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Structural AndOptical Studies of $Pb_{1-X}Zn_xS$ Semiconductor Compounds Prepared By Co-Precipitation Method

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Abstract:$Pb_{1-x}Zn_xS$ *(x=0 to 0.4 in steps of 0.1) ternary semiconductor compounds were synthesized by co-precipitation method. XRD studies revealed that the compounds are polycrystalline in nature and their crystal structure was Cubic. Variation in density and porosity percentage, dislocation density, lattice strain and average grain size of all samples, with respect to change in Zinc concentration, were calculated. Surface morphology and chemical homogeneity studies were carried out by using SEM and EDAX. Energy gap* (E_g) *of each sample wasestimated from optical absorption studies and found that*E_g *increases with Zn concentration. The results are explained in terms of ionic size of Pb and Zn and the defects formed in the matrix.*

Keywords:$Pb_{1-x}Zn_xS$ *semiconductor; co-precipitation technique; XRD; SEM; Grain size; EDAX; Energy gap.*

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I. Introduction

In the field of materials science,compound semiconductors are always in focus due to their outstanding electronic and optical properties. These materials have extensive applications in various electronic devices including optical converters, photoconductors, optical sensors, field effect transistors, solar cells [1-5]. Recent reports showed that ternary compounds act as better window material for the preparation of p-n junctions without lattice mismatch which was encountered, usually, with quaternary compounds such as $CuLn_xGa_{1-x}Se_2$[6] and $CuLn(SSe)_2$[7].

Interest in PbS based nanomaterials was also initiated because of their unique optical absorption and emission properties which in turn found to have tremendous applications in lasers[8], light-emitting devices[9,10], detectors[11], nonlinear optics[12-14], single electron devices[15], optical switches, thermoelectric generators[16] etc. As such PbS, metal sulphide belonging to IV-VI group, has narrow energygap of 0.4eV at300K [17] with a cubic rock salt structure and itsoptical propertiespromote it as a useful agent in filter, reflector and planer waveguide [18] preparations. Likewise, Zinc Sulphide is also another important semiconductor of II-VI group having large bandgap (3.5eV at 300K), mostly available in Hexagonal structure. Consequently,the importance of these compounds promoted an idea of the preparation of a mixture of ZnS and PbS / incorporation of Zn in PbS matrixforming various mixed compounds of PbZnS with varied lattice parameters that lie in between the lattice parameters of ZnS and PbS and exhibits different optical and electrical properties. PbZnS, a ternary chalcogenide semiconductor, is shown to have direct energy bandgap wider than PbS. The mixed compound also has attracted the attention of scientists for its wide usage in solar cells and photovoltaic devices [19]. Subsequently, $Pb_{1-x}Zn_xS$ compounds were found to have less window absorption loss and greater short circuit current [20, 21].

Some studies on ternary semiconductorsprepared in thin film form such as$Zn_{1-x}Pb_xS$, $Zn_xPb_{1-x}S$, $PbZn_xS_{1-x}$ etc.were reported by other researchers[22] but studies on bulk material are limitedas their preparation involves high temperatures and melt technique requires further controls on different vapour pressures of the constituent elements[23]. As an alternate method, PbZnS compounds are prepared by controlled co-precipitation method. With this method, a series of $Pb_xZn_{1-x}S$ ternary compounds are prepared and their structural, morphological and optical studies at room temperature are made. Optical studies on these samples revealed a systematic variation of energy bandgap between the energy gaps of PbS (0.4eV) and ZnS (3.6eV) as x varies from 0 to 0.4. The results are presented and explained with suitable mechanisms.

II. Experimental

2.1 Preparation method;

$Pb_{1-x}Zn_xS$ (x=0 to 0.4 in steps of 0.1) bulk compounds were prepared by controlled co-precipitation method [24-28]. In this method, equimolar solutions of Lead acetate, Zinc acetate and Thiourea were taken in specific volumes so as to get the desired composition. The solution mixture was made alkaline by adding 25%

of Ammonium hydroxide under constant stirring process. The solution was heated at 80^0C for one hour. The colour of the solution changed from pale yellow to grey indicating the formation of precipitation. The bath was further heated for 3 hours to complete the reaction. The dried precipitate was heated for 2 hours at 300^0C under Nitrogen gas atmosphere and then cooled slowly to room temperature. The dried precipitate was ground to fine powder to obtain particles with nearly uniform size. The powder was made in to pellets and then the pellets were heated at 800^0C for 2 hours in Nitrogen gas atmosphere. The samples were cooled slowly to room temperature and are used for structural and optical studies. The process of preparation of the similarternary compounds like $Cd_xZn_{1-x}S$ by co-precipitation method was given in detail in our previous publications [25-28].

2.2 Characterization

X-ray diffraction of all samples were obtained using analytical X' pert powder X-ray diffractometer with CuK_α radiation(λ=1.5405Å) in the angular range 20 to 80 degrees at a scan speed of 0.02^0/sec. SEM and EDAX micrographs were recorded using Scanning electron microscope model ZEISSEVO-18. Optical absorption studies were carried out at room temperature in the wavelength range 200- 900nm by UV-VIS spectrometer model SHIMADZU, UV-3100.

Results and Discussion

3.1 X-Ray diffraction:

Crystal structure of $Pb_{1-x}Zn_xS$ semiconductor compounds was studied using X-Ray diffraction. Fig.1 shows X-ray diffraction patterns of all samples with x = 0 – 0.4.. The observed diffraction peak positions of $Pb_{1-x}Zn_xS$ compounds are compared with the peak positions of Cubic PbS (JCPDS 05-0592) published in literature[29] and found that, the crystal structure of $Pb_{1-x}Zn_xS$ is Cubic and assigned hkl values for all the observed peaks. The presence of cubic phase in $Pb_{1-x}Zn_x$ S with x=0 to x=0.4 may be due to the insertion of Zinc atoms of less atomic radius (compared to Lead atoms) in place of Lead atoms. Substitution of some Pb sites with Znmay cause slight alteration in the lattice parameter.

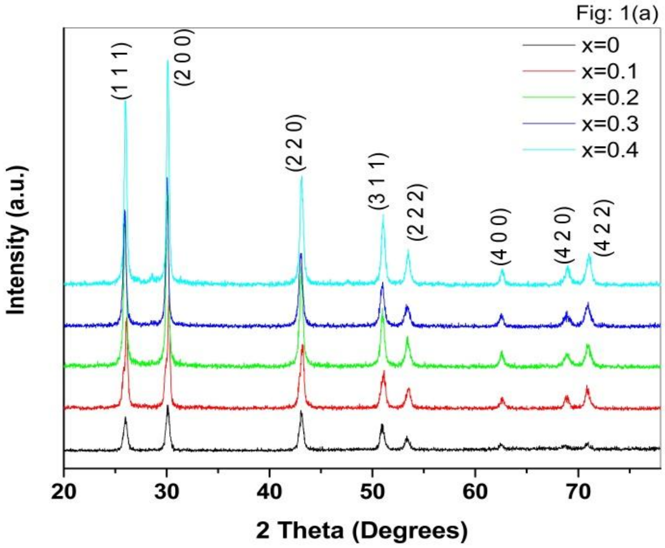


Figure 1: Cubic phase

3.2. Lattice parameter:

The lattice parameter 'a' of Cubic structure in $Pb_{1-x}Zn_x$ S was calculated according to the standard relations [30]between lattice parameter, interplanar spacing and miller indices.

$$\frac{1}{d_{hkl}^2} = \left(\frac{h^2+k^2+l^2}{a^2}\right) \quad \text{------------------ (1)}$$

The lattice parameter values, thus calculated, are in good agreement with the values reported by other researchers[31]. Estimated lattice parameters of $Pb_{1-x}Zn_x$ S are given in Table 1. Graphical variation of these lattice parameters with Zn concentration is also shown in Figs (2). The graph shows clearly that the variation of lattice parameter with Zn concentration is linear and obeys Vegards law [32, 33]. A similar variation was also observed by Hasan [22] in $Zn_{1-x}Pb_xS$ thin films. The increase in the value of ***a***may be due to the occupation of few Zn ions in the interstitial positions of the lattice.

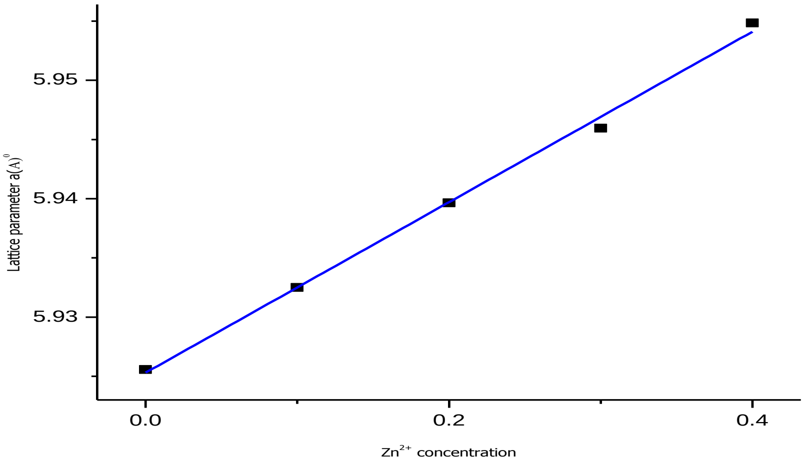


Figure2: Variation of Lattice parameter 'a' with Zn^{2+} concentration.

S No	Sample $Pb_{1-X}Zn_xS$	Lattice constant a (A^0)	FWHM	Crystallite size (D) (nm)
1	x=0	5.9256	0.33	42.7
2	x=0.1	5.9325	0.34	41.5
3	x=0.2	5.9397	0.35	40.2
4	x=0.3	5.9461	0.37	38.1
5	x=0.4	5.9549	0.39	36.1

Table 1: Variation of Lattice parameter and Crystallite sizewith Zn^{2+} concentration.

3.3 Crystallite size:

The average crystallite size (D) of $Pb_{1-x}Zn_xS$ compounds is calculated using Debye Scherer's formula [30] and present in table 1.

$$D = \frac{0.94\lambda}{\beta\cos\theta} \quad \text{-------------} \ (2)$$

Where β is FWHM of an X-ray diffraction peak at the angular position θ.

Variation of the crystallite size with Zn concentration is shown in Figs (3). From figure (3) one may notice that the crystallite size decreases with the composition parameter x. A similar result was also observed in CdZnS by Awodugba [34] et.al.

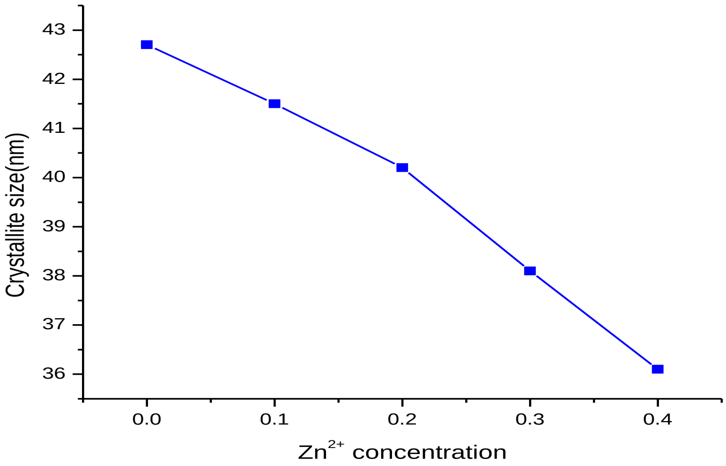


Figure 3: Plot of Grain size with Zn^{2+} concentration

3.4 Scanning electron micrographs and EDAX:

Figure (4) shows the SEM images of all Pb$_{1-x}$Zn$_{x}$S samples, taken at the same magnification. It is observed from these micrographs that the grain size of Pb$_{1-x}$Zn$_{x}$S compounds decreases with increase in Zinc concentration [35]. As Zn concentration increases the inter grain barrier height increases [36] which may cause decrease in grain size.EDAX spectra of all the samples studied are recorded in order to understand the chemical homogeneity. Figure(5) shows typical EDAX spectra of Pb$_{1-x}$Zn$_{x}$S (x=0–0.4) samples. The analysis revealed that peaks corresponding to all the elements in the compounds along with their percentages match with which they were mixed at the time of preparation. The corresponding atomic weight percentages of all the compounds taken from the EDAX spectra are given in Table (2).

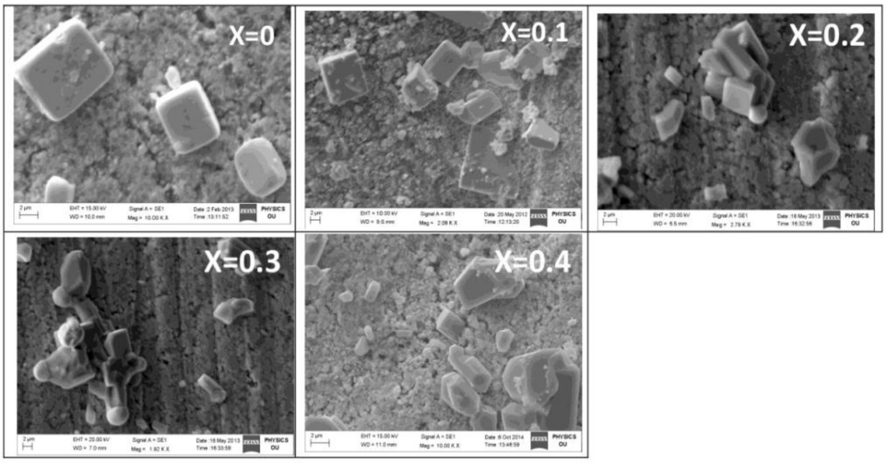


Figure 4: SEM images of Pb$_{1-x}$Zn$_{x}$S compounds.

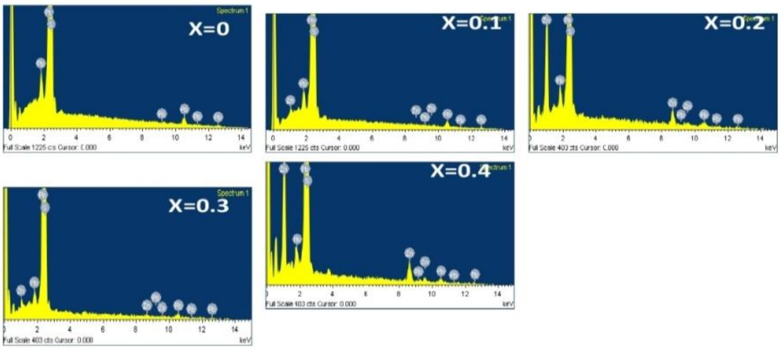


Figure 5: EDAX spectra of Pb$_{1-x}$Zn$_{x}$S compounds.

S No	Sample	Element	Weight (%) at the time of preparation	Weight (%) from EDAX
1	X=0	Pb	86.6	86.47
		S	13.4	13.53
2	X=0.1	Pb	82.85	82.63
		Zn	2.91	2.97
		S	14.25	14.4
3	X=0.2	Pb	78.6	77.83
		Zn	6.2	6.27
		S	15.2	15.9
4	X=0.3	Pb	73.7	74.26
		Zn	9.9	9.41
		S	16.3	16.33
5	X=0.4	Pb	68	67.29
		Zn	14.2	15.15
		S	17.6	17.56

Table 2: Comparison of weight percentages at the time of preparation and weight percentages obtained from EDAX.

3.5. Optical Properties:

Optical absorption of $Pb_{1-x}Zn_xS$ compounds was studied at room temperature in the wavelength range 200nm to 600nm by UV-VIS spectrometer. Absorption spectra was recorded by passing light through homogenous colloidal solution of the sample powder obtained by dispersing the powder in Glycerine by stirring it for one hour at room temperature.Fig. (6) Shows absorption spectra of all $Pb_{1-x}Zn_xS$ compounds. From these spectra it is observed that the absorption peak shifts towards shorter wavelength with the increase of Zinc concentration. The absorption studies also reveal that the prepared compounds are very low absorptive and therefore are more suitable for the fabrication of solar cells. Similar results were also obtained in CdZnS by Premkumar [37] et.al.

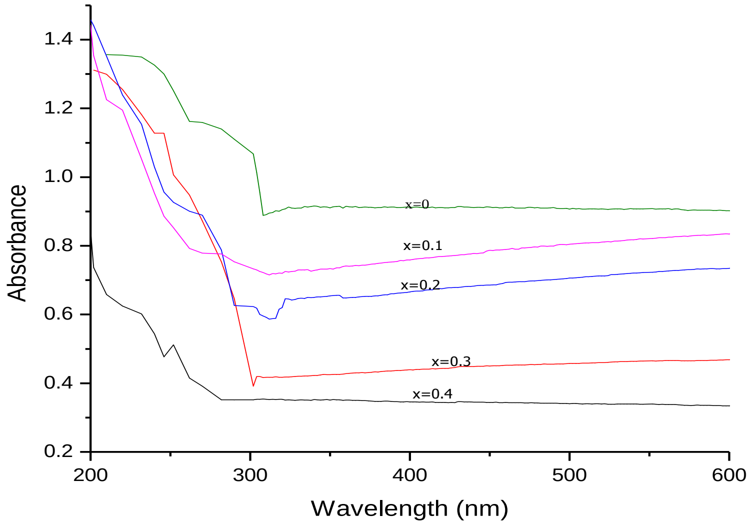


Figure 6: Absorption spectra of $Pb_{1-x}Zn_xS$ compounds.

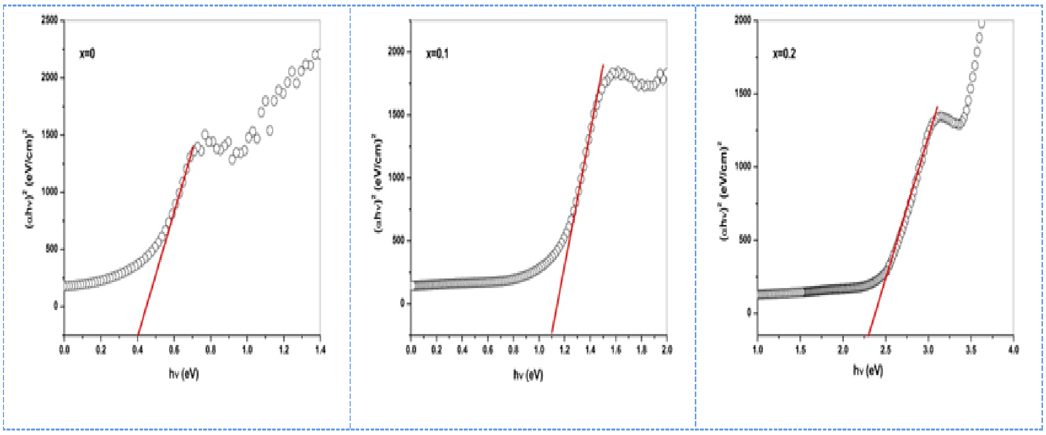
Tauc's relation [38-40]was used to calculate the energy gap of the compounds

$$\alpha h\nu = B(h\nu - E_g)^n \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(9)$$

where Eg represents energy gap, B is a constant, n is the power factor which assumes values 0.5, 2, 1.5 and 3 for allowed direct, allowed indirect, forbidden direct and forbidden indirect transitions respectively. As PbZnS compounds have direct allowed transition we consider n=0.5 for the analysis. From the absorbance relation the equation for absorption coefficient may be written as

$$\alpha = 2.303(A/L) \quad \text{------------} \ (10)$$

Where, A is absorbance and L is optical path length. Plots of $(\boldsymbol{\alpha h\nu})^2$ vs**hν** for all $Pb_{1-x}Zn_xS$ compounds are shown in Fig.(10).



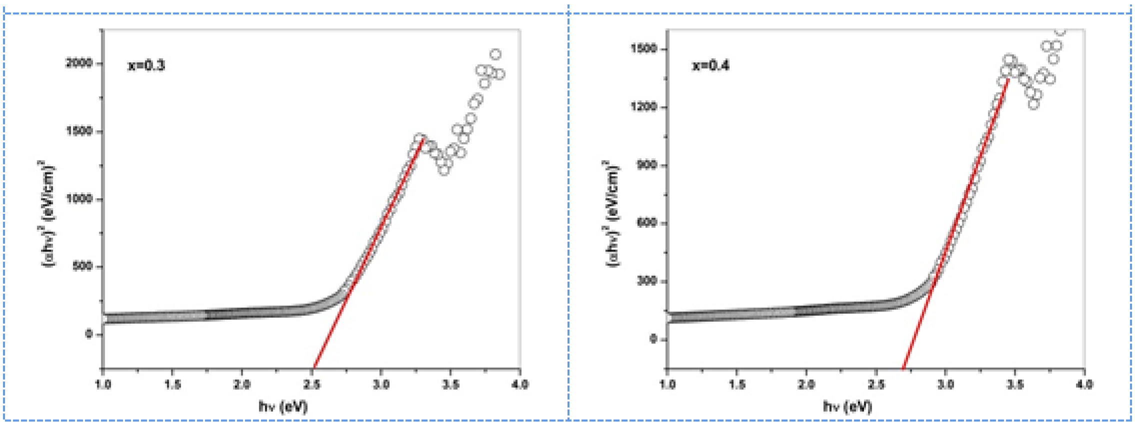


Figure 10: Plots of $(\alpha h\nu)^2$ Vshν of $Pb_{1-x}Zn_xS$ compounds.

From these graphs, direct bandgap energies are evaluated by extrapolating the linear portion of the plots for zero absorption coefficients. The energy bandgap (E_g)values thus obtained for each $Pb_{1-x}Zn_xS$ sampleis given in Table (3). It is seen that the bandgap is 0.35 eV for the compound with x=0 and the bandgap is 2.70eV for the compound with x = 0.4, more precisely, bandgap increases with increase in Zn concentration (Table 3). This may be attributed to the difference in electro negativity corresponding to the bonds S-Pb (0.3) and S-Zn (0.9). This implies that the energy required to break the bond S-Zn is higher than that of S-Pb. Moreover, the interaction energy of S-Zn is more than that of S-Pb which may in turn cause more separation between valence and conduction bands formed in the system. A similar variation of energy gap with composition was also observed in PbCdS compound [41]. The variation of Eg with composition was found to be large which confirms the formation of ternary $Pb_{1-x}Zn_xS$ alloy and the presence of strong quantum confinement in the system [41]. In the present study the energy gap variation with Zn concentration is also shown in Fig. (12)., The variation seems to have a linear behaviour with $0 \le x \le 0.4$.

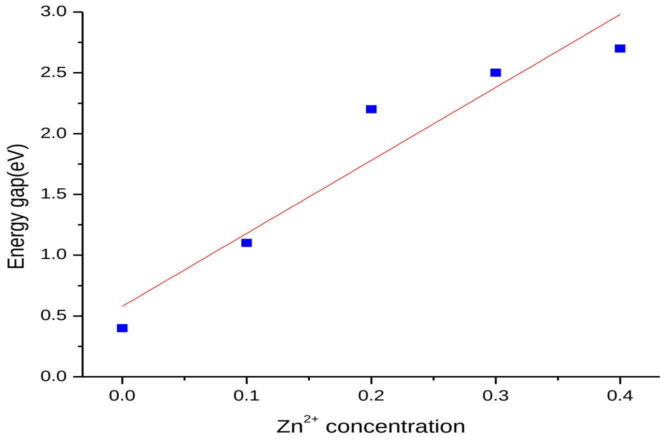


Figure 11: Variation of Energy gap with Zn^{2+} concentration.

Zn^{2+} concentration	Energygap(eV)
0	0.4
0.1	1.1
0.2	2.2
0.3	2.5
0.4	2.7

Table 4: Variation of Energygap with Zn^{2+} concentration.

From the optical studies it may be concluded that the energy gap value of a compound in PbZnS series could be controlled with the Zn concentration and this has been an important useful aspect in the fabrication of PbZnS solar cells. It is well understood that the minimum energy gap required for a material to be useful in solar cell fabrication is 1.2eV and the efficiency of a solar cell is maximum with the energy gap of 1.5eV. Also for LED to emit visible light the energy gap required is 2eV. These energy gap values (1.2eV, 1.5eV, 2eV) are not possible with the series $Cd_{1-x}Zn_xS$. Premkumar [42] et.al reported energy gap values of CdZnS thin films with annealing temperature in the range 2.27 to 3.25eV, Raymond [43]et.al reported energy gap values of ZnCdS nano clusters in the range of 2.5 to 3.73eV, Mahadi [44]et.al reported energy gap values of CdZnS in the range of 2.42 to 3.7eV and similarly Di Xia[45] et.al reported energy gap value of $Cd_{0.8}Zn_{0.2}S$ thin films as 2.59eV. So, $Pb_{1-x}Zn_xS$ compounds are found to be more suitable for the fabrication of solar cells and as well as LEDs to emit visible light.

II. Conclusions

1. $Pb_{1-x}Zn_xS$ samples are prepared by co-precipitation method to study the influence of Zn on the physical properties of the samples.
2. XRD studies revealed that $Pb_{1-x}Zn_xS$ compounds have polycrystalline nature.
3. The percentage of density decreases and porosity increases in $Pb_{1-x}Zn_xS$ with the increase in Zinc concentration.
4. Average crystallite size decreases with the increase in Zinc concentration.
5. Energy gap of $Pb_{1-x}Zn_xS$ increases with Zn concentration. Absorption measurement and Energy gap determination of the prepared samples confirm its applicability in the fabrication of solar cells and LEDs.

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