

Fabrication of CdSe Thin Film for Photosensor Applications

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Abstract - Cadmium selenide (CdSe) thin films are deposited onto precleaned glass substrate using ammonia free Cd and Se precursor solutions employing solution growth technique (SGT) at 80 °C bath temperature to obtain good quality films. The as-deposited films were characterized for structural, morphological, optical and electrical properties. X-ray diffraction (XRD) studies revealed the film to be polycrystalline in nature with cubic crystal structure. Scanning electron microscopy and atomic force microscopy studies revealed uniform deposition of CdSe grains with uneven spherical shapes distributed over the substrates. The stoichiometric composition of the film was confirmed by EDAX analysis. The optical studies revealed direct allowed transition with optical band gap energy 1.74 eV and high transmission coefficient (~ 70%). Semiconducting behavior was observed from resistivity measurements, while activation energy of the film was found to be 0.37 eV. The I-V characteristics reveal good photoresponse demonstrating its suitability for photosensor applications.

Keywords: Thin films; Chemical synthesis; Atomic Force Microscopy; Electrical Properties; Optical Properties; Photosensor.

I. INTRODUCTION

Unique properties of nanomaterials in semiconducting thin films have been found to be useful in the fabricating them into solid state devices. Cadmium selenide (CdSe) is a promising II-VI semiconductor material and further doping of CdSe with suitable impurities form an important class of semiconductor, which finds potential applications in low cost devices such as thin film solar cells [1,2], photoelectrochemical cell [3], heterojunction solar cells [4], photodetectors [5], optoelectronic devices [6], photoconductive cell [7] etc.

Variety of methods have been employed in the fabrication of high quality CdSe thin films such as thermal evaporation [8], electrochemical deposition [9,1], successive ionic layer adsorption and reaction (SILAR) [10,11], close-space vacuum sublimation [12], molecular beam epitaxy (MBE) [13], spray pyrolysis [14], RF magnetron sputtering [15], solution growth technique (SGT) [16-18] etc. SGT is advantageous technique because it is low cost, low temperature operated and no costly instrumentations are required [19]. In this technique, substrates are immersed in an alkaline solution containing the chalcogenide source, the metal ion, added base and complexing agent. Furthermore, in SGT controlled chemical reactions play important role during the deposition of thin film and the rate of deposition can be controlled by adjusting the parameters like bath temperature, pH of solution, stirring rate, immersion time and relative concentration of solutions in the bath.

In the present investigation, we report a simple and economic solution growth technique for the deposition of CdSe thin films, using ammonia free precursors at 80°C bath temperature. The chemically deposited CdSe films were studied using several techniques to determine their physical properties. The results show that this process provides CdSe thin films with high structural and optical characteristics, which are viable candidates for application in solar energy conversion devices.

Previous reports [16-18] on deposition of CdSe thin film employed ammonia containing precursors and furthermore, the as-deposited thin films lacked well defined grains. High volatility of ammonia, concern of its

harmful impact on human beings and environment, difficulty in maintaining constant pH and volume of bath solution throughout the deposition was detrimental in achieving perfect stoichiometric thin films. Thus, with this motivation most researchers have been opting for developing ammonia-free SGT processes to obtain good quality cadmium chalcogenide films. However, very few reports are available wherein ammonia free solution growth techniques have been employed for the fabrication of CdSe thin films [20, 21]. Herein, we have deposited CdSe thin films using mixture of aqueous solutions of Cadmium sulphate, selenium powder, sodium sulphite, triethanolamine (TEA), and sodium hydroxide (NaOH), where TEA is used as the complexing agent and sodium hydroxide for adjusting pH.

II. EXPERIMENTAL

A. Thin Film Preparation

The CdSe thin films were deposited using mixture of aqueous solutions of cadmium sulphate, selenium powder, sodium sulphite, trisodium citrate, triethanolamine (TEA), hydrazine hydrate, and sodium hydroxide (NaOH), where TEA was used as the complexing agent and sodium hydroxide for adjusting the pH. SGT enables decomposition of sodium selenosulphate in an alkaline solution containing cadmium salt and a suitable complexing agent. The deposition process is based on the slow release of Cd^{2+} and Se^{2-} ions in the solution, which then condenses onto the glass substrate. The deposition of CdSe occurs when the ionic product of Cd^{2+} and Se^{2-} exceeds the solubility product of CdSe. Control of Cd^{2+} and Se^{2-} ions in the solution ultimately controls the rate of precipitation and hence the rate of film growth.

The substrates used for the deposition of CdSe thin films were commercial microscope glass slides (Blue Star) with the size of 75 x 25 x 1.35 mm. Before deposition, the substrates were degreased in HNO_3 solution for 24 h, cleaned by commercial detergent and finally rinsed with de-ionized water and dried in desiccator. This process was carried to ensure clean surface, essential for formation of nucleation centers required for thin film deposition. All chemicals used in the present investigations were AR grade. Aqueous solutions of 0.25 M cadmium sulphate (CdSO_4), 0.25 M sodium selenosulphate (Na_2SeSO_3), 0.2 M trisodium citrate, triethanolamine (TEA), 80 % hydrazine hydrate and 4 M sodium hydroxide (NaOH) were used to prepare thin films. Sodium selenosulphate was prepared by refluxing 0.25 M selenium powder mixed with 1 M sodium sulphite in de-ionized water, which was heated to 80 °C for 8 h [22]. Cadmium sulphate solution (20 mL) was taken in a 50 mL glass beaker to which 30 drops of TEA, 5 mL NaOH and 5 drops of hydrazine hydrate solutions were added slowly under continuous stirring. Initially, the solution was milky and turbid due to the formation of $\text{Cd}(\text{OH})_2$ suspension. Addition of excess NaOH led to the dissolution of turbidity and made the solution clear and transparent. Then 5 mL trisodium citrate and 20 mL freshly obtained sodium selenosulphate solutions were added slowly with constant stirring. pH of the final mixture was adjusted to ~ 11. Pre-cleaned glass substrates were inserted into the reaction mixture standing parallel with the walls of the beaker, which was kept in constant temperature bath for 4 h at 80 °C.

Thereafter, the substrate coated with CdSe was removed, rinsed with distilled water, and dried in desiccator. It was observed that the film was uniform, well adhered, and reddish in color [17]. Adhesion of the film was confirmed by centrifugal method. Thickness of the film was found to be ~ 240 nm by using weight difference method. It is noted that the growth is highly dependent on temperature and concentration of complexing agent.

B. Characterization techniques

As-deposited thin film of CdSe was characterized for structural, optical and electrical properties. Glancing incidence angle X-ray diffraction (GIXRD) pattern of the film was recorded on a Bruker AXS, Germany (D8 Advanced) diffractometer. The scanning range of diffractometer used is 20–80° (2 θ), using $\text{Cu-K}_{\alpha 1}$ radiations with wavelength 1.5405 Å at 0.5° glancing angle. The elemental composition was determined by energy dispersive X-ray analysis (EDAX). The surface morphology was studied by scanning electron microscopy (SEM, JOEL-JSM-5600) and atomic force microscopy (AFM, Nanoscope IIIa, Veeco digital instruments). Transmittance and absorbance spectra were recorded in the range 300–1000 nm by means of Jasco V630 spectrophotometer. The resistivity of the films was determined by dc two probe method. The photosensitivity property of CdSe was studied by I-V characteristics in dark and under illumination (visible spectra) with different intensities using lab equipment unit over the range $\pm 6\text{V}$. Silver paste was employed to CdSe thin films to ensure good electric ohmic contacts.

III. RESULTS AND DISCUSSIONS

A. Compositional and Structural Studies

Fig. 1 shows the EDAX spectrum of as-deposited CdSe thin film. The elemental analysis was carried out only for Cd and Se and the average atomic percentage of Cd:Se was found to be 52:48 as shown in inset of Fig. 1. It shows that elemental composition in film is almost same as that in the reaction bath mixture justifying the fact that it is possible to grow nearly stoichiometric film using SGT. However, in addition to Cd and Se, there are other peaks corresponding to Si, O, Ca, Mg, etc., and can be attributed to those originating from amorphous glass substrate [22].

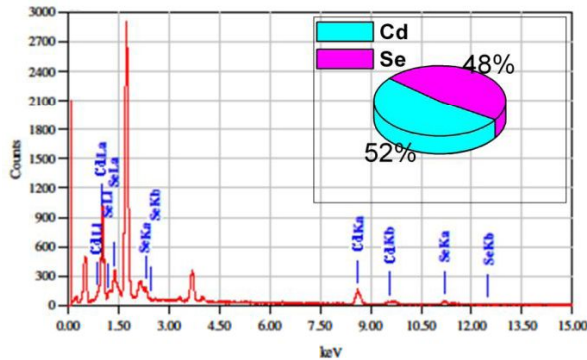


Fig. 1. Representative EDAX spectrum obtained from as-deposited CdSe thin film.

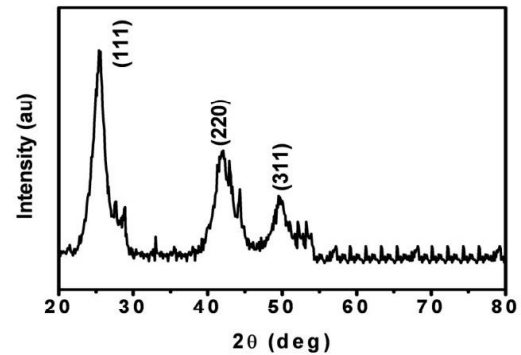


Fig. 2. GI-XRD pattern obtained from the as-deposited CdSe thin film

Fig. 2 shows X-ray diffraction pattern of as-deposited CdSe thin film. The XRD peaks indicate that the film is polycrystalline in nature. The 2θ peaks at 25.39° , 42.19° , and 49.74° correspond to reflections from (111), (220), and (311) planes, respectively. The (111) plane is the preferred orientation and it is the close-packing direction of the zinc-blend structure of cubic CdSe phase (JCPDS card No 91-0191). Crystallite size (D) of the film was calculated using Scherrer's formula [22] from the full width at half maximum (β) of the peaks expressed in radians,

$$D = \frac{K\lambda}{\beta \cos \theta} \quad (1)$$

where 'K' is constant dependent on crystallite shape (0.89), ' λ ' is wavelength of $\text{CuK}\alpha_1$ radiation, and ' θ ' is angle between the incident and scattered X-ray. The average crystallite size (derived from Fig. 2) is found to be ~8 nm. Earlier, H.E. Esparza-Ponce, et al. [20] deposited ammonia free CdSe thin film by SGT; however, they got higher bandgap (1.88 eV) and higher crystallite size (22 nm) than our results. This might have happened due to the use of sodium hydroxide, trisodium citrate, TEA and higher deposition temperature used in our case.

B. Surface Morphological and Topographical Studies

Fig. 3 shows SEM image of as-deposited CdSe thin film. The fine grains were well defined, spherical with different sizes and were not uniformly distributed over a smooth homogeneous background, shows porous nature of as-deposited CdSe film. Generally, semiconductor films with porous structures showed improved performance of solar cells [23]. Not only this, but also for various applications like optical band pass filters [24] and high sensitive chemical sensors also require the surface to be porous in nature [25]. Therefore porous nature of the film improves the photovoltaic performance by enhancing light trapping ability (anti-reflection coating property) [26].

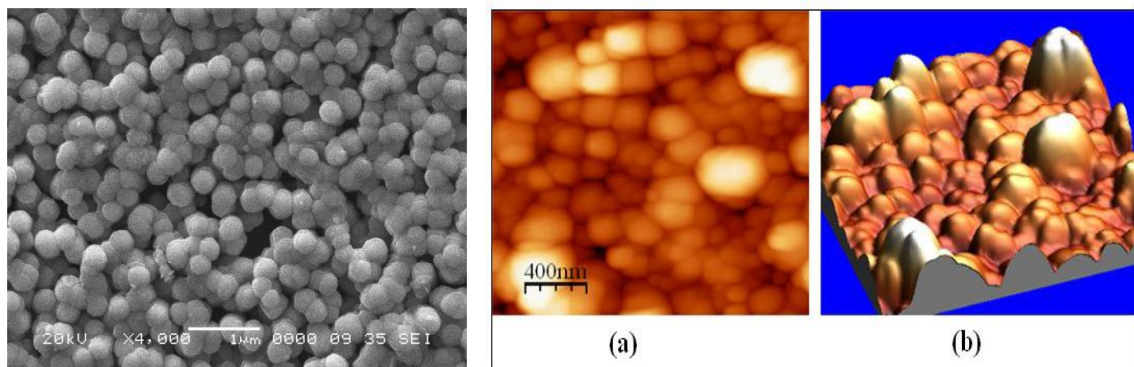


Fig. 3. Representative SEM image of as-deposited CdSe thin film.

Fig. 4. AFM images obtained from as-deposited CdSe thin film (a) 2-D and (b) 3-D.

The AFM profile is used to get surface topography information of thin films. Figs. 4 (a) and 4 (b) shows two-dimensional (2-D) and three-dimensional (3-D) AFM images obtained from CdSe thin film, respectively. The 2D image of the film resembles very well with the morphology of SEM micrograph. The 3D image shows that the as-deposited CdSe film surface has non-uniform mountainous features with sharp domes at the top and a broad bottom (pyramid type grain growth with sharp top surface). Coagulation of small crystallites into big clusters is observed and is in good agreement with the SEM micrograph (Fig. 3). From Fig. 4, the average cluster size and surface roughness was determined to be $\sim 150 \pm 20$ nm and 4 nm, respectively (confirmed by WSxM and SPIP AFM softwares). The surface roughness is unavoidable due to three dimensional growth of thin film [14].

C. Optical Studies

Fig. 5(a) shows transmittance and absorbance spectra obtained from as-deposited CdSe thin film. The optical transmittance of over 70% is noted in the near infrared region. The relation between the absorption coefficient α and the incident photon energy ($h\nu$) can be written as [14],

$$\alpha h\nu = A(h\nu - E_g)^n \quad (2)$$

where 'A' is constant, $n = \frac{1}{2}$ for direct allowed transition, ' E_g ' is optical band gap of the material

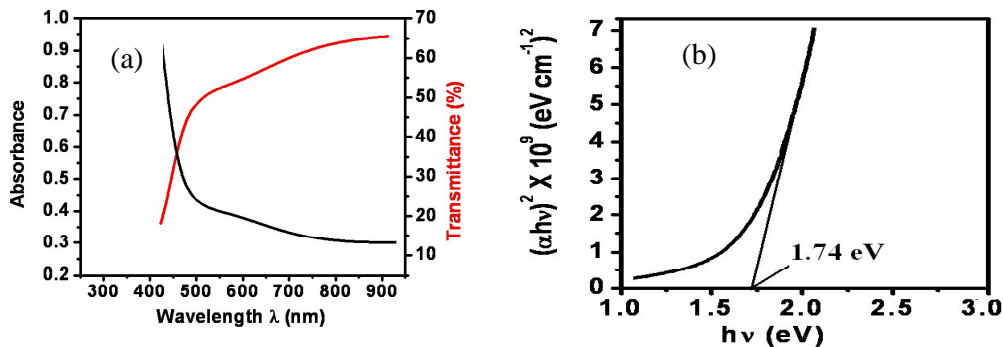


Fig. 5. (a) Plot of absorbance and transmittance versus wavelength (color online) and (b) Plot of $(\alpha h\nu)^2$ versus $(h\nu)$ obtained from as-deposited CdSe thin film.

Fig. 5 (b) shows the plot of $(\alpha h\nu)^2$ against $(h\nu)$ for CdSe thin film derived from the optical spectra. The plot is linear at the absorption edge, indicating a direct allowed transition. The straight line portion was extrapolated to the energy axis and when $(\alpha h\nu)^2 = 0$, the intercept gives the band gap energy of CdSe thin film [16]. The band gap energy is found to be 1.74 eV at room temperature (RT).

D. Electrical Studies

The electrical resistivity of the CdSe thin film in dark was measured using DC two-probe method in the temperature range 300–500 K. A plot of inverse absolute temperature versus $\log(\rho)$ for cooling cycle is shown in Fig. 6. The dependence is almost linear indicating the presence of only one type of conduction mechanism in the film. Our experimental data fit into the Arrhenius equation [22],

$$\rho = \rho_0 \exp\left(\frac{E_a}{kT}\right) \quad (3)$$

where ' ρ ' is resistivity at temperature T, ' ρ_0 ' is a constant, 'k' is Boltzman's constant, 'T' is absolute temperature and ' E_a ' is activation energy.

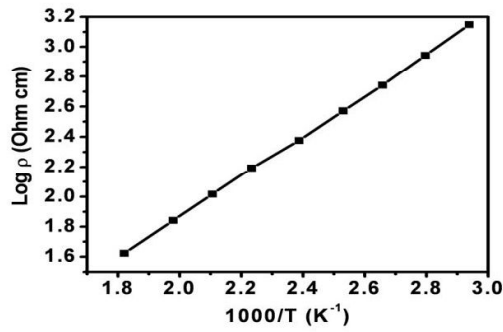


Fig. 6. Plot of $\log(\rho)$ versus $1000/T$ (K^{-1}) obtained from as-deposited CdSe thin film.

The high-temperature conductivity is a thermally activated excitation of charge carriers from nanocrystallite to nanocrystallites. In this case, activation energy was calculated from linear portion of the graph and is found to be 0.37eV. This value of the activation energy indicates that the prepared samples are semiconductor in nature [14]. Also it suggests that the conduction in these thin films is due to the thermally assisted tunneling of the charge carriers through the grain boundary barrier and transition from donor level to conduction band [27] energy charge carriers are trapped at the nanocrystallites, giving rise to interfacial polarization. The decrease in resistivity with increase in temperature confirms the semi-conducting behavior of the film.

E. Photosensitivity Studies

Fig. 7 shows I-V characteristics curve obtained from the as-deposited CdSe thin film for different illumination intensities. An area 1 cm^2 of CdSe thin film on glass substrate was selected and silver paste was applied (two Ag contacts separated by a distance of 1 cm) to ensure the good neutral electrical contacts to the films. The linearity of the plots suggests the formation of ohmic contact at metal-semiconductor (Ag/CdSe) junction, indicating that the work function of metal Ag is higher than semiconductor CdSe. This aligns the metal Fermi level of Ag with the upper valence band edge. Further resistivity decreases with increase in illumination intensity. This suggests that the incident photon energy breaks some of the covalent bonds in the CdSe semiconductor and as a result free electron-hole pairs are available for current conduction. The dark resistivity is found to be $0.52 \times 10^{11}\ \Omega\text{ cm}$ and it decreases to $0.08 \times 10^{11}\ \Omega\text{ cm}$ (calculated from fig.7) for light of intensity 9800 Lux in the visible region

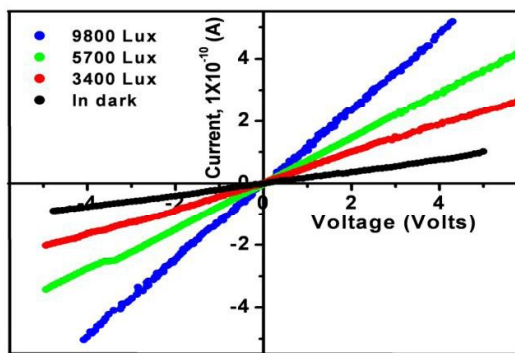


Fig.7. Current–Voltage (I–V) characteristics curve obtained from as-deposited CdSe thin film with different illumination intensities.

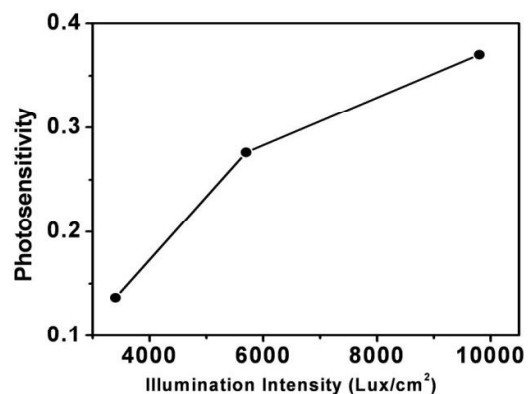


Fig. 8. Photosensitivity plot of as-deposited CdSe thin film.

For the photosensor characterization of the film, photosensitivity is an important parameter which gives the quality of the photosensor. The photosensitivity (S) is calculated by using the equation as [28].

$$S = \frac{R_d - R_L}{R_d} \tag{4}$$

Where, R_d and R_L is the resistance in dark and under light, respectively. Fig. 8 shows the plot of photosensitivity versus illuminated light intensities. It can be concluded that the enhancement of the photoconductive sensitivity is due to the electron-hole pairs excited by the incident light. Thus, there occurs a steady increase in the photosensitivity. This shows that photo carrier generation in these films may be understood on the basis of the simplest picture for a photoconductor with band to band generation of carriers and a set of species of recombination centers [29]. These characteristics are important for the application of these films in photosensors or other optoelectronic applications.

IV. CONCLUSION

CdSe thin film were grown using ammonia free precursor solutions with appropriate selection of the growth parameters by inexpensive SGT. The as-deposited film present excellent adherence, uniform deposition, smooth morphological and nanocrystalline properties, confirmed by SEM, AFM and XRD analysis. It is found that the as-deposited CdSe films are highly oriented with cubic zinc blende structure, and the preferred crystal orientation is (111) plane. The XRD FWHM also suggests that the crystal quality of the as-deposited CdSe films is reasonably good. The EDAX study shows almost stoichiometric deposition. Energy band gap of the as-deposited CdSe film is 1.74 eV and it is quite close to the reported value of 1.88 eV, which shows a blue emission. From I-V study with different illumination intensity shows almost linear photoresponse. The study of physical, optical and electrical properties reveal that the CdSe thin film can be suitably employed in photosensor and/or optoelectronic applications.

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