Research Article

Rutherford Backscattering Spectrometry Analysis and Structural Properties of $\text{Zn}_x\text{Pb}_{1-x}\text{S}$ Thin Films Deposited by Chemical Spray Pyrolysis

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Zinc lead sulphide ternary thin films were prepared by chemical spray pyrolysis on soda lime glass substrates using zinc acetate, lead acetate, and thiourea sources precursor. The films were characterized using Rutherford backscattering (RBS) spectrometry, energy dispersive X-ray (EDX) spectroscopy, scanning electron microscopy (SEM), and X-ray diffractometry (XRD). RBS studies revealed variation in thickness and stoichiometry of the films with respect to compositional substitution between Zn and Pb, thereby giving effective composition $\text{Zn}_x\text{Pb}_{1-x}\text{S}$, where $x = 0, 0.035, 0.069, 0.109, 0.176$, and 0.217 . Film thickness obtained by length conversion ranged from 81.02 nm to 90.03 nm. Microstructural analyses also indicated that the growth and particle distribution of the films were uniform across substrate's surface. Diffraction studies showed that the films possess FCC crystalline structure. Crystallite size reduced from 14.28 to 9.8 nm with increase in Zn^{2+} in the $\text{Zn}_x\text{Pb}_{1-x}\text{S}$ samples.

1. Introduction

Thin films of metal chalcogenides particularly Pb, Cd, and Zn have received much attention due to their importance in photovoltaic and optoelectronic semiconductor devices mostly due to the fulfillment of some of the requirements that are essential for device fabrication [1–3]. Most importantly, ternary chalcogenide semiconductors are of special interest in recent time based on the fact that their energy gap and lattice parameters can be adjusted to enhance their optoelectronic and photovoltaic characteristics [4, 5]. Many studies have been carried out on ternary chalcogenide semiconductor thin films. These include $\text{Zn}_{1-x}\text{Cd}_x\text{Se}$ [1], $\text{Zn}_{1-x}\text{Fe}_x\text{S}$ [6], $\text{Cd}_{1-x}\text{Fe}_x\text{Se}$ [7, 8], $\text{Cd}_{1-x}\text{Zn}_x\text{S}$ [9, 10], and $\text{Cd}_x\text{Pb}_{1-x}\text{S}$. However, to the authors' knowledge little or no reports are currently available on synthesis of $\text{Zn}_x\text{Pb}_{1-x}\text{S}$ chalcogenide thin films using spray pyrolysis method. Despite the promising and novel characteristics of chalcogenide semiconductors thin films, the use of this class of material in optoelectronic

devices may be held back by the difficulty in preparing chemically stable thin films with desired properties. Hence, some technical challenges have to be surmounted in order to prepare chemically stable thin films that could be widely used in optoelectronic device fabrication such as solar cell, light emitting diode, and photodiode. Various deposition techniques such as chemical vapor deposition, vacuum deposition, solution growth, and sputtering have been widely reported for thin film preparation. Limitations such as high cost and stringent growth process conditions (e.g., temperature, pressure, environment, and time) attributed to most of these techniques have negative impacts on the desired film's properties. Spray pyrolysis is essentially thermally stimulated reaction between clusters of ions or atoms of different chemical species. In spray technique a solution containing soluble salts of the constituent atoms of the desired compound is sprayed on a substrate maintained at elevated temperatures. Spray pyrolysis is a useful alternative to the traditional methods for obtaining zinc lead sulphide ternary compound thin films.

TABLE 1: Summary of $\text{Zn}_x\text{Pb}_{1-x}\text{S}$ deposition precursor.

Sample	Precursors (concentration, volume)
ZPS1	$\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$ (0.1 M, 0 mL) + $\text{Pb}(\text{CH}_3\text{COO})_2 \cdot 3\text{H}_2\text{O}$ (0.1 M, 10 mL) + $\text{CS}(\text{NH}_2)_2$ (0.1 M, 20 mL)
ZPS2	$\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$ (0.1 M, 0.2 mL) + $\text{Pb}(\text{CH}_3\text{COO})_2 \cdot 3\text{H}_2\text{O}$ (0.1 M, 9.80 mL) + $\text{CS}(\text{NH}_2)_2$ (0.1 M, 20 mL)
ZPS3	$\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$ (0.1 M, 0.4 mL) + $\text{Pb}(\text{CH}_3\text{COO})_2 \cdot 3\text{H}_2\text{O}$ (0.1 M, 9.60 mL) + $\text{CS}(\text{NH}_2)_2$ (0.1 M, 20 mL)
ZPS4	$\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$ (0.1 M, 0.6 mL) + $\text{Pb}(\text{CH}_3\text{COO})_2 \cdot 3\text{H}_2\text{O}$ (0.1 M, 9.40 mL) + $\text{CS}(\text{NH}_2)_2$ (0.1 M, 20 mL)
ZPS5	$\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$ (0.1 M, 0.8 mL) + $\text{Pb}(\text{CH}_3\text{COO})_2 \cdot 3\text{H}_2\text{O}$ (0.1 M, 9.20 mL) + $\text{CS}(\text{NH}_2)_2$ (0.1 M, 20 mL)
ZPS6	$\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$ (0.1 M, 1.0 mL) + $\text{Pb}(\text{CH}_3\text{COO})_2 \cdot 3\text{H}_2\text{O}$ (0.1 M, 9.0 mL) + $\text{CS}(\text{NH}_2)_2$ (0.1 M, 20 mL)

It has several advantages over other deposition processes which include scalability of the process, cost effectiveness, allowance of intentional doping, and low or moderate growth temperatures (100–400°C). These unique properties create different possibilities such as use of variety of substrates, easy control of thickness, variation of film composition, and possibility of multilayer growth [11, 12].

In this study, we present results on zinc lead sulphide ($\text{Zn}_x\text{Pb}_{1-x}\text{S}$) thin films deposited on soda lime glass substrate using chemical spray pyrolysis. Rutherford backscattering spectroscopy (RBS) was essentially used to study thickness profile and stoichiometric compositions of the films. EDX was used as complementary technique for identification of films constituents. Surface microstructure and crystal orientation were examined by scanning electron microscopy (SEM) and X-ray diffractometer (XRD).

2. Materials and Methods

2.1. Film Deposition Procedure. Soda lime glass substrates were cleaned by washing with detergent and rinsed with distilled water before ultrasonic cleaning in acetone, methanol, and isopropyl alcohol (IPA) bath each for 20 minutes, respectively. The substrates were dried and kept in the vacuum oven in order to minimize extraneous contamination. The precursors for the $\text{Zn}_x\text{Pb}_{1-x}\text{S}$ thin films were obtained by preparing equal molar concentration of commercially available lead acetate, zinc acetate, and thiourea in varying proportions depending on the expected concentration of each cation in the thin films. The mixing ratio of the precursors corresponding to each sample designation (ZPS1–ZPS6) is summarized in Table 1. The freshly prepared solutions were stirred thoroughly for several minutes before stepwise spraying onto preheated clean substrates maintained at $(250 \pm 5)^\circ\text{C}$. Suitable $\text{Zn}_x\text{Pb}_{1-x}\text{S}$ thin films were obtained by maintaining deposition parameters such as substrate temperature, carrier gas flow rate, and pressure that were at $(250 \pm 5)^\circ\text{C}$, $(3\text{--}3.5)\text{ dm}^3/\text{min}$, and 2.3 bar, respectively. The chemical reaction mechanism of $\text{Zn}_x\text{Pb}_{1-x}\text{S}$ thin film is as shown below in

2.2. Characterization Techniques. Ion beam analysis (IBA) of films was carried out at the Centre for Energy Research and Development (CERD), Obafemi Awolowo University, Ile-Ife, Nigeria. The facility has a general purpose end station for particle induced X-ray emission (PIXE), Rutherford backscattering spectrometry (RBS), elastic recoil detection analysis (ERDA), and particle induced gamma-ray emission (PIGE). Film composition, thickness, and depth profile were concurrently obtained by a 2.2 MeV helium particle. SIMNRA fitting code was used to carry out the spectra analyses of the RBS spectra. Scanning electron microscope (Carl Zeiss MA-10 SEM) with attached EDX facility was used to investigate surface morphology of the films. Structural studies were carried out using GBC EMMA X-ray diffractometer with $\text{CuK}\alpha$ radiation ($\lambda = 1.5418 \text{ \AA}$).

3. Results and Discussions

3.1. RBS Analyses. The elemental composition, stoichiometry, and thickness of the deposited $\text{Zn}_x\text{Pb}_{1-x}\text{S}$ thin films were determined using RBS technique. The SIMNRA *Simplex* code was used to fit the simulation over experimental data and give information regarding the stoichiometry and areal concentration. Previous study [13] has shown that composition of compound semiconductors has a strong influence on their properties. Therefore, exact control of composition and accurate analysis of elements of these thin films are prerequisite for preparing high quality and chemically stable thin films. Both the heavy elements (Pb, Zn) and the relatively light element (S) were detected concurrently via the backscattering process. The spectra of simulated one-layer structure of $\text{Zn}_x\text{Pb}_{1-x}\text{S}$ films with varying concentration of Zn are presented in Figures 1(a)–1(f). From distinct nature of the peaks shown in the spectra, it can be inferred that the thin films are relatively uniform and adhere to the substrate well and without diffusion between the layer of the films and the substrate. The results further suggested a thermal decomposition of the precursors to produce stable Zn-Pb-S compound with little or no trace of impurities such as carbon and hydrogen which were part of the constituents of the starting chemicals. Presence of such impurities affects film functionality. In all the spectra, it is also observed that the peaks corresponding to each element are well separated. This is due largely to the differences in the mass of the elements making up the target nuclei, namely, zinc, lead, and sulphur. Peak corresponding to Pb is more enhanced than that of S despite comparable

FIGURE 1: RBS spectra of deposited thin films of $\text{Zn}_x\text{Pb}_{1-x}\text{S}$ for (a) ZPS1, (b) $x = \text{ZPS2}$, (c) $x = \text{ZPS3}$, (d) $x = \text{ZPS4}$, (e) $x = \text{ZPS5}$, and (f) $x = \text{ZPS6}$.

TABLE 2: Elemental compositions and thickness profiles of $\text{Zn}_x\text{Pb}_{1-x}\text{S}$ thin films.

Sample code	Compositions (%)			RBS thickness (10^{15} atoms/cm)	Linear thickness (nm)
	Zn	Pb	S		
ZPS1	—	40.58	59.42	155.220	81
ZPS2	1.65	47.37	50.98	156.147	82
ZPS3	3.15	45.83	51.02	156.121	82
ZPS4	4.84	44.57	50.59	156.318	82
ZPS5	7.45	42.30	50.25	157.452	82
ZPS6	8.84	40.82	50.34	172.465	90

concentrations during preparation. This can be attributed to distinctive interaction of the projectile ion ($^4\text{He}^+$) with Pb nuclei which is much heavier than sulphur in the target, thereby yielding improved backscattering signal. Compositional results of the $\text{Zn}_x\text{Pb}_{1-x}\text{S}$ films from the RBS experiment are presented in Table 2. Notable changes are observed in the concentration of elemental composition of the films. This is as a result of incremental variation of number of Zn ions with respect to volume of the zinc acetate in the precursor solution. It therefore implies that using spraying pyrolysis, it is convenient to build into the precursor the desired concentration of dopants in thin films. Expectedly, as the concentration of zinc acetate was increased in the solution, the concentration of zinc ions increased, reaching 8.84% per mL in the 30 mL precursor. Film thickness was obtained in RBS unit (atoms/cm²) as shown in Table 2. In order to obtain it in S.I unit, areal density unit was approximated into the length unit using (2) [14]. Since the densities of the deposited films are not known directly and may differ from that of bulk Zn-PbS, therefore, Zn due to its relatively low concentrations in the films could as well be taken as a dopant element. Based on the foregoing, a density similar to stoichiometric PbS (7.60 g/cm³) was assumed and a factor of 0.523 was obtained for the conversion of the atoms area density (10^{15} atoms cm⁻²) into nanometer. The corresponding length unit values are given in Table 2:

$$\text{Thickness} = \frac{\text{atoms per unit area}}{\text{atoms per unit volume}}. \quad (2)$$

In order to complement the composition results obtained from backscattering experiments, full scale EDX scan was done at beam kinetic energy of 20 keV. Analysis was carried out using INCA Point ID software for quant optimization. Since the facility is not exclusive in quantitative study, it could only be used for identification of elements. Hence, three samples were selected to understand composition of all the deposited films. Spectrum shown in Figure 2(a) representing samples ZPS2–ZPS6 confirmed the presence of lead, zinc, and sulphur while Figure 2(b) confirmed lead and sulphur for sample ZPS1 with no contamination detected. Figure 2(c) showed the elemental characteristics of the soda lime glass slide used as the substrate. These results further justify the composition results obtained from the RBS analysis.

3.2. Morphological Analysis. Surface uniformity, particle distribution, and porosity (microstructural properties) of

the deposited $\text{Zn}_x\text{Pb}_{1-x}\text{S}$ thin films were appraised from SEM micrographs. The images shown in Figures 3(a)–3(f) correspond to each of the samples ZPS1 to ZPS6, respectively. From the micrographs, it can be seen that all the films are composed of distinct crystallites that are well distributed across the substrate. Considering image in Figure 3(a) (sample without zinc), it can be seen that the film is well crystalline and its particles are closely packed. There is no observable crack or pinhole. Images of other samples (see Figures 3(b)–3(f)) possess similar features with a number of observations: (i) particle size appears smaller than in Figure 3(a) and (ii) particles are coalesced and formation of polycrystal is enhanced as percentage contribution of Zn increases. Thus, it can be suggested that introduction of zinc can cause slight reduction in the particle size. Increasing the Zn^{2+} in the $\text{Zn}_x\text{Pb}_{1-x}\text{S}$ films caused an increase in surface diffusion.

3.3. Structural and Crystallographic Analysis. The XRD spectra were used to determine the structure and crystallographic orientation of the samples. The diffraction patterns of the $\text{Zn}_x\text{Pb}_{1-x}\text{S}$ thin film prepared by CSP are shown in Figure 4. The diffraction patterns indicate that the prepared $\text{Zn}_x\text{Pb}_{1-x}\text{S}$ thin films are polycrystalline in nature with the existence of sharp and well defined peaks.

As shown in Figure 4, all the prominent peaks of each spectrum are indexed to the rock salt (NaCl) structure of PbS, according to the value of the reference standard JCPDS (card number 5-592) [15, 16]. The prominent peaks indicate that the crystals have preferred orientations at the planes observed at (111), (200), (220), (311), and (222). The relative diffraction peaks intensities of the most three prominent peaks (I_{111}/I_{200}) and (I_{220}/I_{200}) of the prepared $\text{Zn}_x\text{Pb}_{1-x}\text{S}$ thin films are shown in Table 3. Both the intensity ratios (I_{111}/I_{200}) and (I_{220}/I_{200}) for thin films grown are within the range (0.14–0.24) and (0.12–0.20); these values are lower than the JCPDS standard of 0.84 and 0.57, respectively, showing that the samples prepared have (200) preferred orientation with strong intensity.

In addition, the diffraction angle of the peaks shifts slightly with the addition of Zn^{2+} . Also, as the concentration of Zn^{2+} increases, the intensity of diffraction peaks decreases. The slight shifts in the diffraction peaks and decrease in peak intensity with increase of Zn^{2+} in the $\text{Zn}_x\text{Pb}_{1-x}\text{S}$ thin films can be attributed to induced structural disorder in the films [17]. The slight shift of the diffraction peak angle could also be as a result of the increase in heterogeneity of the films due to

FIGURE 2: EDX spectrum of deposited thin films on soda lime glass substrate: (a) ZPS2, (b) ZPS1, and (c) soda lime glass.

TABLE 3: I/I_0 ratio of prominent peaks of the prepared films and that of the JCPDS standard.

Relative intensity ratio	JCPDS standard	ZPS1	ZPS2	ZPS3	ZPS4	ZPS5	ZPS6
I_{111}/I_{200}	0.84	0.14	0.15	0.22	0.24	0.24	0.24
I_{220}/I_{200}	0.57	0.12	0.14	0.17	0.21	0.22	0.22

the occupation of Zn^{2+} into the host lattice. Crystal size of the samples was estimated using Debye Scherer's formula [18] given by

$$D = \frac{K\lambda}{\beta \cos \theta}, \quad (3)$$

where D is the grain size, λ is the wavelength (1.5406 Å for $\text{CuK}\alpha$ radiation), β is the full width at half maximum (FWHM), and $K = 0.94$ is the shape factor approximately equal to unity.

The lattice parameters were estimated using the combination of Bragg's law and plane spacing equation for the rock salt (NaCl) structure given by (4) [19, 20]. The computed crystals size and the lattice constants (a) are presented in Table 3:

$$\sin^2 \theta = \frac{\lambda^2}{a^2} (h^2 + k^2 + l^2). \quad (4)$$

TABLE 4: Computed crystals size and the lattice constants for thin film samples.

Sample	Lattice constant (Å)	Crystal size using Scherer's formula (nm)	Crystal size using modified Scherer's formula (nm)	Residual strain
ZPS1	5.942	14	15	3.125×10^{-3}
ZPS2	5.952	13	13	3.22×10^{-3}
ZPS3	5.961	12	13	3.85×10^{-3}
ZPS4	5.973	12	12	5.75×10^{-3}
ZPS5	5.989	11	11	6.70×10^{-3}
ZPS6	5.995	10	10	7.00×10^{-3}

The crystal size decreases from 14.28 to 9.8 nm as the concentration of Zn increases in the $\text{Zn}_x\text{Pb}_{1-x}\text{S}$ thin film prepared. The lattice constant increased from 5.942 to 5.995 Å as the Zn^{2+} in $\text{Zn}_x\text{Pb}_{1-x}\text{S}$ increases from ZPS1 to ZPS6 (Table 4).

However, during thin film deposition lattice mismatch between the film and the substrate with the influence of the deposition conditions such as the temperature, composition, and deposition rate can also create strain [21]. Therefore, the

FIGURE 3: SEM micrographs of deposited thin film of $\text{Zn}_x\text{Pb}_{1-x}\text{S}$ for (a) ZPS1, (b) ZPS2, (c) ZPS3, (d) ZPS4, (e) ZPS5, and (f) ZPS6.

residual strain on the deposited films was calculated using the modified Scherer's equation [22] expressed in

$$\beta = \frac{K\lambda}{D \cos \theta} + 4\epsilon \tan \theta, \quad (5)$$

where ϵ is the residual strain and D is the grain size. From the data obtained from the diffraction patterns of the prepared $\text{Zn}_x\text{Pb}_{1-x}\text{S}$ thin film shown in Figure 4, the grain size D and residual strain (ϵ) are deduced from the intercept and slope of the plot of $\beta \cos \theta$ versus $\sin \theta$. Hence the increase of Zn in

FIGURE 4: XRD pattern of deposited thin film of $\text{Zn}_x\text{Pb}_{1-x}\text{S}$.

the $\text{Zn}_x\text{Pb}_{1-x}\text{S}$ causes slight increase in residual strain in the film from 3.125×10^{-3} to 7.0×10^{-3} . This may be due to the replacement of some larger lead ions (1.21 Å) by smaller zinc ions (0.74 Å). Similar results were reported for Mn-doped CdS films [23].

4. Conclusion

Zinc lead sulphide thin films have been synthesized successfully by chemical spray pyrolysis without any complexing agent, otherwise using common chemical reagents. The desired composition was built into the precursors prior to deposition. Rutherford backscattering experiment revealed changes in the thickness with stoichiometry. Direct observation from the micrographs showed that the films are well crystalline with closely packed particles. Film diffraction analysis indicated direct relationship between lattice constant, residual stress, and Zn compositions. Thus, by suitably controlling the Zn composition in the layers, the $\text{Zn}_x\text{Pb}_{1-x}\text{S}$ films may have minimum lattice mismatch compared to that of pure PbS in forming heterojunction device.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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