

## Structural and Optical Properties of Nanocrystalline Pbs Thin Films Prepared by Microwave-Assisted Chemical Bath Deposition

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**Abstract:** Nanocrystalline lead sulfide (PbS) thin films have been successfully deposited onto glass substrate via microwave assisted chemical bath deposition. Aqueous solutions of lead nitrate  $Pb(NO_3)_2$  and thiourea  $[SC(NH_2)_2]$  were used as lead and sulfur ion sources respectively. Structural and morphological studies revealed a high synthesized nanocrystalline PbS thin films quality. Optical studies showed a significant blue shifting in the energy gap of the prepared nanocrystalline PbS thin films. Compared with bulk PbS, the prepared nanocrystalline PbS showed large energy gap, value of about 2.4 eV. This could be attributed to the confinement effect of nanocrystalline PbS thin films. The study introduced a simple and cost-effective method to further investigate other properties of nanocrystalline PbS thin films such as electrical and morphological properties.

**Keywords:** Nanocrystalline PbS Thin Films, Properties, Microwave CBD, Blue Shift, Energy Gap

### 1. Introduction

Narrow band-gap semiconductors are of interest for photovoltaic (PV) solar energy conversion as they can absorb the infrared (IR) tail of the solar spectrum, which is not absorbed by commonly used PV materials (Yang et al., 2006). The use of such absorbers in semiconductor sensitized solar cells allows the integration of low-cost device configurations and broad spectral response, which may also be utilized in IR and near-IR (NIR) photodetectors. Lead sulfide is an important direct narrow energy gap semiconductor material with an approximate energy band gap of 0.4 eV at 300K. Lead sulfide is an inorganic compound with the chemical formula PbS. It is also known as galena, which is the principal ore and important compound of lead. It is one of the earliest materials to be used as a semiconductor as it tends to crystallize in sodium chloride structure. PbS is widely used in photo optic applications, mainly as IR detectors due to its band gap value (Butson et al., 2019).

PbS thin films can be deposited using various techniques such as screen printing, chemical vapor deposition (CVD) and thermal evaporation (Nanda et al., 1998; Banerjee et al., 2000). Such techniques require high cost and sophisticated instrument. Chemical bath deposition (CBD) is a promising technique that produces homogeneous and high quality PbS thin films (Husham et al., 2017). It is a simple and cost-effective method. However, PbS thin films prepared using CBD require a long preparation time.

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Heat, usually transmitted from the outside to the inside of the material, leads to uneven distribution of temperature in the solution which may affect the quality of the deposited thin films. Microwave irradiation has been widely applied as a heating method in chemistry and material syntheses. Using microwave as a heating source, the heat is created from inside the material unlike conventional methods where heat flows inward (Husham et al., 2015). The direct interaction between the radiation and materials facilitates very short reaction times, moreover, less energy is consumed. This work aims to investigate structural and optical properties for nanocrystalline PbS thin films prepared using chemical bath deposition in a relatively short deposition time.

## 2. Experimental Procedure

Nanocrystalline PbS thin films were deposited onto microscopic glass slides substrates. Prior to the deposition, substrates were first cleaned with soap and hot distilled water. After they were rinsed with distilled water, substrates were soaked in an aqua regia solution (Pitt, 1999) for 30 minutes then they were rinsed again thoroughly with distilled water and dried with nitrogen gas. Aqueous solutions of lead nitrate  $\text{Pb}(\text{NO}_3)_2$  and thiourea  $[\text{SC}(\text{NH}_2)_2]$  were used as sources of  $(\text{Pb}^{2+})$  and sulfur ( $\text{S}^{2-}$ ) ions, respectively. Ammonium acetate ( $\text{NH}_4\text{CH}_3\text{COO}$ ) was added as a buffer solution to control the release of ions and to ensure slow deposition rate (Obaid et al., 2012).

For the preparation of PbS thin films, first, 10 ml of lead nitrate (0.1M) added into a 100 ml beaker, after that, appropriate quantity of 0.5 M ammonium acetate was added under continuous stirring. After 5 min 10 ml of thiourea (0.1 M) was added dropwise to the solution. Deionized (DI) water (resistivity~18.2  $\text{M}\Omega\cdot\text{cm}$ ) was then added to the beaker solution to achieve total volume of 100 ml. pH of the final solution was fixed at 12 by adding 0.5 M sodium hydroxide (NaOH) solution. The cleaned substrates were then fixed vertically inside the beakers; beakers were covered and heated in a microwave oven at 70 °C for 20 min. After the deposition process was done, samples were washed and ultrasonicated with DI water for 1 min to remove the remaining salts and loosely adherent particles, and then they were dried with nitrogen gas. Mirror-like dark gray PbS thin films with highly adherent to the substrates were obtained. Preparation steps illustrated in Figure 1, started with mixing solutions (step a) followed by other steps (b-d). Morphological, structural, and optical properties for the deposited thin films were studied using field emission scanning electron microscopy (FESEM) with energy dispersive X-ray spectroscopy (EDX) installed, high-resolution X-ray diffraction (HR-XRD), and UV-Vis-NIR spectrometer at wavelengths ranging from 300 to 2500 nm, respectively.

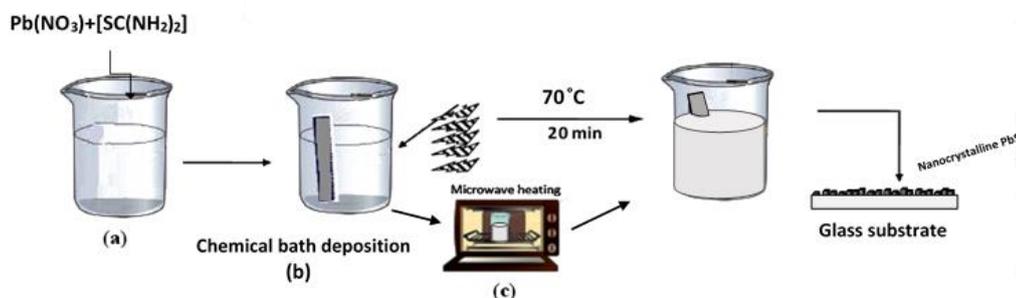


Figure 1: Preparation steps for PbS thin films using microwave heating-CBD

### 3. Results and Analysis

#### 3.1 Structural Properties

Figure 2 shows XRD patterns for the grown PbS thin films. The pattern showed the formation of a polycrystalline phase along (111) preferred orientation. All the observed diffraction peaks were indexed within a cubic (rock salt) structure as confirmed using a standard JCPDS card (ICCD-PDF4 No. 00-005-0592). No oxidation occurred during the preparation which resulted in good quality of the formed thin films. The crystalline size for the prepared PbS thin films was calculated using the Debye–Scherrer formula (Wang et al., 2014):

$$D = 0.94\lambda / \beta \cos \Theta \quad [1]$$

Where D is the average crystalline size (nm),  $\lambda$  is the wavelength of the X-ray (0.15 nm),  $\beta$  is the full width at half maximum (FWHM) of the XRD spectrum of PbS thin films (radians), and  $\Theta$  is the diffraction angle (radians). The estimated D of the PbS thin films was found to be 20 nm.

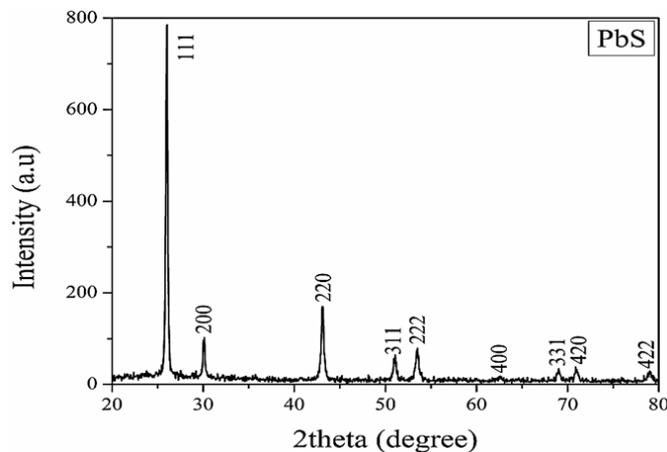


Figure 2: XRD patterns for the grown PbS thin films

#### 3.2 Morphological Study

Surface morphology of the prepared PbS thin films was observed through FESEM image analysis. As shown in Figure 3, the prepared PbS thin films were compact and uniform throughout the entire regions, and no voids and cracks were present. EDX analyses confirmed the presence of lead and sulphur for the prepared samples. Lead and sulphur ratios in the prepared thin films are summarized in Figure 4.

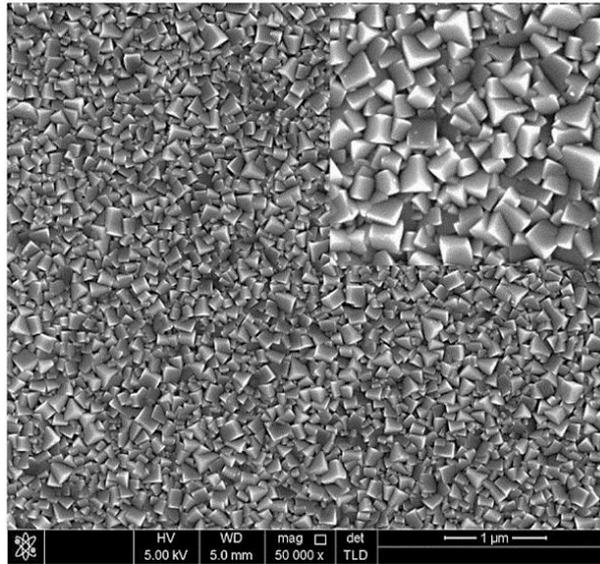


Figure 3: FESEM for the grown PbS thin films

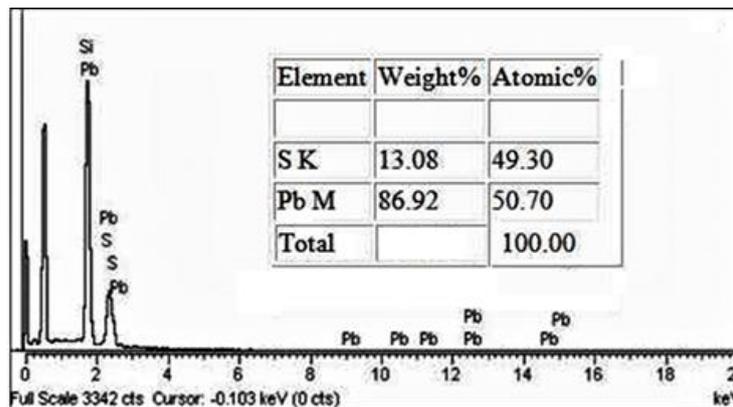


Figure 4: EDX spectrum for the prepared PbS thin films

### 3.3 Optical Properties

The optical properties of the deposited PbS thin films were investigated. Figure 5 shows light transmission and absorption spectra for the prepared PbS thin films. As shown in the figures, the transmission curve shows a sharp absorption edge at a wavelength of 540 nm. Using Tauc formula, the energy gap ( $E_g$ ) of the prepared PbS thin films was determined and found to be 2.3 eV (see Fig 5b). Results revealed a significant blue shift in the energy gap for the prepared PbS thin films. The prepared PbS thin films exhibited a wide band gap compared with that for bulk PbS (0.41 eV). Therefore, the significant increase in the energy gap may indicate nanostructured PbS thin films.

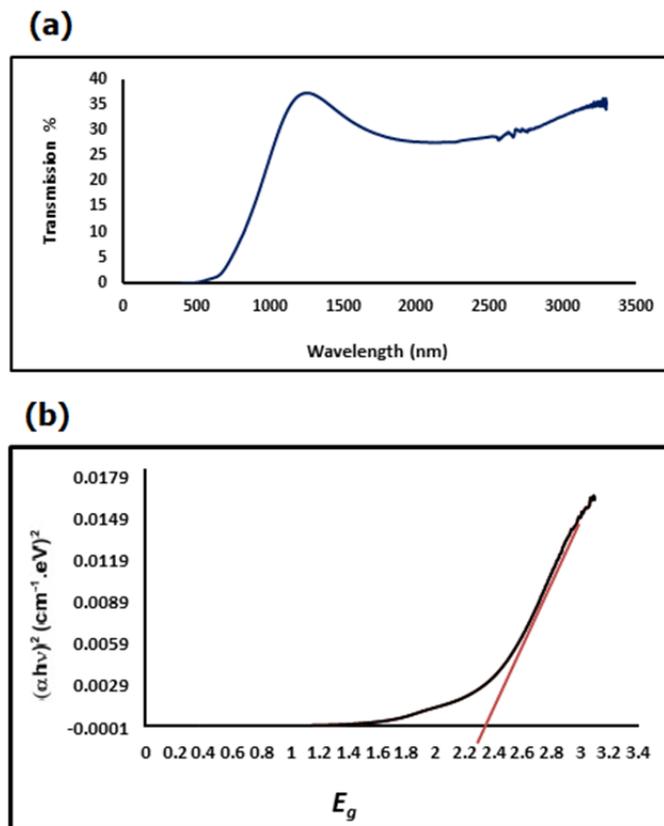


Figure 5: (a) Transmission spectrum for the prepared nanocrystalline PbS thin films  
(b) Tauc plot showing the absorption coefficient spectrum with energy gap value

#### 4. Conclusion

In summary, nanocrystalline PbS thin films were grown onto glass substrates via CBD with the aid of microwave heating. Results revealed the growth of well crystalline nano structured PbS thin films. Morphological analyses showed compact and uniform nanocrystalline PbS thin films throughout the whole surface. Besides, the prepared PbS thin films exhibited a significant blue shift in their energy gap, value of 2.3 eV which indicate their nano structured properties. The study may introduce a facile way to prepare nanocrystalline PbS thin films for further studies.

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