

**“Growth and Characterization of $\text{Cd}_x\text{Pb}_{(1-x)}\text{Se}$
Ternary Compound Thin Films.”**

Minor Research Project

47 – 1275/09



Principal Investigator



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NEW DELHI – 110 002.

**Annual/Final Report of the work done on the Major/Minor Research Project.
(Report to be submitted within 6 weeks after completion of each year).**

1. Project report No. 1st / 2nd / 3rd / final : Final Report.
2. UGC Reference No : 47 – 1275/09 dated 17/11/2009
3. Period of report: from : 20/03/2010 to 20/03/2012
4. Title of research project: “Growth and Characterization of Cd_xPb_(1-x)Se Ternary Compound Thin Films.”
5. (a) Name of the Principal Investigator: Dr Umeshkumar Prabhakar Rao Khairnar
(b) Deptt. and University/College where work has progressed: Department of Physics, S.S.V.P.S. Dhule’s Arts, Commerce and Science College, Shindkheda.
6. Effective date of starting of the project : 20/03/2010.
7. Grant approved and expenditure incurred during the period of the report:
 - a. Total amount approved :Rs. 1, 55, 000.
 - i) 1st Installment :Rs. 1, 22, 500.
 - b. Total expenditure :Rs. 1, 58, 236.

Report of the work done: (Please attach a separate sheet)

- (i). Brief objective of the project:-
 - (a) To study Cd_xPb_(1-x)Se Ternary compound semiconductor thin films as wide and low band gap materials using vacuum evaporation technique.
 - (b) To study their structural, optical and electrical properties.
 - (c) The main aim in doing this study to explore the possibility of developing this technique for the growth of device grade films and in particular for application to thin film photoelectrochemical solar cell.

- (ii) Work done so far and results achieved and publications, if any, resulting from the work (Give details of the papers and names of the journals in which it has been published or accepted for publication)

To start-with preparation of ternary semiconductors $Cd_xPb_{(1-x)}Se$ the constituent compounds CdSe, and PbSe have been taken in molecular stoichiometry proportional weights and crushed mixed homogeneously compositions 'x' were deposited on to the amorphous precleaned glass substrates at the desired substrate temperature by physical evaporations. A number of trials were taken for optimizing deposition parameters of each type of sets. All the samples are deposited under the similar optimized condition. These samples were then used for characterizations.

- 1) **Substrate cleaning:** - In general, to obtain a good quality of the films with respect to uniformity, durability and adherent the very basic requirement is that the substrate surface must be free from contaminants such as dust particles, absorbed water or gas molecules and greases etc. The gross contaminants are first removed by detergent solution. These slides were cleaned with redistill water and then agitated in ultrasonic cleaner for about 20 minutes. The slides were then rinsed thoroughly several times in redistilled water and latter subjected to vapour degreaser containing pure alcohol. A.R. Grade isopropyl alcohol was used for this purpose. These clean microscopic glass slides dimension 75mmx25mmx1mm (Blue star make) were used as substrates for the film deposition.
- 2) **Heating sources:** - The molybdenum boats of maximum current carrying capacity of 200 ampere were used as heating sources.
- 3) **Substrate temperature:** - During the deposition of thin films of $Cd_xPb_{(1-x)}Se$ substrate temperature was kept at 300 K for all the sets of samples.
- 4) **Working pressure:** - All films were deposited under the similar condition of vacuum. The pressure was 10^{-5} mbar.
- 5) **Power used:** - Electrical power was supplied for heating sources using 20 Volts and 200-Ampere transformer.
- 6) **Deposition rate:** - For the preparation of Ternary-evaporated films the deposition rate was optimized and kept about 15-20 Å/sec.

- 7) **Thickness measurement:** - The thickness of the film was measured during deposition by Quartz crystal thickness monitor Model No. DTM-101 Hi-Tech instruments, Bangalore. The thicknesses of samples are crosschecked using Tolansky's technique. Film thickness found correct within $\pm 75 \text{ \AA}$.
- 8) **Source to substrate distance:** - Source to substrate distance was kept constant throughout the preparation of complete set of films and it was 15 cm.
- 9) **Sample preparation:** -

For the preparation of ternary semiconductors, $\text{Cd}_x\text{Pb}_{(1-x)}\text{Se}$ the constituent compounds CdSe and PbSe have been taken in molecular stoichiometry proportional weights and crushed and mixed homogeneously. The different sets of samples of varying compositions ($x = 0.1$ to 0.9) were deposited onto the amorphous pre-cleaned glass substrates at the temperature of 300 K. All the samples are deposited under the similar optimized condition. These samples were annealed at reduced pressure of 0.1 micron for the duration of 3 hours at the temperature of 403 K and maintained carefully.

These samples were then used for characterization by transmission and reflection spectra, XRD for structural characteristics.

CHARACTERIZATION OF $\text{Cd}_x\text{Pb}_{(1-x)}\text{Se}$ THIN FILMS.

STRUCTURAL STUDIES

XRD diffractograms (Rigaku, Miniflex, Japan) with $\text{CuK}\alpha$ radiation (1.5418 \AA) were recorded for all samples of various compositions and are presented in Fig- 1 - 2 and Scanning electron micrographs (JEOL JSM 6360 [LA]) are presented in Fig- 3 - 11.

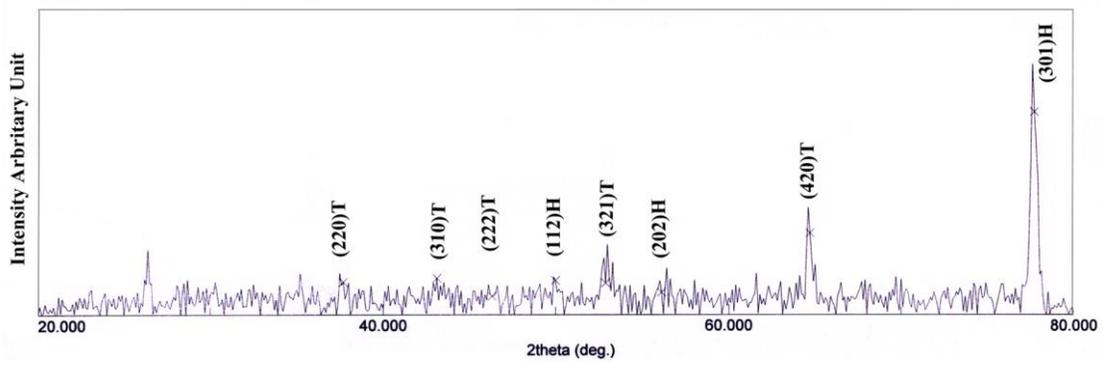


Fig - 1 X-Ray Diffractogram of $Cd_xPb_{(1-x)}Se$ Thin Films ($x = 0.1$) ($d = 1400 \text{ \AA}$)

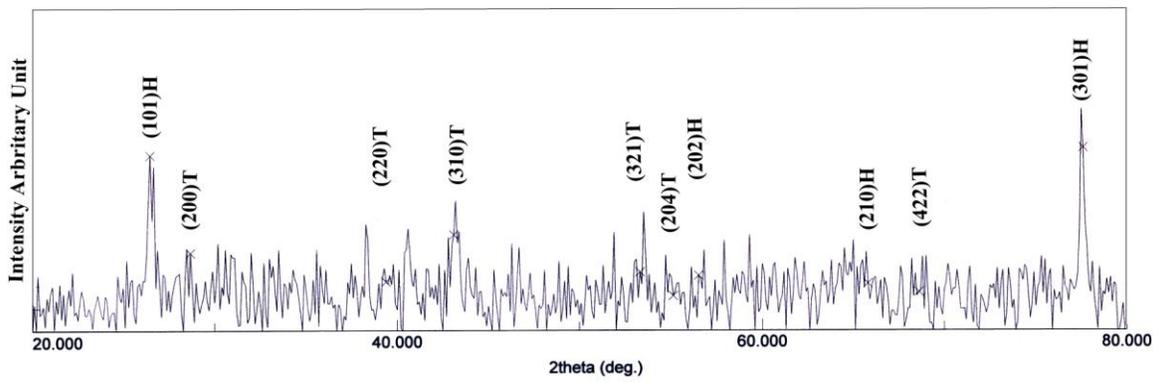


Fig - 2 X-Ray Diffractogram of $Cd_xPb_{(1-x)}Se$ Thin Film ($x = 0.2$) ($d = 1250 \text{ \AA}$)

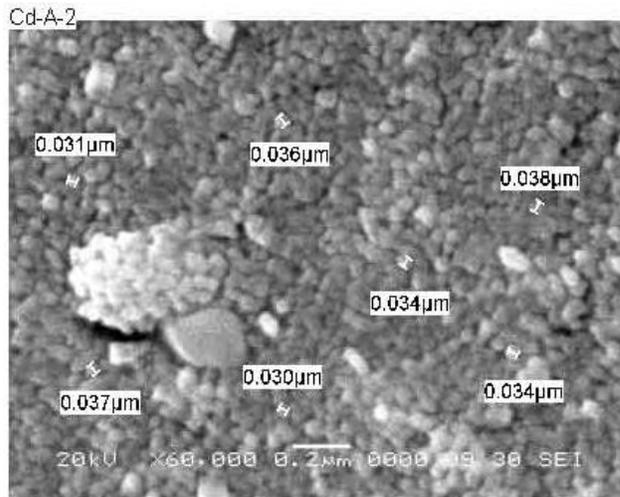


Fig-3 Scanning Electron Micrograph for $x = 0.1$ composition

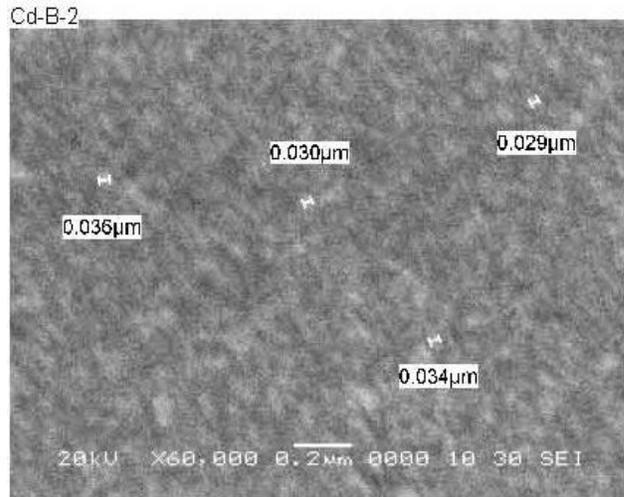


Fig-4 Scanning Electron Micrograph for $x = 0.2$ composition

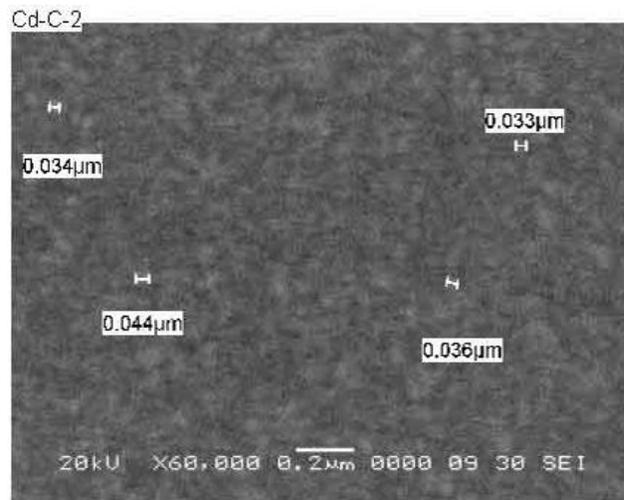


Fig-5 Scanning Electron Micrograph for $x = 0.3$ composition

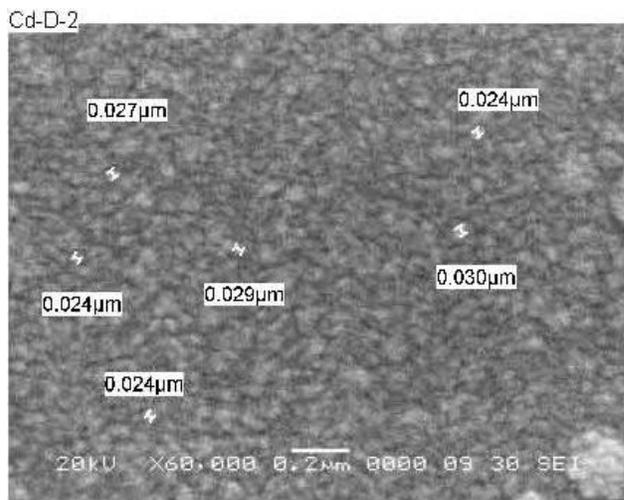


Fig-6 Scanning Electron Micrograph for $x = 0.4$ composition

Cd-E-2

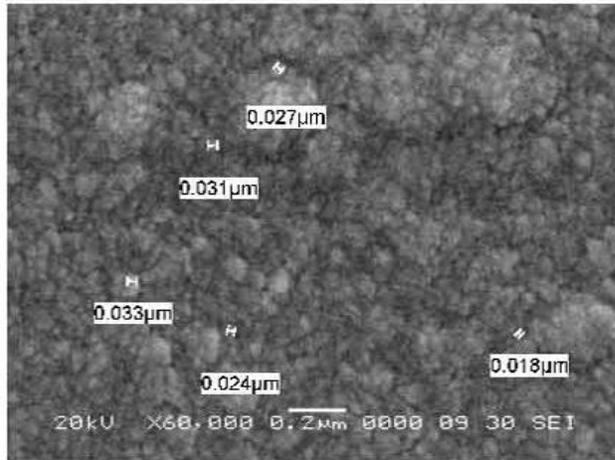


Fig-7 Scanning Electron Micrograph for $x = 0.5$ composition

Cd6-2

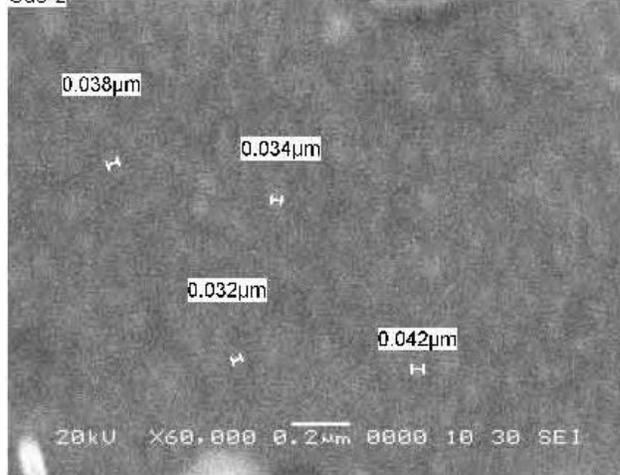


Fig-8 Scanning Electron Micrograph for $x = 0.6$ composition

Cd7-2

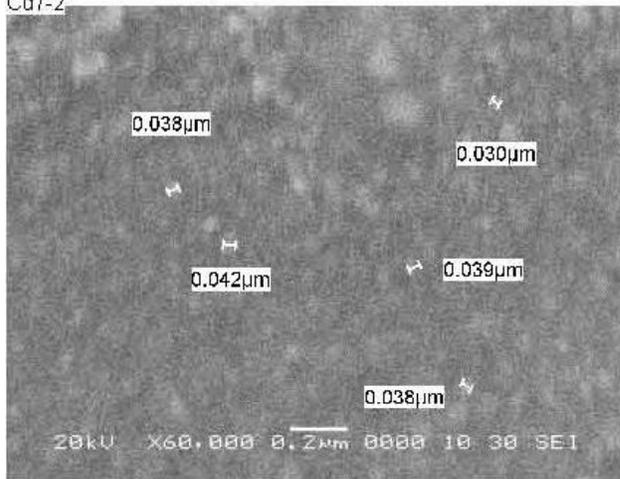


Fig-9 Scanning Electron Micrograph for $x = 0.7$ composition.

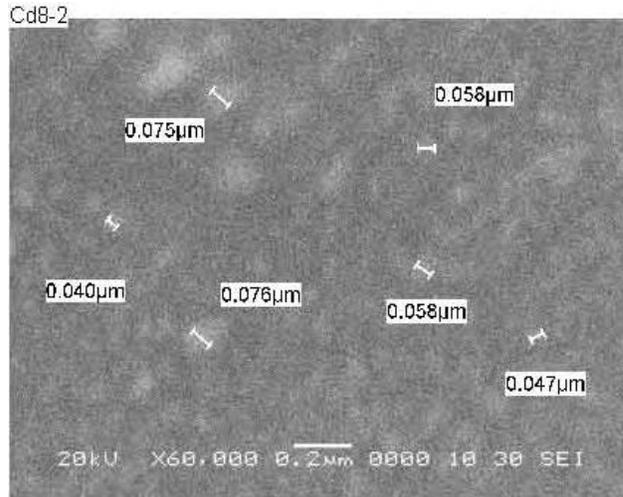


Fig-10 Scanning Electron Micrograph for $x = 0.8$ composition

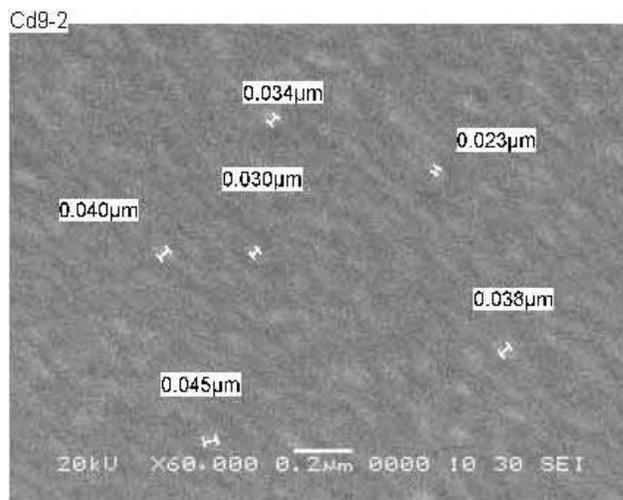


Fig-11 Scanning Electron Micrograph for $x = 0.9$ composition

OPTICAL MEASUREMENTS

Transmittance and reflectance of all the samples of various compositions of $\text{Cd}_x\text{Pb}_{(1-x)}\text{Se}$ have been measured as a function of the wavelength over the wavelength range 200 – 2500 nm, at normal and near normal incidence respectively using (JASCO MODEL V-670) spectrophotometer. These spectra are presented in Fig- 12 - 13. These spectra exhibit characteristic features described as below.

For all compositions i) transmittance and reflectance curves are oscillatory, sinusoidal nature. ii) They are complementary i. e. one increases as a function of wavelength other decreases.

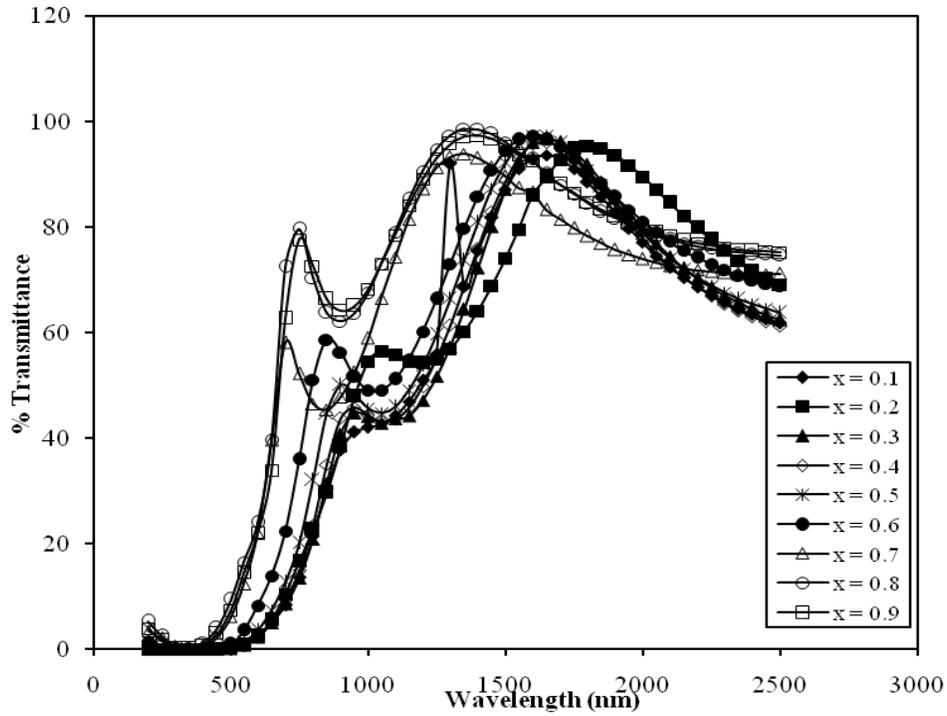


Fig-12 Spectral behaviour of transmittance with wavelength of $Cd_xPb_{(1-x)}Se$ thin films.

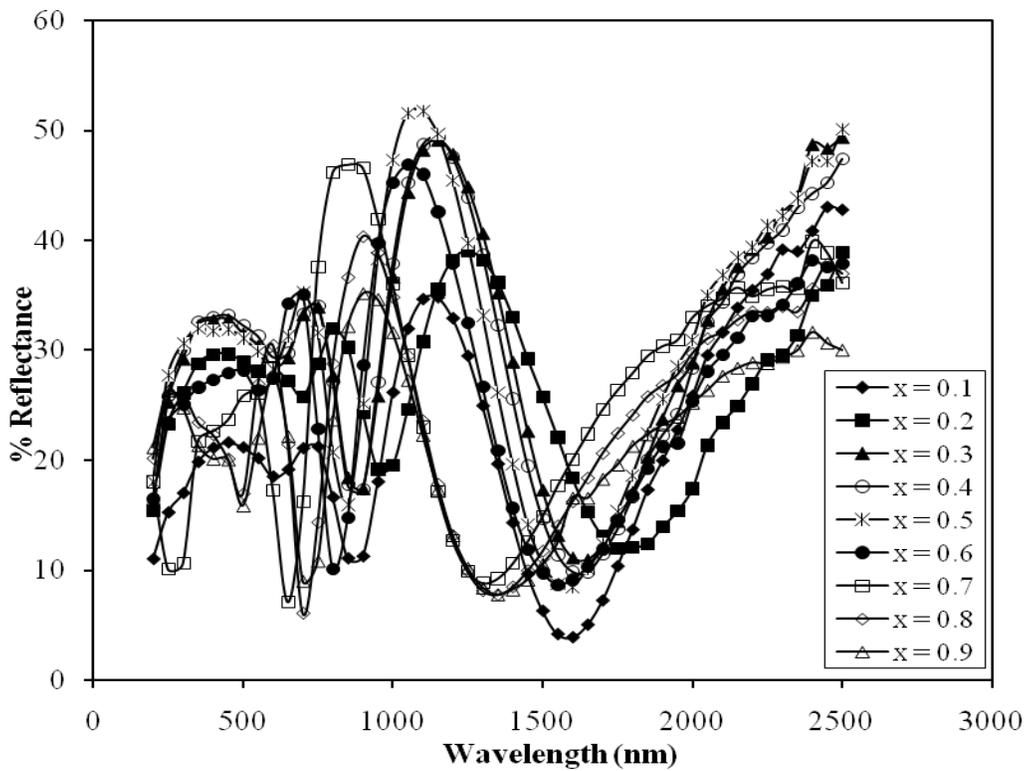


Fig-13 Spectral behaviour of reflectance with wavelength of $Cd_xPb_{(1-x)}Se$ thin films.

TRANSPORT PROPERTIES

Resistivity of all the samples was measured by four-probe technique as a function of compositions and temperature. The variations of resistivity as a function of compositions are presented in Fig-14. The plots of log of resistivity Vs reciprocal of temperatures ($\log \rho$ Vs $1/T$) have been plotted for the samples ($x=0.1$ and $x=0.9$) and are presented in Fig- 15 – 16.

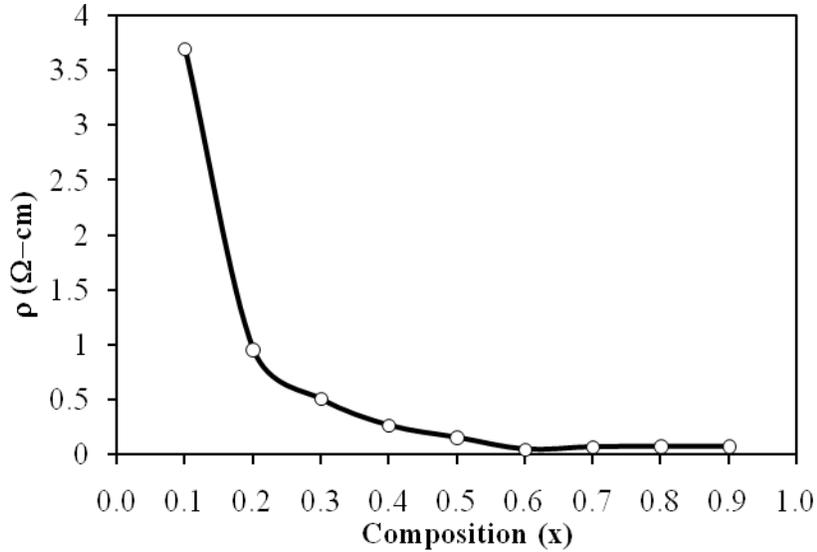


Fig-14 Variation of Resistivity with composition (x) of $\text{Cd}_x\text{Pb}_{(1-x)}\text{Se}$ thin films.

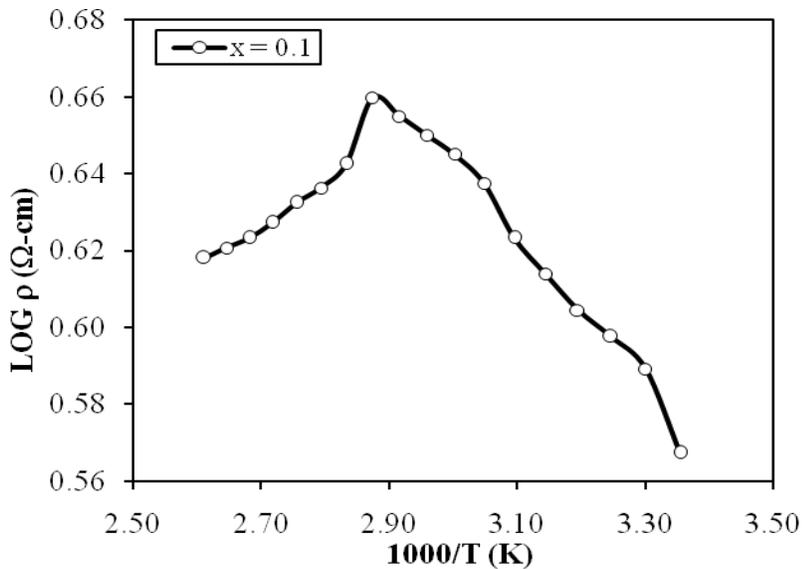


Fig -15 Plot of Reciprocal of Temp. Vs $\text{LOG } \rho$ for $\text{Cd}_x\text{Pb}_{(1-x)}\text{Se}$ ($x=0.1$) thin film.

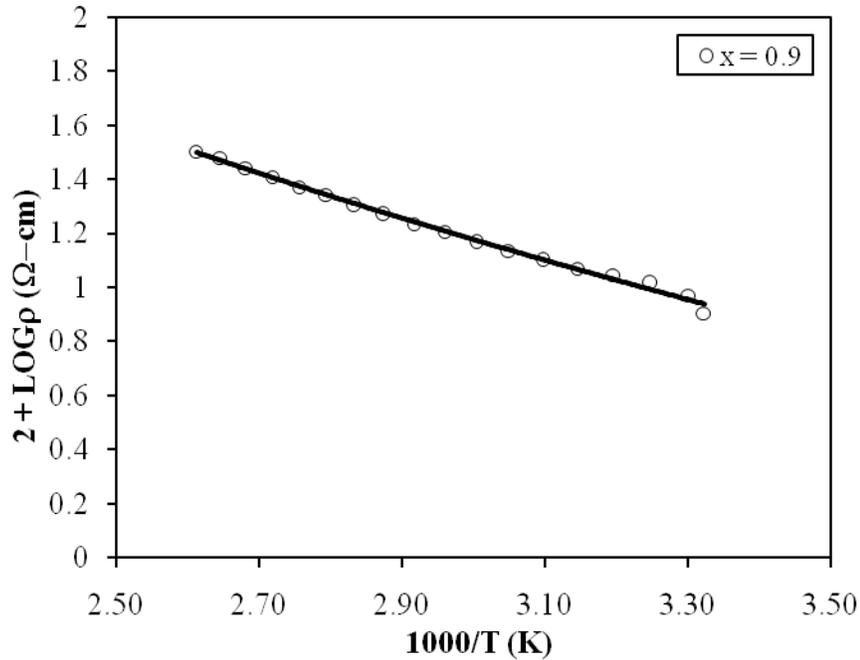


Fig -16 Plot of Reciprocal of Temp. Vs LOG ρ for Cd_xPb_(1-x)Se (x=0.9) thin film.

RESULTS AND DISCUSSION:

STRUCTURAL INFORMATION

Diffraction analysis suggests that all the samples of various compositions are polycrystalline nature. However the characteristics peak of hexagonal CdSe and tetragonal PbSe changes their angular position and relative intensities for different compositions suggesting multi phases and inhomogeneity in the growth of films. The samples of compositions (x = 0.1, 0.2, 0.4, 0.5, 0.7, 0.8 and 0.9) exhibit predominant diffraction lines corresponding to (301) plane of CdSe (H) may be attributed to the characteristics growth with (301) reflecting plane as a preferred orientation. While the sample (x =0.3 and 0.6) exhibits predominant diffraction lines corresponding to (222) and (220) plane of PbSe (T) is again attributed to the characteristics growth with the (222) and (220) reflecting plane as preferred orientation. The shifting of peak positions of these prominent diffraction lines suggests the formation of solid solution corresponding to Cd_xPb_(1-x)Se material from the basic starting compounds CdSe and PbSe.

Diffraction analysis indicates that Cd_xPb_(1-x)Se films are polycrystalline and grain sizes determined by using Scherrer formula [1]

$$C_s = K\lambda/B\cos\theta \quad \dots\dots\dots (1)$$

Where the constant K is a shape factor usually $\cong 1$, λ is the wavelength of the X-ray (1.5418 \AA), θ is the Bragg's angle and B is the corrected FWHM. The range of crystallite size estimated for composite thin films ($x = 0.1$ to 0.9) samples are 115.74 \AA to 160.81 \AA respectively. The crystallite size is by definition, measured in direction normal to the reflection plane (301), i.e. in the $\langle 301 \rangle$ direction and consequently perpendicular to the substrate [1, 2, 3].

Surface topography-

Scanning electron microscopy (SEM) is for surface topography of the films. The particles are nearly spherical in nature. The films therefore are expected to be porous in nature having larger surface to volume ratio. Such nanostructure films could give high photoresponse.

Fig-3 Shows the SEM image of $x = 0.1$ composite thin film. It is clear from figure that the film is nanostructure. The particle size of film was measured at seven different locations, as indicated on image. The average particle size was calculated to be 37 nm .

Fig-11 Shows the SEM image of $x = 0.9$ composite thin film. It is clear from figure that the film is nanostructure with elliptical shaped particles. The particle size of film was measured at six different locations, as indicated on image. The average particle size was calculated to be 35 nm . This may be relatively smaller growth rate of grains. Atomic radius of Pb is larger (1.21 \AA) as compared to Cd (0.97 \AA). Pb [4] may have smaller mobility as compared to Cd and it would therefore act as nuclear centers to situate the material around.

The composition of starting basic ingredients and film composition comparison is presented in the table No.-1 and expressed in atomic percentages. The atomic percentage of basic ingredient seems to be in agreements with that obtain from EDAX analysis. From this table it is remarkable point to note that for composition ($x = 0.5$) the atomic percentage of basic ingredient taken is very close to atomic percentage obtain from EDAX spectra.

Table No- 1 EDAX data for Cd_xPb_(1-x)Se composite thin films.

Basic Ingredient Taken				EDAX Composition		
Composition 'x'	At% Cd	At% Pb	At% Se	At% Cd	At% Pb	At% Se
0.1	5	45	50	8.13	48.93	42.94
0.2	10	40	50	20.86	35.41	43.73
0.3	15	35	50	21.41	35.09	43.50
0.4	20	30	50	23.57	29.48	46.95
0.5	25	25	50	27.91	25.41	46.69
0.6	30	20	50	32.85	20.43	46.72
0.7	35	15	50	37.87	16.48	45.65
0.8	40	10	50	42.77	9.76	47.46
0.9	45	5	50	42.64	6.84	50.52

OPTICAL PROPERTIES

Determination of Optical Band Gap –

Using the transmission data absorption coefficient ' α ' has been calculated and curves of $(\alpha h\nu)^2$ Vs $h\nu$ have been plotted. These curves shows clearly linear dependence for the value of $p=1/2$. This clearly indicates that the optical transitions are allowed and direct band gap. The evaluated band gap energies are 2.05, 2.10, 2.15, 2.35, 2.25, 2.55, 2.75, 2.80 and 2.85 eV for the composite samples of Cd_xPb_(1-x)Se (x varies from 0.1 to 0.9) thin films respectively. Band gap energies are to be composition dependent. Band gap energy increases with the increasing 'x', this is as expected as band gap energy for PbSe is 0.25 eV and band gap energy for CdSe is 1.7 eV. Enhancement in the band gap observed (average band gap \cong 2.42 eV) may be attributed to nanocrystalline nature (average particle size is smaller than \cong 34 nm) of the composite thin films. Variation of band gap energy as function of composition is shown in Fig- 17.

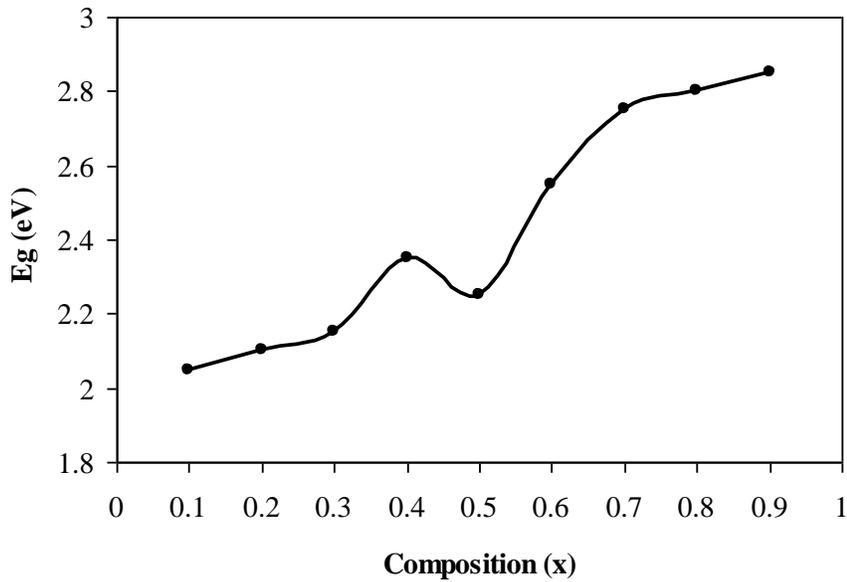


Fig-17 Variation of Optical Band Gap with composition of $Cd_xPb_{(1-x)}Se$ thin films.

P P Hankare et. al. [5] studied chemosynthesis of technologically important $Cd_{1-x}Pb_xSe$ thin films with varying lead concentrations ($0 \leq x \leq 1$). The effect of lead composition x on properties such as optical band gap, absorption coefficient and dark dc electrical conductivity has also been studied.

It was also attempted to plot $(\alpha hv)^{1/2}$ Vs hv for all the samples. These plots did not show any linear dependence indicating that the optical transitions are not indirect.

In the present work, all the film compositions investigated are of mixed phases of hexagonal and tetragonal structure. Therefore the larger optical band gap shift (2.05 to 2.85 eV) may be attributed to this nanostructures (average particle size = 34 nm) nature of solid solution thin films [6].

Determination of Optical constant ‘n’ and ‘k’ –

Near normal incidence reflectance and transmittance data have been used to determine optical constant ‘n’ and ‘k’. The variation of refractive indices and extinction coefficients as a function of wavelength as represented in Fig- 18 – 19 for the samples of compositions ($x = 0.1$ to 0.9) respectively. It is found that variations in refractive indices and extinction coefficients are oscillatory in nature. Secondly variation in n and k seems

to be complementary i. e. maxima of one and minima of the other at the same wavelength. This is presented in table No- 2. It is also observed that number of maxima and minima depend upon the compositions of film samples. This comparative study reveals the following facts.

- 1 For the composition of $x = 0.1$ sample there is no well define maxima or minima in variation of 'n' and 'k' as a function of wavelength.
- 2 For the composition of $x = 0.2$ to 0.6 samples there are two well define maxima and one minima in the variation of 'n', while there is only one maxima and two minima in variation of 'k'.
- 3 For the composition of $x = 0.7$ to 0.9 sample there are two minima and two maxima in variation of 'n' and 'k' as a function of wavelength.
- 4 In the sample of higher compositions the nature of variation of 'n' and 'k' as a function of wavelength is similar having two well defined maxima and two minima in the variation of 'n' and 'k'. Refractive index varies in the range from 1.16 to 2 while the range of variation of extinction coefficient is from 0.001 to 0.418. The table No.- 2 represents these features of variation of 'n' and 'k' i.e. for each sample, the values of maxima minima and the corresponding wavelengths [7, 8].

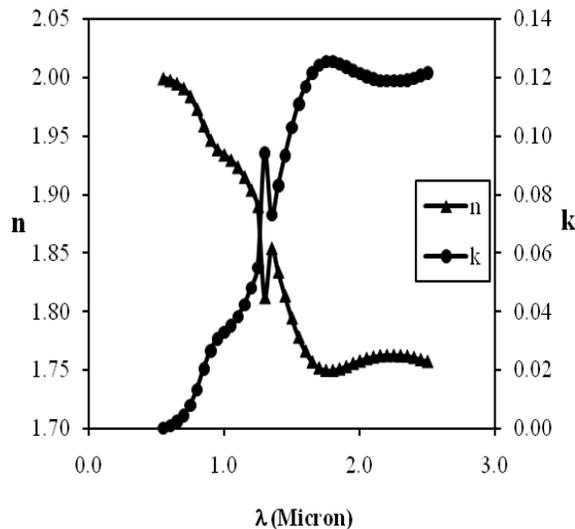


Fig.-18 Variation of 'n' and 'k' with Wavelength ($x = 0.1$).

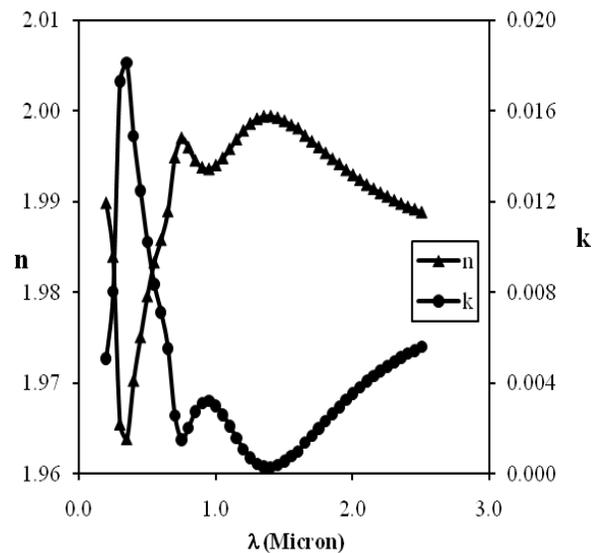


Fig.-19 Variation of 'n' and 'k' with Wavelength ($x = 0.9$).

Table No- 2 Well defined Maxima and Minima in variation of ‘n’ and ‘k’ for $(\text{Cd}_x\text{Pb}_{(1-x)}\text{Se})$ composite thin films.

Sr. No.	Sample Composition (x)	λ (μ)	Maxima		Minima	
			‘n’	‘k’	‘n’	‘k’
1	0.1	0.5	1.99			0.004
		1.3		0.094	1.81	
		1.35	1.85			0.073
		1.75		0.125	1.74	
2	0.2	0.5		0.217	1.56	
		1.05	1.92			0.037
		1.25		0.046	1.90	
		1.80	1.98			0.005
3	0.3	0.45		0.418	1.16	
		0.95	1.88			0.057
		1.20		0.070	1.86	
		1.65	1.99			0.004
4	0.4	0.45		0.419	1.16	
		0.95	1.88			0.058
		1.10		0.071	1.85	
		1.65	1.98			0.005
5	0.5	0.45		0.311	1.37	
		0.90	1.90			0.048
		1.10		0.067	1.86	
		1.65	1.99			0.003
6	0.6	0.4		0.303	1.39	
		0.85	1.92			0.035
		1.05		0.058	1.88	
		1.6	1.99			0.003
7	0.7	0.20	1.89			0.052
		0.35		0.197	1.60	
		0.70	1.94			0.029
		0.85		0.052	1.89	
		1.35	1.98			0.006
8	0.8	0.20	1.90			0.045
		0.30		0.172	1.65	
		0.75	1.97			0.013
		0.95		0.033	1.93	
		1.35	1.99			0.001
9	0.9	0.20	1.989			0.005
		0.35		0.018	1.963	
		0.75	1.997			0.001
		0.95		0.003	1.993	
		1.40	1.999			0.001

In the present work however it is observed oscillatory behavior of 'n' and 'k' as a function of wavelength in the transparent region. To our knowledge no such oscillatory behavior was reported earlier. However the spectral behavior of absolute values of normal transmittance and reflectance is in closely agreement with that of observed by P. Gupta et. al.[4]. It is observed that all films are transparent in wavelength range 200-600 nm. At higher wavelengths the experimental results T and R satisfy the relationship $T+R= 1$. This indicates that neither absorption nor scattering of light occurs beyond the absorption edge. The appearance of maxima and minima results from interference effect and their number increases with increases film composition.

TRANSPORT PROPERTIES

It can be seen from the Fig-14 that resistivity decreases almost linearly with increasing composition parameter 'x'. From approximately 3.69 Ω -cm ($x = 0.1$) to about 0.080 Ω -cm ($x = 0.9$).

The nature of variation of resistivity as a function of reciprocal of temperature is similar for all compositions with $x \geq 0.2$ to 0.5 and is almost linear for these compositions resistivity increases with decreasing temperature suggesting film material is semiconductor with negative temperature coefficient of resistivity. Composition ($x = 0.1$) figure-15 variation of resistivity exhibit quite differently. Initially resistivity increases with increasing temperature attaining maximum value at 348 K and further decreases with increasing temperature. This indicates that in low temperature range i. e. from room temperature to 348 K film material has metallic behavior. It transforms to semiconducting behavior around above 348 K. The compositions with $x = 0.6$ to 0.9 and is almost linear for these compositions resistivity decreases with decreasing temperature suggesting film material is metallic behavior [9 - 12].

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SUMMARY

The Minor Research Project entitled, “Growth and Characterization of $\text{Cd}_x\text{Pb}_{(1-x)}\text{Se}$ Ternary Compound Thin Films.” consists of synthesis and characterization of films deposited on glass substrate by thermal evaporation (reactive evaporation) technique. The properties measured are structural, transmittance, reflectance, I-V characteristics, resistivity, etc. From the data obtained, material parameters have been derived such as optical band gap, refractive index, extinction coefficients, resistivity, activation energy, etc.

In the present work, all the $\text{Cd}_x\text{Pb}_{(1-x)}\text{Se}$ composite thin films were characterized and structural information have been derived by X-ray diffraction study. Diffraction

analysis suggests that all the films of various compositions are polycrystalline nature. However the characteristics peak of hexagonal CdSe and tetragonal PbSe changes their angular position and relative intensities for different compositions suggesting multiphase and in homogeneity of films. From the scanning electron micrographs it is found that the particles are nearly spherical in nature. It is clear from figure that the film is nanostructure. The average particle size was calculated to be $\cong 34$ nm. This may be relatively smaller growth rate of grains. However films surfaces are very smooth. The composition of starting basic ingredients and film composition comparison seems to be in agreement with that obtaining from EDAX analysis. The salient features of transmittance and reflectance were described and the absorption coefficients, type of transition, optical band gap and optical constants have been evaluated from this study for all the composite films. From the current voltage characteristics the resistivity of sample materials have been evaluated. Resistivity has been measured as a function of temperature coefficient of resistivity indicating that film material is semiconducting. Activation energies increase with increasing composition parameter 'x' from 0.056 to 0.358. These results are satisfactory and as theoretically expected.

Scope of research work has been amongst chalcogenides of II and IV group metals Selenides binary have been extensively studied while ternary of these materials have been comparatively less attended. Several techniques have been used for the preparation of thin films and are characterized for their electrical, optical and structural properties. In the present work it has synthesized ternary $\text{Cd}_x\text{Pb}_{(1-x)}\text{Se}$ in the form of thin films by physical evaporation technique such as thermal evaporation. This synthesized ternary compound thin films of the form $\text{A}_x\text{B}_{(1-x)}\text{Se}$ [$\text{Cd}_x\text{Pb}_{(1-x)}\text{Se}$, $x = 0.1$ to 0.9] are also characterized for their structural (TEM) properties, different optical constants and thermo electric properties and various parameters. The attempt is made to device fabrication of the promising material $\text{Cd}_x\text{Pb}_{(1-x)}\text{Se}$, since it has many such applications as in Solar cells, IR detectors, In filters, Switching devices, High speed stable integrable thin film Transistors, Lasers, Schottky diodes etc

List of Publications:-

1. Composition dependence of optical constants of $Zn_xCd_{(1-x)}Te$ thin films.
U. P. Khairnar, S. S. Behere, P. H. Pawar and S. R. Jadhav.
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(iii) Has the progress been according to original plan of work and towards achieving the objective. If not, state reasons:

Progress of the work is mostly according to the original plane. However there were scarcity of power and water supply mostly in period of May-June every year that causes to do work towards device fabrication and characterization of device remained uncompleted within stipulated period of the project.

(iv) Please indicate the difficulties, if any, experienced in implementing the project:

In our college laboratory there has been not facility of Vacuum evaporation systems. So I will deposited the samples from district Place College. Therefore the large time is spent for sample preparation.

- (v) If project has not been completed, please indicate the approximate time by which it is likely to be completed. A summary of the work done for the period (Annual basis) may please be sent to the Commission on a separate sheet
- (vi) If the project has been completed, please enclose a summary of the findings of the study. Two bound copies of the final report of work done may also be sent to the Commission
- (vii) Any other information which would help in evaluation of work done on the project. At the completion of the project, the first report should indicate the output, such as (a) Manpower trained (b) Ph. D. awarded (c) Publication of results (d) other impact, if any

**SIGNATURE OF THE PRINCIPAL
INVESTIGATOR**

REGISTRAR/PRINCIPAL

Dr U. P. Khairnar
Principal Investigator,
Minor U.G.C. Research Scheme,
Ref. No. 47 – 1275/09.
Department of Physics
Shindkheda-425406.
Date:

To,
University Grants Commission,
Western Regional Office,
Ganeshkhind,
Pune- 411007.

Sub: - Final Report of the work has done on the Minor Research Project.

Sir,

Please find here with the Final report of U.G.C. Minor Research Scheme-
Ref. No. 47 – 1275/09. Here I am attached the Utilization Certificate, Statement
of Expenditure, Detail audited statements and Two copies of Final report
(**Annexure –III**) along with this letter.

I, therefore request you to release remaining amount of grant **Rs 35736/-**
of Minor Research Project at an earliest.

Thanking you,

Yours faithfully

Forwarded Through

(Dr U. P. Khairnar)
Principal Investigator,

Principal,
S.S.V.P.S. Dhule's Arts, Commerce
and Science College, Shindkheda.