

Characterization of chemically deposited nanocrystalline PbS thin films

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Received: 24 February 2005 / Accepted: 11 October 2005 / Published online: 16 May 2006
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Nanocrystalline materials are novel materials, which are not only scientifically interesting but also held great potential for various applications. Their properties are different and often superior to those of conventional coarse-grained materials and also amorphous alloys of the same configuration. Nanocrystalline materials exhibit increased strength, hardness, enhance diffusivity, improved quality, roughness, reduced elastic modulus, higher thermal expansion coefficient, lower thermal conductivity and superior soft magnetic properties to the conventional coarse-grained materials [1].

Recently, lead sulfide (PbS) nanocrystalline thin films have received considerable attention because of their actual and potential application in optoelectronic devices. In solar energy research PbS thin films have been investigated for photo thermal conversion application [2–4]. The PbS thin films have been prepared using various chemical methods including electrodeposition [5–7], SILAR [8–10], spray pyrolysis [11] etc. In our previous study, we have reported the deposition of PbS at 80 °C using chemical bath deposition (CBD) [12]. CBD is most popular technique for PbS synthesis and thin film deposition because the CBD method is a relatively simple, inexpensive and convenient for large area deposition [12–15]. This method has been used to deposit various semiconductor nanocrystalline thin films by adjusting deposition temperature [16, 17]. In the present

investigation, we have obtained nanocrystalline PbS films due to the room temperature deposition and their structural, surface morphological, and electrical properties have been reported.

For the preparation of nanocrystalline PbS thin films, solution of lead acetate (0.025–0.15 M) and thiourea (0.1 M) were added in a beaker at room temperature (300 K). The pH of the bath was adjusted to 9 by dropwise addition of aqueous ammonia. The ultrasonically cleaned glass substrate was dipped in the solution. The preparative parameters such as concentration of lead acetate and deposition time were optimized.

To study the structural properties of the films, X-ray diffraction analysis was performed on Philips (PW-3710) diffractometer with chromium target ($\lambda = 2.2896 \text{ \AA}$). The surface morphological study of the PbS film was carried out by scanning electron microscopy using Cambridge stereoscan 250 MK-3 model. A two-probe method was used for electrical resistivity measurement. Thermo-emf measurement was carried out to determine the type of electrical conductivity of the PbS thin film.

By making several trials for different concentration, deposition time, etc. was optimized. Figure 1 shows variation of PbS thin film thickness as a function of concentration of lead acetate for 0.1 M thiourea and deposition time 6 h. Initially, PbS film thickness was increased with lead ion concentration, and reached maximum value (290 nm) at 0.1 M. After this, the film thickness was decreased, which is due to the formation of outer porous layer, as a film peeled off the substrate.

Figure 2 shows the variation of PbS film thickness as a function of deposition time. Initially film thickness was increased linearly and then remained constant. Such behavior can be understood by film formation and continuous

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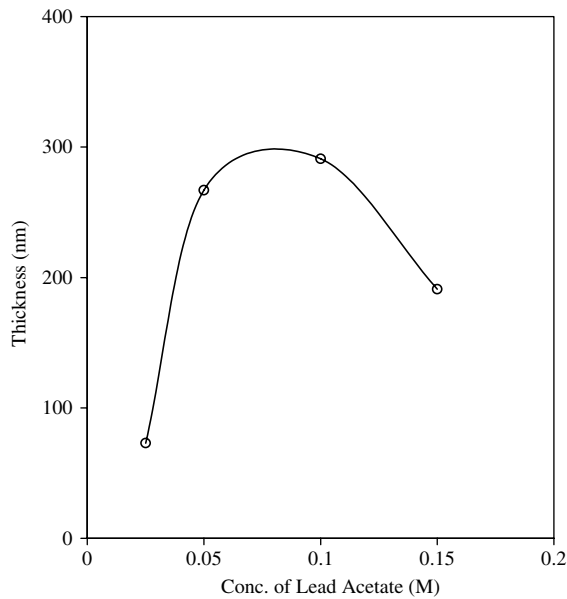


Fig. 1 Variation of PbS film thickness as a function of concentration of lead acetate for concentration of thiourea 0.1 M and deposition time 6 h

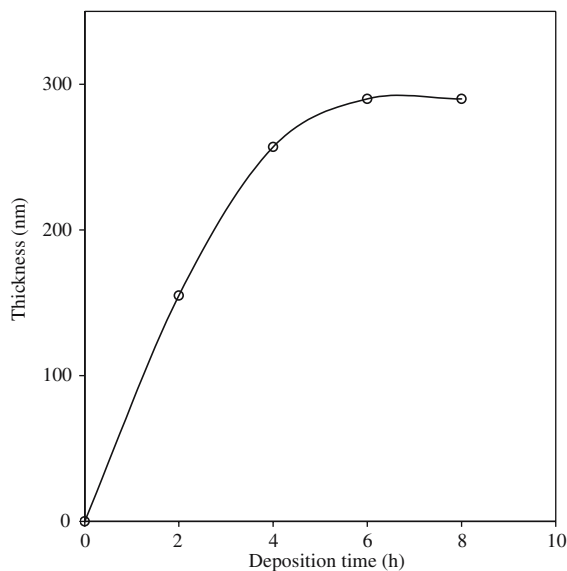


Fig. 2 Variation of PbS film thickness as a function of deposition time

precipitation in the bulk of solution. The thickness of PbS film was found to vary between 150 and 290 nm with deposition time 2–6 h.

Figure 3 shows XRD pattern of PbS thin film. The XRD pattern fairly matched with the peak position (2θ) of the standard X-ray powder diffraction data of the FCC structure (ASTM data File No. 5-592). It is noteworthy that the (200) the intense peak as compare to others, the similar type of orientation was observed by Gadave et al. [12] for PbS films deposited by CBD at 80 °C and by Puiso et al.

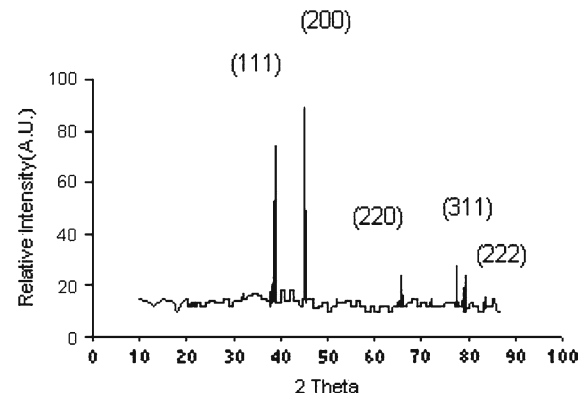


Fig. 3 The XRD pattern of PbS thin film

[18] for SILAR deposited PbS films on Si substrates. The crystallite size was calculated for (200) plane by using Scherrer's equation

$$d = 0.9\lambda / \beta \cos \theta \quad (1)$$

where λ is the wavelength of X ray used, β is the full width at half maxima in radian and θ is the Bragg's diffraction angle. The crystallite size was 32 nm indicates the film is nanocrystalline [19].

PbS thin film deposited on to glass substrate with 290 nm thickness was used for the study of surface morphology using scanning electron microscope. Figure 4 shows a scanning electron micrograph of PbS thin film deposited onto a glass substrate at 30,000 \times magnification. The scale bar length is 100 nm. It is observed that the film is smooth, homogeneous, fine grained and well covered to the substrate.

The optical absorption of PbS films was studied in the wavelength range of 350–850 nm. The optical absorption in the visible region (400–850) was between 70% and 75% indicating that PbS films are useful as absorber in the visible region.

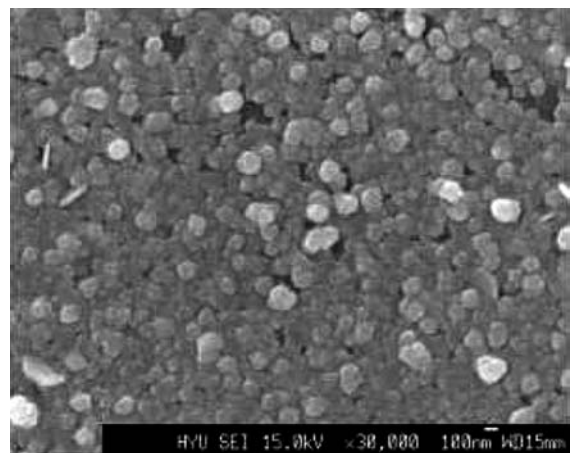


Fig. 4 The SEM of PbS thin film at 30,000 \times magnification

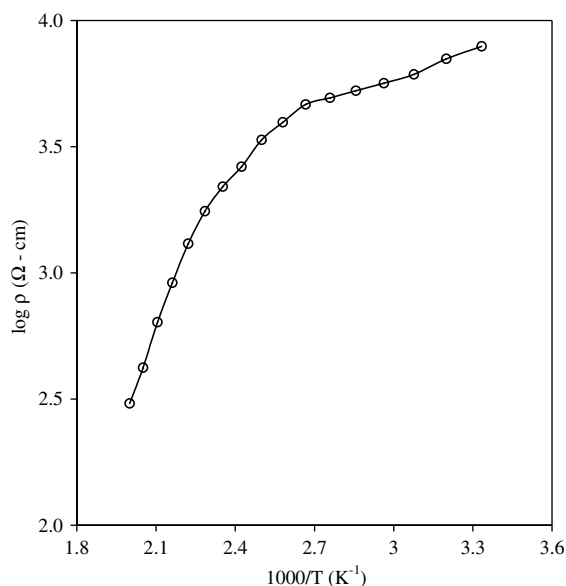


Fig. 5 Variation of $\log \rho$ with reciprocal temperature ($1000/T$) for PbS thin film

The dark dc electrical resistance of the as deposited PbS film was measured in 300–600 K temperature range. The resistivity at room temperature was found to be of the order of $10^4 \Omega \text{ cm}$. Figure 5 shows variation of electrical resistivity with inverse of absolute temperature. The electrical resistivity of PbS film was decreased with increase in temperature, indicating the semiconducting nature. The nature of the plot indicated the presence of two types of conduction mechanism.

The type of electrical conductivity exhibited by PbS thin film was determined by thermo-emf measurement. The temperature difference causes a transport of carriers from hot end to cold end and, thus creates an electric field which gives rise to thermal voltage. It was found that the polarity of thermoelectric voltage for nanocrystalline PbS thin film was in favor of p-type conductivity [12].

From above studies, it is concluded that the nanocrystalline PbS thin film can be deposited at room tem-

perature from the bath containing lead acetate and thiourea. The XRD study confirmed the nanocrystalline nature of PbS thin film. From SEM micrograph, it is observed that the film is smooth, homogeneous and well covered to the glass substrate. The room temperature electrical resistivity of nanocrystalline PbS thin film was $10^4 \Omega \text{ cm}$. The p-type electrical conductivity was confirmed from thermo-emf measurement.

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