

PHOTOCONDUCTIVITY AND PHOTOLUMINESCENCE STUDIES OF CHEMICALLY DEPOSITED LA DOPED Cd (S-Se) FILMS

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Cd(S-Se):CdCl₂,La films were prepared by chemical deposition method on glass substrates at a temperature 60 °C in a water bath (WB) . Results of Scanning electron microscopy (SEM) and X-ray diffraction (XRD) studies, optical absorption, photoluminescence (PL) emission spectra and photoconductivity (PC), rise and decay studies are reported . SEM studies show ball-type structures along with voids which are related to layered growth. A crystalline nature of the films is observed in the XRD studies. Formation of solid solution of the mixed base is also observed. The values of strain (ϵ), grain size (D) and dislocation density (δ) are evaluated from XRD studies. Optical absorption spectra also show the presence of La in the lattice. From the results of optical absorption spectra, the band gaps are determined. The effect of flux, impurities photo to dark current ratio I_{pc}/I_{dc} is observed in the PC rise and decay studies. The maximum value of $I_{pc}/I_{dc} \sim 10^7$ is obtained for the impurity doped films. In PL, observed two peaks are related to transitions between excitonic levels and levels due to La.

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1. Introduction

The PC and PL studies of CdS type materials are quit important because of their wide technological applications while the PC properties are used as sensitive photoconductor ,IR detector and solar cells, the PL properties are useful in lamp phosphors and display devices.The II–VI binary semiconducting compounds belonging to cadmium chalcogenide family e.g.CdS,CdSe,CdTe etc are considered to be very important materials for wide spectrum ,opto-electronic application ,particularly CdSe having band gap 1.75eV and CdS with band gap 2.42eV make them appropriate candidates 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 [1].The films were prepared by chemical bath deposition technique ,utility of which were already demonstrated by the contribution of Mane and Lokhande[2]and earlier work of Bhushan and coworkers[3,4,5,6]. Rare earth ions when incorporated into a solid show distinct spectral lines of absorption and emission as a result of the electronic transitions within the 4f^N shell configuration[7].The rare-earth ions are characterized by a partially filled 4f shell that is well shielded by 5s² and 5p⁶ orbitals. Although there are no 4f electrons in La [remaining configuration being same].Bhushan and coworkers reported interesting photoconducting properties in La doped CdS and CdPbS films,photocurrent to darkcurrent ratio of 10⁵ were reported in CdS:La films[8]. SEM studies presented crystalline nature of undoped CdS.In presence of NaF layered growth took

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place which finally resulted in cabbage type growth. In presence of La cabbage and crystalline growth both were observed [9]. In (Cd-Pb)S :La photo to dark current ratio 10^6 were observed. The reason for high sensitization were increased in life time as well as carrier concentration. The increase in carrier concentration may be associated to release of valance electrons from the trivalent La [10]. Photoluminescence [PL] were also recorded in La doped [Cd -Pb] S films [11]. Due to such interesting observation on the doped CdS and [Cd-Pb] S this impurity was selected with Cd[S-Se] base for the present work. Results of SEM, XRD, optical absorption, PL emission spectra and PC rise and decay studies are reported.

2.Experimental details

2.1 Film preparation

The Films were prepared on substrates of microscopic glass slides of dimension 24mm \times 75mm. The substrates were cleaned with acetone and distilled water. Such cleaned glass slides were dipped vertically into a mixture of solutions of 1M cadmium acetate, appropriate ratio of thiourea and sodium selenosulphate [Na_2SeSO_3] solution (prepared by heating elemental selenium (99.9% pure) in aqueous solution of sodium sulphite [Na_2SO_3] at 90°C for 5 h), triethanolamine (TEA) and 30% aqueous ammonia. All the chemicals used were of AR grade. All the solutions were prepared in double distilled water. For preparing doped films, calculated proportions of 0.01 M solutions of CdCl_2 and La_2O_3 were added to the original mixture. After the depositions, the films were cleaned by flushing with distilled water and then dried by keeping the samples in open atmosphere at room temperature.

TEA and ammonia solution were used to adjust pH of the reaction mixture and to increase film adherence. To obtain good quality films, time, temperature of deposition and pH of the solution mixture were optimized. The optimum time, temperature and pH were observed to be 1 h, 60°C and about 11 respectively. The thickness of the films was measured by interferometry method and was found to lie between 0.313 μm to 0.349 μm .

2.2 PC and PL cells

For PC studies coplanar electrodes (1.5mm wide and 24mm long at a separation of 1mm) were formed by applying colloidal silver paint to the surface of the film. The photocurrents were measured by exposing the total area of the film. The PL cell consisted of the film deposited on the glass substrates.

2.3 Measuring instruments

The excitation source for PC rise-decay studies is 100W incandescent bulb. The corresponding measurements of the photocurrents were made using a nanometer (DNM-121), and for measurement of voltages, a digital multimeter (Scientific HM 5011-3) was used. The PL excitation was done by 365 nm line of Hg which was obtained from a high pressure mercury vapour lamp filtered by Carl Zeiss interference filter. Light output was detected by an RCA 6217 photomultiplier tube which was operated by a highly regulated power supply (EHT-11 Scientific Equipment, Roorkee). The integrated light output in the form of current was recorded by a sensitive polyflex galvanometer. For spectral studies a prism monochromator was used. XRD and SEM studies were performed at IUC -DAE, Indore using Rigaku RU: H2R Horizontal Rotaflex X-ray machine and JEOL JSM 5600 scanning electron microscope equipped with Oxford Link EDS respectively. The optical absorption spectra were recorded by using a 1700 Pharmespec Shimadzu Spectrophotometer over the wavelength range 300-700 nm.

3. Results and discussion

3.1. SEM studies

The SEM micrograph of $\text{Cd}(\text{S}_{0.95}\text{-Se}_{0.05})$ & $\text{Cd}(\text{S}_{0.7}\text{-Se}_{0.3})$ are presented in fig.1(a)&1(b) respectively. Both consist of large number of smaller grain particles along with some leafy structure. More voids are seen in films of higher Se concentration. SEM micrograph of $\text{Cd}(\text{S}_{0.95}\text{-Se}_{0.05})\text{:CdCl}_2(3\text{ml}),\text{La}(4\text{ml})$ & $\text{Cd}(\text{S}_{0.7}\text{-Se}_{0.3})\text{:CdCl}_2(2\text{ml}),\text{La}(8\text{ml})$ film are shown in figure 1(c)&1(d) respectively. A similar nest type structure was reported by Hosono et al [12] in Zinc oxide. Earlier worker[13] in this laboratory found well developed cabbage structure which they interpreted in terms of layered growth structure forming cabbage due to overlap of different layers formed under continue growth. The bigger structure in the present case also shows overlap of such structures. The scattered leafy structure may also be expected when overlap does not take place. Presence of voids are also seen in such growth.

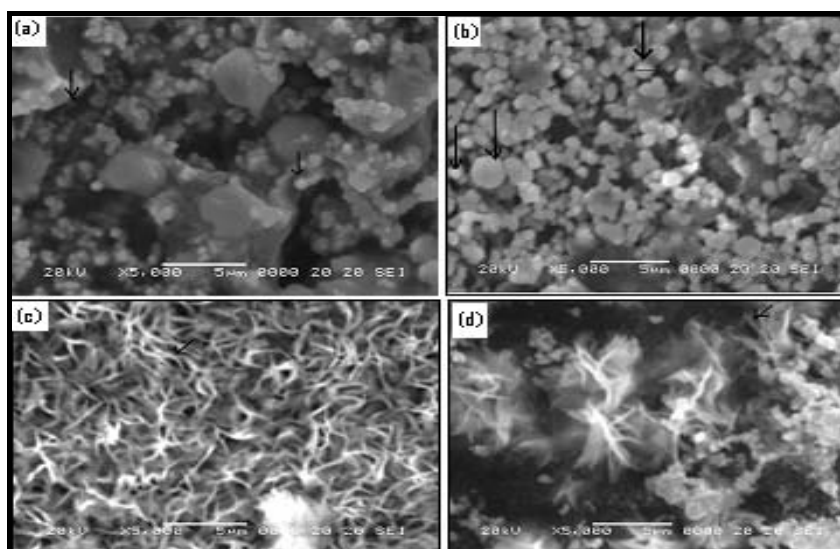


Fig. 1. SEM micrographs of (a) $\text{Cd}(\text{S}_{0.95}\text{-Se}_{0.05})$ film, (b) $\text{Cd}(\text{S}_{0.7}\text{-Se}_{0.3})$ film, (c) $\text{Cd}(\text{S}_{0.95}\text{-Se}_{0.05})\text{:CdCl}_2(3\text{ml}),\text{La}(4\text{ml})$ film and (d) $\text{Cd}(\text{S}_{0.7}\text{-Se}_{0.3})\text{:CdCl}_2(2\text{ml}),\text{La}(8\text{ml})$ film.

XRD studies.

The XRD pattern of $\text{Cd}(\text{S}_{0.95}\text{-Se}_{0.05})$; $\text{Cd}(\text{S}_{0.7}\text{-Se}_{0.3})$; $\text{Cd}(\text{S}_{0.95}\text{-Se}_{0.05})\text{:nCdCl}_2(3\text{ml}),\text{La}(4\text{ml})$; $\text{Cd}(\text{S}_{0.7}\text{-Se}_{0.3})\text{:CdCl}_2(2\text{ml}),\text{La}(8\text{ml})$ are shown in 2(a), (b), (c) and (d), respectively. The corresponding data are presented in table 1. The assignment of peaks were done by comparing with ASTM data and also by determination of lattice constant values and their comparison from reported values. It is observed that peaks corresponding to CdS and CdSe appear, changes in intensity of different peaks along with appearance of new peaks and disappearance of some of the peaks are observed due to change in concentrations of CdS to CdSe and incorporation of impurity. CdS is formed in cubic as well as hexagonal phases. Such a phases arise due to difference in arrangement of atomic layers. The cubic and hexagonal phases arise due to atomic arrangements as ABCABC.....and ABAB....[14] It is also possible to find mixed forms with random stacking of very long period repeats as is found in polytypes of SiC [15]. The total crystal consists of different atomic layers of CdS in cubic as well

as hexagonal phases along with some atomic layers of CdSe in hexagonal phases. According to Langer et al [16], one might think of solid solutions as mixtures of microcrystalline regions of the pure CdSe and CdS, where each microregion might consist of a number of layers.

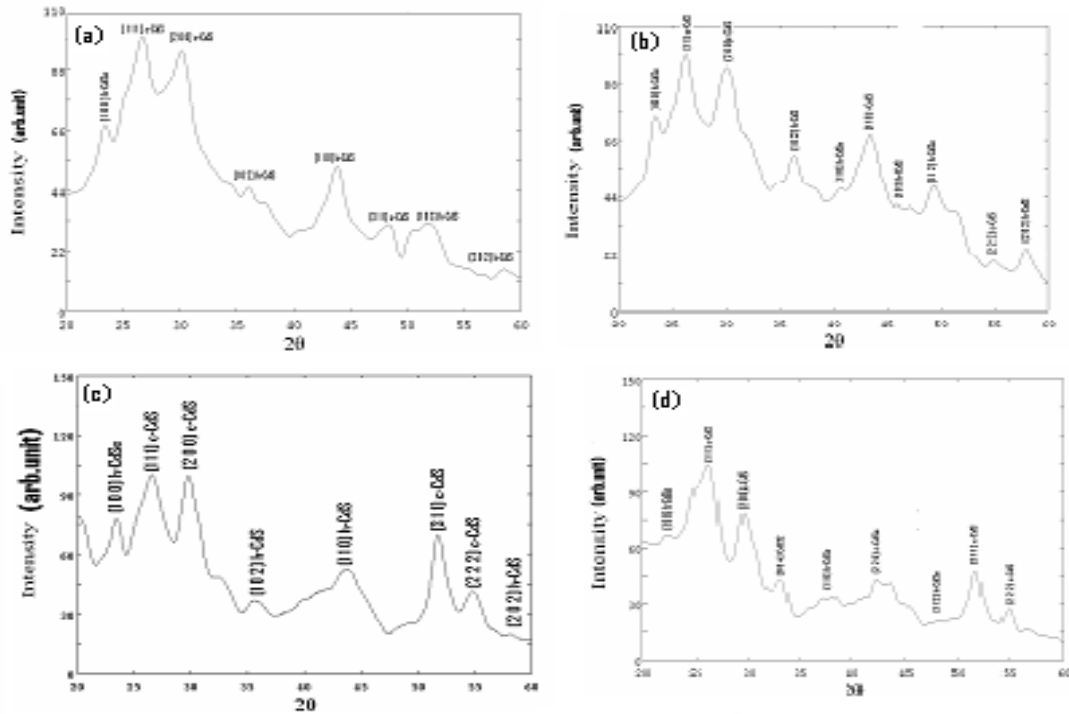


Fig. 2. X-ray diffractograms of (a) $\text{Cd}(\text{S}_{0.95}\text{Se}_{0.05})$ film, (b) $\text{Cd}(\text{S}_{0.7}\text{Se}_{0.3})$ film (c) $\text{Cd}(\text{S}_{0.95}\text{Se}_{0.05}) : \text{CdCl}_2$ (3 ml), La (4 ml) film and (d) $\text{Cd}(\text{S}_{0.7}\text{Se}_{0.3}) : \text{CdCl}_2$ (2 ml), La (8 ml) film.

Table 1 - XRD diffraction spectra of different Cd (S-Se) Film
(Preparation time =60 min; Preparation temperature =60°C).

d value		Intensity		h k l	Lattice constant	
Obs.	Rep.	Obs.	Rep.		Obs.	Rep.
(a) Cd(S _{0.95} -Se _{0.05}) films.						
3.792	3.72	68	100	(100) h-CdSe	a=4.37	a=4.30
3.331	3.36	100	100	(111) c -CdS	a =5.759	a=5.818
2.963	2.90	95	40	(200) c- CdS	a =5.92	a=5.818
2.493	2.45	45	25	(102)h- CdS	a =4.127	a=4.135
2.061	2.06	52	57	(110)h- CdS	a =4.12	a=4.135
1.87	1.89	32	42	(103) h- CdS	a =4.12	a=4.135
1.761	1.753	31	60	(311) c- CdS	a =5.814	a=5.818
1.577	1.581	15	7	(202)h-CdS	a =4.12	a=4.135
(b) Cd(S _{0.7} -Se _{0.3}) films						
3.77	3.72	75	100	(100) h-CdSe	a =4.35	a=4.30
3.39	3.36	100	100	(111) c -CdS	a =5.871	a=5.818
2.96	2.90	94	40	(200) c- CdS	a=5.919	a=5.818
2.46	2.45	60	25	(102)h- CdS	a =4.237	a=4.135
2.21	2.15	32	82.49	(110)h- CdSe	a =4.49	a=4.30
2.08	2.058	47	80	(110)h- CdS	a =4.127	a=4.135
1.92	1.89	67	42	(103)h- CdS	a=4.127	a=4.135
1.84	1.833	40	55.99	(112) h-CdSe	a=4.35	a=4.30
1.67	1.68	48	10	(222) c- CdS	a=5.788	a=5.818
1.59	1.58	49	7	(202) h- CdS	a=4.127	a=4.135
(c) Cd(S _{0.95} -Se _{0.05}): CdCl ₂ (3ml),La(4ml) films.						
3.788	3.72	78	100	(100) h-CdSe	a =4.36	a=4.30
3.343	3.36	100	100	(111) c -CdS	a =5.79	a=5.818
2.99	2.90	99	40	(200) c- CdS	a =5.98	a=5.818
2.515	2.45	37	25	(102)h- CdS	a =.139	a=4.135
2.07	2.06	52	57	(110)h- CdS	a =4.139	a=4.135
1.76	1.753	68	60	(311) c- CdS	a =5.828	a=5.818
1.67	1.68	41	10	(222) c- CdS	a=5.798	a=5.818
1.580	1.581	19	7	(202) h-CdS	a=4.139	a=4.135
(d)Cd(S _{0.7} -Se _{0.3}): CdCl ₂ (2ml),La(8ml) films.						
3.98	3.72	65	100	(100) h-CdSe	a=4.59	a=4.30
3.38	3.36	100	100	(111) c -CdS	a=5.854	a=5.818
3.01	2.90	75.5	40	(200)c- CdS	a=5.98	a=5.818
2.697	2.648	42	90	(004)CdCl ₂	-----	a=3.851
2.31	2.15	33	82.49	(110)h- CdSe	a=4.61	a=4.30
2.052	2.058	41	80	(220) c- CdS	a=5.803	a=5.818
1.88	1.833	20	50	(112) h-CdSe	a=4.61	a=4.30
1.764	1.753	45	60	(311)c- CdS	a=5.855	a=5.818
1.667	1.68	26	10	(222)c-CdS	a=5.775	a=5.818

Table 2-Values of Particle size, Strain and Dislocation density for Cd (S-Se): CdCl₂, La films (S_{0.95}Se_{0.05}):CdCl₂(3ml),Gd(4ml)Film

Sample	Particle size D(nm)	Strain ϵ (lin ⁻² m ⁻⁴)	Dislocation density(x10 ¹⁵ lin/m ²)
Cd(S _{0.95} -Se _{0.05}):CdCl ₂ (3ml),La(4ml)	4.28	.00843	54.61
Cd(S _{0.7} -Se _{0.3}):CdCl ₂ (2ml),La(8ml) film	3.39	.0106	87.03

It is observed that the average particle size of the different sample lie in the range 5.125 nm-7.123 nm. The shift in spectrum reported earlier may also be due to nano crystalline effect in addition to other effect mentioned earlier. The strain value ϵ [17] and dislocation density δ [18] were obtained for (111)c peak of CdS by using the well known formulae and corresponding values are listed in table-2.

3.3.Optical absorption Studies

The optical absorption measurements of the undoped and doped Cd(S-Se) films were carried out at room temperature by placing an uncoated identical glass substrate in the reference beam. The optical spectra of the films were recorded in the wavelength range 300–700 nm. The energy band gaps of the materials in these films were determined from the absorption spectra. The optical absorption spectra of different Cd(S_{0.95}-Se_{0.05}), Cd(S_{0.7}-Se_{0.3}) films are shown in fig 3(a),(b)respectively. Shift in absorption ages are observed in the both cases. No other major changes are observed. Since for direct band gap materials the absorption coefficient ' α ' and the band gap ' E_g ' are related by [19]

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

Thus from the extrapolation of non-linear plot between $(\alpha h\nu)^2$ & $h\nu$ (Tauc's plot) gives the band gap value ' E_g ' of the corresponding material. From such plots the band gaps are found as Cd(S_{0.95}-Se_{0.05}) = 2.33 eV; Cd(S_{0.95}-Se_{0.05}): CdCl₂(3ml) = 2.35 eV; Cd(S_{0.95}-Se_{0.05}):CdCl₂(3ml),La(4ml) = 2.36 eV; Cd(S_{0.7}-Se_{0.3}) = 2.14 eV; Cd(S_{0.7}-Se_{0.3}): CdCl₂ (2ml) = 2.13 eV; Cd(S_{0.7}-Se_{0.3}): CdCl₂(2ml),La (8ml) = 2.16 eV. The band gap of CdS is known to be 2.4 eV and that of CdSe 1.7 eV [20], thus as expected the band gaps reduced due to increasing concentration of Se. A Common lattice of Cd (S-Se) is formed whose band gap lies in between the two values. A slight increase in band gap is observed due to presence of La which may be different ionic radius as compared to that of Cd in place of which it is substituted.

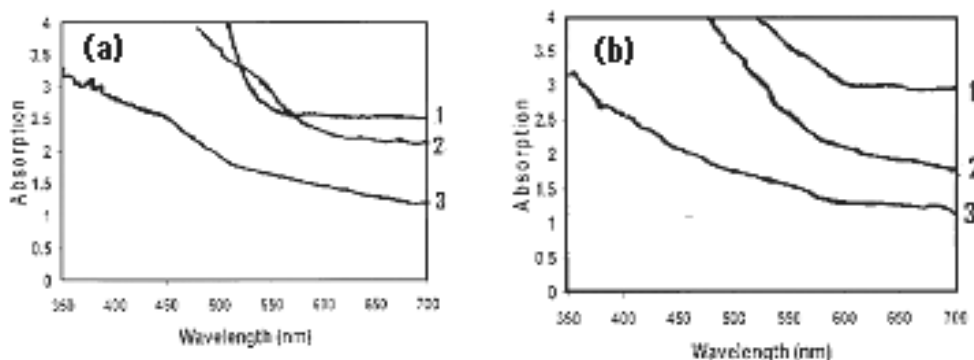


Fig. 3. Absorption spectra of (a) different Cd (S_{0.95}-Se_{0.05}) films. 1. Cd (S_{0.95}-Se_{0.05}); 2. Cd (S_{0.95}-Se_{0.05}): CdCl₂(3ml), 3. Cd (S_{0.95}-Se_{0.05}): CdCl₂(3ml),La (4ml) (b) different Cd (S_{0.7}-Se_{0.3}) films. 1. Cd (S_{0.7}-Se_{0.3}); 2. Cd (S_{0.7}-Se_{0.3}): CdCl₂(2ml), 3. Cd (S_{0.7}-Se_{0.3}): CdCl₂(2ml), La (8ml)

3.4. PL spectra

PL emission spectrum of CdS was found to consist of a peak at 516nm. This emission is quite close to the band gap (2.43 eV) as observed from the absorption spectrum. Thus, the emission may be due to edge emission of CdS. Thomas and Hopfield [21] attributed this emission to the excitonic transition involving free excitons. Maximum PL emission was observed for 0.7:0.3 combination of CdS:CdSe and hence this combination was used for PL studies. In presence of CdCl₂ the highest emission was found at a volume of 2ml. Therefore this concentration was used in the presence of impurities. Plot of PL intensities & wavelength for different Cd(S-Se) films are shown in fig 4. The peak position of different films are presented in table 3. Cd(S-Se) of 0.7:0.3 films shows two broad emission peaks. The lower wavelength emission is associated to a annihilation of free excitons because of similar excitonic nature of edge emission of CdS and CdSe[22]. The broadening of this peak can be attributed to the localized excitonic state with a broad distribution of energies[23]alongwith exciton-lattice coupling and scattering. The second peak was found to shift towards higher wavelength with increasing percentage of Se. This emission can be associated with donar –accepter transitions formed by incorporations of cations (excess Cd in present case) introducing deep acceptor levels (like S vacancies with ionization energy ~0.03eV) 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). Existence of excess Cd in such preparation was already confirm by EDX measurements[24].

Table 3-Peak position of PL emission spectra of different Cd(S-Se) films

System	Peak position (nm)	Intensities
Cd(S _{0.7} -Se _{0.3})	500,570	55, 49
Cd(S _{0.7} -Se _{0.3}): CdCl ₂ (2ml)	500 ,570	80 , 63
Cd(S _{0.7} -Se _{0.3}): CdCl ₂ (2ml),La (4ml)	505 ,570	85 , 64
Cd(S _{0.7} -Se _{0.3}): CdCl ₂ (2ml),La (6ml)	507 ,575	89 , 72
Cd(S _{0.7} -Se _{0.3}): CdCl ₂ (2ml),La (8ml)	510 ,577	115 , 100
Cd(S _{0.7} -Se _{0.3}): CdCl ₂ (2ml),La (10ml)	512 ,579	107 , 93
Cd(S _{0.7} -Se _{0.3}): Cd Cl ₂ (2ml), La (12ml)	514 ,583	101 , 91

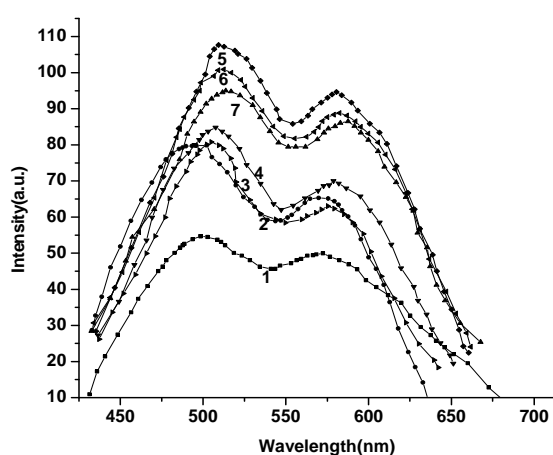


Fig. 4. PL emission spectra of different Cd(S_{0.7}-Se_{0.3}) films:

1. Cd (S_{0.7}-Se_{0.3}): 2. Cd (S_{0.7}-Se_{0.3}): CdCl₂ (2ml),3. Cd (S_{0.7}-Se_{0.3}): CdCl₂ (2ml), La (4ml) 4. Cd (S_{0.7}-Se_{0.3}): CdCl₂ (2ml), La (6ml) 5. Cd (S_{0.7}-Se_{0.3}): CdCl₂ (2ml), La (8ml) 6. Cd (S_{0.7}-Se_{0.3}): CdCl₂ (2ml), La (10ml) 7. Cd (S_{0.7}-Se_{0.3}): CdCl₂ (2ml), La (12ml).

In presence of La, these peak positions are found to be shifted on longer wavelength side on increasing concentration of La. This shows these emission are not due to direct transitions in energy band of La. However, the origin of emission peaks in presence of La may be related to transitions between the excitonic levels of undoped Cd(S-Se) and the levels due to La, which produce slight shift in emission peaks. Further improvement in peak intensities are also observed in presence of La, which may be due to energy transfer from levels of La. Improvement in intensities of emission peaks in presence of CdCl₂ may be due to better crystallization in its presence.[25]

3.5.PC studies

Fig. 5 shows the rise and decay curves of different Cd(S-Se) films. Under visible excitation maximum photocurrent was found for the composition Cd(S_{0.95}-Se_{0.05}). In presence of CdCl₂, after varying its volume, maximum photocurrent was found at 3ml. Earlier worker found the role of CdCl₂ as improving crystallinity and as flux [25] because in its absence rare earth impurities were not that effective. Showing maximum photocurrent at a particular concentration shows that probably excess Cd may be incorporated in the lattices which help in increase in photocurrent. After a particular concentration, concentration quenching may take place.

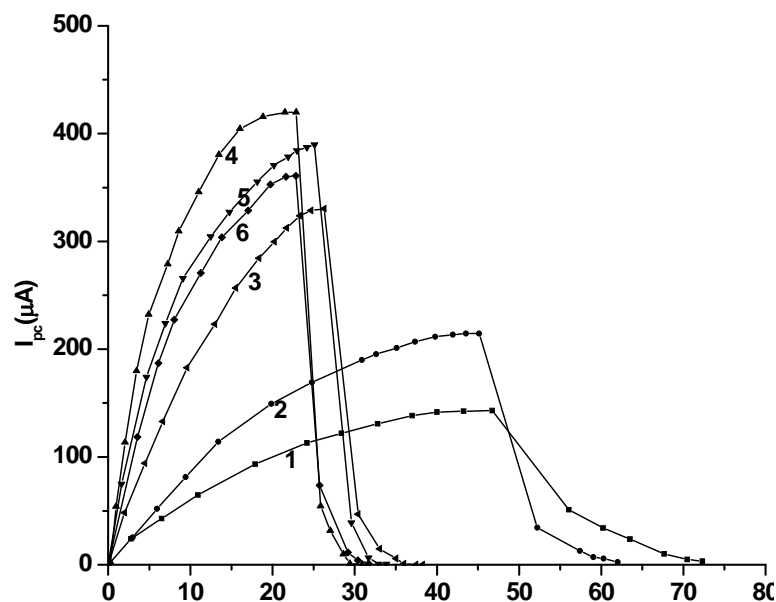


Fig. 5. rise and decay curves of different Cd(S-Se) films

1. Cd (S_{0.95}-Se_{0.05})
2. Cd (S_{0.95}-Se_{0.05}):CdCl₂ (3ml),
3. Cd (S_{0.95}-Se_{0.05}): CdCl₂ {3ml}, La (2ml)
4. Cd (S_{0.95}-Se_{0.05}): CdCl₂ {3ml}, La (4ml),
5. Cd (S_{0.95}-Se_{0.05}):CdCl₂ {3ml}, La (6ml)
6. Cd (S_{0.95}-Se_{0.05}):CdCl₂ {3ml}, La (8ml)

It should be noted that excess Cd is already produced in such preparation. Such observation was already confirmed by EDAX measurements. Impurity La shows a maximum photocurrent at 8ml volume. This obviously comes from release of triple valence electron from La. The general nature of the curve shows a rapid rise of photocurrent in the early part, which is followed by a slower rise finally leading to a saturation of photocurrent. The early part of rise is due to generation of carriers which is followed by recombination due to which the rate of increase is lowered. Finally due to balance between two process saturation appears, further initial part of decay curves consist of fast decreases followed by very low decrease.

Table 4. Values of dark current (I_{dc}), photocurrent (I_{pc}) and the ratio (I_{pc}/I_{dc}) for different Cd(S_{0.95}-Se_{0.05}) films.

System	I_{dc} (nA)	I_{pc} (μ A)	I_{pc}/I_{dc}	LifeTime (τ) Sec	Mobility cm^2/Vs	Trap Depth	
						E1	E2
Cd(S _{0.95} -Se _{0.05})	0.17	143	8.4×10^4	18.6	30.1	0.709	0.711
Cd(S _{0.95} -Se _{0.05}): CdCl ₂ (3ml)	0.1	214	2.14×10^6	32.1	44.4	0.727	0.737
Cd(S _{0.95} -Se _{0.05}):	0.05	370	0.74×10^7	65.5	75.3	0.841	0.840
Cd(S _{0.95} -Se _{0.05}):	0.04	420	1.05×10^7	84.5	82.8	0.879	0.874
Cd(S _{0.95} -Se _{0.05}):	0.06	400	0.66×10^7	60.8	72.3	0.801	0.832
Cd(S _{0.95} -Se _{0.05}):	0.05	360	0.72×10^7	74.7	64.2	0.834	0.841

Fast decreases is related to recombination effect after switching off the source whereas slow variation is related to a slow release of trapped electron from traps formed in Cd(S-Se) system. The values of dark current (I_{dc}), the photocurrent (I_{pc}) and their ratio (I_{pc}/I_{dc}) are summarized in table 4, from where it can be observed that maximum value of (I_{pc}/I_{dc}) is 8.4×10^4 which improves to 2.14×10^6 in presence of CdCl₂ which finally increases to maximum value 1.05×10^7 in presence of La. Reasons for improvement are already discussed earlier. The values of lifetime mobility and trap depth are obtained by method described earlier (10) and such values are also listed in table 4. It is noticed that lifetime of carriers improves in presence of impurities which is again a cause for light photo sensitization.

4. Conclusions

High photosensitive material can be prepared by chemical deposition of Cd(S-Se) films doped with rare earths. SEM studies shows layered growth of films. XRD pattern shows diffraction lines due to CdS, CdSe and CdCl₂. From absorption spectra direct band gap nature is found along with band gap value equal to 2.35eV to 2.15 eV for corresponding to increasing proportion of Se in Cd(S-Se) films. PL emission spectra of chemically deposited Cd (S_{0.7}-Se_{0.3}), Cd (S_{0.7}-Se_{0.3}): CdCl₂, La (8ml) films consists of two peaks which are related to the transition between excitonic levels and levels due to La. The life time and mobility determined from the decay curve and the I_{pc}/I_{dc} ratio are found to increase under doping condition.

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