



ORIGINAL ARTICLE

Effect of La on optical and structural properties of CdS–Se films



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Abstract La doped Cd (S–Se):CdCl₂ films are co-deposited on glass slide substrates through chemical deposition technique at room temperature (RT). Results of optical absorption spectra, photoluminescence (PL) emission spectra, scanning electron microscopy (SEM) and X-ray diffraction (XRD) studies are reported and discussed. SEM studies exhibit non-uniform distribution of grains with some layered growth while XRD results show polycrystalline nature of the films. The values of some important parameters like strain (ϵ), grain size (D) and dislocation density (δ) evaluated through XRD results are discussed. Optical absorption spectra confirm the presence of La in the lattice. From these results, the band-gap values are calculated, which show direct band-gap nature of the materials undertaken. In PL spectra, the observed two peaks are attributed to transitions between excitonic levels and energy levels of La.

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1. Introduction

Among the various wide band-gap II–VI binary semi conducting compounds, cadmium sulphide (CdS) (band-gap = 2.42 eV at RT) is a promising and widely used material for applications like solar cell (Acharya et al., 2010), light emitting (Xuan et al., 2006) and photonic devices (Ullrich et al., 1999).

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On the other hand CdSe having band-gap of 1.75 eV, when mixed with CdS, turns into an appropriate candidate for the conversion of low energy light into electricity. The homogeneous alloys formed over the entire composition range by combination of these compounds allow the production of very interesting ternary Cd (S_{1-x}–Se_x) (0.1 < x < 1) system (Murali and Venkatachalam, 2008). The films are prepared through chemical deposition technique, which is a well established technique (Bhushan and Shrivastava, 2006; Chandra and Bhushan, 2004; Mane and Lokhande, 2008). Reisfeld et al. (2003) reported that the rare earth ions, when incorporated into a solid, show distinct absorption and emission spectral lines as a result of the electronic transitions within the 4f^N shell configuration. They are characterized by a partially filled 4f shell well shielded by 5s² and 5p⁶ orbitals. In the recent times Bhushan and co-workers reported interesting photoconducting properties in La doped CdS and CdPbS films. The photocur-

rent to dark current ratio $\sim 10^5$ was reported in CdS:La films (Bhushan and Sharma, 1990) and SEM studies presented crystalline nature of undoped CdS films. In presence of NaF, layered growth takes place, which finally results in cabbage type structure. In presence of La, both cabbage and crystalline growth were found by Bhushan and Sharma (1992). The increase in carrier concentration is associated to release of valance electrons from the trivalent La (Bhushan et al., 2001). Photoluminescence [PL] was also recorded in La doped [Cd–Pb] S films (Bhushan et al., 2002). Above interesting observations have led to the selection of La as impurity with base Cd (S–Se) for the present work.

2. Experimental details

2.1. Sample preparation

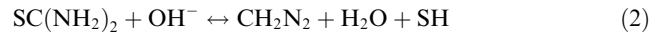
The samples for various studies are prepared by vertically dipping the cleaned substrates of highly transparent glass plates (dimension = $75 \times 24 \times 2$ mm) at RT for 18 hours. Prior to deposition, the substrates are cleaned with double distilled water, acetone, and ultrasonic cleaner. A 25.0 ml beaker is used as a container for reaction of chemicals. The aqueous solution taken in the beaker contains highly pure and analytical reagent grade 1 M cadmium acetate $[\text{Cd}(\text{CH}_3\text{COO})_2]$, appropriate ratio of thiourea $[\text{SC}(\text{NH}_2)_2]$ to sodium selenosulphate $[\text{Na}_2\text{SeSO}_3]$ solutions [prepared by heating elemental selenium (99.9% pure) in aqueous solution of sodium sulphite $[\text{Na}_2\text{SO}_3]$ at 90°C for 5 h], triethanolamine (TEA) $(\text{HOCH}_2\text{CH}_2)_3\text{N}$ and 30% aqueous ammonium hydroxide (NH_4OH) . For preparing doped films, calculated proportions of 0.01 M solutions of cadmium chloride (CdCl_2) and lanthanum oxide (La_2O_3) are also added to the original mixture. The beaker containing aqueous solution of the chemicals is well covered with another inverted bigger beaker to prevent possible ammonia loss to some extent.

In the beginning solution is stirred for few minutes and no further stirring is done during the deposition. The deposition is made in the static condition by placing glass substrates inclined vertically to the walls of the beaker. Thereafter, the substrate is removed from the beaker and treated with distilled water to wash out the uneven overgrowth of grains at the surface and dried by keeping in open atmosphere under sun light until it completely dries. This helps in achieving longer operating life of PC and PL devices. The dried film is quite adherent to the substrate surface and is irremovable. Bhushan and Chandra (2008) reported that chemically deposited films have operating life of more than 2 years. In the above, the role of TEA and ammonia solution is to adjust pH of the reaction mixture and to increase film adherence. The thickness of the film is measured by interferometry method and is found to lie between 0.76 and 0.85 μm .

The mechanism of deposition of CdS–Se films is similar to that of CdS, which is based on the slow release of Cd^{++} and S^{--} ions in aqueous basic bath and subsequent condensation of these ions on the substrates suitably mounted in the bath. The slow release of Cd^{++} ions is achieved by dissociation of a complex species of cadmium $\text{Cd}(\text{TEA})^{++}$. The availability of Cd^{++} ions is governed by the following dissociation equilibrium:



The S^{--} ions are provided by the dissociation of thiourea $[\text{SC}(\text{NH}_2)_2]$ in the ammoniacal medium.



Since the solubility of CdS is low, even the low concentration of Cd^{++} and S^{--} yield the solid phase.

2.2. Measuring instruments

The PL excitation is done with 365 nm line of mercury, obtained from a high pressure mercury vapour lamp filtered by Carl Zeiss interference filter. The light output is detected by an RCA-6217 photomultiplier tube operated by a highly regulated power supply. The integrated light output in the form of current is recorded by a sensitive polyflex galvanometer. The spectral studies are performed using a prism monochromator. The optical absorption spectra are recorded using a 1700 Pharmespec Shimadzu Spectrophotometer over the wavelength range 350–700 nm. The XRD and SEM studies are performed at Inter University Consortium, Indore, India using Rigaku X-ray diffractometer and JEOL JSM-5600 scanning electron microscope, respectively.

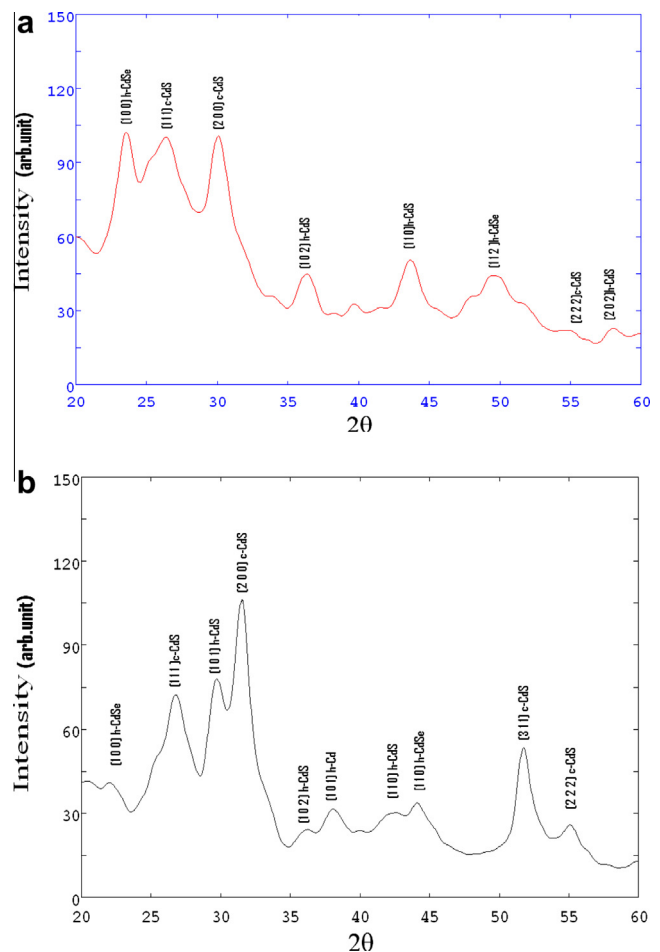


Figure 1 (a) X-ray diffractograms of Cd $(\text{S}_{0.7}\text{Se}_{0.3})$. (b) X-ray diffractograms of Cd $(\text{S}_{0.7}\text{Se}_{0.3})\text{:CdCl}_2$, La films.

3. Results and discussions

3.1. Structural analysis

3.1.1. XRD studies

The samples are characterized by X-ray diffraction. The XRD diffractograms of Cd ($S_{0.7}Se_{0.3}$) and Cd ($S_{0.7}Se_{0.3}$):CdCl₂, La films are presented in Fig. 1 and corresponding data are listed in Table 1. The assignment of diffraction lines is made by comparing with American Standards for Testing Materials (ASTM) data and also by determination of parameters like lattice interval, lattice constant and Miller indices.

XRD patterns show diffraction peaks corresponding to CdS and CdSe both. The peak intensities change due to incorporation of La as impurity. CdS is found in both cubic and hexagonal phases, which arise due to difference in arrangement of atomic layers. The possible reason for this rising of cubic and hexagonal phases is atomic arrangements as ABC ABC ... and AB AB ... (Kittle, 1995). Mixed phases are also found as a result of random stacking of very long period (Lopez et al., 2009).

Langer et al. (1966) reported that a solid solution is a mixture of microcrystalline regions of pure CdSe and CdS, where each micro region is composed of a number of unit cells of

each material with the lattice-constant of CdS stressed by surrounding CdSe and vice-versa. Such a model explains uniform shift of absorption edge with composition. These researchers found possibility of solid solution consisting of statistical distribution of CdSe and CdS with respect to their overall concentration. Present investigation witnesses the shift of absorption edge. The average particle size is evaluated using famous Debye Scherer's Formula (Ubale et al., 2007):

$$D = 0.94\lambda/\beta_{1/2} \cos \theta \quad (3)$$

where $\beta_{1/2}$ is the full width at half maximum (FWHM) for the peaks expressed in radians, λ – the wavelength of X-rays and θ – the Bragg's angle. The average particle size evaluated for different samples lie in the range 5.125–7.123 nm. The shift in spectrum reported earlier is due to nano crystalline effect in addition to other effects mentioned earlier. The values of related parameters like strain ε (Senthilkumar et al., 2005) and dislocation density δ (Singh and Bhushan, 2009) obtained for cubic (1 1 1) peak of CdS are listed in Table 2.

3.1.2. SEM studies

The SEM micrographs of Cd ($S_{0.7}Se_{0.3}$) and Cd ($S_{0.7}Se_{0.3}$):CdCl₂, La films at a magnification of 5 k are presented in Fig. 2. In both the micrographs, large numbers of clusters

Table 1 XRD diffraction data of Cd ($S_{0.7}Se_{0.3}$) films (preparation time = 16 h; preparation temperature $\sim 28^\circ\text{C}$).

Inter planner spacing (d values)		Intensity		Miller indices (hkl)	Lattice constant	
Obs. values (\AA)	Rep. values (\AA)	Obs. values (\AA)	Rep. values (\AA)		Obs. values (\AA)	Rep. values (\AA)
<i>(a) Cd ($S_{0.7}Se_{0.3}$) films</i>						
3.77	3.72	100	100	(1 0 0) _h – CdSe	$a = 4.35$	4.30
3.37	3.36	100	100	(1 1 1) _c – CdS	$a = 5.837$	5.818
2.96	2.90	99	40	(2 0 0) _c – CdS	$a = 5.920$	5.818
2.35	2.45	44	25	(1 0 2) _h – CdS	$a = 4.137$	4.14
					$c = 6.31$	6.71
2.07	2.06	50	80	(1 1 0) _h – CdS	$a = 4.137$	4.135
1.839	1.833	44.3	50	(1 1 2) _h – CdSe	$a = 4.35$	4.30
					$c = 7.25$	7.02
1.672	1.68	22	10	(2 2 2) _c – CdS	$a = 5.791$	5.818
1.58	1.581	22	7	(2 0 2) _h – CdS	$a = 4.137$	4.135
					$c = 6.82$	6.71
<i>(b) Cd ($S_{0.7}Se_{0.3}$):CdCl₂ (2 ml), La (8 ml) films</i>						
4.06	3.72	40	100	(1 0 0) _h – CdSe	$a = 4.67$	4.30
3.32	3.36	71	100	(1 1 1) _c – CdS	$a = 5.74$	5.818
3.0	3.16	77	100	(1 0 1) _h – CdS	$a = 4.1$	4.135
					$c = 6.81$	6.71
2.83	2.90	100	40	(2 0 0) _c – CdS	$a = 5.66$	5.818
2.47	2.45	24	25	(1 0 2) _h – CdS	$a = 4.1$	4.135
					$c = 6.79$	6.71
2.05	2.06	33	57	(1 1 0) _h – CdS	$a = 4.1$	4.135
						6.71
2.16	2.15	30	82	(1 1 0) _h – CdSe	$a = 4.32$	4.30
1.76	1.753	51	60	(3 1 1) _c – CdS	$a = 5.837$	5.818
1.66	1.68	25	10	(2 2 2) _c – CdS	$a = 5.749$	5.818

Table 2 Values of particle size, strain and dislocation density for Cd (S–Se):CdCl₂, La films at RT ($\sim 28^\circ\text{C}$).

System	Particle size D (nm)	Dislocation density ($\times 10^{15} \text{ lin m}^{-2}$)	Strain ε ($\text{lin}^{-2} \text{ m}^{-4}$)
(Cd ($S_{0.7}Se_{0.3}$):CdCl ₂ (3.00 ml), La (4.00 ml)	4.28	54.61	0.00843
Cd ($S_{0.7}Se_{0.3}$):CdCl ₂ (2.00 ml), La (8.00 ml)	3.39	87.03	0.0106

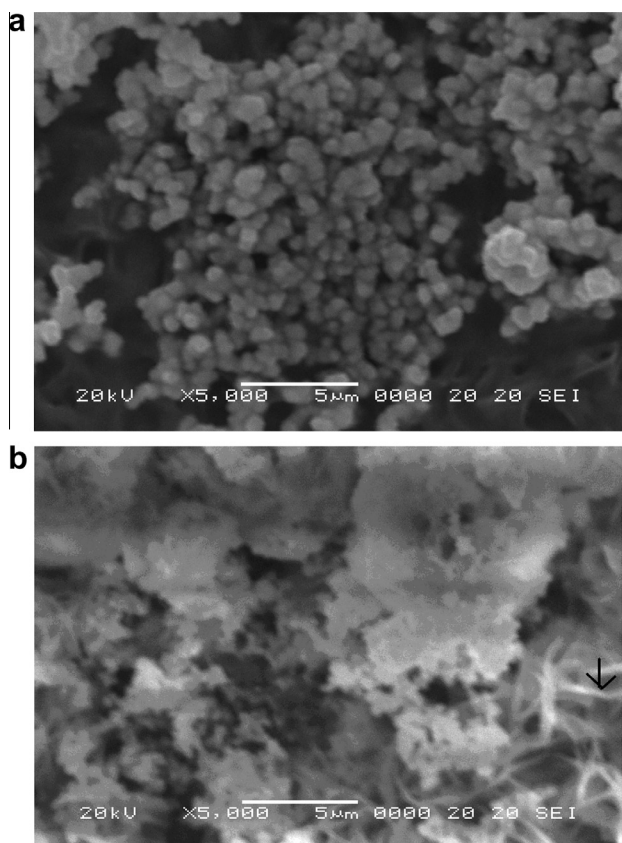


Figure 2 (a) SEM micrographs of Cd (S_{0.7}-Se_{0.3}) film. (b) SEM micrographs of Cd (S_{0.7}-Se_{0.3}):CdCl₂, La films.

comprising of small grains along with some leafy structures are seen. There is some vacant space in fig. (a) between the clusters of non-homogenous spherical grains while fig. (b) shows layered growth along with few fibers. Earlier workers (Bhushan and Shrivastava, 2007) found well developed cabbage structure, which they interpreted in terms of overlapping of different layers formed under continuous growth. Karanjai and Dasgupta (1988) found that CdS grains have greater tendency to coalesce with the increasing concentration of CdCl₂. CdCl₂ is also known to promote recrystallization of CdS grains (Mukherjee and Bhushan, 2003a,b). The average grain size is calculated using Heyne's intercept method (Subba Rao et al., 1972) in which instead of estimating the number of grains per unit area, the grains intercepted by a theoretical line on the specimen surface are counted. In the present case, the average particle size is found to lie between 8.3 and 10.7 nm.

3.1.3. Optical absorption studies

The study of optical absorption is important in understanding the behavior of semiconductor nano crystals. A fundamental property of semiconductors is band-gap energy separation between the filled valence band and the empty conduction band. Optical excitations of electrons across the band-gap are strongly allowed, producing an abrupt increase in absorption at the wavelength corresponding to the band-gap energy.

Results of optical absorption spectra recorded in the wavelength range 340–660 nm and carried out at RT for pure and doped Cd (S–Se) films are presented in Fig. 3. From these

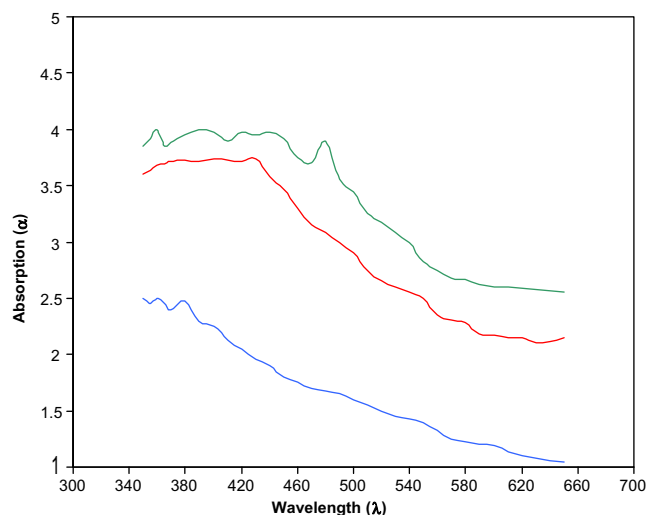


Figure 3 Absorption spectra of different Cd (S_{0.7}-Se_{0.3}) films: — Cd (S_{0.7}-Se_{0.3}); — Cd (S_{0.7}-Se_{0.3}):CdCl₂ (2 ml), — Cd (S_{0.7}-Se_{0.3}):CdCl₂ (2 ml), La (8 ml).

curves it is noticed that absorption is dominant mainly in blue region. As the impurities are added to pure Cd (S_{0.7}-Se_{0.3}) films, absorption peak is blue shifted. This shift is due to the quantization effect according to which the band-gap value increases with the size reduction of crystallites. The band-gap values of these materials determined from the Tauc's plots (Fig. 4) are found as Cd (S_{0.7}-Se_{0.3}) = 2.16 eV; Cd (S_{0.7}-Se_{0.3}):CdCl₂ (2.00 ml) = 2.15 eV; Cd (S_{0.7}-Se_{0.3}):CdCl₂ (2.00 ml), La (8.00 ml) = 2.41 eV. The materials of present study are of direct band-gap nature and a slight increase in band-gap value is noticed in La doped sample, which is due to its different ionic radii as compared to that of Cd where it is substituted.

The absorption coefficient (α) and the band-gap (E_g) are related by the following expression in direct band-gap materials (Dawar et al., 1990):

$$\alpha = C(h\nu - E_g)^{1/2}/h\nu \quad (4)$$

where ' E_g ' is the optical band-gap and ' C ', the speed of light.

3.2. PL spectral studies

The PL spectra of different Cd (S_{0.7}-Se_{0.3}) films at varied concentrations of CdCl₂ and La are presented in Fig. 5. PL emission spectrum of CdS peaks at 516 nm (\sim 2.39 eV), which is quite close to the band-gap (2.43 eV) of pure CdS as observed from the absorption spectrum. Thus, the emission is attributed to edge emission of CdS. Thomas and Hopfield, (1962) correlated this emission to the excitonic transitions involving free excitons. Maximum PL emission is observed for 0.7:0.3 combinations of CdS: CdSe and hence this combination is used for PL studies. In presence of CdCl₂, the highest emission is obtained at a volume of 2.00 ml. Therefore, this concentration is used in the presence of impurities.

The peak positions of different films are listed in Table 3. Cd (S–Se) films with ratio of 0.7:0.3 show two broad emission peaks. The lower wavelength emission is associated to a annihilation of free excitons because of similar excitonic nature of

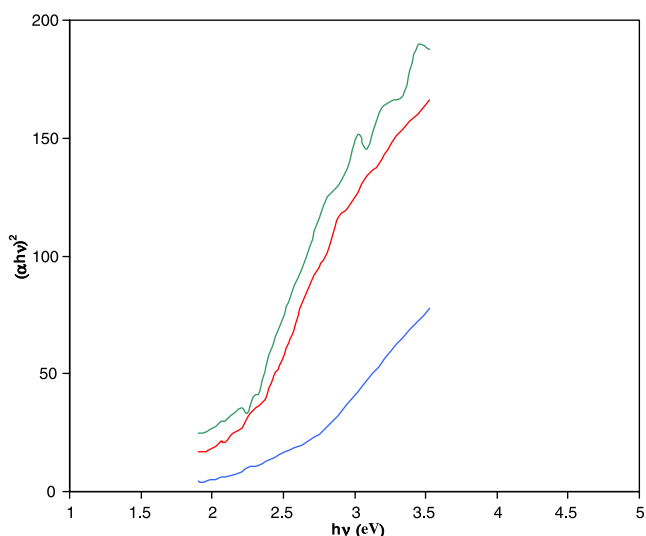


Figure 4 Tauc's plots of different Cd ($S_{0.7}Se_{0.3}$) films: — Cd ($S_{0.7}Se_{0.3}$); — Cd ($S_{0.7}Se_{0.3}$): CdCl₂ (2 ml), — Cd ($S_{0.7}Se_{0.3}$): CdCl₂ (2 ml), La (8 ml).

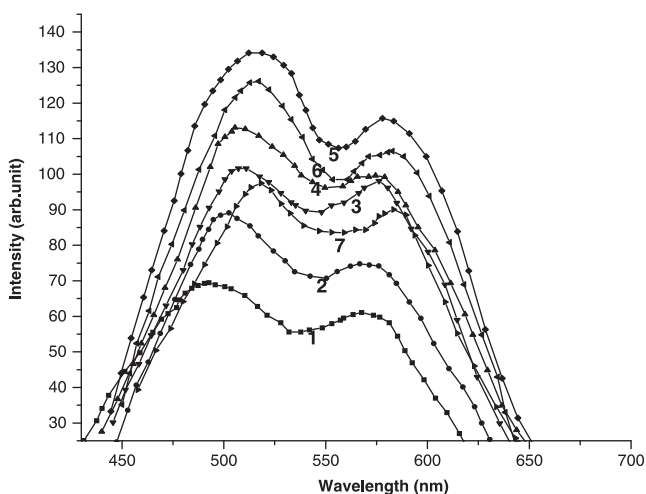


Figure 5 PL emission spectra of different Cd ($S_{0.7}Se_{0.3}$) films: (1) Cd ($S_{0.7}Se_{0.3}$), (2) Cd ($S_{0.7}Se_{0.3}$): CdCl₂ (2 ml), (3) Cd ($S_{0.7}Se_{0.3}$): CdCl₂ (2 ml), La (4 ml), (4) Cd ($S_{0.7}Se_{0.3}$): CdCl₂ (2 ml), La (6 ml), (5) Cd ($S_{0.7}Se_{0.3}$): CdCl₂ (2 ml), La (8 ml), (6) Cd ($S_{0.7}Se_{0.3}$): CdCl₂ (2 ml), La (10 ml), (7) Cd ($S_{0.7}Se_{0.3}$): CdCl₂ (2 ml), La (12 ml).

edge emission of CdS and CdSe (Bhushan and Oudhia, 2009). The broadening of this peak is attributed to the localized excitonic state with a broad distribution of energies (Shevel et al., 1987) along with exciton-lattice coupling and scattering. The second peak is found to shift towards higher wavelength with increasing percentage of Se. This emission is associated to donor–acceptor transitions formed by incorporations of cations (excess Cd in present case) introducing deep acceptor levels (like S vacancies with ionization energy ~ 0.03 eV) and that of anions (S or Se in present case) introducing deep acceptor levels (Cd vacancies with ionization energy typically ~ 1.1 eV for sulphides and ~ 0.6 eV for selenides). Bhushan and Pillai (2008), through EDX measurements have already reported that in such preparation excess CdS exists as compared to its counter part. With increasing concentration of La, these peak positions get shifted towards longer wavelength side. This supports that these emissions are not due to direct transitions in energy bands of La.

However, the origin of emission peaks, in presence of La is related to transitions between the excitonic levels of pure Cd (S–Se) and the energy levels due to La, which cause slight shift in emission peaks. Further improvement in peak intensities is also observed in presence of La, which is due to energy transfer from energy levels of La. Improvement in intensities of emission peaks in presence of CdCl₂ is due to better crystallization in its presence (Mukherjee and Bhushan, 2003a, b).

4. Conclusions

- La doped Cd ($S_{0.7}Se_{0.3}$):CdCl₂ films are prepared through CBD technique, which is a simple and suitable method for obtaining smooth, uniform, high reflecting and strong adherent thin films.
- Sufficiently intense PL spectra are observed in Cd ($S_{0.7}Se_{0.3}$):CdCl₂ films prepared on glass substrate at RT. These samples can be used as a PL device.
- PL emission spectra of chemically deposited Cd ($S_{0.7}Se_{0.3}$), Cd ($S_{0.7}Se_{0.3}$):CdCl₂, La (8 ml) films consist of two peaks, which are related to the transition between excitonic levels and levels due to La. Thus, La can be preferred as dopant to enhance PL intensity.
- SEM studies show layered growth of films while XRD patterns exhibit diffraction lines corresponding to CdS, CdSe and Cd.
- Absorption spectra exhibit direct band-gap nature of material.

Table 3 Peak positions of PL emission spectra of different Cd (S–Se) films at RT (~ 28 °C).

System	Peak positions (nm)	Intensities
Cd ($S_{0.7}Se_{0.3}$)	495, 570	70, 60
Cd ($S_{0.7}Se_{0.3}$):CdCl ₂ (2.00 ml)	504, 573	89, 72
Cd ($S_{0.7}Se_{0.3}$):CdCl ₂ (2.00 ml), La (6.00 ml)	508, 576	102, 98
Cd ($S_{0.7}Se_{0.3}$):CdCl ₂ (2.00 ml), La (8.00 ml)	516, 580	134, 116
Cd ($S_{0.7}Se_{0.3}$):CdCl ₂ (2.00 ml), La (10.00 ml)	515, 581	126, 107
Cd ($S_{0.7}Se_{0.3}$):CdCl ₂ (2.00 ml), La (12.00 ml)	519, 583	97, 91

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