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Influence of Lead Precursor Concentration on Properties of Spray Deposited Lead Sulphide Thin Films

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Abstract

In this present paper we report the application of stoichiometric lead sulphide thin films, prepared using chemical spray pyrolysis technique. The concentration of łead acetate precursor is varied from 1:1 to 2:1 (łead acetate: thiourea) to achieve stoichiometry and to tune the concentration of lead ions in the lead sulphide thin films. Lower concentration of lead acetate yields PbS thin films with (200) crystallite orientation. On increasing the concentration, there is shift in the orientation from (200) to (111) indicating three dimensional growth of thin films and its crystallite size increases from 5 nm to 40 nm. Morphological studies using SEM reveals that lower lead ion concentration yields smooth and dense films while, increasing the concentration improves the crystallinity of the films. The absorbance of the stoichiometric lead sulphide thin films posses two absorption thresholds: one at 1000 nm and another at 3000 nm. The tauc plot indicates the presence of both direct and indirect energy band gap. Band gap energy of the PbS thin films reduces from 1.2 eV to 0.4 eV as the lead ion concentration is increased from 45 % to 55%. The PbS thin films have resistivity in the order of 10^2 Ω cm for stoichiometric thin film and reduces from 10 to 0.5 x 10^2 Ω cm for increased lead ion concentration. The reported resistivity values are suitable for applications in solar cells and photodiodes. The results suggest that the optical and electrical properties of the lead sulphide thin films prepared using chemical spray pyrolysis method can be tuned by varying the lead acetate precursor concentration.

Keywords: Thin films; Spray pyrolysis; Crystallite size; UV-VIS-NIR spectrometer; direct and indirect band gap;

1. INTRODUCTION

Chalcogenides are a class of semiconducting materials having one or more chalcogens such as S, Se, Ge, As and Te as a main constituent. The semiconducting properties of these chalcogenide depend on the composition and crystallinity of the materials and typically have energy band gap of 1 – 3 eV [1]. Chalcogenides are widely studied for their functionality and strong response to various optical, electrical and thermal stimuli. They possess the ability to bend, switch and self-organize along with inherent infrared transparency and optical nonlinear properties [1]. Thin films of chalcogenides (such as Ge, As, Se etc.) find widespread applications in optoelectronics such as photodetectors, solar cells, light-emitting diodes, optical memories, gratings, waveguides, optical sensors, holography and light amplifiers [1-2].

Lead sulphide is one such chalcogenide material belongs to IV-VI family which is widely investigated for its tuneable semiconducting properties that results from the ability to control the optical constants such as refractive index, extinction coefficient and dielectric constant. The lead sulphide thin films have rock salt cubic structure with partial ionic bonding. The semiconducting nature of thin films can be tuned to be both n-type and p-type having narrow energy band gap of 0.41 eV with positive temperature coefficient of the fundamental band gap [3-6]. Further, the lead sulphide thin films have high mobility, small effective mass of electron and holes. The large exciton Bohr radius of 18 nm gives thin films the nanocrystalline form which exhibits quantum properties that are utilized in the development of modern microelectronics devices with nano-sized elements [7,8]. The quantum confinement effects are more pronounced in PbS and controlling the crystallite size of the nanocrystalline thin films influences the band gap and hence, broadband tuneable emission in visible to infrared region can be achieved [30]. Reports have shown that lead sulphide nanocrystalline thin films on glass possess characteristics that are suitable for solar cell and photodetecting applications [6, 9-12].

Lead sulphide thin films are fabricated using various techniques that can be classified into physical methods and chemical methods. Physical methods include vacuum evaporation, molecular beam epitaxy, pulse laser deposition and sputtering. Chemical methods of fabrication include electrodeposition, spray pyrolysis, photochemical deposition, SILAR, ionothermal method etc. Chemical methods of fabrication are advantageous over the physical counterpart due to its simplicity, requirement of simple equipments, low-cost and high efficiency [8, 13-21]. Spray pyrolysis is one of the simple, advanced, economical, fast and vacuum less chemical method which has good control over the rate of deposition. This method of fabrication yields homogenous, pin hole free and smoother thin films. Stoichiometric nanocrystalline thin films with desired thickness and properties can be fabricated – with ease of doping – by optimizing spray parameters such as substrate temperature, spray rate, concentration of solution, annealing temperature and composition of the film [7-8, 13-14] .

In this paper, we report the preparation of stoichiometric lead sulphide thin films by chemical spray pyrolysis method. From the literature it was found that, the physical properties of Pbs was enhanced by varying the concentration of both cation and anion precursor concentration. But our objectives are to fabricate stoichiometric lead sulphide thin films by varying the lead acetate concentration and to study the effect of increased lead ion concentration on electrical and optical properties of films [3, 22-25]

2. EXPERIMENTAL DETAILS

2.1. Precursor preparation and deposition parameters

Initially the glass substrates are dipped in chromic acid for 24hrs then successively cleaned in an ultrasound bath, using acetone and distilled water, further dried in atmospheric air. Lead sulphide semiconductor thin films were deposited on the thoroughly pre cleaned glass substrates by spray pyrolysis method at 225˚C substrate temperature. Neutral solutions of $Pb(CH_3COO)_2$ and $SC(NH_2)_2$ at $Pb(CH_3COO_2)$: $SC(NH_2)_2$ molar ratios of 1:1, 1.25:1, 1.5:1, 1.75:1 and 2:1 were prepared.

1.2. Thickness measurement

The thickness of the films was measured by a well – known gravimetric weight difference method. It was maintained as 500±50 nm. It was found that thickness of deposited films increased with an increase in lead ion concentration. The thickness of the films was maintained constant by adjusting deposition time.

1.3. Characterization

The structural characterization of the films was carried out by analyzing the X-ray diffraction patterns obtained using XRD (Rigaku mini flex 600) Cu K_α radiation in the 2θ range of angles between 20˚ to 60˚. The surface morphology and compositional analysis were carried out using scanning electron microscope (SEM, JSM - 6380 LA from JEOL, Japan) interfaced with EDAX. The UV-VIS-NIR spectrometer (UV-NIR 3600) was used to record the optical absorption spectra of the films in the wavelength range 300-3000 nm. The DC electrical resistivity of the films was measured using Kiethly source measure unit. Silver electrodes were used as ohmic contacts. Hot probe method was used to identify the type of conductivity in lead sulphide thin film.

2. Results and discussion

Initially, the precursor concentration of lead was first optimized to achieve Pb_xS_{1-x} (x=0.5) 50:50 stoichiometric thin films. The stoichiometric films of the PbS thin films were varied from 45(Pb):55(S) to 55(Pb):45(S) by varying only the precursor concentration.

3.1. Structural studies

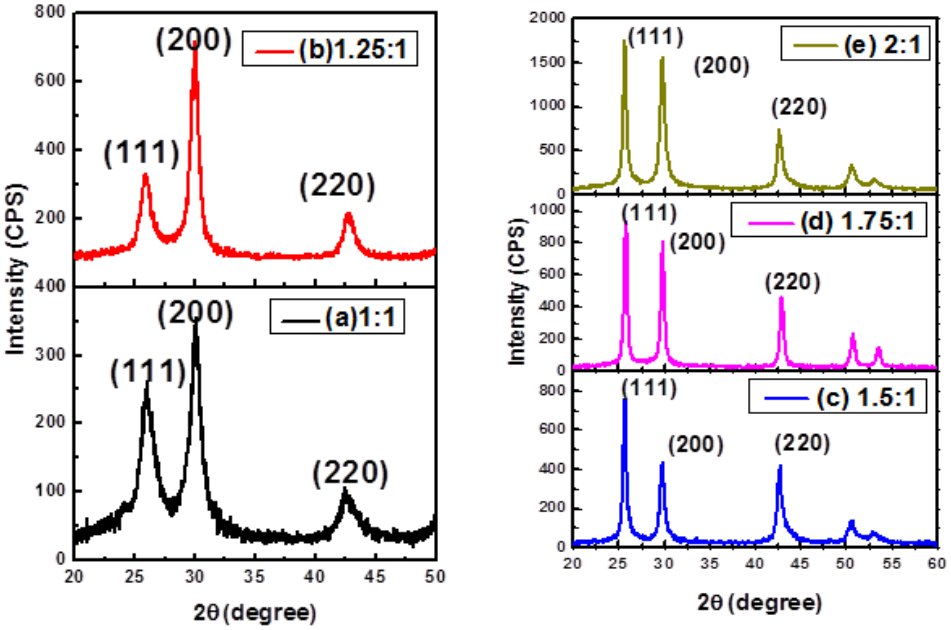


Figure 1 X-ray diffraction pattern of the Pb:S thin films with different precursor concentration of Pb (a) 1:1 (b) 1.25:1 (c) 1.5:1 (d) 1.75:1 (e) 2:1.

XRD pattern of lead sulphide thin films with various lead precursor concentration are shown in **Fig. 1**. All PbS thin films have face centered cubic structure with polycrystalline nature as evidenced from the presence of higher intensity peak (**figure 1b**) at 30.07˚ which corresponds to PbS as observed in standard JCPDS file (00-005-0592) [8]. The 50:50 stoichiometric thin films sharp peak at 30.07˚ confirming the presence of PbS. Other (111) and (220) at 25.64˚ and 42.75˚ correspond to different orientations of the crystallites in the thin films. The purity and homogeneity of the PbS thin films could be evidenced by the absence of peaks other than that correspond to PbS. The increase in lead precursor concentration from 1:1 to 2:1 (in steps of 0.25) results in shifting of crystallites orientation in PbS thin films from (200) to (111) indicating a three dimensional growth of the crystallites. This change in crystallite orientation could be attributed to the variation in number of Pb^{2+} and S^{2-} ions in the reactant solution at higher molar concentration. The results suggest that the micro structural characteristics of the sprayed lead sulphide films is dependent on the lead precursor concentration and hence, the orientation of the crystallites can be tuned by varying the cationic precursor concentration. The crystallite size (D) of deposited thin films was calculated using Scherer's formula [3].

$$D = {0.9\lambda}/{\beta cos\theta} \quad (1)$$

where λ is the wavelength of the X-ray (1.5406 Å), β is the full width at half maximum (FWHM) of the peak , ϑ is the Bragg angle. The formula was made

used on (111) diffraction peak for the determination of crystallite size of the films. It reveals that the increase in lead precursor concentration results in increased crystallite size as tabulated in **Table1**. The increase in crystallite size and thickness is due to the enhancement of the horizontal velocity of the adatoms and condensation of energetic lead and sulphur vapour on the substrate surface. The crystallites are found to be nanocrystalline in nature which explains that the PbS thin films have larger nucleation rate than the growth rate due to more number of nucleation centers that exist on the surface of the substrate as explained by Rajathi et al [8].

The increase in nanocrystallite sizes results in narrowing of the crystallite orientation peaks as observed in **figure 1(a-c)**. While the lead acetate exceeds 2:1 precursor ratio, the thickness of lead sulphide film decreases. The result seems to confirm that the nucleation rate is mainly regulated by the concentration of Pb^{2+} ions in solution which results in the development of PbS complex.. As reported by Seghaier et al for lead sulphide by varying lead nitrate precursor concentration using CBD technique [23]. Further the lattice constant (a) of the films was determined by the following relation [26].

$$\boldsymbol{a = d(h^2 + k^2 + l^2)} \qquad (2)$$

Table 1: Structural parameters of PbS thin films with different precursor concentration of Pb.

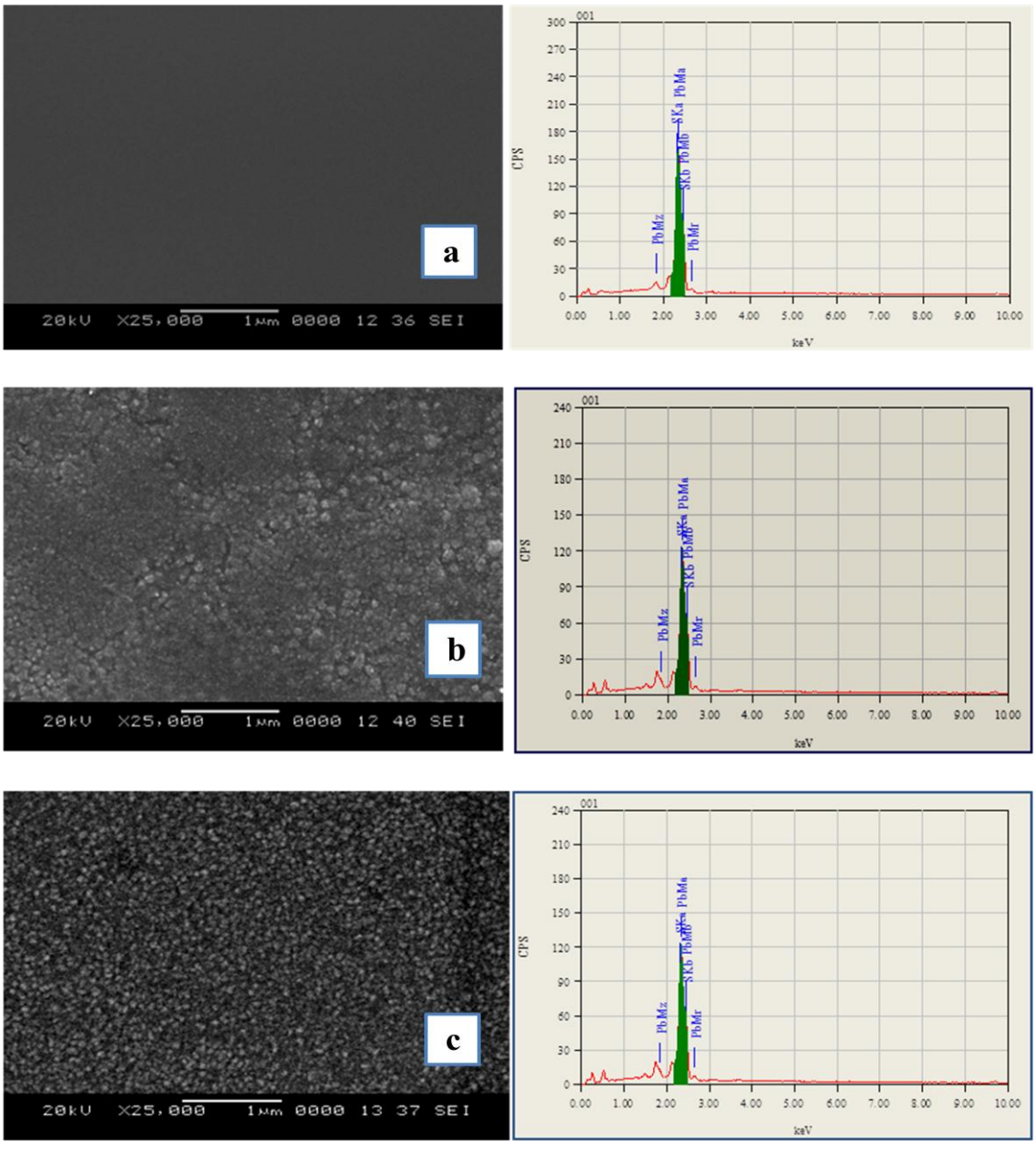
Pb:S ratio	Maximum intensity		For (111) plane				
	2θ	(hkl) Plane	2θ	d (Å)	a (std) (Å)	a (cal) (Å)	Crystallite size (D) (nm)
1:1	30.09	(200)	25.86	3.445	5.939	5.944	5.00
1.25:1	30.07	(200)	25.96	3.429		5.939	10.42
1.5:1	25.64	(111)	25.64	3.451		5.977	18.58
1.75:1	25.64	(111)	25.64	3.451		5.977	26.94
2:1	25.64	(111)	25.64	3.469		6.008	39.14

2.2.Morphological studies

The morphology of the nanocrystalline PbS thin films is shown in **figure 2**. The stoichiometric Pb_xS_{1-x} (x=0.5) lead sulphide thin film (**figure 2b**) shows a uniform and smooth surface morphology over the entire substrate. For 1:1 molar concentration of lead precursor solution the resultant PbS thin films are found to be smooth and grains formation is not observed considerable grains formation appears to occur at 1.5:1 molar concentration. The grains are small and densely packed throughout the surface. The grains grow larger as the molar concentration of the lead precursor is increased. Larger grains of 40 nm have been observed for 2:1 molar concentration. The results suggest that the micro structural characteristics such as grain size, grain density and grains packing of the lead sulfide thin films are influenced by the lead precursor concentration. It also validates the analysis from Scherer's formula that the nanocrystallite size increases as the lead precursor concentration increases (as given in **Table 1**).

2.3.Compositional studies

Quantitative chemical analysis of the spray deposited lead sulphide thin films using EDAX spectroscopy is given in **Table 2**. The data shows that near stoichiometric PbS thin films are achieved at 1.25:1 molar concentration of lead precursor. The results indicate that the concentration of Pb ions in the PbS thin films can be varied from 45% to 55% by increasing the lead precursor concentration. The absence of additional peaks in EDAX spectra reveals that the films are purely homogeneous in nature.



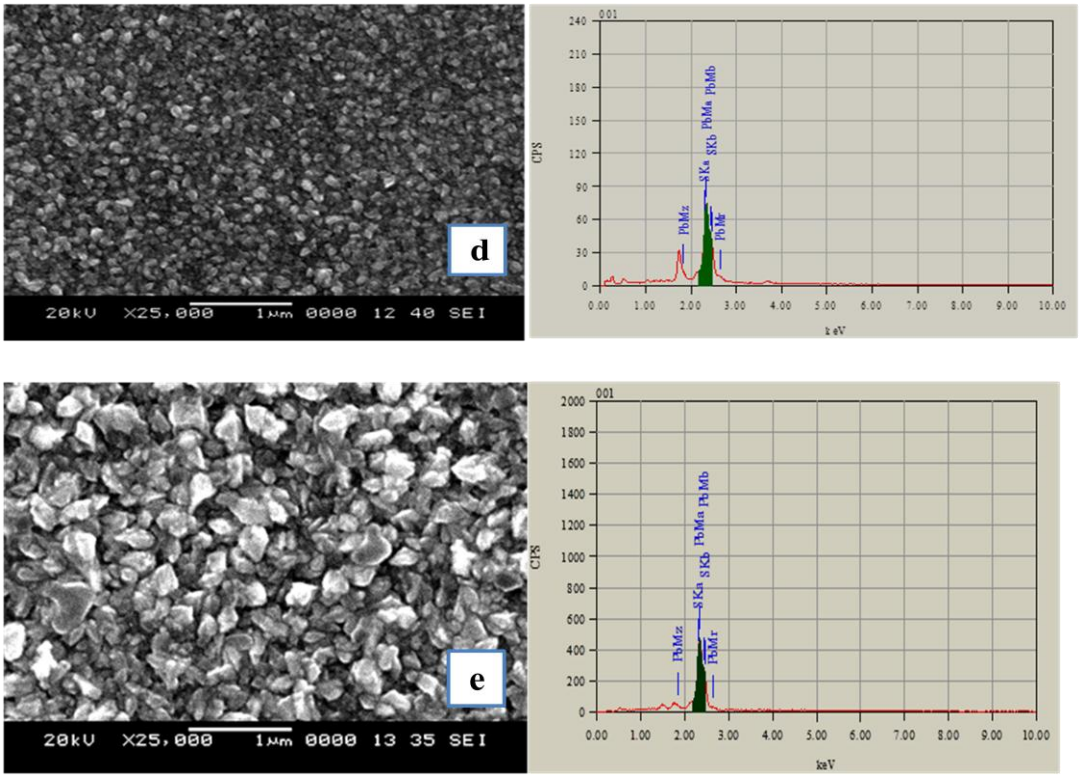


Figure 2 SEM image and EDAX spectra of PbS thin films with different precursor concentration of Pb (a) 1:1 (b) 1.25:1 (c) 1.5:1 (d) 1.75:1 (e) 2:1

Table 2 Pb:S atomic % for films deposited with various precursor solution concentration of Pb.

Pb:S precursor solution concentration ratio	atomic % of lead	atomic % of sulphur
1:1	44.03	55.97
1.25:1	50.35	49.65
1.5:1	51.49	48.51
1.75:1	53.25	46.75
2:1	55.54	44.46

2.4.Optical studies

Figure 3 shows the optical studies of the PbS thin films performed using absorption spectra recorded for different lead precursor concentration as a function of wavelength ranging from 500 nm-3000nm. The absorbance spectrum of stoichiometric PbS thin films shows the presence of two absorption thresholds one in NIR region and the other mid-IR region. From **Fig. 3** it is evident that the absorption increases linearly from 3000 nm to 1100 nm. At the second threshold near 1100 nm,

the absorption increases steeply and peaks at ˜330nm. The absorption does not reach to zero in visible region indicating that the sample is partially transparent. as the lead ion concentration is increased from 45% to 55% by increasing the molar concentration of lead precursor solution the absorption threshold wavelengths get lead shifted as observed in **Fig. 3**.

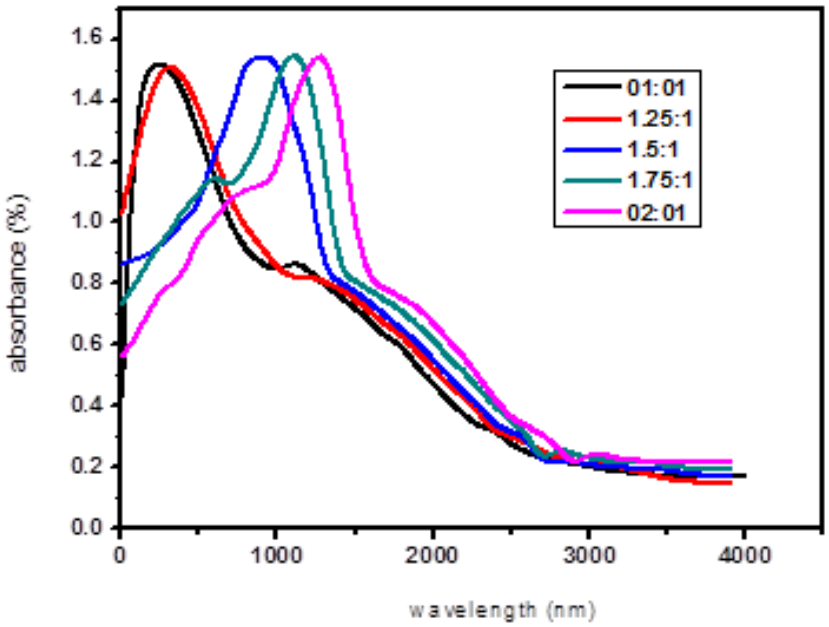


Figure 3. The absorption spectra of the films deposited with different precursor concentration of Pb.

The absorption peak shifts hugely from 330 nm (for 50% Pb) to 880 nm (for 52% Pb). Although the change in lead concentration is feeble, the micro structural characteristics of both the films show marked difference i.e., the film (with 52% Pb) looks more granular with dense packing. The granularity possibly induces strain in the film resulting in red shift of the absorption threshold and maximum absorption. The shift in absorption peaks is attributed to the increase in dangling bonds for higher Pb concentration intern elevates the density of localized states for the films as reported by douri et al for thermal evaporated PbS films [25]. The trend of red shifting in absorption peaks continues as the lead precursor concentration is increased to 2:1 (in step of 0.25). The absorption threshold at mid IR wavelength region remains almost unaltered by change in lead ion concentration.

The absorption coefficient α of the film was found to be of the order 10^6 cm^{-1} was calculated using the formula [27]

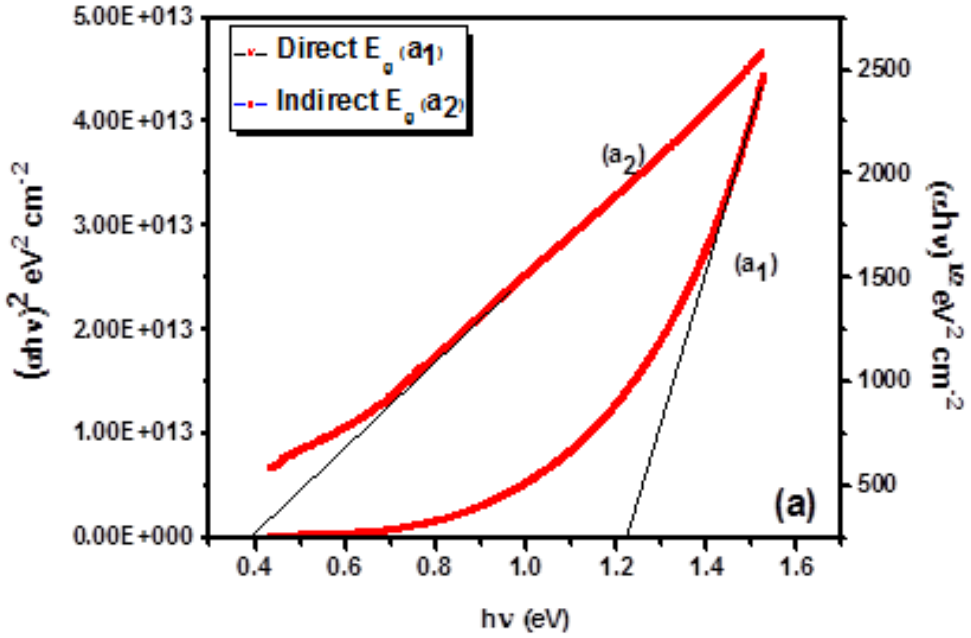
$$\alpha = {2.303A}/{t} \tag{3}$$

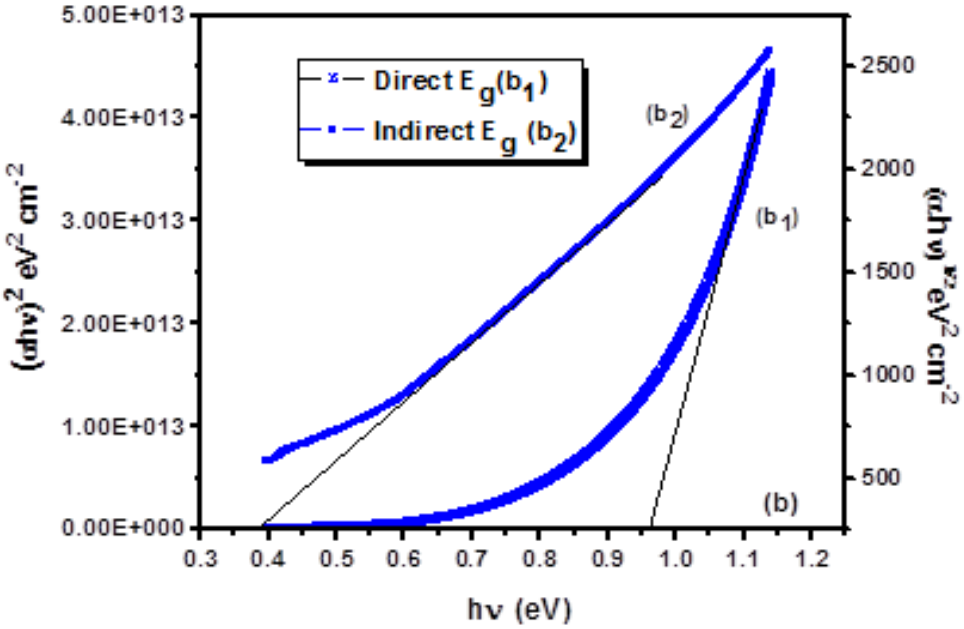
where *t* is the thickness and *A* is the optical absorbance of the films. The band gap of the material was determined using the Tauc's relation [28]

$$\alpha h\vartheta = K(h\vartheta - E_g)^n \quad (4)$$

where K is constant depends on transition probability, $h\vartheta$ is a photon energy, E_g is a band gap energy n=1/2 for allowed direct transition and n=2 for indirect transition. The optical band gap of the films has been calculated by extrapolating the linear part of $(\alpha h\vartheta)^{1/n}$ versus photon energy plot . **Fig. 4** shows the Tauc's plot for both direct and indirect transition of the films deposited with various Pb concentration. It also might be due to the creation of new states in the E_g that leads to an increase in the density of localized states, which causes a shift of E_g to lower photon energy [25]. The band gaps of lead sulphide thin films as calculated from tauc's plot are tabulated in **Table 3**. Tauc's plot shows the presence of direct and indirect band gap in the PbS thin film. This might be due to the creation of new states in E_g that leads to an increase in the density of localized states which causes the shift in E_g to lower photon energy [8, 25-23] as shown in **Table 3**.

The direct band gap energy of PbS thin films varies from 1.22 for 45% Pb to 0.78 for 55% Pb concentration. However, the indirect band gap energy remains almost constant for all the Pb ion concentrations. The optical studies reveal that as the lead precursor concentration is increased from 1:1 to 2:1. The band gap energy of the PbS films reduces from 1.2 eV to 0.78 eV. As a result optical absorption gets red shifted from visible to near-IR range.




Direct $E_g(b_1)$
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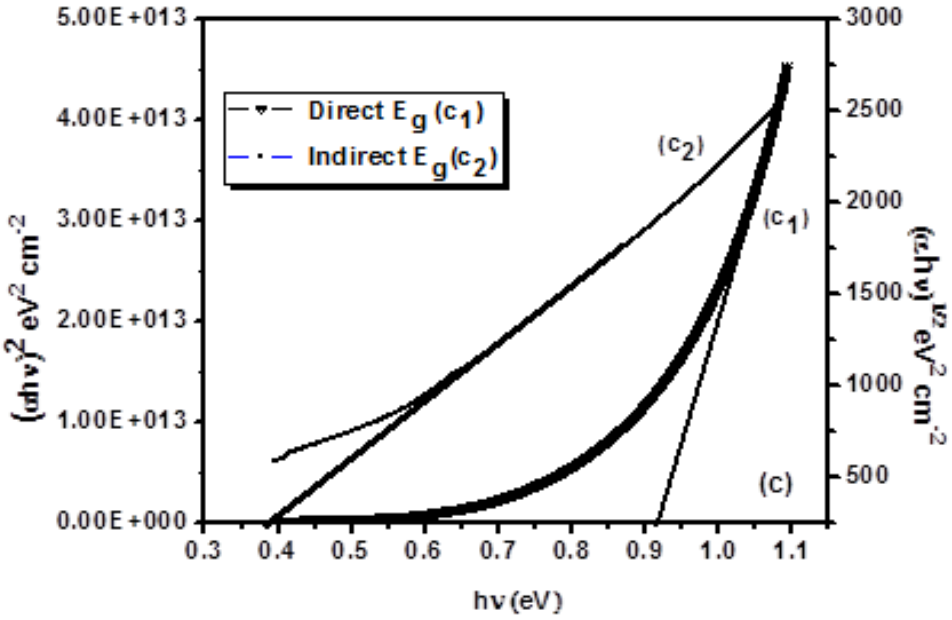
Direct E_g (c_1)
Indirect $E_g(c_2)$
(c_2)
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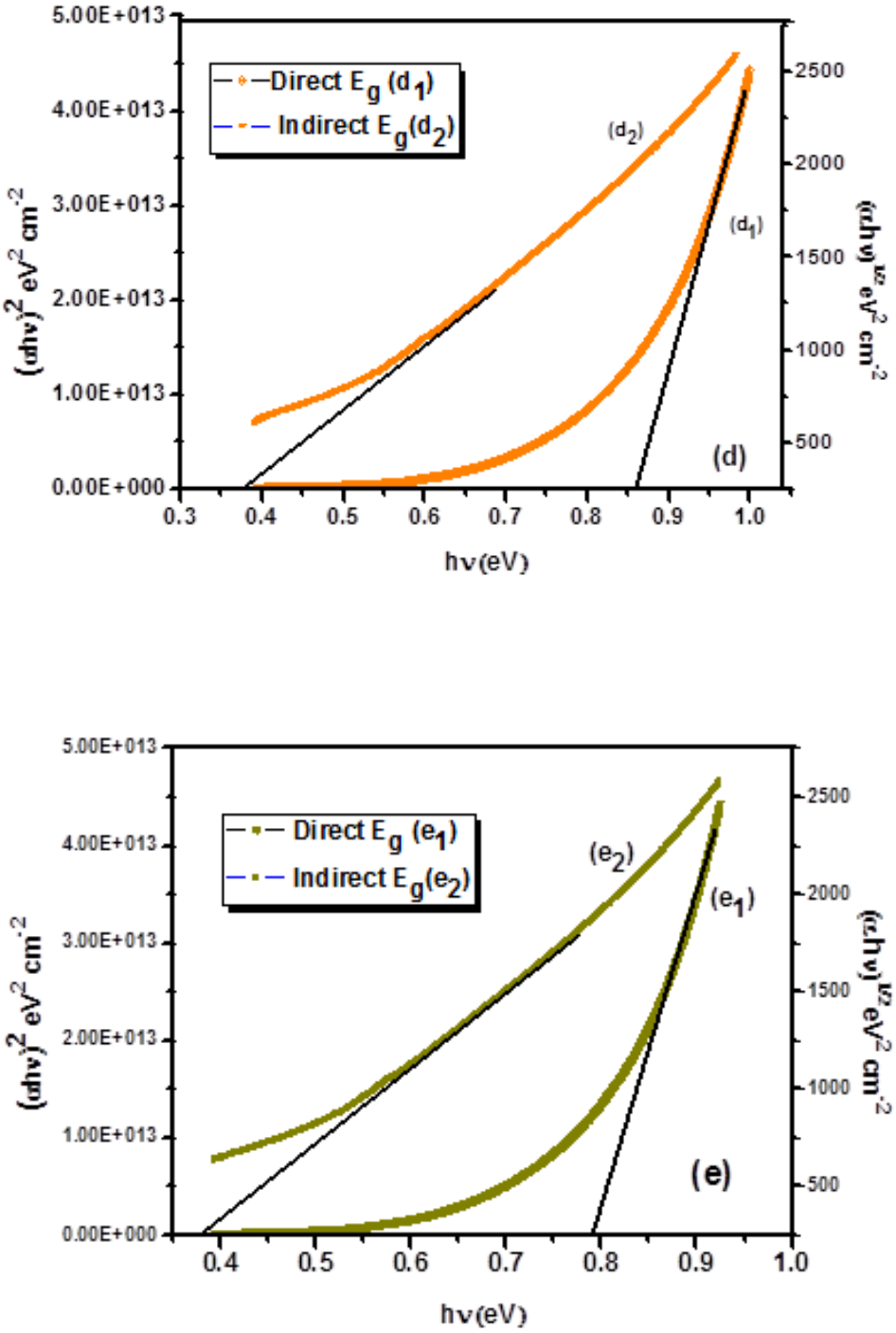

Figure 4. Tauc's plot of Pb:S thin films (a) 1:1 (b) 1.25:1 (c) 1.5:1 (d) 1.75:1 (e) 2:1 precursor concentration ratio.

Table 3: Variation of direct and indirect energy band gap with different precursor concentration of Pb.

Pb:S precursor solution ratio	Direct band gap energy (eV)	Indirect band gap energy (eV)
1:1	1.22	0.41
1.25:1	0.96	0.40
1.5:1	0.91	0.39
1.75:1	0.86	0.38
2:1	0.78	0.37

2.5.Electrical studies

The current –voltage (I-V) characteristics of PbS thin films are shown in **Fig. 5**. The PbS thin films show linear current voltage response indicating that the conduction mechanism is ohmic with silver contact. The resistivity of the PbS thin films were calculated using the relation

$$\boldsymbol{\rho = {RA}/{L}} \quad (5)$$

where *R* is the resistance of the films, *A* is the area of the film and *L* is the spacing between the electrodes. The resistivity of films found to decrease with increase in lead precursor concentration, this is due to the improvement in crystallinity and larger crystallite sizes of thin films. The resistivity of PbS thin films found to be in the order of 10^2 Ω cm which are listed in **Table 4**.

The **Fig. 6** shows Arrhenius plot (variation of log R with 1/T) of the PbS films. The resistance decreased with increased temperature shows the semiconducting nature of the films. The nature of the plot gives single slope which indicate the presence of one type of conduction mechanism in intrinsic region. The thermal activation energy was calculated using below equation [3, 22]

$$\boldsymbol{\sigma = \sigma_0 e^{-E_a/KT}} \quad (6)$$

where E_a is the activation energy, σ_0 is a constant, K is the Boltzmann's constant and T is the absolute temperature. The activation energy (as listed in **table 4**) of the films are found to decrease with increase in lead ion concentration. The decrease in activation energy also reflects in the decrease in band gap of the thin films. Further, the resistivity of the PbS thin films also decrease with decreasing activation energy and band gap. The observed resistivity values of the spray deposited PbS thin films are better for wet chemically prepared PbS thin films. The Hot probe measurement showed +ve value of V_{DC} which indicates PbS thin films were n type semiconductors with different lead precursor concentration.

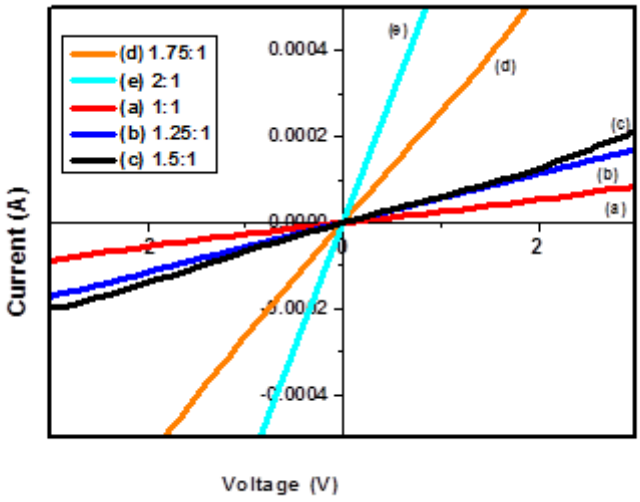

Fig. 5. I-V characteristics of the Pb:S thin films with different precursor solution concentration of Pb.

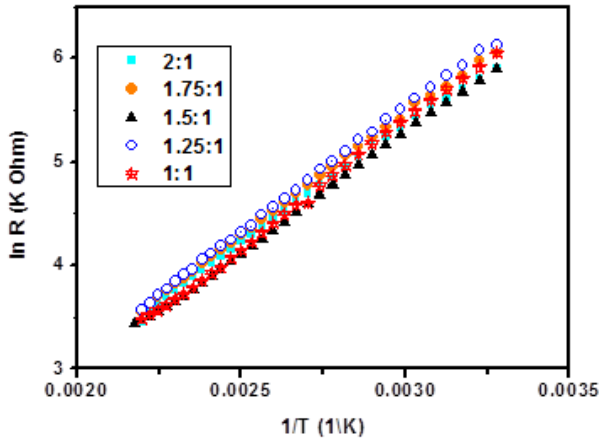

Figure 6. Variation of ln R with 1\T for PbS thin films with different lead precursor solution concentration of Pb.

Table 4 : Activation energy and resistivity measured for PbS thin films with different precursor concentration of Pb.

Pb:S precursor concentration ratio	**Activation energy (eV)**	**Resistivity ρ ($X10^2$) in Ω cm**
1:1	0.205	10.600
1.25:1	0.2	05.117
1.5:1	0.195	04.348
1.75:1	0.19	01.060
2:1	0.185	00.497

CONCLUSIONS

Lead sulphide thin films are grown by chemical spray pyrolysis technique. The lead precursor concentration is varied from 1:1 to 2:1 to improve the crystallinity and micro structural properties of PbS thin films. Properties of PbS thin films. Thin films prepared with higher lead precursor concentration exhibits better crystallinity with larger crystallite sizes as seen from XRD and SEM micro structural analysis. Further the optical properties such as the band gap and activation energy of the prepared PbS thin films are found to be influenced by the lead precursor concentration. The optical absorbance measurements indicate the presence of direct and indirect transitions with a corresponding energy gap in the range 0.78 – 1.22 eV and 0.37-0.41 eV respectively. It is also seen that the electrical band gap obtained is equal to the bulk value. The PbS thin films have resistivity as the order of 10^2 Ω cm which is highly suitable for applications such as photodiode and solar cells. Hence, the optical and electrical properties of the lead sulfide thin films prepared using chemical spray pyrolysis technique can be tuned by varying the lead precursor concentration of the spray precursor.

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