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Synthesis of CdSe Thin Film by Chemical Bath Deposition and Characterization

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Abstract— Nanocrystalline cadmium selenide (CdSe) thin film is deposited on transparent conducting Fluorine doped tin oxide (FTO) substrates from chemical bath containing cadmium sulphate, liquid ammonia and sodium selenosulfate at a bath temperature of 70° C, and 11 pH by chemical bath deposition. The as-deposited CdSe thin film is annealed at 250° C for five hours. The annealed CdSe thin film characterized by X-ray diffraction study revealed the hexagonal phase of the film with preferred orientation along (002) plane. The optical studies of CdSe thin film showed the band gap of 1.78 eV and the transmittance of 60 % in the visible region. The Scanning electron micrograph of CdSe thin film showed grains of uniform size. The wettability test showed the hydrophobic surface nature of the film. The photoactivity of CdSe thin film was confirmed by measuring its photoelectrochemical properties under dark and illumination.

Index Terms—II-VI compound semiconductors, chemical bath deposition, CdSe thin films, contact angle, photoelectrochemical studies.

I. INTRODUCTION

The II^A-VI^B compound semiconductors (A=Cd, Zn, Hg, B=S, Se, Te etc) have remarkable properties because of their wide band gap [1]. CdSe thin films with wide direct band gap (1.7-1.8 eV), n-type semiconducting nature, high photosensitivity, and high transmittance in the visible region are important in the research field due to its wide applications in fabrication of optoelectronic devices. These films can be grown in cubic [2], hexagonal [3] or mixed form (polymorphism) [4] and find application in the field of solar cells [5], thin film transistors [6], gas sensors [7], photoconductors [8], lasers [9] etc.

CdSe thin films are obtained by different techniques viz. Electrodeposition (ED) [10], successive ion layer adsorption and reaction (SILAR) [11], chemical bath deposition (CBD) [12]-[15], molecular beam epitaxy (MBE) [16], thermal evaporation [17], metal oxide chemical vapor deposition (MOCVD) [18] spray pyrolysis [19] etc. Among these, CBD is an important deposition technique for thin films of compound semiconductors like CdSe thin films. It is a slow process with competing mechanisms of deposition [20] viz. ion by ion condensation, cluster by cluster condensation in an aqueous medium which leads to controlled precipitation of the desired compound on the substrate in the presence of a suitable complexing agent. The process is sensitive to precursor concentrations, deposition time, pH of the chemical bath and to the nature of substrate used. CBD has many advantages; it is a convenient low cost technique for producing large area thin films. The technique is quite simple, sophisticated instrumentation and vacuum are not required, less incorporation of impurities etc. The film thickness, band gap, morphology can be controlled by tuning and optimizing various deposition parameters, like bath temperature, concentration of reactants, deposition time, complexing agent. In this work we have investigated the growth of CdSe thin films by CBD. The results of investigation of the structural, morphological, optical properties of the deposited films are presented in this paper.

II. EXPERIMENTAL DETAILS

In the present work cadmium sulfate (3CdSO₄.8H₂O) is used as a cadmium precursor and sodium selenosulfate (Na₂SeSO₃) is used as selenium precursor. All chemicals used were analytical reagents (AR) grade and were used without any further purification. Sodium selenosulfate solution is highly unstable and hence it has to be freshly prepared at the time of synthesizing CdSe thin films. 0.05 M Na₂SeSO₃ was prepared by dissolving 1.25 g of selenium (Se) powder and 5 g anhydrous sodium sulfite (Na₂S) in 250 mL double distilled water (DDW). The



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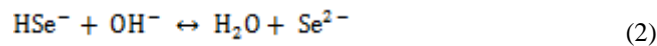
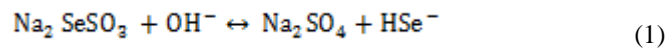
solution was then refluxed at 80 °C for 10 hour without stirring followed by filtration to remove excess, undissolved selenium [21]. The chemical bath comprises of 20 mL, 0.05 M $3\text{CdSO}_4 \cdot 8\text{H}_2\text{O}$ in a 50 mL beaker. The pH of the solution was adjusted to 11 by adding 30% liquid ammonia slowly drop by drop. To this freshly prepared 20 mL 0.05 M Na_2SeSO_3 solution was added and the contents of the beaker stirred well. Pre cleaned FTO substrates (5 cm x 0.3cm x 1.0 cm) were then introduced into the chemical bath in the vertical position using a substrate holder. The chemical bath placed in water bath which was maintained at 70° C and simultaneously stirred using heater cum magnetic stirrer. After 1 hour the substrates were removed from the chemical bath washed in DDW and dried in air.

A. Annealing of CdSe thin film

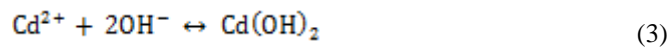
Annealing of thin films reduces the defects and increases crystallite size during the recrystallization process. As-deposited CdSe thin film was subjected to annealing in vacuum using a furnace at 250 °C (523 K) for 5 hours.

B. Reaction mechanism of formation of CdSe thin film

The following steps describe the reaction process for the formation of CdSe thin film [22]. In the anionic precursor solution the sodium selenosulfate hydrolyses in an alkaline solution to give selenide ion (Se^{2-}).

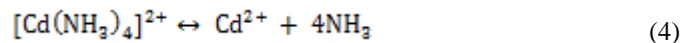


When ammonia was added to Cd^{2+} salt solution, white precipitate of cadmium hydroxide ($\text{Cd}(\text{OH})_2$) was formed.



$\text{Cd}(\text{OH})_2$ precipitate dissolves in excess ammonia solution to form the complex tetra-ammine-cadmium (II) $[\text{Cd}(\text{NH}_3)_4]^{2+}$

The cat ionic precursor solution releases Cd^{2+} from $[\text{Cd}(\text{NH}_3)_4]^{2+}$



Film formation occurs when ionic product of Cd^{2+} and Se^{2-} exceeds the solubility product CdSe ($K_{sp} = 10^{-33}$) Finally CdSe thin film formation takes place as



C. Characterization

The thickness of CdSe thin film is measured using Surface profiler XP-1 Ambios Technology. Structural characterization of CdSe thin film is carried out by X-ray diffractometer (Bruker model D2 Phasor of XAS analytical instruments) with $\text{CuK}\alpha$ radiation $\lambda = 1.54056 \text{ \AA}$, in the 2θ range from 20° to 80°. Room temperature optical measurement of CdSe thin film is carried out using UV-Visible spectrophotometer (UV-1800 Shimadzu) in the wavelength range 300-900 nm. Surface morphology of CdSe thin film is observed in the SEM JSM-6360, JEOL. Contact angle measurement of CdSe thin film is performed using contact angle goniometer rame-hart instrument. The photoelectrochemical properties of the film were performed using electrochemical setup autolab potentiostat 302N, 32FRA, metrohm Switzerland.

III. RESULTS & DISCUSSION

A. Structural studies by X-ray Diffraction (XRD)

The X-ray diffraction pattern of the CBD-CdSe thin film is as shown in Fig. 1. The XRD plot exhibits well defined peaks (002), (110), and (201) at $2\theta = 25.79^\circ$, 41.82° , and 50.8° of the hexagonal (wurtzite) phase (JCPDS Data



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Sheet No. 03-065-3415) along with peaks due to FTO substrate (JCPDS Data Sheet No. 01-070-0377). The most intense reflection peak at $2\theta = 25.79^\circ$ corresponding to a hexagonal structure [23] having the (002) plane as the preferred orientation.

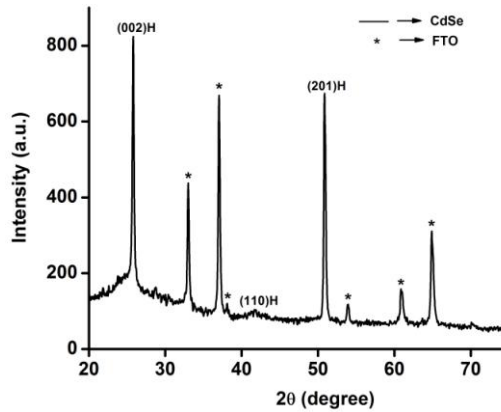


Fig. 1: XRD plot of CdSe thin film

The crystalline size (D) of the thin film is calculated using Debye Sherrer's equation [24],

$$D = 0.9 \lambda / (\beta \cos \theta) \quad (6)$$

Where λ is wavelength of X-ray used, β is full width at half maximum of the peak and θ is Bragg's angle

The interplanar spacing (d) can be calculated using Bragg's equation

$$d = \lambda / (2 \sin \theta) \quad (7)$$

The dislocation density can be calculated using Williamson & Smallman's equation

$$\rho = 1 / (D^2) \quad (8)$$

The induced strain in the sample can be calculated by

$$\epsilon = \beta \cos \theta / (4) \quad (9)$$

The number of crystallites per unit area can be calculated using the equation

$$N = t / (D^2) \quad (10)$$

Where t is the thickness of the film. Table I gives the comparison of interplanar distance (d) and 2θ between their experimental and standard values. There is a very good agreement of the experimental data with standard JCPDS (Data Sheet No. 03-065-3415). The calculated structural parameters of the CdSe sample are tabulated in Table II.

Table I: Comparison of Interplanar distance (d)

(hkl)	2θ (std)	2θ (exp)	Interplanar spacing d (std) (Å)	Interplanar spacing d (exp) (Å)
(002)	25.34°	25.79°	3.512	3.451
(110)	41.89°	41.82°	2.154	2.158
(201)	50.57°	50.80°	1.803	1.795

Table II: Structural parameters of CdSe thin film

D (nm)	ρ (lines/m ²)	N (m ⁻²)	ϵ	Lattice constant (Å)	
				Std	Exp
34.13	8.584×10^{14}	4.424×10^{15}	1.015×10^{-3}	a = 4.309	a = 4.316
				c = 7.024	c = 6.902



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B. Optical studies by UV-Visible spectrophotometer

The thin film of CdSe is subjected to UV-VIS transmittance spectral analysis. The transmittance (T) (Fig. 2) range is found to be 30-70 % [25] in the wavelength range 300 nm to 900 nm. At 550 nm the transmittance is 60 %. The thickness (t) of the film measured using a surface profiler XP-1 is found to be 175.9 nm.

Absorption coefficient (α) of the film is calculated using the formula [26]

$$\alpha = -\ln(T)/(t) \tag{11}$$

The optical band gap was calculated using the formula

$$\alpha = A(h\nu - E_g)^p / (h\nu) \tag{12}$$

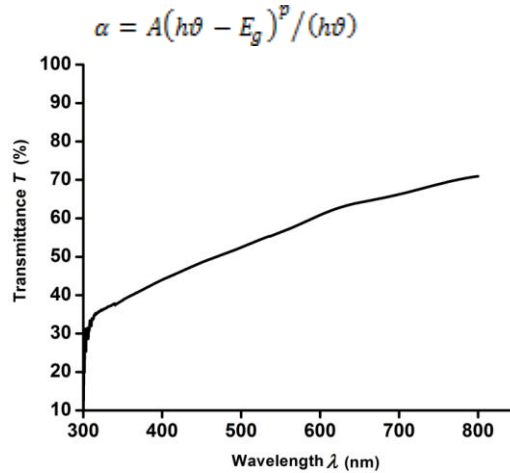


Fig. 2: Optical transmission spectrum of CdSe thin film

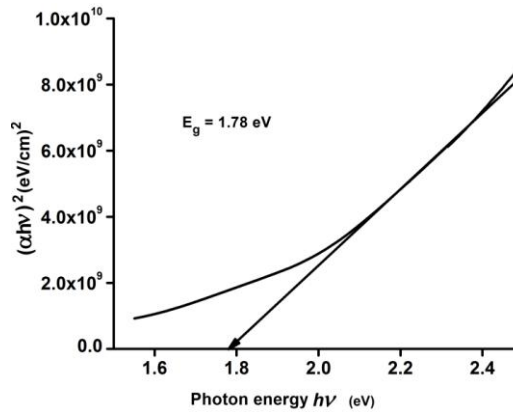


Fig. 3: A plot of $(\alpha h\nu)^2$ as a function of $h\nu$

Where $h\nu$ is the photon energy, E_g is the optical band gap of the material, A is a constant, and $p = 0.5$ for direct band gap material. $(\alpha h\nu)^2$ is plotted as a function of $h\nu$. Linearity of the plots indicates that the material is of direct band gap in nature. The linear portion of the curve is extrapolated to $(\alpha h\nu)^2 = 0$ and the band gap is found to be 1.78 eV (Fig. 3) [27] which is slightly greater than (1.73 eV) for bulk CdS [28].

C. Morphological studies by Scanning Electron Microscope (SEM)

Scanning electron microscopy is the most useful technique for the study of surface morphology, microstructure features of the thin films as these would influence their optical properties. Fig. 4 shows SEM micrographs at 25 kx and 50 kx magnifications. The film shows thick, uniform surface without cracks and well covered on to the glass substrate [25, 29]. The micrograph indicated the presence of well adherent film with grains of uniform size.

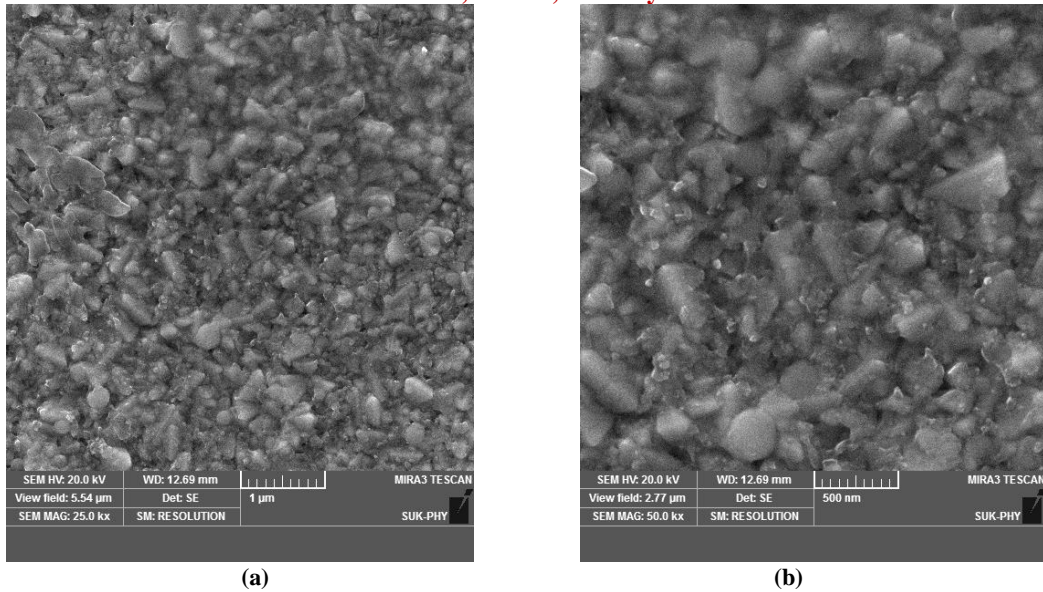


Fig. 4: SEM images of CdSe thin film (a) at 25 kx and (b) at 50 kx magnification

D. Surface wettability study of CdSe thin film

The wetting nature of the surface is characterized by the water contact angle measurement (θ). If the wettability is high then contact angle will be small ($\theta < 90^\circ$) then the surface is hydrophilic [30] and if the wettability is low contact angle will be high ($\theta > 90^\circ$) then the surface is hydrophobic [31]. The wetting of the surface is responsible for the formation of better interfacial region in the PEC cell [32]. Surface wettability of CdSe thin film is studied by means of measuring the water contact angle to the CdSe thin film. Fig. 5 shows the image of water drop on the CdSe sample. The left contact angle of CdSe sample is $100^\circ \pm 1^\circ$ while its right contact angle is $105^\circ \pm 1^\circ$. The mean contact angle is $102.5^\circ \pm 1$. This shows CdSe thin film at 11 pH exhibits the hydrophobic property [33] and hence poor wetting nature of the surface of CdSe.

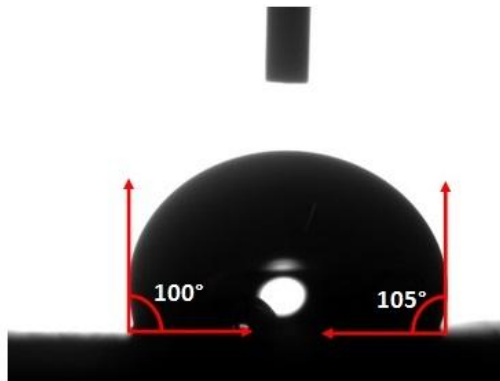


Fig. 5: Surface wettability study of CdSe thin film by measuring the water contact angle

E. Photoelectrochemical studies of CdSe thin film

The photoelectrochemical (PEC) study of CdSe thin film having average area 1 cm^2 and graphite were employed as the working electrode and counter electrode respectively. The photo electrode and counter electrode were separated by 1 cm distance. Polysulfide electrolyte, an aqueous redox electrolyte of 1 M sodium hydroxide (NaOH) + 1 M sodium sulphite (Na_2S) + 1 M sulphur (S) is used to fabricate the PEC cell along with two electrodes. The photoelectrochemical performance of the CdSe thin film electrode was examined by measuring the current density (J) versus Voltage (V) characteristics on FTO substrate in polysulfide electrolyte in dark as well as under illumination shown in the Fig. 6. The measurements are performed under input power (P_{in}) 50 mWcm^{-2} illumination and the current density and voltage measurements are performed on autolab potentiostat using two electrode cell configuration. The solar cell parameters such as short circuit current density (J_{sc}) and open circuit



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voltage (V_{oc}) are obtained from the Fig. 6. Fill-factor (FF) and power conversion efficiency of cell (η) [34] are calculated using the following equations

$$\eta = J_{sc} * V_{oc} * FF / (P_{in}) \quad (13)$$

Fill- factor can be calculated by using the following equation,

$$FF = I_m * V_m / (I_{sc} * V_{oc}) \quad (14)$$

Where I_{sc} is the short circuit current, I_m and V_m are the maximum current density and maximum voltage respectively chosen in such a way that the power becomes maximum. All the calculated solar cell parameters from the J-V characteristics [35, 36] are tabulated in Table III. The power conversion efficiency of the cell is found to be 0.03%. The reported efficiency of the photo electrode is in the range of 0.01-2.01% [37]. Under illumination of the PEC cell the magnitude of voltage increase with negative polarity towards the working electrode showing the cathodic behavior of the semiconductor thin film. This indicates that CdSe thin film has n-type semiconducting nature [38]. From the water contact angle measurement it is evident that the obtained CdSe film has shown hydrophobic surface nature and thus resulted in the formation of poor interfacial region in the PEC cell. This would lead to less charge transfer between polysulfide electrolyte and the working electrode which may result in reduction of efficiency of the PEC cell.

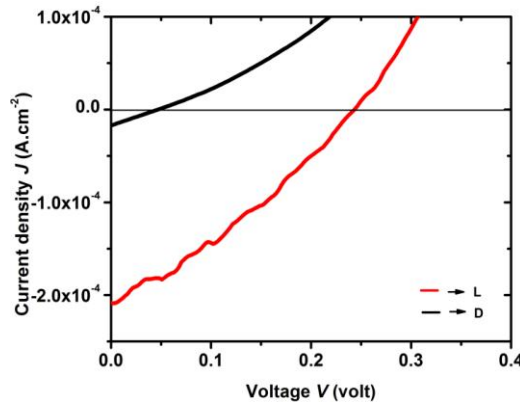


Fig. 6: Current density (J) versus Voltage (V) measurement of CdSe thin film under dark and illumination

Table III: Experimental values of PEC solar cell parameters

P_{in} (mW/cm ²)	V_{oc} (V)	J_{sc} (mA/cm ²)	I_m (A)	V_m (V)	FF	η (%)
50	0.243	0.21	1×10^{-4}	0.152	0.30	0.03

IV. CONCLUSION

A well adherent CdSe nanocrystalline thin film has been successfully deposited on transparent conducting FTO substrate by CBD method. XRD study revealed stable hexagonal phase of CdSe thin film with average crystallite size 34.13 nm. The optical band gap determined from optical transmission spectroscopy was found to be 1.78 eV with direct transition. The SEM micrographs show the surface morphology composed of grains of uniform size. The surface wettability test by measuring contact angle showed the hydrophobic surface nature of the CdSe thin film. The PEC performance of CdSe thin film indicates its n-type semiconducting nature and the power conversion efficiency of the PEC cell is found to be 0.03 %. The results presented in this paper continuous to be the area of current interest in the field of synthesis and characterization of new materials with stringent physical and chemical properties for application in the fabrication of optoelectronic devices and heterojunction solar cells with enhanced efficiency to render the devices cost effective.

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