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Proceedings of the Nigerian Society of Physical Sciences

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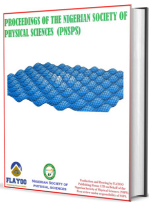

Effect of concentration on the crystallographic and FTIR properties of lead sulphide (PbS) thin films

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ABSTRACT

This study reports the use of the successive ionic layer adsorption and reaction (SILAR) technique as a low-cost and rapid method for depositing lead sulphide (PbS) thin films. The films were deposited on soda-lime glass (SLG) substrates by varying the molar concentrations of lead ions (0.3, 0.5, and 0.7 M) at a constant SILAR cycle number of 25. X-ray diffraction (XRD) patterns showed that the deposited PbS thin films were crystalline and had a cubic structure. Fourier-transform infrared (FTIR) spectroscopy indicated nanocrystalline PbS thin films with weak-to-medium M–O bonding. The O–H stretching vibration appeared at 3419.10 cm^{-1}. Bands at 1338.90 and 1220.10 cm^{-1} were associated with improved crystallinity and C–O stretching vibrations. N–H group vibrations were observed at 1686.42 and 1597.87 cm^{-1}, whereas CH_2 bending vibrations occurred between 1482.79 and 1441.44 cm^{-1}.

Keywords: PbS thin film, SILAR cycle, Nanocrystallinity.

DOI: 10.61298/pnspsc.2026.3.339

1. INTRODUCTION

Lead sulphide (PbS) thin film is a narrow-band-gap semiconductor used in sensors, infrared (IR) detectors, and optics. PbS is a group IV–VI semiconductor with a narrow direct band-gap energy of 0.41 eV at room temperature and a relatively large exciton Bohr radius of 18 nm [1]. It has been widely used in applications such as Pb^{2+}-ion-selective sensors, photography [2], and IR detectors [3].

Thin films are nanostructured materials with thicknesses on the order of 1 μm or less. They are distinct from bulk materials because of their special properties and are important because they offer exceptional functional qualities. Their chemical composition can be determined using methods such as X-ray photoelectron spectroscopy (XPS) and Rutherford backscattering spectroscopy (RBS). These methods reveal details about the constituents of the film and their relative amounts [2]. The crystalline structure of a thin film can vary depending on the deposition method and process variables. Techniques such as transmis-

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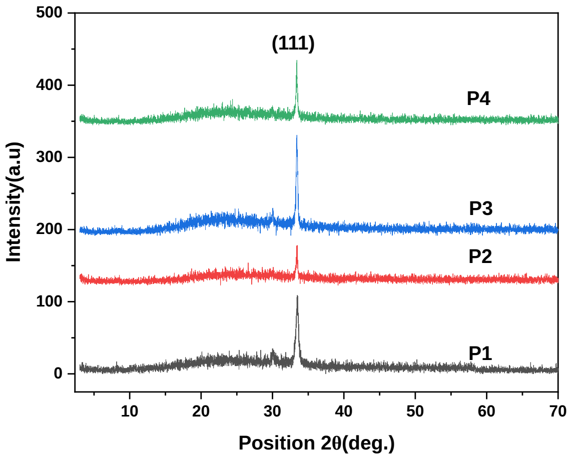

Figure 1. X-ray diffraction pattern of PbS thin films at varied molar concentrations.

sion electron microscopy (TEM) and XRD can be used to characterize the structure. Specific optical characteristics, such as high or low reflectivity, can also be engineered into thin films. Applications including solar cells, displays, waveguides, and photodetector arrays depend on these characteristics. The substrate and material type, whether insulator, metal, or semiconductor, affect the electrical properties of thin films. Because of size effects, shorter mean free paths, and increased scattering at structural defects and grain boundaries, thin films may be less electrically conductive than bulk materials [4]. The thickness and microstructure of thin films also affect their mechanical characteristics. In contrast to their bulk equivalents, thin films can behave differently and may have improved mechanical properties, such as yield strength and hardness. Grain boundaries, dopants, and dislocations are examples of microstructural features that can contribute to increased hardness. Magnetic thin films are used in applications such as memory disks, and their magnetic properties can be adjusted in different ways [1, 2]. Because of their thermal properties, thin films can also be used to make heat sinks and barrier layers. Depending on the application, such films may provide heat dissipation or thermal insulation.

Successive ionic layer adsorption and reaction (SILAR) is a chemical deposition technique that offers a low-cost, fast, and scalable route for depositing PbS thin films [1]. Few studies have reported the effect of concentration on the crystallographic and FTIR properties of PbS thin films deposited via the SILAR technique; this gap provides the justification for the present study. Accordingly, this study deposits PbS thin films via the SILAR technique and investigates how the concentration of $Pb(NO_3)_2$ affects the crystallographic and FTIR properties of the deposited films.

2. EXPERIMENTAL DETAILS

The deposition technique used in this study was the successive ionic layer adsorption and reaction (SILAR) method. Lead nitrate [$Pb(NO_3)_2$] and thiourea [$SC(NH_2)_2$] were used as precursors. Thiourea was used as the sulphide ion source, while lead nitrate was used as the lead ion source. Sodium hydroxide (NaOH) and soda-lime glass (SLG) substrates were used as the complexing agent and substrates, respectively. All analytical-grade chemicals were sourced from Sigma-Aldrich and used as received without further purification.

The deposition of the thin film was carried out using SLG substrates. A four-beaker system, labelled A, B, C, and D, was used for deposition. To remove dirt from the glass slides and provide nucleation centres for thin-film growth, the slides were washed with detergent and cotton wool, then allowed to air-dry. The PbS bath was prepared using 20 mL of 0.5 M $Pb(NO_3)_2$ and 15 mL of 0.5 M NaOH in a 50 mL beaker (beaker A). The solution was stirred for 3 min with a magnetic stirrer at room temperature to obtain a homogeneous mixture. Another 50 mL beaker (beaker C) was filled with 20 mL of 0.5 M thiourea [$SC(NH_2)_2$]. Two additional 50 mL beakers (beakers B and D) were each filled with 20 mL of distilled water. The solutions in beakers A and C represented the cationic and anionic precursors, respectively, while NaOH served as the complexing agent.

The bath was prepared by varying the $Pb(NO_3)_2$ concentration as 0.3, 0.5, and 0.7 M to obtain different bath concentrations. The substrates were placed vertically in the beaker during each immersion to prevent tilting or falling. Each deposition cycle had a total dip time of 120 s. One SILAR cycle comprised four stages: (i) adsorption of lead species for 30 s; (ii) rinsing with distilled water for 30 s to remove excess loosely bound lead species; (iii) formation of stable PbS by reaction with the thiourea precursor solution for 30 s; and (iv) final rinsing with distilled water for 30 s to remove excess, unreacted, or powdery PbS species. PbS thin films were deposited on SLG substrates for 25 cycles at lead ion concentrations of 0.3, 0.5, and 0.7 M and were labelled P1, P2, and P3, respectively. The undeposited SLG substrate served as the control and was labelled P4.

3. RESULTS AND DISCUSSION

3.1. CRYSTALLOGRAPHIC ANALYSIS OF THE FILMS

The structural phases of the PbS thin films identified using XRD are shown in Figure 1. The reflection peak occurs at $2\theta = 33.400°$ for the (111) orientation plane [5]. P1, P2, P3, and P4 are the PbS samples deposited at molar concentrations of 0.3, 0.5, and 0.7 M, and the undeposited SLG control, respectively, for 25 SILAR cycles. From Figure 1, the highest peak occurs for P3, corresponding to the sample deposited at the maximum concentration of 0.7 M, whereas the lowest deposited-film peak occurs for P2 at 0.5 M. The undeposited SLG sample has the lowest overall peak. This diffraction pattern shows that the film is nanocrystalline. No characteristic peaks of impurity phases, such as PbO or $PbSO_4$, were detected. The diffraction patterns of the films agree well with the Joint Committee on Powder Diffraction Standards (JCPDS) card no. 05-0592, and the observed peak positions are consistent with the cubic structure of lead sulphide [3].

The Bragg equation relates the diffraction angle (θ) of X-rays from a crystal lattice to the spacing between planes of atoms in the crystal (d) and the X-ray wavelength (λ):

$$n\lambda = 2d\sin\theta. \tag{1}$$

Using the diffraction angle ($2\theta = 33.400°$), the d-spacing was calculated from Eq. (1). For a plane in a PbS crystal structure,

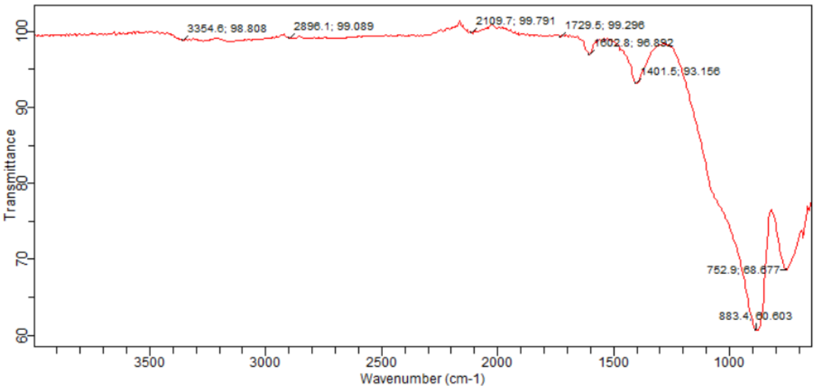

Figure 2. FTIR spectrum for the prepared P1 sample.

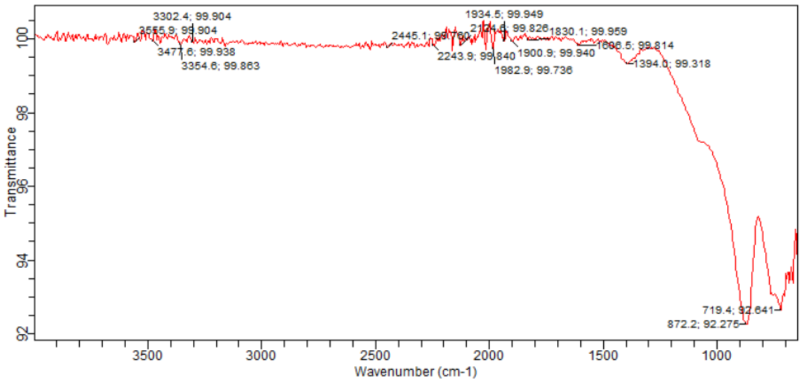

Figure 3. FTIR spectrum for the prepared P2 sample.

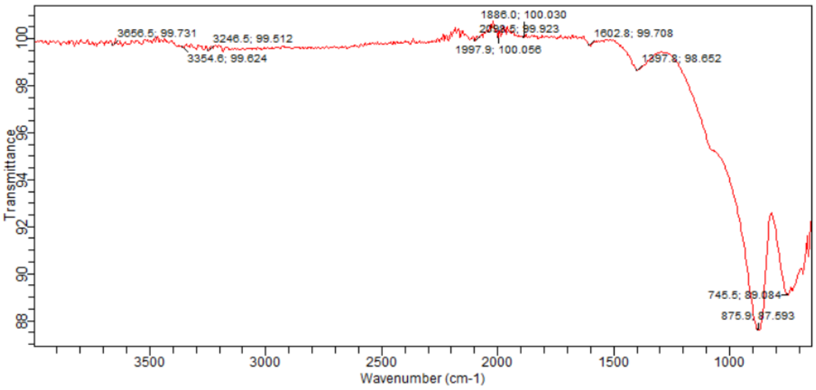

Figure 4. FTIR spectrum for the prepared P3 sample.

the d-spacing is given by

$$d = \frac{a}{\sqrt{h^2 + k^2 + l^2}}, \tag{2}$$

where a is the lattice constant and h, k, and l are the Miller indices that describe the orientation of the plane. The preferred crystalline orientation is the (111) plane.

3.2. FTIR ANALYSIS OF THE FILMS

Figures 2–5 show the functional groups and their peak bands. The FTIR spectra of PbS thin films deposited at precursor concentrations of 0.3, 0.5, and 0.7 M are labelled P1, P2, and P3, respectively. These spectra provide important information about chemical bonding and surface functional groups as a function of concentration. A systematic increase in absorbance intensity and improved peak definition is observed with increasing molarity, indicating enhanced film growth, better surface coverage, and increased bond density within the films.

Across all deposited samples, the broad absorption band observed around 3200–3600 cm^{-1} is attributed to O–H stretching vibrations [5, 6], which are typically associated with adsorbed moisture or hydroxyl groups on the film surface. The presence of this band is common in chemically deposited thin films and tends to increase slightly with concentration [7], suggesting greater

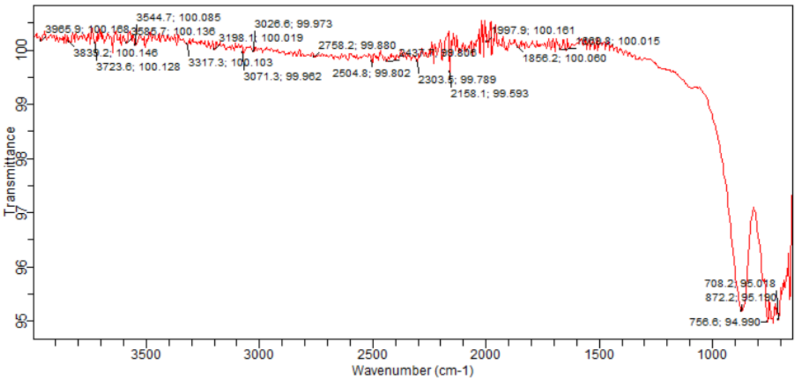

Figure 5. FTIR spectrum for the prepared P4 sample.

surface interaction with atmospheric species due to increased film thickness. In addition, weak bands near 2850–2950 cm^{-1} correspond to C–H stretching vibrations, possibly arising from residual organic precursors or contaminants during the deposition process [7].

In the fingerprint region, absorption bands around 1600–1650 cm^{-1} are assigned to H–O–H bending vibrations of molecular water, while peaks in the range 1400–1500 cm^{-1} may be linked to carbonate (CO_3^{2-}) groups formed by atmospheric CO_2 adsorption. More importantly, characteristic Pb–S stretching vibrations are typically identified in the lower wavenumber region around 600–700 cm^{-1}. In this study, these bands become increasingly pronounced and sharper as the precursor concentration increases from 0.3 to 0.7 M, confirming improved formation of the PbS lattice. At 0.3 M, the Pb–S band appears weak and broad, suggesting incomplete film formation and lower crystallinity. At 0.5 M, the band becomes more distinct, indicating improved nucleation and bonding interactions. At the highest concentration, 0.7 M, the Pb–S peak exhibits greater intensity and sharper definition, reflecting enhanced crystallinity, thicker film formation, and a more ordered structure. These observations are consistent with the principle that infrared absorbance is proportional to the number of active vibrational bonds present in the material [4, 8, 9].

In comparison, the P4 sample shows dominant peaks associated with silicate (Si–O–Si) vibrations, typically around 1000–1100 cm^{-1}, with no significant Pb–S features. The emergence and progressive intensification of Pb–S bands in the P1, P2, and P3 samples confirm successful deposition and highlight the role of concentration in modulating film properties.

4. CONCLUSION

PbS thin films were successfully deposited onto SLG substrates using the SILAR technique. The crystallographic results support the stoichiometric synthesis and crystallinity of the PbS films, as indicated by the XRD peak positions and the calculated lattice and *d*-spacing values. The FTIR spectra demonstrated that increasing the precursor concentration from 0.3 to 0.7 M enhanced the formation and quality of the PbS thin films. This enhancement was evidenced by the progressive increase in Pb–S bond intensity, improved peak sharpness, and stronger absorption features. The results highlight the critical role of concentration in optimizing thin-film deposition for improved structural and functional properties.

4.1. RECOMMENDATIONS

Future studies should explore annealing of PbS films and alternative deposition techniques for improved film quality. UV–Vis and higher-magnification SEM analyses should be used for detailed optical and surface-morphology characterization. In addition, X-ray fluorescence (XRF) spectrometry and TEM should be considered to provide information on the chemical properties of PbS thin films.

DATA AVAILABILITY

The datasets analysed during the current study are not publicly available due to the experimental nature of the research. However, the data are available from the corresponding author on reasonable request.

DECLARATION OF COMPETING INTEREST

The authors declare that they have no competing interests.

FUNDING

This research did not receive funding from any public, private, or non-profit agency.

ACKNOWLEDGMENT

The authors are grateful to the staff of the Crystal Growth Laboratory, Department of Physics, Tai Solarin Federal University of Education, Ijebu-Ode, Nigeria, for the technical support provided during this research.

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