



Effective role of thickness on structural, electrical and optical properties of lead sulphide thin films for photovoltaic applications



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ABSTRACT

The n-type lead sulphide thin films were deposited at 350 °C substrate temperature on glass substrates using advanced spray pyrolysis technique. The thickness of the thin films played an important role to improve the properties of lead sulphide and to use in device fabrication apart from various deposition parameters. The films deposited at thickness of 520 nm resulted in a well oriented polycrystalline with face-centered cubic structure. An enhancement in the crystallite size with increase in film thickness was evidenced by XRD and SEM. The variation in crystallite size of films associated with different thickness provides a significant control over optical and electrical properties. The resistivity of the thin films decreased with an increase in thickness was of the order of $10^2 \Omega \text{ cm}$. The activation energy and optical band gap of the films deposited at optimized condition were found to be 0.20 eV and 1.22 eV, respectively. The absorption coefficient of the films was found to be 10^6 cm^{-1} . Results prove that the lead sulphide films synthesized using spray technique appeal its adaptability for potential photovoltaic applications in solar cells.

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1. Introduction

The IV–VI group semiconductors have attracted considerable attention from the research community. Among them, PbS exhibits both n-type and p-type conductivity with a narrow band gap of 0.41 eV. In addition, it has a large exciton Bohr radius of 18 nm making it suitable to use in various applications [1]. Moreover, lead sulphide possesses a moderate conductivity, high mobility, small effective masses of electrons and holes [2], solar control characteristics and photoconductive properties [3] which have been greatly related to use in wide variety of applications such as photo resistors, diode lasers, photothermal conversion application, solar cells and thermoelectric applications [4]. Fabrication of lead sulphide based photovoltaic devices has been reported since 1970. Further, the property of multiple carrier generations possessed by PbS makes it a suitable candidate for next generation solar cells. Reportedly, the PbS thin films are synthesized using various deposition techniques such as vacuum evaporation [5,6], molecular beam epitaxy [1], successive ionic layer adsorption and reaction [7], chemical vapor deposition (CVD) [8], spray pyrolysis [9], chemical bath deposition (CBD) [10,11] and electrodeposition [7].

The limitations such as high cost and stringent growth process conditions (e.g. temperature, pressure, environment and time) attributed to most of these techniques have negative impacts on the desired film properties. Spray pyrolysis technique is suitable to prepare doped thin film with required ratios of dopants. In addition, this technique is also helpful to yield well adhered, pin hole free, homogeneous and smoother thin films with the required thickness [12,13]. The thickness of the films plays a vital role in achieving a good quality device and exhibits remarkable efficiency [14]. The optimization of the film thickness towards achieving bulk properties of the lead sulphide material is a concern for photovoltaic in solar cell device application. The variation in the thickness of the film alters the properties of PbS films have been well reported by various research groups [15,16]. Gaiduk et al. (2008) prepared PbS nanocrystalline layers on single crystalline wafers of Si, Ge and GaAs using CBD technique. They observed that, the thickness of the PbS layers strongly depends on the chemical nature of the substrate [15]. Abbas et al. (2011) showed that in all the chemical methods, the thickness of the films depends on deposition time and the films at 600–660 nm thickness with tuned optical properties were optimized for solar window applications [17]. Faraj (2015) varied the thickness of the films from 100 nm to 300 nm using spray pyrolysis technique and reduced the resistivity of the p-PbS films to $10^3 \Omega \text{ cm}$ which can be used in solar cell applications [18]. In the present work, we report that the optimiza-

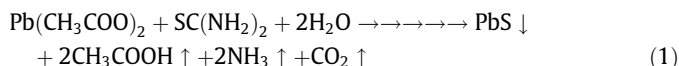
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tion of thickness to achieve n-PbS with the reduced resistivity in the order of $10^2 \Omega \text{ cm}$ give an attention to using PbS films for photovoltaic applications in solar cells.

2. Experimental details

PbS films were synthesized on a glass substrate by spray pyrolysis technique using lead acetate and thiourea as precursors. Substrates were cleaned with distilled water and dipped in chromic acid for 24 h then rinsed with acetone and dried in air. The precursor solution was prepared by mixing 99.99% aqueous lead acetate ($\text{Pb}(\text{CH}_3\text{COO})_2$) and aqueous thiourea ($(\text{NH}_2)_2\text{CS}$) supplied by Alfa Aesar. To study the effect of thickness on structural, electrical and optical properties of PbS films, films of different thickness were prepared by varying the deposition time while keeping the all other parameters constant as mentioned in Table 1. The thickness of the films was calculated using gravimetric method and measurements were verified using cross-sectional view SEM micrographs. After deposition, the films were allowed to cool at room temperature. Deposited films were extremely resistive to oxidation up to 350 °C and did not decompose over time as reported in our earlier work. This is due to the tight crystalline structure which prevents degradation of the films [19]. The adhesion of the films onto the substrate was quite good. The chemical reaction taking place in the process of synthesizing the lead sulphide thin film is



Though the PbS is harmful and a hazardous material, it is a potential candidate in semiconductor device technology. While depositing the PbS thin films using spray pyrolysis technique the operator has to be conscious of safety measures. Therefore, PbS films were deposited using spray pyrolysis technique closed and controlled environment fitted with exhaust window to exhaust hazardous gases produced during deposition. Gloves and mask used to avoid touch and inhale the poisonous gas respectively. Since gases are not hazardous at room temperature, the door of the system should be closed till compound get cooled to room temperature.

The structural characterization of the films was carried out using XRD (Rigaku mini flex 600) Cu-K α radiation within the range of 20°–60°. The surface morphology determination and compositional analysis were carried out using scanning electron microscope (CARL Zeiss) interfaced with EDAX. The UV–VIS–NIR spectrometer (Shimadzu 3600 UV–NIR 3600) was used to record the optical absorption spectra of the film in the wavelength range 300–3000 nm. The electrical resistivity of the film was determined using two probe setup (Keithley source measure unit 2400 and Keithley multimeter 2002) at room temperature that confirmed the nature of the ohmic contact. Silver film is deposited using physical vapor deposition technique and coplanar geometry has been used with thickness ~150 nm, area 1 cm × 1 cm and a gap

Table 1
Preparative parameters and optimized values for the synthesis of PbS thin films.

Parameter	Optimized value
Precursor	Lead acetate and thiourea
Concentration	0.1 M
Volume	20 ml
Substrate temperature	350 °C
Spray rate	1.5 ml/min
Nozzle to substrate distance	16 cm
Carrier gas	Air
Pressure of carrier gas	1.8 bar

of 1 mm. Resistivity measurements were performed at various temperatures and the activation energy was calculated from $\ln R$ vs $1/T$ plot. It is a simple method, which is widely used to determine the type of conductivity of a semiconductor specimen. Two fine metal probes are placed on the semiconductor sample and a multimeter is connected between them to measure the voltage. One of the probes is kept at room temperature and the other is heated to 80 °C. Hot probe heats the semiconductor immediately beneath it so that the kinetic energy of free carriers in this region is increased. Therefore, the carriers diffuse out of the hot region at a faster rate than they diffuse into this region from adjacent low-temperature regions. If the semiconductor is n-type, the electron will move away from the hot probe leaving a positive charge region of donors and the hot probe becomes positive with respect to the cold probe. The current will flow from the hot probe to the cold probe. In a p-type semiconductor, the direction of the current flow is reversed. Thus the polarity of the hot probe indicates whether the semiconductor is n-type or p-type. Hot probe and Hall Effect methods were used to find the type of conductivity in PbS thin film. Carrier concentration and mobility of the films were measured using Hall Effect method.

3. Results and discussion

3.1. Structural studies

XRD patterns of PbS thin films with different thickness are shown in Fig. 1. It is observed that the films have f.c.c structure with prominent (111), (200), (220) and (311) planes being observed in the diffractogram. The comparison of observed lattice parameters with the standard (JCPDS No. 05-0592) confirms the formation of lead sulphide thin films. The position of the diffraction peaks is independent of the film thickness; however, maximum intensity peak changes with film thickness as observed from the relative intensities of the peaks. The modifications in the peak intensity may be due to the re-alignment of crystallites with the increase in thickness [20]. This can be understood easily; in the samples which are sufficiently thick there is much more matter which can diffract X-rays and the peak is more intense. This causes an improvement in crystallinity with an increase in film thickness as reported earlier from researcher groups [21,22]. The average crystallite size (D) of PbS thin films has been calculated using Scherer's formula [23].

$$D = 0.9\lambda/\beta \cos \theta \quad (2)$$

where λ is the wavelength of the X-ray (1.5406 Å), β is the full width at half maximum (FWHM) of the peak, θ is the Bragg angle. The variation of average crystallite size with film thickness is summarized in Table 2. It is seen that crystallite size increases up to 520 nm film thicknesses; however for the higher film thickness crystallite size seems to be saturated. This clearly shows that, the thickness effect upon the crystallite size appears only in the thinnest films, the thickness of which is lower than approximately 600 nm–700 nm. In this range, the crystallinity is closely related to the film thickness. But, a tendency to the saturation and independence versus film thickness was observed beyond the range indicating that the space required for the thermal movement is optimum at 520 nm film thicknesses [22]. Further, the lattice constant 'a' of the films was determined by the following relation [16].

$$a = d(h^2 + k^2 + l^2) \quad (3)$$

Fig. 2 shows representative EDAX spectra of the film with the optimized thickness 520 nm. It has been observed that the at.% of lead and sulphur were 51.20 and 48.80 respectively with an error 0.5% for the films with thickness 520 nm. An excess of Pb results in

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