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Influence of precursor molar concentration on the structural, morphological, optical and electrical properties of PbS thin films deposited by spray pyrolysis technique using perfume atomizer

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ABSTRACT

PbS thin films were deposited onto glass substrates at 400 °C using lead nitrate and thiourea as sources of Pb and S respectively. The molar concentration of precursor salts (both cationic and anionic) was varied as 0.025, 0.05, 0.075 and 0.1 keeping S/Pb ratio equal to 1. The structural studies reveal that the S:Pb concentration has a strong influence on the microstructural characteristics of the sprayed PbS films. The films were adherent to the substrate and well crystallized according to orthorhombic structure with the preferential orientation making a transition from (0 0 4) plane to (4 1 3) plane when S:Pb molar concentration increases. All the films have high optical transmittance > 70%. The optical band gap values are found to be in the range of 2.44 – 2.55 eV. The refractive index dispersion of the films obeys the single oscillator model. Electrical resistivity of all the films was found to be in the order of 10^2 Ωcm. PbS films fabricated by this simplified technique are found to be good in structural, optical and electrical properties which are desirable for window layers in solar cell applications.

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1. Introduction

Lead sulfide (PbS) belongs to the IV – VI family and is an important semiconductor widely studied due to its extensive applications in diode lasers, optical detectors, optoelectronic devices, photo resistors, sensors etc. [1–3]. PbS with direct band gap of 0.4 eV and absorption coefficient continuously increasing from the infrared through the visible region, has been used in infrared detectors [4]. It is important in electronic devices, infrared photography, photo thermal conversion applications etc. The emission and absorption lines of PbS thin films are consequently broader, but by monitoring its crystallites size, tunable emission can be obtained in a large spectral region, ranging from visible to near infrared. This spectral range is of great interest for fabricating light sources (including lasers) or optical amplifiers. Many reports have shown that PbS films deposited on glass substrates possess excellent solar control characteristics comparable to the metallic films [5]. PbS films have a NIR reflection of 10 – 45% and an optical transmittance for visible spectrum of 10 – 50%. The PbS thin films was deposited by various chemical methods, like chemical bath deposition [6], electro deposition [7], spray pyrolysis [8], photo chemical deposition [9] and successive ionic layer adsorption and reaction (SILAR) method [10]. Among these techniques, spray pyrolysis offers the possibility of relatively simple, quick, economical and suitable for large area deposition of any configuration. Quality of the as deposited PbS thin films can be improved by optimizing deposition parameters such as precursor concentration, substrate temperature, spray rate, volume of the solution sprayed, etc. In the present work, a further simplified spray technique, using perfume atomizer has been employed to fabricate PbS films with different S:Pb molar concentrations 0.025:0.025, 0.05:0.05, 0.075:0.075 and 0.1:0.1 respectively. Seghaier et al. [11] studied the structural and optical properties of PbS thin films deposited by CBD method at various concentration of the precursors and different deposition time. They found that PbS film with better crystallinity is obtained for the film with Pb:S molar ratio equal to 0.17:0.1. The film showed preferential orientation along the (2 0 0) plane corresponding to fcc cubic structure. To the best of our knowledge, there are no similar studies on the effects of precursor molar concentration on the characteristics of spray deposited PbS films. In this paper, we report the effects of precursor molar concentration on the structural, morphological, optical and electrical properties of

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PbS films grown on glass substrates by the simplified spray pyrolysis method. Accurate determination of the optical constants of PbS films is important, not only in order to know the basic mechanisms underlying these phenomena, but also to exploit and develop their interesting technological applications [12]. Therefore, the optical absorption parameters, such as optical dispersion energies, E_o and E_d, the average value of oscillator strength S_o, wavelength of single oscillator λ_o, have been evaluated under the effect of S:Pb molar concentrations.

2. Experimental details

PbS films were fabricated by an inexpensive spray technique using a perfume atomizer from aqueous solutions of lead nitrate, $Pb(NO_3)_2$ (source for lead ions) and thiourea, $SC(NH_2)_2$ (source for sulphur ions). The molar concentrations of lead and sulphur (S:Pb) in the original water solutions are kept as 0.025:0.025, 0.05:0.05, 0.075:0.075 and 0.1:0.1. To prepare the starting solution, required proportions of the source materials were dissolved in doubly deionized water and stirred thoroughly using a magnetic stirrer for 30 mins. The clear starting solutions thus prepared was sprayed manually using a perfume atomizer on pre-heated glass substrates (micro-slides of dimensions $76 \times 25 \times 1.5\,mm^3$) kept at 400 °C. The spray procedure employed in this work involves the following two steps: a spray and a 5 s wait. The spray intervals in this spray sequence allow the substrates to attain the required temperature before the start of the next spray. Before deposition, the substrates were boiled with detergent, soaked in chromic acid, cleaned in isopropyl alcohol, rinsed in distilled water and dried. The film thicknesses were measured by weight difference method using a sensitive microbalance. X-ray diffraction patterns, SEM images and transmission spectra were obtained using X-ray diffractometer (PANalytical – PW 340/60 X'pert PRO), scanning electron microscope (HITACHI S – 3000H) and UV – Vis-NIR double beam spectrophotometer (LAMDA – 35), respectively. X-ray diffractometer was operated at 40 kV and 30 mA with X-ray source of CuK_α radiation having wavelength 1.5406 Å. Transmission spectra were recorded in the range of 300 – 1100 nm. The elemental analyses of the films were performed using energy dispersive X-ray spectroscopy (EDS). The film thickness was found to vary from 175 nm to 391 nm as S:Pb molar concentration varies from 0.025 to 0.1.

3. Results and discussion

3.1. Structural studies

Fig. 1 shows the X-ray diffraction spectra of spray deposited PbS films deposited with different S:Pb molar concentrations 0.025:0.025, 0.05:0.05, 0.075:0.075 and 0.1:0.1. It is seen that all the films deposited from solutions having different S:Pb molar concentrations have polycrystalline nature. The crystallography of the films is good and characterized by three principal peaks at 2θ values of approximately 19, 22 and 30° corresponding to (0 0 4), (1 2 1) and (4 1 3) orientations. Along with these peaks some small peaks (5 1 1), (0 3 4), (2 1 8) and (8 1 2) were also observed. By comparison with the data from JCPDS Card No. 78 – 0792, all diffraction peaks can be indexed as orthorhombic structure of PbS. The peaks in the XRD patterns clearly depict that the preferential orientation changes gradually from (0 0 4) to (4 1 3) when the molar concentration of sulphur and lead (S:Pb) in the starting solution increases. For PbS films prepared from starting solution having lower S:Pb molar concentration (0.025: 0.025), the preferential orientation is along the (0 0 4) plane. But, for higher S:Pb molar concentrations (0.05:0.05, 0.075:0.075 and 0.1:0.1), the (4 1 3) plane is observed as the preferential orientation plane. The preferential orientation

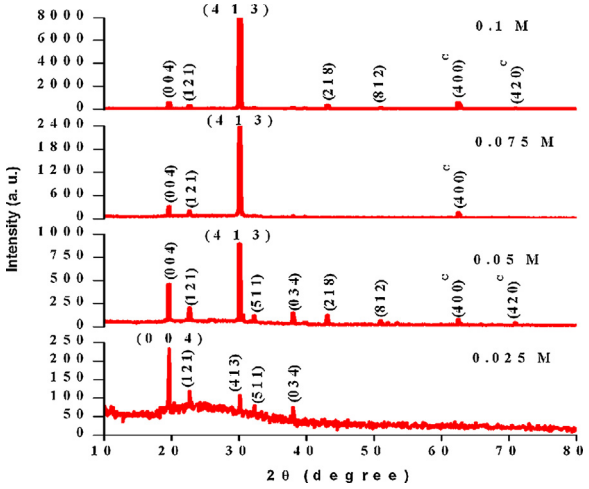

Fig. 1. XRD spectra of PbS thin films deposited with different S:Pb molar concentrations.

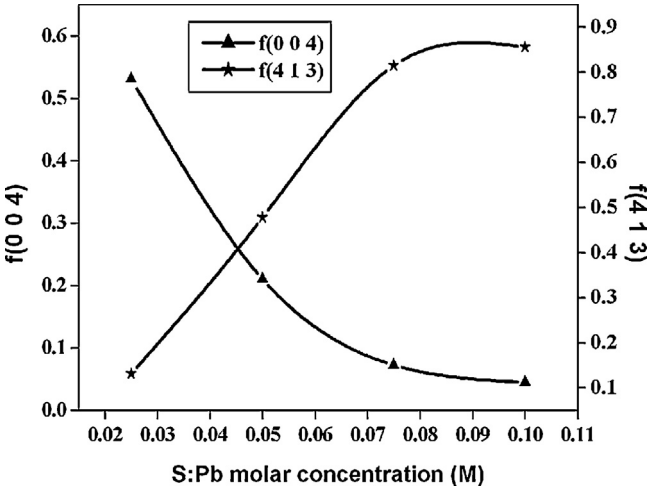

Fig. 2. Variations of f(0 0 4) and f(4 1 3) as a function of S:Pb molar concentrations.

Table 1
d-spacing (d) values of the PbS films.

S:Pb molar concentration	*d*(Å) (0 0 4) 4.5215*	(1 2 1) 3.9129*	(4 1 3) 2.9655*
0.025: 0.025	4.5200	3.9171	2.9631
0.05: 0.05	4.5240	3.9181	2.9657
0.075: 0.075	4.5215	3.9174	2.9637
0.1: 0.1	4.5217	3.9166	2.9634

* Standard *d*-spacings (JCPDS 78 – 0792) for PbS orthorhombic structure.

factor f(*h k l*) of the peaks ((0 0 4) and (4 1 3)) of the sprayed PbS films relative to the other peaks were calculated and the variation of f(0 0 4) and f(4 1 3) as a function of S:Pb molar concentrations are presented in Fig. 2. These variations reveal that the concentration of sulphur and lead in the starting solution has a strong influence on the microstructural characteristics of sprayed PbS films. The observed d-spacings of the ((0 0 4), (1 2 1) and (4 1 3)) planes of all the PbS films are listed in Table 1.

The standard d-spacing values (JCPDS 78 – 0792) of the PbS orthorhombic structure are also given for comparison. Traces of cubic phase PbS (JCPDS 65 – 0307) were observed for the films with higher S:Pb molar concentrations (0.05:0.05, 0.075:0.075 and 0.1:0.1).

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