the recent past, electrodeposition of CdTe thin films has gained a huge attention on metallic [11,12] as well as conducting glass substrates [13]. The advantages of using non-vacuum electrodeposition include simplicity of the process, economic viability and large scalability [14,15]. From early days, researchers have investigated the electrodeposition of CdTe from acidic aqueous solutions [16–18] in the pH range of 1–3 as well as in alkaline aqueous solutions (pH10-12) [19-21]. Nearly stoichiometric CdTe thin films have thus been prepared and used for solar cell applications. However, owing to the limited solubility of Te species in acidic and alkali aqueous solutions and the inhalation of toxic Cd and Te vapours during electrodeposition process, the need to use non-aqueous [22,23] and ionic liquid medium [24–28] became inevitable. To overcome the drawback of high temperature process in non-aqueous bath, the concept of low/high viscous room temperature ionic liquids (ILs) for electrochemical deposition of CdTe films has gained a lot of interest among the research groups. The major advantages of using "green" IL solvent are: (i) wide electrochemical window, (ii) low vapour pressure, (iii) high chemical and thermal stability, (iv) good ionic mobility [29–31] and good solubility of inorganic salts. In our previous research effort, we have been able to deposit CdTe needle-like structure from BmimCl IL bath at 80 °C by electrochemical technique [32]. However, the effect of substrate surface and presence of O<sub>2</sub> in the ionic liquid on the overall morphology of the deposits has not been explored.

In this paper, we present the influence of substrate and electrolytic bath content on the surface morphology and elemental composition of CdTe films obtained by electrodeposition from an air stable 1-butyl-3-methyl imidazolium chloride (BmimCl) ionic liquid medium at 80 °C.

#### 2. Experimental section

The electrodeposition of CdTe thin film was carried out using a

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three electrode conventional cell setup. Fluorine doped tin oxide (FTO) with an effective area of 0.25 cm<sup>2</sup> was used as the working electrode, platinum (Pt) wire as a quasi-reference electrode and Pt mesh as a counter electrode. FTO was ultrasonically cleaned in trichloroethylene, iso-propanol, acetone and De-ionized water successively whereas Pt wire and mesh were ultrasonically cleaned in HCl and thoroughly rinsed with DI water prior each electrodeposition. The precursors and solvent were purchased from Sigma Aldrich and used as received. The details for the preparation of active electrolytic IL bath has been discussed elsewhere [32]. The sheet resistivity of the FTO substrate used is 7  $\Omega$ /sq. The influence of substrate on the electrodeposits was studied by heat treating FTO substrate over a petri dish (PD) and in DI water bath (HW), at 80 °C for 15 min using a digital PID controlled hot plate. The change in the electrolytic bath content on the deposition process was carried out by purging nitrogen (N2) and adding surfactant cetyltrimethylammonium bromide (CTAB) in active IL electrolyte. The bath temperature was kept at 80 °C for each deposition conditions as IL coexists in liquid form only above 70 °C. Potentiostatic electrodeposition of CdTe was carried out by using a potentiostat/galvanostat (PGSTAT302N, Autolab). The amount of net charge transfer on modified FTO and cyclic voltammogram during electrodeposition of CdTe under various conditions were also studied. The change in the surface morphology and elemental composition of CdTe films obtained at various conditions were carried out by using FE-SEM (Ultra-55, Zeiss) equipped with EDX (Oxford Inca). The pH of active electrolytic IL bath was measured using Auto pH system (PM300, Welltronix).

#### 2.1. Surface pre-treatment & characterization of FTO substrate

The electrochemical deposition of CdTe thin films were carried out under different conditions: pre-treatment of substrate and that of electrolytic IL bath. The treatment of the FTO substrate surface was done in two ways: heat treatment of substrate (i) over a petri dish and (ii) in DI water bath, at 80  $^{\circ}\text{C}$  for 15 min. The effect of pre-treatment on the physical properties of the FTO substrate has been studied by several measurements.

#### 2.1.1. Electrical studies

Fig. 1 presents the sheet resistivity of pristine and pre-treated FTO substrates. The measurement was done using four probe technique, a handmade setup attached to a Dynamic Measurement DC Source (Agilent 6632B)

The sheet resistivity of pristine FTO was found as  $\sim 10.7\,\Omega/\text{sq}$ . which is in well accordance with the tagged data sheet of the product

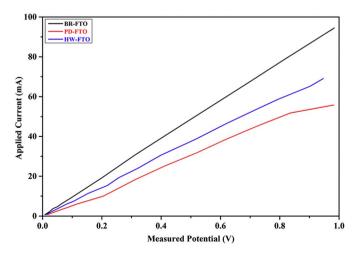
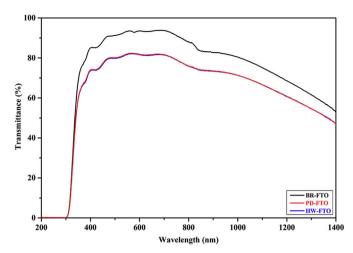


Fig. 1. I–V plot for determining the sheet resistance of (i) pristine FTO (BR-FTO) (ii) FTO heat treated over petri dish (PD-FTO) (iii) FTO heat treated in hot DI water bath (HW-FTO).



**Fig. 2.** Comparison of the transmittance spectrum of FTO substrates: (i) pristine FTO (BR-FTO) (ii) FTO heat treated over petri dish (PD-FTO) (iii) FTO heat treated in hot DI water bath (HW-FTO), obtained after 15 min of heat treatment.

 $(7\,\Omega/sq).$  As the substrate was heated to about 80 °C for duration of 15 min, an increase in the sheet resistivity of FTO surface was observed. This may be attributed to the metallic property of FTO substrate. When FTO was heat treated over a petri dish kept on a digital PID controlled hot plate, the sheet resistivity was measured to be  $\sim\!17.7\,\Omega/sq.$  whereas for FTO heat treated in hot DI water bath, it was measured to be  $\sim\!13.7\,\Omega/sq.$  We observe a significant difference in the sheet resistivity for modified FTO substrates. The difference in the sheet resistivity of modified and pristine FTO substrate may be due to the decrease in the electron mobility of FTO films [33]. Moreover, heating over petri dish results in anisotropic heating and hence, lead to higher resistivity. Thus, FTO substrate shows a positive temperature coefficient.

#### 2.1.2. Optical studies

The immediate optical transmittance spectra of FTO substrates on treatment prior to electrodeposition of CdTe is shown in Fig. 2. As can be seen from the spectrum, the transmittance of pristine FTO in the visible region is found to be around  $\sim 91\%$  whereas, for FTO heat treated over a petri dish and in hot DI water bath, the transmittance was around  $\sim 80\%$  and  $\sim 79\%$ , respectively. It is to be noted that the absorption and reflectance by the glass substrate are well included in the optical transmittance data. The visible transmittance of the treated FTO substrates decreased by  $\sim 10\%$ . This may be due to a decrease in the carrier density of FTO films after heat treatment [33]. The transmittance of modified substrate almost remained unaltered suggesting that the difference in the carrier density of such films were almost negligible.

#### 2.1.3. Structural studies

Effect of heat treatment on the crystalline nature of the FTO substrate prior deposition has been studied by XRD measurements. The XRD pattern of pre-treated and pristine FTO substrates is shown in Fig. 3.

The diffraction peaks for all the samples fit well with the tetragonal Cassiterite phase of tin oxide. The average crystallite size for pristine FTO surface was found to be 20.37 nm. The average crystallite size increased to 20.45 nm for the FTO substrate heat treated over petri dish. However, for FTO substrate heat treated in hot DI water bath, it was found to be 20.35 nm. There is no significant change in the average crystallite size for pristine and heat treated FTO surface over petri dish and in hot DI water bath. Fig. 4 presents the crystallite size along various lattice planes for modified and pristine FTO substrates. The lattice constants 'a' and 'c' calculated from the diffraction peaks along with the average crystallite size (D) are presented in Table 1. It is

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