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Synthesis and Characterization of Hydrophobic PbS Thin Films

P. D. More

Materials Research Laboratory, Department of Physics, Ahmednagar College, Ahmednagar, India

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ABSTRACT

In this article, structural, optical, electrical transport and wettabilty studies of PbS thin films deposited by spray pyrolysis have been investigated. The as deposited PbS films exhibit cubic crystal structure. The optical studies revealed a high absorption coefficient (10^4-10^5 /cm). The dc electrical conductivity and thermoelectric power were measured by two probe method. The wettability test shows hydrophobic behaviour of PbS thin films.

Key words: PbS, Spray pyrolysis, Hydrophobic.

INTRODUCTION

PbS is an important direct narrow band gap semiconductor material (≈ 0.41 eV at room temperature). During the last few years, the research on the growth of PbS thin films has increased, for its technological applications in photo resistance, diode lasers, decorative coatings, and optoelectronic devices [1-2]. Therefore, PbS semiconductor have been focused in experiments and theory [3]. But PbS in bulk form is a material of potential application for infra red detection. Therefore it is important to investigate the bulk properties of PbS. Carrier densities derived from Hall measurements are equal to those derived from thermoelectric power measurements, and are therefore interpreted to be equal to the carrier density in the grains of the PbS layer [4]. Considering the fact that stoichiometric excess of sulfur in PbS (bulk) imparts p-type character whereas deficient sulfur makes it n-type [5]. Thin films of PbS were prepared by different techniques. The most simple, inexpensive deposition technique is spray pyrolysis. By employing spray pyrolysis, deposition rate and thickness of the film can easily be controlled for a wide range.

MATERIALS AND METHODS

When a droplets of the spray solution of lead chloride ($PbCl_2$ of 0.1M) and thiourea (NH_2CSNH_2 of 0.1 M) at 300^0C substrate temperature reaches the hot substrate, owing to the pyrolytic decomposition of the solution, well adherent films were deposited by spraying 30 ml prepared solution at the rate of 5 ml/min using air as a carrier gas. In this process, the solution is pulverized by means of air and arrives on the substrate placed inside the chamber in the form of fine drops known as aerosols which form a thin layer on the substrates. The as deposited PbS films was of black color. Thickness and angle of contact of PbS thin films were measured by contact profiler meter. The structural properties of PbS was studied by X-ray diffraction (XRD) using filtered CuK_α radiation (λ=1.5406 A^0). The surface morphologies of the PbS thin films were observed by scanning electron microscope operating at 25 KV. The optical studies carried in the range of 300-1100 nm wavelengths. The dc electrical conductivity and thermoelectric power were measured by two probe method.

Table.1. Interplanar spacing's deduced from XRD patten and compared with the corresponding ones obtained from literature data. Numbers in brackets (n) represent the labels of reflections in diffraction patterns

(n)	Interplanar spacings determined	Interplanar spacing's reported in literature*	
	d (A^0)	d (A^0)	h k l
1	3.3117	3.420	111
2	2.8710	2.9618	200
3	2.0552	2.0943	220
4	1.7568	1.7860	311
5	1.6359	1.7100	222
6	1.4639	1.4809	400
7	1.3462	1.3589	331
8	1.3120	1.3245	420

**Joint Commission Powder Diffraction File No. 78-1899*

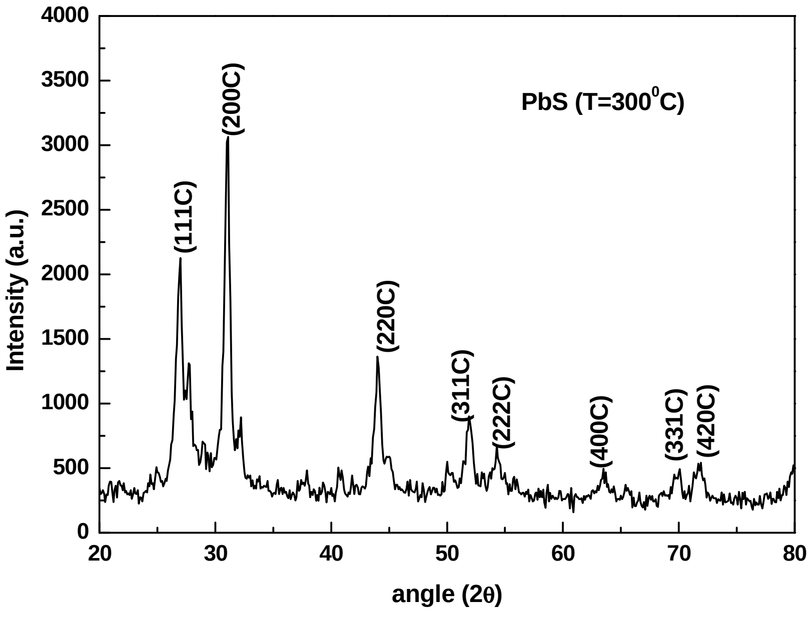
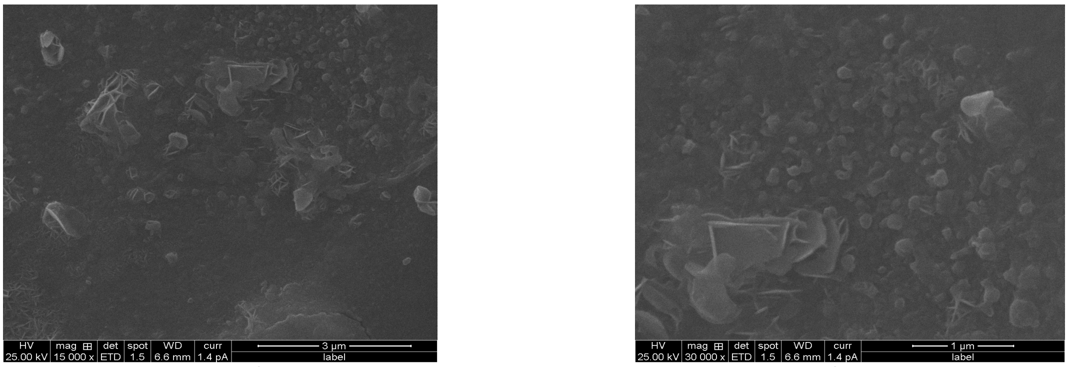


Fig. 1. X-ray diffraction pattern of PbS thin films



a. Magnification at 15000 X *b. Magnification at 30000 X*

Fig. 2. SEM of PbS thin films at different magnifications

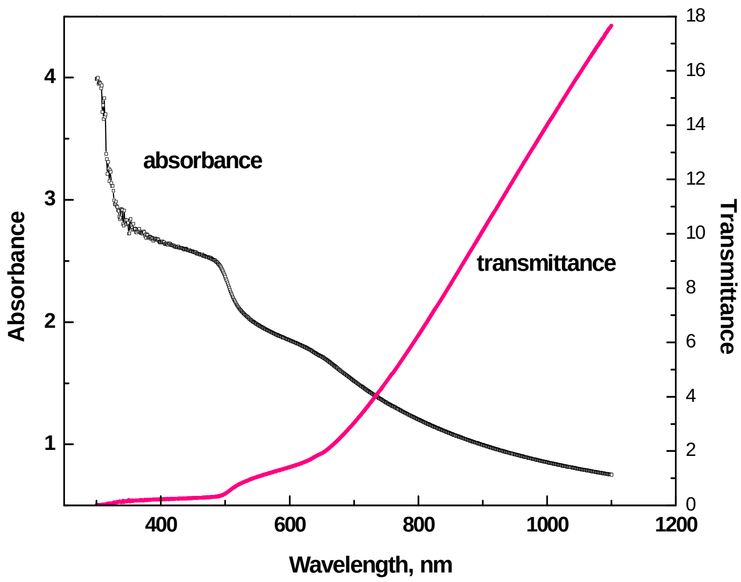


Fig. 3. Shows optical absorbance and transmittance vs wavelength of PbS thin films

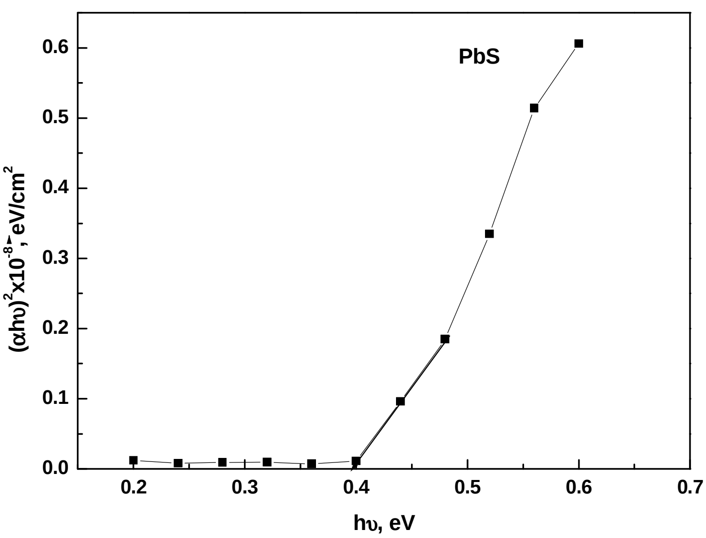


Fig. 4. Plot of $(\alpha h\nu)^2$ vs hν

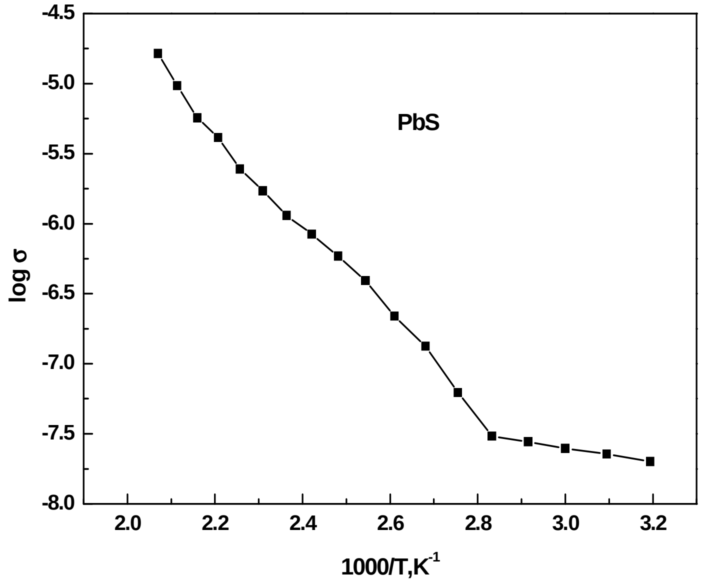


Fig. 5. Shows electrical conductivity of Pbs thin films

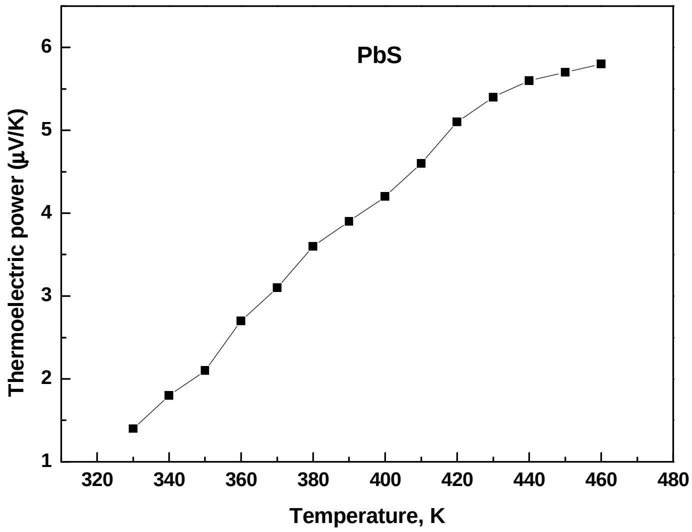


Fig. 6. Thermoelectric power of PbS thin films

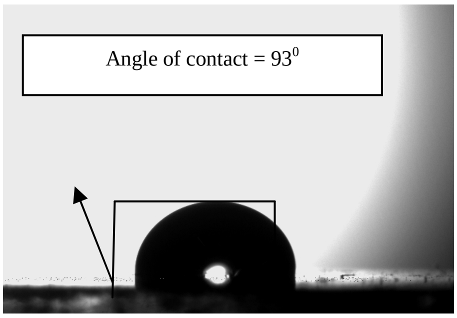


Fig. 7. Contact angle of PbS thin films

RSEULTS AND DISCUSSION

3.1) X-ray Diffraction studies:

The crystallographic properties have been investigated by X-ray diffraction technique using CuKα radiation. Figure. 1 shows X-ray diffraction (XRD) patterns of films at temperatures 300°C. The film prepared shows several peaks with high intensity occurred at $2\theta = 26.9^o$, 31.13°, 44.03^o, 52.01^o, 54.51^o. Diffraction along the [111] and [200] plane shows the highest intensity with well-defined sharp peak indicating high crystallinity of the material prepared. All these peaks (table 1) corresponding to cubic phase of PbS were well matched with the standard JCPD [6]. The lattice parameter was calculated from the XRD data a=5.79685 and are found to be close agreement with the JCPD data. The grain size and lattice constant of the films were determined by fitting the more intense diffraction peaks to Gaussian functions and using the Debye Scherrer formula. It was found to be 38 nm.

3.2) Microscopic studies:

Scanning electron microscopy (SEM) is an extremely versatile technique that provides a direct vision of the structural information's of the materials. SEM (fig.2) shows cubic crystal which is good agreement with XRD studies.

3.3) Optical studies:

The absorption spectra of PbS data were analyzed using the Stern relation for near edge optical absorption of semiconductors as [7-8]

$$A= [K (h\nu-Eg)^{n/2}] / h\nu \qquad (1)$$

where, h- Planck's constant, ν- frequency, K-constant, n=1 for direct type band gap or 4 for indirect type band gap.

Fig.3. shows absorbance and transmittance spectra recorded at 300 to 1100 nm wavelength. Fig. 4 shows the linear nature of the plot near absorption edge indicates the existence of the direct transition. Extrapolating the straight line portion of each curve in fig. 4 intersects the zero absorption coefficients (energy axes). The value of energy at this interesting point indicates the energy of band gap $E_g = 0.39$ eV.

3.4) Electrical studies:

The electrical conductivity of PbS thin films was measured in the 300-500K temperature range using two probe techniques and its temperature dependence can be expressed by usual Arrhenius equation [9].

$$\sigma_{(T)} = \sigma_o \exp (-E_{a\sigma} /KT) \qquad (2)$$

where $E_{a\sigma}$ is the activation energy; T is the absolute temperature and K is the Boltzmann constant.

The room temperature electrical resistivity of this sample is of the order of 10^5cm. The temperature dependence of an electrical conductivity is shown in fig.5. It is seen that there are two distinct conducting regions indicating more than one conduction mechanisms [9]. The localized states responsible for this conduction process are the direct consequence of the imperfections associated with polycrystalline thin films [10]. Two distinct regions corresponding to two activation energies are clearly seen. The activation energies of an electrical conduction have been determined in both low and high temperature regions and are 0.39 eV and 0.11 eV respectively.

3.5) Thermoelectric power:

The TEP for PbS samples was therefore measured and recorded in the range of 300- 480K temperature range. The temperature dependence of TEP is shown in fig.6. It is seen that TEP increased with temperature.

3.6) Wettability test:

The wetting of solid with water is dependent on the relation between the interfacial tensions. The ratio between water/air, water/solid and solid/air determines the contact angle between a water and droplet on a given surface. Measurement of surface water contact angle is inversely proportional to the wettability and can be determined by young's relations [11].

$$\cos \theta = (\gamma_s - \gamma_{sl})/ \gamma_{lv} \qquad (3)$$

Fig.7. Indicating hydrophobic behaviour ($\theta < 90°$) means the solid has a less affinity for water.

CONCLUSION

The preparative parameters can easily be optimized to produce good quality of Pbs thin films by spray pyrolysis. As deposited films showed sharp peaks and cubic structure. The lattice parameter (a) is nearly matched with standard value. The PbS thin films exhibit both type of conduction mechanisms. The wettability test showed hydrophobic behaviour.

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