Available online at www.joac.info

ISSN: 2278-1862

Journal of Applicable Chemistry

2015, 4 (4): 1227-1236

(International Peer Reviewed Journal)

XRD and SEM Characterization of Chemically Deposited $Pb_xCd_{1-x}S$ Thin Films

K. E. Suryavanshi [1], A. M. Patil[2] and R. B. Dhake*[3]

1. R. C. Patel Institute of Technology, Shirpur, **INDIA**
2. R. C. Patel Arts, Commerce and Science College, Shirpur, **INDIA**
3. Dadasaheb D.N. Bhole College, Bhusawal, **INDIA**

Email: kiran4suryavanshi@gmail.com

Accepted on 11th July 2015

ABSTRACT

$Pb_xCd_{1-x}S$ thin films ($0.0 \leq x \leq 1.0$) have been deposited on glass substrate by Chemical Bath Deposition technique at 75°C temperature. The source material Cadmium Acetate, Lead Acetate and Thiourea were used to prepare thin films. Ammonia was used as complexing agent. For good quality of uniform films the various preparative parameters such as deposition temperature, time and pH of the reaction mixture were optimized. The deposited samples were characterized through structural, surface morphological and compositional studies. The Structural property of film is studied by using x-ray diffraction technique. The X-ray diffraction results show that the films are of $Pb_xCd_{1-x}S$ composite with individual PbS and CdS planes. XRD analysis shows that all thin films are polycrystalline with a hexagonal (CdS), Cubic (CdS) and orthorhombic (PbS) structure. Scanning electron micrograph of different films was also investigated. The crystallite size measured by XRD and FESEM studies was found to be varied with composition 'x'.EDXS shows compositional analysis of prepared films.

Keywords: $Pb_xCd_{1-x}S$ Thin Films, XRD, FESEM, EDXS.

INTRODUCTION

The II-VI group chalcogenide compounds have been greatly used for deposition of thin films due to their application of advanced technology. Among these metal chalcogenides, PbS and CdS are more promising material used to deposit thin films for its applications [1-4]. Lead Sulphide (PbS) is p- type material with wide direct band gap 0.41 eV. The PbS materials have many applications for infra-red detectors [5-6], Solar cells [7].Cadmium Sulphide (CdS) is n- type material with wide direct band gap (2.42 eV) has been used in many optoelectronic applications.

$Pb_xCd_{1-x}S$ is an important semiconductor material prepared at 75°C temperature. Various techniques have been employed to deposit $Pb_xCd_{1-x}S$ thin films such as spray pyrolysis [8-9], chemical bath deposition [10-13], CVD [14], Layer coating [15], Thermal Evaporation [16] successive ionic layer and reaction (SILAR) [17].

Among these, the CBD technique is much more useful because it possesses a number of advantages over the other thin film deposition methods. It does not require an expensive apparatus. The chemical bath deposition process uses a controlled chemical reaction to deposit a thin film by precipitation. The major factor for growing thin film depending upon deposition conditions such as bath temperature, stirring rate, pH, solution concentration etc. the film growth take place by ion-by-ion condensation of materials or by adhesion of colloidal particles from the solution on the substrate. The use of chemical bath deposition (CBD) technique has received much attraction due to its great potential application to fabricate high quality films. It is a preparative method for large area fabrication of film, which is simple and low cost technique.

In this present paper, we report the deposition of $Pb_xCd_{1-x}S$ thin films ($0.0 \leq x \leq 1.0$) on the corning glass substrates by using Chemical Bath deposition technique.The deposited samples were characterized through structural, surface morphological and compositional studies. The results are discussed and compared with standards.

MATERIALS AND METHODS

Preparation of thin film: We use commercially available corning glass substrate for deposition of thin film. $Pb_xCd_{1-x}S$ thin films were grown on glass substrates by the Chemical Bath deposition technique at 75°C temperature. The bath solution was prepared by mixing the appropriate volumes of 0.1M lead acetate $[Pb(CH_3COO)_2.2H_2O]$, 0.1M cadmium acetate $[Cd(CH_3COO)_2.2H_2O]$, 6M ammonia (NH_3), 1M Thiourea $[(NH_2)_2.CS]$.

First glass substrate is ultrasonically cleaned and used for deposition of $Pb_xCd_{1-x}S$ thin film. Then substrate washed by using distilled water and acetone, after that completely dried.

Take depositing solution of 0.1M lead acetate, 0.1M cadmium acetate, 6M ammonia in glass beaker. Then place this beaker in chemical bath and set temperature 75°C. When temperature reaches to 50°C place glass substrate vertically in solution and adds 20mL of 1M Thiourea drop by drop in a beaker until temperature increases from 50°C to 75°C. After deposition cool it to room temperature and rinsed with distilled water and dried in air. All $Pb_xCd_{1-x}S$ films exhibited yellowish to deep black color.

Reaction mechanism of $Pb_xCd_{1-x}S$ thin film: The CBD is based on sequential reaction at the surface of substrate. Similarly the formation of $Pb_xCd_{1-x}S$ thin film depends on the rate of release of Pb^{2+}, Cd^{2+} and S^{2-} from the bound state. The formation of $Pb_xCd_{1-x}S$ may involve the following steps [10, 18-20].

$$_x[Pb(NH_3)_4]^{2+} + {}_{1-x}[Cd(NH_3)_4]^{2+} + (NH_2)_2CS + 2OH^- \text{ -----> } Pb_XCd_{1-X}S + \text{Waste product}$$

The color of the prepared $Pb_xCd_{1-x}S$ films exhibited yellowish to black as the composition parameter x has increased from 0 to 1.

Structural characterization of $Pb_xCd_{1-x}S$ thin film: The structural property and orientation of the $Pb_xCd_{1-x}S$ thin films was investigated by means of X-ray diffraction (XRD) technique using Bruker D8 Advance diffracto-meter with CuKα1 radiation. The $Pb_xCd_{1-x}S$ thin films were scanned continuously between range 20˚ and 90˚. The scanning of thin film is done for 20 min. Scanning Electron Micrographs (SEM) of different films were also investigated by using Hitachi S-4800-Type-II (Hitachi High Technology Corporation). Energy-dispersive X-ray Spectroscope (EDXS) used for Compositional analysis and is done by using X-Flash detector 5030, Bruker (Berlin-Germany).

RESULTS AND DISCUSSION

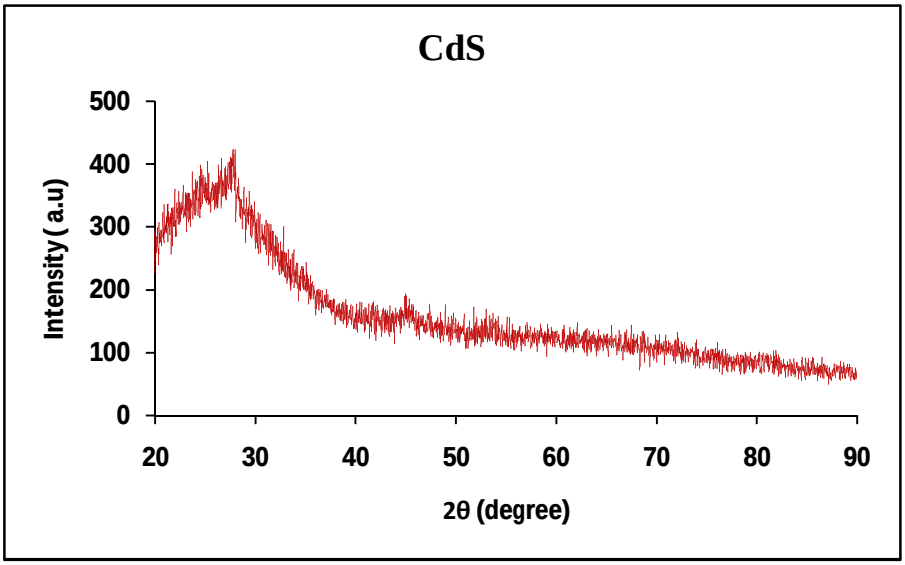
X-ray diffraction (XRD) Studies: In order to study crystalline nature of prepared $Pb_xCd_{1-x}S$ thin films, the XRD pattern were recorded in the 2θ range 20° to 90°. The XRD results for films with different composition show that the films are polycrystalline. The X-ray diffraction of $Pb_xCd_{1-x}S$ thin films ($0.0 \leq x \leq 1.0$) prepared by chemical bath deposition is shown in figure.1.It is evidentfrom figure.1 $Pb_xCd_{1-x}S$ thin films are polycrystalline with broad hump, which is due to amorphous glass substrate. It is observed that CdS exhibits zinc blend (Hexagonal) and Wurtzite (Cubic) crystal structure match with JCPDS (PDF00-041-1049) and JCPDS (PDF03-065-8873) respectively. This CdS hexagonal and CdS Cubic structure good matches with earlier papers [21]. Also PbS is present in orthorhombic crystal form match with JCPDS (PDF 01-079-4716) which reports in some earlier papers [22-26]. All the match values for prepared thin films are given in table.1.

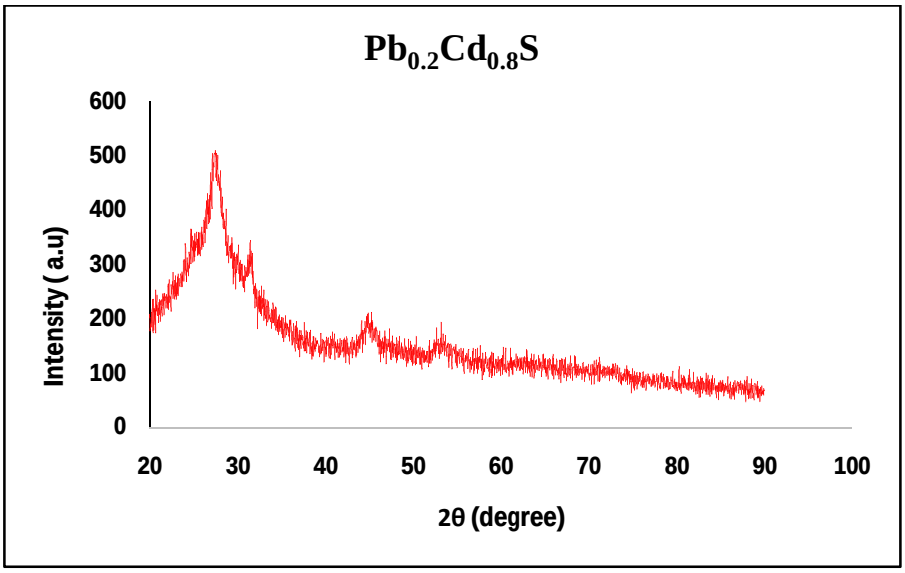
All major diffraction peaks for all samples were chosen to estimate the average size of the crystallites by using DebyeScherer's equation 1.

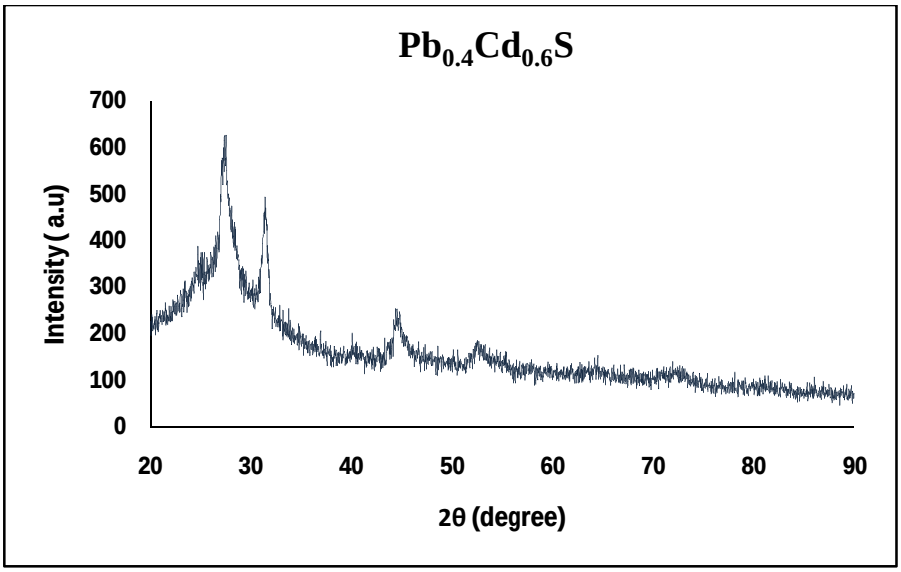
$$D = 0.94 \lambda / \beta \cos\theta \qquad \text{.....(1)}$$

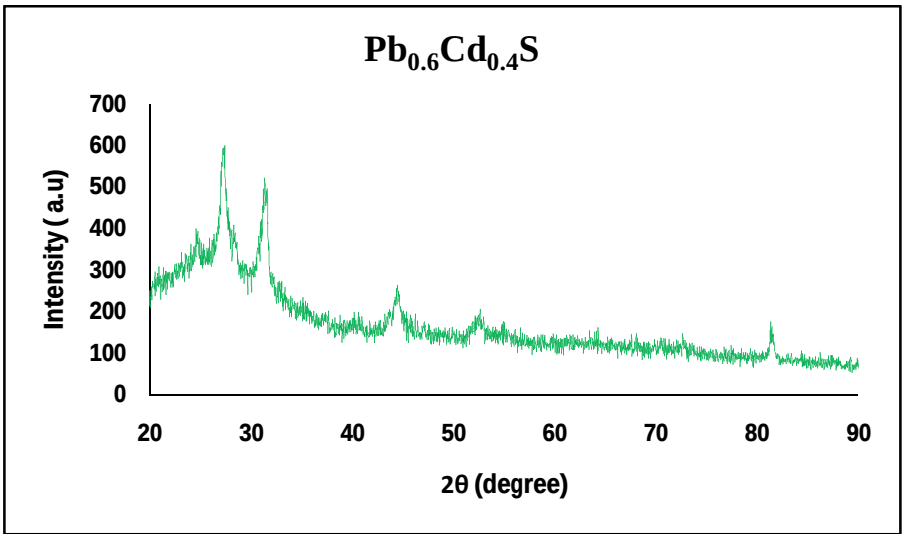
Where, λ is the wavelength of X-ray used,
β is the full width at half maximum in radian, and
θ is the Bragg's diffraction angle.

The average grain sizes for prepared $Pb_xCd_{1-x}S$ thin films ($0.0 \leq x \leq 1.0$) are in between11.85 nm to 272.79 nm.




$Pb_{0.2}Cd_{0.8}S$
Intensity (a.u)
600
500
400
300
200
100
0
20
30
40
50
60
70
80
90
100
2θ (degree)


$Pb_{0.4}Cd_{0.6}S$
Intensity (a.u)
700
600
500
400
300
200
100
0
20
30
40
50
60
70
80
90
2θ (degree)


$Pb_{0.6}Cd_{0.4}S$
Intensity (a.u)
700
600
500
400
300
200
100
0
20
30
40
50
60
70
80
90
2θ (degree)

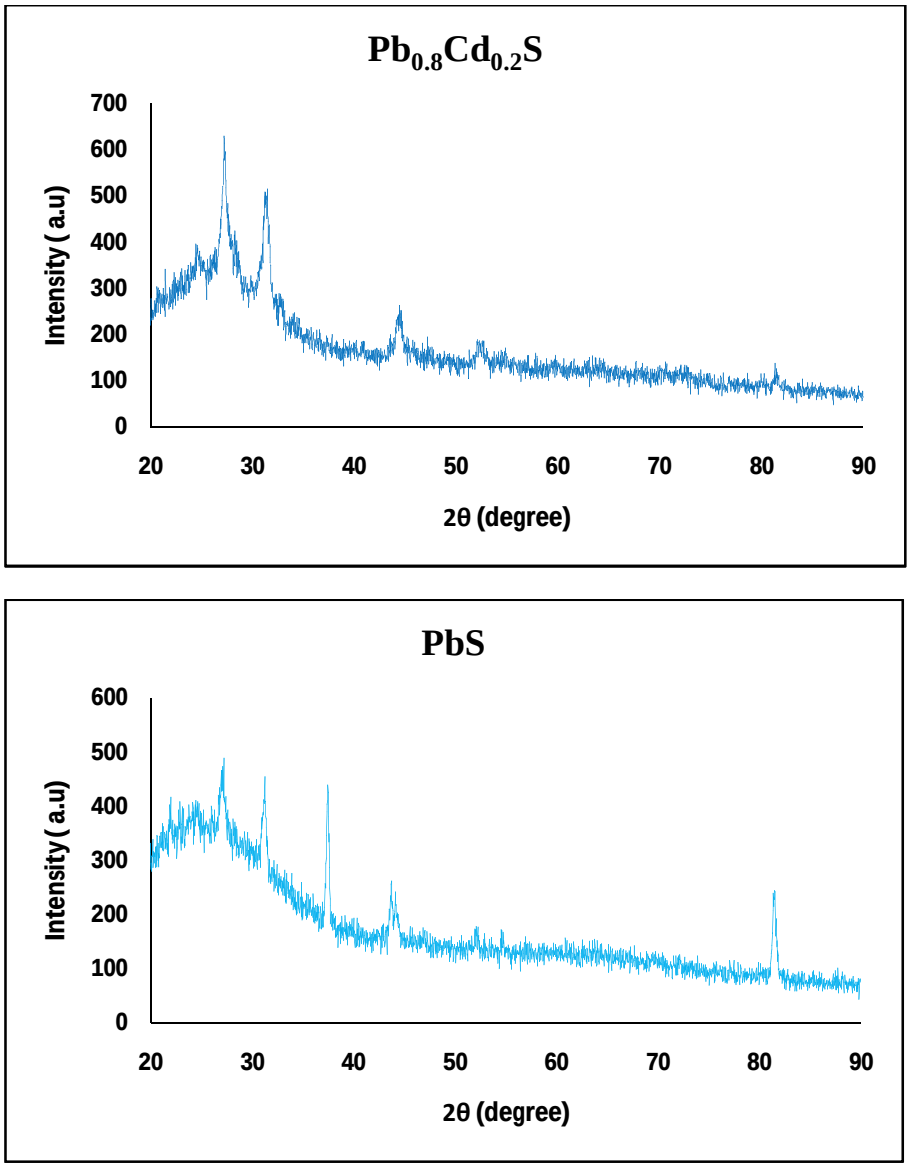


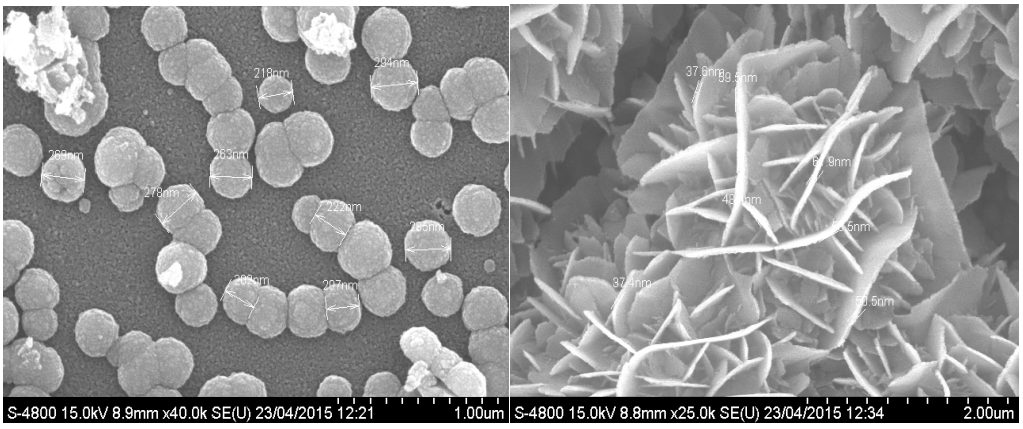
Figure.1. X-ray diffraction of $Pb_xCd_{1-x}S$ thin films ($0.0 \leq x \leq 1.0$)

Table.1. X R D Analysis of the of $Pb_xCd_{1-x}S$thin films ($0.0 \leq x \leq 1.0$)

Composition (x)	2θ (Deg)	d (A°) (Std)	d(A°) (Obs)	hkl planes
CdS	28.21	3.16078	3.20745	101
	43.73	2.0683	2.0144	110
	47.88	1.8985	1.8589	103
	66.84	1.3986	1.3984	203
$Pb_{0.2}Cd_{0.8}S$	24.81	3.5861	3.5915	100
	27.41	3.2519	3.2485	210
	31.92	2.8016	2.8421	111
	54.59	1.6799	1.6777	004

$Pb_{0.4}Cd_{0.6}S$	27.41	3.2519	3.2527	210
	31.25	2.8600	2.8406	200
	31.92	2.8016	2.8406	111
	44.78	2.0223	2.0331	220
	61.57	1.5050	1.4990	701
$Pb_{0.6}Cd_{0.4}S$	27.41	3.2519	3.2671	210
	31.92	2.8016	2.8444	111
	31.25	2.8600	2.8444	200
	44.78	2.0223	2.0310	220
	70.31	1.3370	1.3344	801
	82.56	1.1676	1.1807	422
$Pb_{0.8}Cd_{0.2}S$	27.41	3.2519	3.2729	210
	26.98	3.3024	3.2729	111
	31.25	2.8600	2.8542	200
	31.70	2.8000	2.8542	111
	43.71	2.0693	2.0421	102
	44.78	2.0223	2.0400	220
PbS	27.05	3.2519	3.2944	210
	31.27	2.8016	2.8583	111
	31.20	2.8200	2.8648	400
	37.44	2.3430	2.4001	401
	43.69	2.0693	2.0703	102
	81.46	1.1715	1.1805	802

Scanning Electron Microscopy (SEM)Studies: Surface morphology of the ternary alloy $Pb_xCd_{1-x}S$ thin films ($0.0 \leq x \leq 1.0$) were carried out by using scanning electron microscopy (SEM).The SEM images of deposited $Pb_xCd_{1-x}S$ thin films ($0.0 \leq x \leq 1.0$) is shown in figure. 2. The SEM micrographs of all the films show uniform surface morphology over the entire glass substrate. It is also seen from the SEM images that all the samples consists of a dense layer with small crystallites. It was observed that the surface morphology of image (a) and (e) shows is ball like structure [27], for image (b) and (c) shows a sand rose like structure [21],for image (d) it showsa rice-likestructure [12] and for image (f) it shows cubic with hexagon structure [24].The grain size was calculated using standard scale bar method. It observed that grain size is different for every change in composition. The average grain size is calculated was ranging between 42.77 nm and 240.57 nm.



(a) (b)

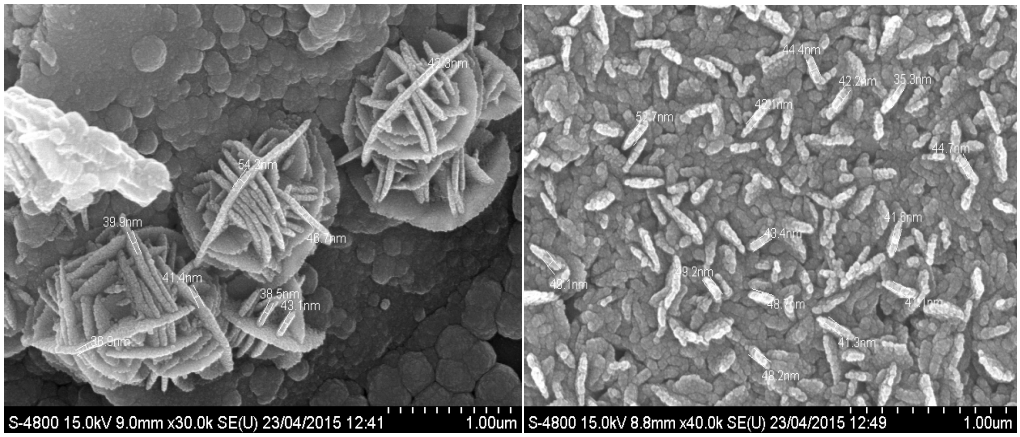
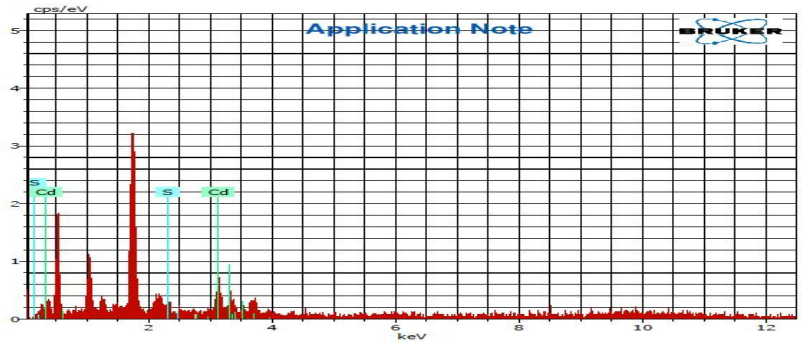


Figure.2. (a-f) SEM images of prepared $Pb_xCd_{1-x}S$ thin films ($0.0 \leq x \leq 1.0$)

Energy-Dispersive X-ray Spectroscope(EDXS) Studies: The EDXS analysis of deposit $Pb_xCd_{1-x}S$ thin films ($0.0 \leq x \leq 1.0$) are shown in figure 3 (a, b and c). The spectrum shows the presence of Pb, Cd and S elements on glass substrates. The compositional analysis of the deposited $Pb_xCd_{1-x}S$ thin films ($0.0 \leq x \leq 1.0$) is done by using EDXS, shown in table 2. The presence of Pb, Cd and S in the EDXS analysis is due to the precursors, Lead acetate, cadmium acetate and thiourea respectively. An atomic percentage of the elements in thin films are in close agreement with the volumetric ratio of the elemental precursor chemical solutions taken in the bath during deposition.



(a)

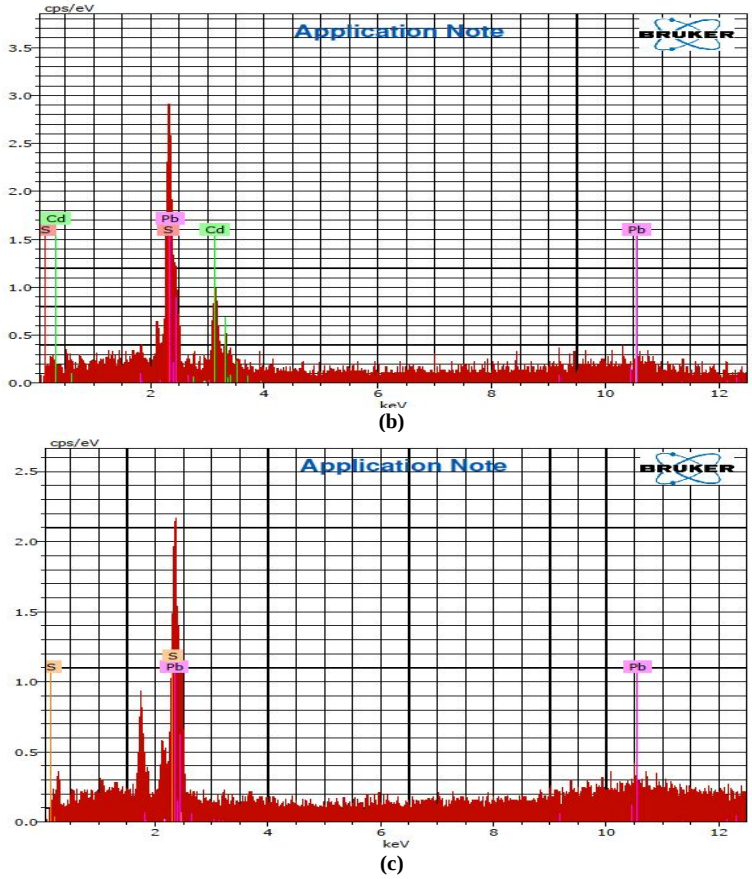


Figure.3. EDXS Spectrum (a) CdS (X = 0.0), (b) PbCdS (X = 0.6) and (c) PbS (X = 1.0)

Table.2. Compositional analysis of deposited $Pb_xCd_{1-x}S$ thin films ($0.0 \leq x \leq 1.0$)

Composition (X)	Bath solution of film (%)			EDXS analysis (%)		
	Pb	Cd	S	Pb	Cd	S
CdS	0	50	50	0	52.13	47.14
$Pb_{0.2}Cd_{0.8}S$	10	40	50	6.15	49.12	43.36
$Pb_{0.4}Cd_{0.6}S$	20	30	50	15.24	36.38	48.16
$Pb_{0.6}Cd_{0.4}S$	30	20	50	28.17	19.63	48.63
$Pb_{0.8}Cd_{0.2}S$	40	10	50	38.54	8.74	47.85
PbS	50	0	50	48.25	0	48.65

Grain Size by XRD and SEM: The grain size for deposited $Pb_xCd_{1-x}S$ thin films ($0.0 \leq x \leq 1.0$) is measured by X-ray diffraction (XRD) and scanning electron microscopy (SEM). The comparative grain sizes for deposited samples are shown in table.3 which varies with composition 'X'.

Table.3. Compared grain size by XRD and SEM

Composition(x)	Grain Size(nm)	
	XRD	SEM
CdS	272.79	240.57
$Pb_{0.2}Cd_{0.8}S$	11.85	51.80
$Pb_{0.4}Cd_{0.6}S$	17.03	43.31
$Pb_{0.6}Cd_{0.4}S$	272.49	42.77
$Pb_{0.8}Cd_{0.2}S$	108.98	50.41
PbS	32.60	75.33

APPLICATIONS

The characterization techniques discussed in present study are helpful to find the crystal structure, morphology, grain size, phases and compositional analysis of the material. It also helps to design new optoelectronics devices like solar cell, sensors, photodiode, IR detector etc.

CONCLUSIONS

Thin films of $Pb_xCd_{1-x}S$ thin film ($0.0 \leq x \leq 1.0$) were deposited by chemical bath deposition technique. XRD studies shows that the deposited thin films are polycrystalline in nature with cubic, hexagonal and orthorhombic phases. SEM micrographs shows uniform morphology with ball like, sand rose like, rice-like and cubic with hexagon structure. Formation of Pb, Cd and S elements in compositions was also confirmed from EDXS spectra. The grain size for deposited thin films is measured by X-ray diffraction (11.85 nm to 272.79 nm) and scanning electron microscopy (42.77 nm to 240.57nm).

ACKNOWLEDGMENTS

The Authors are thankful to, Central Instrumentation Laboratory, University Institute of Chemical Technology, N.M.U, Jalgaon, for providing XRD, FESEM, and EDXS analysis facilities.

REFERENCES

[1] J. F. Mohammad, H. S. Al-jumaili and S. M. Abed, *Int. J. of App. or Inno. inEngg and Mgmt.*,**2014**,3.
[2] R. Y. Mohammed, S. Abduol, A. M. Mousa, *Int.Lett. of Chem., Phy.andAstr.* **2014**, 10, 91.
[3] A. S. Obaid, M.A.Mahdi, Z.Hassan, and M.Bououdina,*Int. Conf. on Edu., App. Sci. and Mgmt.,Dubai (UAE)*, **2012**.
[4] V. Popescu, G. L. Popescu, *J. of optoele. and adv. Mater.***2011**, 13(7), 926.
[5] N. choudhury and B.K.sharma, *Bull.of Mater. Sci.*, **2009, 32** (1), 43.
[6] X.Lui and M. Zhang, *Int. J. of Infra. & Mm. waves*, **2000**, 21(10), 1697.
[7] D.Kumara, G. Agarwal, B.Tripathi, D .Vyas, and V. Kulshrestha,*J. of Alloys and Compds.*, **2009**, 484, 463.
[8] V. Popescu, H. I. Nascu, E. Darvasi, *J.ofOptoele.andAdv.Mater.*, **2006**, 8, 1187.
[9] M. Kamruzzaman, R. Dutta and J. Podder, *Semi's.*, **2012**, 46(7), 979–983.
[10] M. A. Barote, A. A. Yadav, E. U. Masumdar, *Chalco.Lett.*, **2011**, 8(2), 129.
[11] M. A. Mohammed, A. M. Mousa, and J. P. Ponpon, *J. of Semi.Tech.and Sci.*, **2009**, 9, 117.
[12] H. S. AL-Jumaili, *App.Phy. Res.*, **2012**,4,75.

[13] K.E.Suryavanshi, R.B.Dhake, A.M.Patil, *Int. J. of Adv. Sci. and Tech.Res.*, **2014,** 4 (2),858.
[14] E. Omotoso, G. A. Adegboyega, M. A. Eleruja, B. Olofinjana, O. O. Akinwunmia, O. O. Ilori, B. A. Taleatu, E. O. B. Ajayi, *J. of Non-Oxide Glasses*, **2013**,5(1), 9.
[15] J. C Osuwa, C.I Oriaku, E.C Mgbaja, *Chalco.Lett.*, **2010**, 7, 679.
[16] M. M. Abdulla, N.H.Hasan, H.I.Mohammed, G.H.Mohamed, K. A. Al-Hamdani and A.F.Abdulameer, *J. of Elect. Devi*, **2012**, 12, 761.
[17] S. Lindroos, J. Puišo, S. Tamulevičius, M. Leskelä, *Solid State Pheno.*, **2004**, 99,243.
[18] S. Thangavel, S. Ganesan, S. Chandramohan, P. Sudhagar, Yong Soo, Kang & Chang-Hee, Hong, *J. of Alloys and Compds*, **2010**, 495, 234.
[19] Ubale A. U, Junghare, A. R. Wadibhasme, N. A. Daryapukar, A. S. Mankar R. B. & Sangawar V. S, *Turk. J. Phys.*, **2007**, 31, 279.
[20] Zahid, R., Zakaria, A., Ghazali, M. S. M., Jafari, A., Fasih U. D., Din, &Zamiri, R. *Int. J. Mol. Sci.*, **2011**, 12, 1293.
[21] M. A. Barote, A. A. Yadav, L. P. Deshmukh, E. U. Masumdar, *J.of Non-Oxide Glasses*, **2010**, 2(3), 151.
[22] S. Rajathi, K. Kirubavathi, K. Selvaraju, *Arab. J. ofChem.*, **2015**, http://dx.doi.org /10.1016/ j.arabjc.**2014**.11.057
[23] J.O. Rivera –Nieblas, J. Alvarado- Rivera, M.C. Acosta-Enriquez, R. Ochoa-Landin, F.J. Espinoza-Beltran, A. Apolinar-Iribe, M. Flores-Acosta, A. Deleon, S.J. Castillo, *Chalco.Lett*, **2013**, 10(9)349.
[24] O. P. Moreno, M. C. Portillo, M. M. Flores , J. M. Juárez, G. A. Ávila, R. L. Morales and O. Z. Ángel, *J. of Mate. Sci. and Engg.* A 1, **2011,** 759.
[25] R. G. Pérez, G.H.Téllez, U. P. Rosas, A. Moran Torres, J. H.Tecorralco, L. C. Lima and O. P. Moreno, *J. of Mate. Sci. and EnggA 3*, **2013**, 1, 1.
[26] D. Zagorac, J.C. Schön, K. Doll and M. Jansen, *ActaPhysi. Polo. A*, **2011**, 2(120).
[27] N. Murali, K. VijayaBabu, K. Ephraim Babu and V. Veeraiah, *J.Applicable.Chem*, **2014,** 3 (3): 1202.
[28] R S Singh, *Int. J. of Adv.Engg. Res. and Studies*, 2015, 275.

AUTHORS' ADDRESSES

1. **Mr. K. E. Suryavanshi**
Assistant Professor,
Department of Applied Science,
R.C.Patel Institute of Technology, Shirpur, Dist-Dhule, State –Maharashtra,
Mobile: - 9921637860,
Email: -kiran4suryavanshi@gmail.com
2. **Dr. A. M. Patil**
Associate Professor,
Department of Physics,
R.C.Patel Arts, Commerce and Science College, Shirpur, Dist-Dhule, State –Maharashtra,
Mobile: - 9423288371,
Email: -ampatil67@gmail.com
3. **Dr. R. B. Dhake**
Assistant Professor,
Department of Chemistry,
Dadasaheb D.N. Bhole College, Bhusawal, Dist-Jalgaon, State –Maharashtra,
Mobile: - 9860779428, Email: - drrbdhake@yahoo.com