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Chemical synthesis and characterization of CdSe thin films deposited by SILAR technique for optoelectronic applications

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Abstract

CdSe thin films were deposited on the glass substrate by successive ionic layer adsorption and reaction (SILAR) method. Different sets of the film are prepared by changing the number of immersion cycles as 30, 40, 50 and 60 and the effect of a number of immersion cycles on the characteristic structural, morphological, optical and electrical properties of the films are studied. The XRD studies revealed that the deposited films showed hexagonal structure with most prominent reflection along (1 0 1) plane. Moreover, the peak intensity of (1 0 1) plane is found to be increased as the number of immersion cycles is increased. All the thin films look relatively smooth and homogeneous covering the entire surface area in FESEM image. Optical properties of the CdSe thin films for a different number of immersion cycles were studied, which indicates that the absorbance increases with the increase in the immersion cycles. Furthermore, the optical band-gap in conjunction with the electrical resistivity was found to get decreased with increase in the immersion cycles. A good correlation between the number of immersion cycles and the physical properties indicates a simple method to manipulate the CdSe material properties for optoelectronic applications.

Keywords: Cadmium selenide; SILAR; Structural properties; Optical properties and Electrical resistivity
1. Introduction

Thin films of metal chalcogenides have been studied extensively in view of their potential industrial applications [1, 2]. Notwithstanding to this, these materials are also important both academically as well as scientifically. In particular, Cadmium selenide (CdSe) is among the metal chalcogenide materials, which has remarkable optoelectronic properties that make it suitable for various applications in the field of low-cost optoelectronic devices such as solid-state solar cells [3, 4], photoconductors [5], photoelectrochemical cells [6] and solar control coatings [7] etc. Major attention has been given in recent years to the investigation of electrical and optical properties of CdSe thin films in order to improve the performance of the devices and also for finding new applications [8]. CdSe is an n-type material with its band-gap lying in close range with the maximum theoretical range that is attainable for energy conversion efficiency. Moreover, CdSe can be grown with either hexagonal, cubic or mixed (hexagonal-cubic) crystal structures. Accordingly, the optical band-gap can be defined for each structure, which could be suitable for different applications such as solar cells, thin film transistors, sensors, lasers, photoconductors, gamma ray detectors [9, 10].

Till date, thin films of CdSe have been deposited by various techniques such as chemical bath deposition (CBD) [11, 12], electrodeposition [13], cathodic electrodeposition [14], physical vapour deposition [15], spray pyrolysis [16], vacuum evaporation technique [17] etc. Preparation of thin films by a simple SILAR method is currently attracting considerable attention as it is simple, cost-effective and reproducible [18, 19]. Importantly, with this method, one can avoid fast precipitation and the deposition can be done in a controlled manner, which is what rather difficult in other methods, especially, CBD. In concern to this, only a few reports are available on the deposition of CdSe thin films by SILAR method. In the year 2002, Pathan et al. [20] used SILAR method for the deposition of CdSe thin films with cadmium sulphate and sodium selenosulphite as a cationic and anionic precursor, respectively and tartaric acid as a complexing agent. They found the broad hump in the XRD pattern, which they suggest amorphous/fine granular nature of the CdSe thin film deposited with 45 immersion cycles. Optical band-gap was found to be 1.80 eV. Later, in the year 2004, Kale et al. [21] prepared CdSe thin films using SILAR method. For this, they used cadmium acetate solution complexed with tartaric acid and TEA as the cationic precursor solution and anionic precursor solution made up of sodium selenosulphite. Their structural study indicates the nanocrystalline cubic phase for CdSe thin film.
deposited with 150 immersion cycles. From the optical absorption studies, they showed that CdSe thin film has a direct optical band-gap, $E_g$, of 2.1 eV, which is higher as compared to the earlier report [20]. Further in 2011, Akaltun et al. [22] studied the film thickness effect on the characteristics parameters of CdSe thin films prepared by using SILAR method. From XRD and SEM studies they showed that the thin films have a polycrystalline structure with preferential orientation along (0 0 2) plane and the crystalline and surface properties of the prepared films were improved by increasing film thickness.

Therefore, from the aforementioned studies, it could be seen that the characteristic properties of the CdSe thin films and their applications in the field of optoelectronics are closely related to the crystallinity, orientation, grain size, optical band-gap and electrical resistivity that are affected by the film thickness, which will be controlled by varying the immersion cycles in the SILAR deposition method. And hence in this paper, we report the synthesis of CdSe thin film by SILAR technique. The effect of immersion cycles on the structural, morphological, optical and electrical properties of SILAR deposited CdSe thin films was studied.

2. Experimental details

2.1 Preparation of CdSe thin film

In this work, CdSe thin films were deposited on a glass substrate using SILAR method at room temperature and ambient conditions. To deposit CdSe thin films, 0.2M cadmium chloride (CdCl$_2$-H$_2$O) solution at pH ~ 8 and freshly prepared 0.1M sodium selenosulphite (Na$_2$SeSO$_3$) at pH ~11.3 were used as cationic and anionic precursor solutions, respectively. EDTA was used as a complexing agent and ammonia is used to control the pH of the cationic precursor solution to 8. Before, the actual deposition, the glass substrates were thoroughly washed with detergent & chromic acid, rinsed with acetone and finally ultrasonically cleaned with double distilled water.

The following procedure was adopted to deposit CdSe thin films, one SILAR growth cycle involving four steps (see Fig. 1):

(i) Immersion of the cleaned substrate in first reaction beaker containing cationic precursor solution of 0.2M [CdCl$_2$.H$_2$O] for 60 s. This process leads Cd$^{2+}$ ions to get adsorbed on the surface of the substrate.

(ii) This substrate was rinsed by high purity deionized water for 15 s to remove excess Cd$^{2+}$ ions that are loosely adherent to the glass substrate (achieved in the previous step).
(iii) The substrate was then immersed in the anionic precursor solution of 0.1 M Na$_2$SeSO$_3$ for the 30 s. The selenide (Se$^2-$) ions reacted with adsorbed Cd$^{2+}$ ions on the active center of the substrate to give CdSe.

(iv) Again the substrate was rinsed in deionized water for 15 s to remove loosely bound ions present on the substrate and unreacted Cd and Se ions.

This completes one SILAR immersion cycle of CdSe deposition. The scheme for the deposition of CdSe films by SILAR method is represented in Fig. 1. Hence, several repeated immersion cycles can result in the required CdSe compound of desired thickness. The uniqueness of this SILAR method lies in the easy control of the parameters [18]. This allows one to properly control the thickness necessary for various device applications.

2.2 Characterization of the films

To investigate the effect of SILAR immersion cycles on the properties of the CdSe thin films, XRD, FESEM, optical absorption measurements and the two-point-probe methods were used. The XRD pattern of the films were recorded on a Bruker AXS, Germany (D8 Advanced) diffractometer in the scanning range $2\theta = 20-80^\circ$ using Cu $K_{\alpha}$ radiations with wavelength 1.5405 Å. S-4800 Type-II (HITACHI HIGH TECHNOLOGY CORPORATION Tokyo, Japan) field emission scanning electron microscope (FESEM) with an energy dispersive spectrometer (EDS) attachment was used for the determination of morphology and elemental chemical composition of the sample. To study the optical characteristics of the films, absorbance spectra were recorded in the range 450-900 nm by means of JASCO UV-VIS spectrophotometer (V-630). The resistivity of the CdSe thin films was determined by the standard two-probe method.

3. Results and discussion

3.1 Film thickness

In order to study the growth rate, SILAR coated CdSe thin films were deposited for various immersion cycles on glass substrates. For this particular study, we have deposited CdSe thin films with different immersion cycles i.e., 30, 40, 50 and 60 SILAR immersion cycles. Fig. 2 represents CdSe film thickness as a function of the immersion cycles for optimized concentrations of CdCl$_2$ and Na$_2$SeSO$_3$. It is found that the film thickness increases with the immersion cycles. The CdSe film has a maximum terminal thickness of the order of 370 nm at 60 SILAR immersion cycles.

3.2 Structural analysis
XRD pattern of SILAR deposited CdSe thin films with 30, 40, 50 and 60 immersion cycles are as shown in Fig. 3 [(a), (b), (c), (d)], respectively. The XRD patterns clearly showed the influence of the immersion cycles on the crystallinity of the films. For all CdSe films, the hexagonal structure characterized with (1 0 1) plane as preferred orientation, are identified with the standard JCPDS data [23]. This result is different than Akaltun et al. [22] for CdSe thin films prepared by SILAR method. In their case, the CdSe thin films were preferentially grown along (0 0 2) plane. This might be due to a different number of the immersion cycle and/or the film thickness. Apart from this, some other diffraction peaks are also visible in the XRD pattern of CdSe thin films. The peaks at $2\theta = 43.14^{\circ}$ and $50.90^{\circ}$ referred to the (1 1 0) and (2 0 1) orientations, respectively of the hexagonal phase of the CdSe. Importantly, the XRD peaks corresponding to hexagonal CdSe became more intense as the number of immersion cycles increases from 30 to 60 with no significant shift in the peak position. Additionally, the XRD peaks were broadened. This probably may be due to the presence of nanocrystallites of CdSe. The crystallite size ($D$) is calculated (considering the instrumental broadening) using the well-known Scherrer’s formula along the (1 0 1) plane for all the samples [24]:

$$D = \frac{k\lambda}{\beta \cos \theta}$$  \hspace{1cm} (1)

where $K$ is constant (0.9), $\lambda$ is the wavelength of X-ray, $\beta$ is the full width at half of the peak maximum in radians and $\theta$ is Bragg’s angle. It is observed that the crystallite size increases from 3.07 nm to 3.98 nm as immersion cycle increases from 30 to 60. 

Further, to have more information on the amount of defects in the synthesized thin films, the dislocation density ($\delta$) was calculated from Williamson Smallman’s formula as given below [24]:

$$\delta = \frac{n}{D^2}$$  \hspace{1cm} (2)

where ‘$n$’ is a factor, which when equal to unity gives the minimum dislocation density and ‘$D$’ is the average crystallite size.

The average microstrain developed in the prepared thin films is defined as disarrangement of lattice and was calculated by using the relation as given below [24]:

$$\varepsilon = \frac{\beta \cot \theta}{4}$$  \hspace{1cm} (3)
Fig. 4 shows the variation of the microstrain ($\varepsilon$) and dislocation density ($\delta$) of CdSe thin films as a function of immersion cycles. From the figure it has been found that the dislocation density ($\delta$) and microstrain ($\varepsilon$) have similar trends i.e., both decreases with the immersion cycles. That means the dislocation density ($\delta$) and microstrain ($\varepsilon$) are inversely proportional to the number of immersion cycles and crystallite size (D) as well. This shows that the quality of the deposited CdSe thin film improves with the increase of immersion cycles.

3.3 Morphological properties
The surface morphology of CdSe thin films was studied using FESEM. FESEM micrographs of the films deposited with 30, 40, 50 and 60 SILAR immersion cycles are as shown in Fig.5 [(a)-(d)], respectively. From micrographs, it is observed that the prepared films are continuous covering the entire area and uniform without cracks or pinholes. Moreover, the FESEM micrographs for all the samples revealed irregular nanosized grains coagulated together to form bigger globular structures. The broadening in the XRD measurement observed (see Fig. 3) might be due to the presence of such nanosized grains. Mahato et al. [13] also observed similar morphology for CdSe thin films synthesized using simple electrodeposition method on ITO coated glass substrate.

3.4 Elemental analysis
The elemental analysis of CdSe thin films deposited on the glass substrate was performed using EDS analysis. The typical EDS spectra for the 60 SILAR immersion cycles deposited CdSe thin film is shown in Fig. 6. It is observed that the emission lines of ‘Cd’ and ‘Se’ are present in the EDS spectra indicating the formation of CdSe thin films. Fig. 7 shows the average atomic ratio of Cd/Se as a function of SILAR immersion cycles. It is observed that the ‘Cd’ and ‘Se’ ratio is found to be decreased (reaching 1.05) with the increase of immersion cycles, which indicates the stoichiometric CdSe formation. This is also in conjunction with the thickness measurements (Fig. 2), where the thickness starts to saturate indicating the lowering of the CdSe compound on the thin film surface [25].

3.5 Optical properties
Fig. 8 shows the optical absorption spectra of CdSe thin films deposited with different SILAR immersion cycles. It can be observed that the absorption edge of the spectra shifts towards longer wavelength in the higher immersion cycles. Also, the absorbance was found to be increased with
the increase in the immersion cycles. This might be due to the simultaneous increase in the thickness that is being observed.

The theory of optical absorption gives the relation between the absorption coefficient $\alpha$ and the photon energy $h\nu$, especially, for direct allowed transition as,

$$\alpha = \frac{A(h\nu - E_g)^2}{h\nu}$$

(4)

where $h\nu$ is the photon energy, $E_g$ is the optical band-gap, $A$ is a constant.

A typical plot of $(\alpha h\nu)^2$ versus $h\nu$ for 30 SILAR immersion cycles deposited CdSe thin films is as shown in Fig. 9 (a). The linear fit of the plot indicates the existence of the allowed direct band-gap transition. The band gap was found within the range 1.79-1.88 eV for CdSe thin film. These band-gap values were in good agreement with the earlier reported values of band-gap for CdSe nanocrystalline thin films deposited by CBD technique [11]. The direct band-gap of CdSe thin films deposited with various SILAR immersion cycles is determined and is shown in the Fig. 9 (b). It is obvious from the results that the optical band-gap decreases with the increase in the SILAR immersion cycles, which may be due to the quantum size effect, improvement of the crystallization and variation in the stoichiometry of the film.

### 3.6 Electrical properties

The measurements on electrical resistivity of the CdSe thin film as a function of SILAR immersion cycles were carried out in the temperature range 300 - 423 K on samples with a typical size of 1cm x 1cm, using a standard two point probe method. The variation of log $\rho$ versus the inverse of absolute temperature (1000/T) for the films deposited with different SILAR immersion cycles, shown in Fig. 10. The resistivity of all the films decreases with increase in temperature which indicates semiconducting nature of the films [26]. The resistivity of the films decreased from $19.5 \times 10^{11}$ to $0.51 \times 10^{11}$ $\Omega \text{ cm}$ with increasing the SILAR immersion cycles. The reason for the high resistivity value for all samples can be explained with dislocations and imperfections [26]. This decrease of resistivity with the SILAR immersion cycles might be due to the decrease of residual defects and improvement in the crystalline and grain size in the films, which was observed in the XRD studies [27] and due to morphological changes of the films [26].

### Conclusion

CdSe thin films were deposited successfully using SILAR technique with different immersion cycles. From XRD studies, it is confirmed that obtained films have a hexagonal phase with
(1 0 1) as preferential orientation and films are nanocrystalline in nature, which is in corroboration with the FESEM data. The optical band-gap, as well as electrical resistivity decreases with the increase of immersion cycles indicating that the thin films can be easily tailored by simply SILAR immersion cycles.

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Figures Captions:

**Figure 1.** Schematic diagram of the synthesis of CdSe thin films by using SILAR method. (○ - Cd²⁺; ● - Se²⁻): (a) cationic precursor, (b) ion exchange water, (c) anionic precursor and (d) ion exchange water.

**Figure 2.** Plot of thickness of the CdSe thin films as a function of immersion cycles.

**Figure 3.** X-ray diffraction patterns for CdSe thin films deposited with (a) 30, (b) 40, (c) 50 and (d) 60 immersion cycles.

**Figure 4.** The strain and dislocation density of nanocrystalline CdSe thin films as a function of immersion cycles.

**Figure 5.** FESEM images of CdSe thin films deposited with (a) 30, (b) 40, (c) 50 and (d) 60 immersion cycles.

**Figure 6.** Typical representation of EDAX data for CdSe thin films deposited with 60 immersion cycles.

**Figure 7.** Plot of the average atomic ratio of Cd/Se as a function of immersion cycles.

**Figure 8.** Plot of absorbance with respect to wavelength for CdSe thin deposited with a) 30, b) 40, c) 50 and d) 60 immersion cycles.

**Figure 9 (a).** Typical plot of \((ahv)^2\) versus \(hv\) for CdSe thin films deposited with 30 SILAR immersion cycles.

**Figure 9 (b).** The variation in the band-gap versus number of immersion cycles for all deposited CdSe thin films.

**Figure 10.** Temperature-dependent resistivity plot for CdSe thin films deposited with (a) 30, (b) 40, (c) 50 and (d) 60 immersion cycles.
Fig. 2
Fig. 3

Fig. 4
Fig. 6

Fig. 7
\[ E_g = 1.88 \pm 0.01 \text{ eV} \]

Fig. 8

\[ (\alpha h\nu)^2 \times 10^{14} \text{ (eV/cm)}^2 \]

Fig. 9 (a)
Fig. 9 (b)

Fig. 10