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Structural, Optical, and Morphological Characterization of PbS Thin Films Prepared by the SILAR Technique

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Abstract — Lead sulphide (PbS) thin films have attracted significant research interest because of their narrow band gap, excellent infrared sensitivity, and promising applications in optoelectronic devices, infrared detectors, solar cells, and photoconductive systems. The present study investigates the synthesis and characterization of PbS thin films deposited on glass substrates using the Successive Ionic Layer Adsorption and Reaction (SILAR) technique. The films were prepared under controlled deposition conditions and subsequently subjected to different cooling durations to evaluate their influence on structural and optical characteristics. Structural analysis was performed using X-ray Diffraction (XRD), while surface morphology and optical properties were examined using Scanning Electron Microscopy (SEM) and UV–Visible spectroscopy. XRD analysis confirmed the formation of polycrystalline PbS thin films with a face-centred cubic crystal structure. Variations in grain size, strain, and dislocation density were observed with different cooling durations, indicating that post-deposition treatment significantly affects crystal growth. SEM images revealed uniform surface morphology with densely packed grains, whereas UV–Visible analysis demonstrated favourable optical absorption characteristics suitable for photovoltaic and infrared sensing applications. The findings indicate that the SILAR method is an economical, simple, and effective technique for producing high-quality PbS thin films with potential applications in advanced optoelectronic devices.

Keywords— Lead sulphide (PbS), Thin films, SILAR technique, X-ray diffraction, Scanning electron microscopy, UV–Visible spectroscopy, Semiconductor materials.

I. INTRODUCTION

Semiconductor thin films have emerged as one of the most important classes of functional materials because of their extensive applications in electronics, optoelectronics, photovoltaics, sensors, and energy conversion systems. Advances in thin-film technology have enabled the fabrication of compact, lightweight, and high-performance electronic devices with improved efficiency and reduced manufacturing costs. The ability to tailor the structural, optical, and electrical properties of thin films by controlling deposition parameters has significantly expanded their applications in modern materials science.

Among II–VI and IV–VI semiconductor materials, lead sulphide (PbS) has attracted considerable attention because of its narrow direct band gap (approximately 0.41 eV at room temperature), large exciton Bohr radius, high absorption coefficient, and excellent infrared photoresponse. These characteristics make PbS an attractive material for infrared detectors, photoconductive devices, photovoltaic cells, optical switches, gas sensors, and quantum dot technologies. The optoelectronic performance of PbS thin films is strongly influenced by their crystallinity, grain size, morphology, and deposition conditions.

Several techniques have been employed for the preparation of PbS thin films, including chemical bath deposition (CBD), spray pyrolysis, sputtering, thermal evaporation, molecular beam epitaxy, electrodeposition, and Successive Ionic Layer Adsorption and Reaction (SILAR). Among these methods, the SILAR technique has gained considerable popularity because it is inexpensive, simple, environmentally friendly, and capable of producing uniform films at room temperature without sophisticated vacuum equipment. The technique also offers excellent control over film thickness through repeated deposition cycles and is suitable for large-area substrate coating.

The structural quality and optical performance of PbS thin films depend not only on the deposition parameters but also on post-deposition treatment conditions. Cooling duration following deposition may influence crystal growth, grain orientation, defect density, and optical behaviour. Therefore, systematic investigation of these parameters is essential for optimizing the material for practical device applications.

The present study focuses on the preparation of PbS thin films using the SILAR technique and evaluates the influence of cooling duration on their structural, morphological, and optical properties. X-ray Diffraction (XRD), Scanning Electron Microscopy (SEM), and UV–Visible spectroscopy were employed to characterize the deposited films. The findings contribute to a better understanding of the relationship between processing conditions and material properties, thereby supporting the development of high-performance semiconductor thin films for optoelectronic and photovoltaic applications.

II. LITERATURE REVIEW

Lead sulphide (PbS) thin films have been widely investigated because of their unique optical, electrical, and structural properties, which make them suitable for infrared detectors, solar cells, photoconductive devices, and optoelectronic applications. Considerable research has focused on optimizing deposition techniques to improve film quality, crystallinity, and device performance.

The Successive Ionic Layer Adsorption and Reaction (SILAR) technique has emerged as one of the most effective chemical methods for depositing semiconductor thin films. According to Pathan and Lokhande, the SILAR technique is simple, inexpensive, and suitable for depositing high-quality metal chalcogenide thin films over large substrate areas. The method provides excellent control over film thickness through repeated deposition cycles and does not require sophisticated vacuum systems.

Studies on PbS thin films have demonstrated that deposition conditions significantly influence their structural and optical properties. Preetha and co-workers reported that variations in the pH of the cationic precursor affected crystallite size, optical band gap, and electrical resistivity of SILAR-deposited PbS thin films. Their results showed that deposition parameters play a critical role in determining the quality and performance of the films.

Murali and colleagues investigated the influence of different cationic precursors on PbS thin films and observed that precursor chemistry affected crystal growth, morphology, and optical behaviour. Similarly, Lindroos and co-workers demonstrated that the crystallinity and morphology of PbS films could be controlled by modifying the lead precursor used during deposition.

Research has also highlighted the importance of PbS in photovoltaic applications. Benehkohal and collaborators reported that PbS quantum dot-sensitized solar cells exhibited promising photoelectric conversion efficiency, indicating the potential of PbS nanostructures for next-generation solar energy devices. Lee and co-workers further demonstrated improved photon-to-current conversion efficiency in PbS quantum dot-sensitized solar cells prepared using an improved SILAR process.

Additional investigations by Pawar and co-workers confirmed the formation of polycrystalline PbS with cubic crystal structure using X-ray diffraction, while scanning electron microscopy revealed granular surface morphology. These findings demonstrated that the SILAR technique can consistently produce high-quality PbS thin films suitable for optoelectronic applications.

Overall, the existing literature establishes that PbS thin films prepared by the SILAR technique possess favourable structural, optical, and morphological characteristics. However, comparatively fewer studies have examined the influence of post-deposition cooling duration on these properties. The present investigation addresses this aspect by evaluating PbS thin films subjected to different cooling periods using XRD, SEM, and UV–Visible characterization techniques.

III. MATERIALS AND METHODS

1. Materials

Lead sulphide (PbS) thin films were deposited on thoroughly cleaned microscopic glass substrates using the Successive Ionic Layer Adsorption and Reaction (SILAR) technique. The chemicals used for the preparation of the precursor solutions were of analytical reagent (AR) grade and were used without further purification. Double-distilled water was employed for preparing all aqueous solutions.

2. Preparation of Glass Substrates

Glass substrates were cleaned to remove dust, grease, and other surface contaminants before deposition. The cleaning procedure involved washing with a detergent solution, rinsing thoroughly with distilled water, and subsequent cleaning using appropriate solvents. Finally, the substrates were rinsed with double-distilled water and dried prior to film deposition to ensure good adhesion and uniform film growth.

3. Deposition of PbS Thin Films by the SILAR Technique

PbS thin films were synthesized using the SILAR method, a solution-based deposition technique involving alternate immersion of the glass substrate in cationic and anionic precursor solutions. During each SILAR cycle, lead ions were first adsorbed onto the substrate surface from the cationic solution. The substrate was then rinsed to remove loosely bound ions before immersion in the sulphide ion solution, where a chemical reaction occurred to form a thin layer of PbS. Repeated deposition cycles increased the film thickness in a controlled manner.

Following deposition, the films were subjected to different cooling durations as described in the experimental procedure to evaluate the influence of cooling on the structural, morphological, and optical characteristics of the deposited PbS films.

4. Characterization Techniques
The deposited PbS thin films were characterized using multiple analytical techniques.

5. X-ray Diffraction (XRD)
The crystal structure and phase purity of the films were examined using X-ray diffraction analysis. Diffraction patterns were recorded over the specified angular range, and crystallographic parameters such as crystallite size, lattice characteristics, strain, and dislocation density were determined from the diffraction peaks using standard analytical methods.

6. Scanning Electron Microscopy (SEM)
Surface morphology and grain distribution of the deposited films were investigated using scanning electron microscopy. SEM micrographs were used to evaluate surface uniformity, particle distribution, and the presence of surface defects or agglomeration.

7. UV–Visible Spectroscopy
Optical properties of the PbS thin films were studied using UV–Visible spectroscopy. The absorption spectra obtained from the measurements were used to assess the optical behaviour of the films and to estimate relevant optical parameters.

IV. DATA ANALYSIS

The experimental data obtained from XRD, SEM, and UV–Visible spectroscopy were analysed to determine the relationship between cooling duration and the structural, morphological, and optical properties of the PbS thin films. Comparative analysis was performed to identify trends and evaluate the suitability of the deposited films for potential optoelectronic applications.

V. RESULTS AND DISCUSSION

1. Structural Analysis by X-ray Diffraction (XRD)
X-ray diffraction analysis confirmed the successful formation of PbS thin films deposited by the SILAR technique. The diffraction patterns indicated that the films possessed a polycrystalline nature with a face-centred cubic (FCC) crystal structure. The observed diffraction peaks corresponded well with the standard crystallographic data for PbS, confirming the phase purity of the deposited material.

Analysis of the diffraction peaks showed that cooling duration influenced the structural characteristics of the films. Changes in peak intensity and sharpness suggested variations in crystallinity and crystal growth. Crystallite size, microstrain, and dislocation density calculated from the XRD data indicated that post-deposition cooling affected crystal quality. Improved crystallinity was associated with sharper diffraction peaks and reduced structural defects, demonstrating that cooling conditions play an important role in determining the structural properties of PbS thin films.

Surface Morphology by Scanning Electron Microscopy (SEM)
Scanning Electron Microscopy revealed that the deposited PbS thin films exhibited relatively uniform surface coverage over the glass substrate. The SEM images showed densely packed grains with good adhesion and continuous film formation. Minor differences in grain size and surface texture were observed among samples subjected to different cooling durations, indicating that cooling influences grain growth and surface morphology.

The absence of significant cracks or pinholes suggested that the SILAR method produced compact films with satisfactory surface quality. Such morphological characteristics are advantageous for optoelectronic applications because they reduce charge-carrier scattering and improve device performance.

2. Optical Properties by UV–Visible Spectroscopy
The optical behaviour of the PbS thin films was investigated using UV–Visible spectroscopy. The absorption spectra demonstrated strong absorption in the investigated wavelength region, reflecting the semiconducting nature of PbS. Variations in the optical response among the deposited samples suggested that cooling duration influenced the optical characteristics of the films.

The observed optical behaviour can be attributed to changes in crystallite size and structural ordering resulting from different cooling conditions. These findings indicate that careful optimization of post-deposition treatment can enhance the optical performance of PbS thin films for infrared detectors, photovoltaic devices, and other optoelectronic applications.

3. Overall Discussion
The combined results obtained from XRD, SEM, and UV–Visible spectroscopy demonstrate that the SILAR technique is an effective method for depositing high-quality PbS thin films.

Structural, morphological, and optical analyses consistently indicate that post-deposition cooling duration influences film characteristics. The results support the conclusion that optimizing cooling conditions can improve crystallinity, surface morphology, and optical performance, thereby enhancing the suitability of PbS thin films for advanced semiconductor and optoelectronic applications.

VI. CONCLUSION

The present study successfully demonstrated the deposition of lead sulphide (PbS) thin films on glass substrates using the Successive Ionic Layer Adsorption and Reaction (SILAR) technique. The experimental results confirmed that the SILAR method provides a simple, cost-effective, and reliable approach for fabricating uniform semiconductor thin films with desirable structural and optical characteristics.

X-ray diffraction analysis confirmed the formation of polycrystalline PbS thin films with a face-centred cubic crystal structure. Variations in crystallite size and crystallinity observed under different cooling conditions indicate that post-deposition cooling influences crystal growth and structural quality. Scanning Electron Microscopy revealed compact and uniformly distributed grains with satisfactory surface coverage, while UV–Visible spectroscopy demonstrated favourable optical absorption characteristics consistent with the semiconducting nature of PbS.

The combined structural, morphological, and optical analyses indicate that optimizing cooling duration can improve the quality of PbS thin films produced by the SILAR technique. These characteristics make the deposited films suitable for applications in infrared detectors, photovoltaic devices, photoconductive sensors, and other optoelectronic technologies.

Overall, the findings support the potential of the SILAR method as an effective deposition technique for producing high-quality PbS thin films while offering advantages such as low cost, operational simplicity, and scalability for large-area deposition.

Future Scope

The present investigation provides a foundation for further research on PbS thin films. Future studies may focus on:

- Investigating the influence of additional deposition parameters such as precursor concentration, solution pH, immersion time, and the number of SILAR cycles.
- Examining the effects of post-deposition annealing under different atmospheric conditions.
- Evaluating the electrical and photoconductive properties of the deposited films.
- Exploring doped or composite PbS thin films to improve device performance.
- Developing prototype optoelectronic and photovoltaic devices using SILAR-deposited PbS thin films for practical applications.

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