MAKARA, SAINS, VOL. 14, NO. 2, NOVEMBER 2010: 117-120

SYNTHESIS OF PbSe THIN FILM BY CHEMICAL BATH DEPOSITION AND ITS CHARACTERIZATION USING XRD, SEM AND UV-Vis SPECTROPHOTOMETER

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Abstract

Lead selenide thin films were prepared by chemical bath deposition method using aqueous of lead nitrate, sodium selenate and sodium tartrate. The influence of bath temperature towards the properties of the thin films was studied. The films were characterized by X-ray diffraction, scanning electron microscopy and UV-Vis spectrophotometer. The XRD results confirmed the polycrystalline cubic structure of PbSe films. The intensity of major peak at $2\Theta = 25.1°$ which belonged to (111) plane of PbSe, increased with bath temperature from 40 to 80 °C. The SEM micrographs showed that the most homogeneous surface and larger grain sizes could be seen for the films deposited at 80 °C as compared with other bath temperatures.

Keywords: lead selenide, semiconductor, chemical bath deposition, thin films

1. Introduction

The binary [1-2] and ternary [3-4] semiconductor compounds attract considerable scientific attention due to their potential applications in the field of solar cells, sensors, laser materials, thin films polarizers and thermoelectric cooling materials. PbSe thin films can be obtained as polycrystalline thin films by several deposition techniques such as electrodeposition [5], chemical bath deposition [6], electrochemical atomic layer epitaxy [7], photochemical [8], molecular beam epitaxy [9] and pulsed laser deposition method [10]. Chemical bath deposition method is considered as a cheap and simple method to produce large area thin films. So far, the vast preparation and studies of metal chalcodenides thin films using chemical bath deposition method can be classified into two categories, namely binary compounds such as $Cu_{2-x}Se$ [11], FeS_2 [12] and ZnS [13], and ternary compounds such as $CuBiS_2$ [14], $CdS_{1-x}Se_x$ [15] and $Pb_{1-x}Fe_xS$ [16].

However, no attempt is made on chemical bath deposited of the lead selenide thin films by using sodium tartrate as a complexing agent during deposition process. In view of this, an attempt has been made to synthesis lead selenide thin films at different bath temperatures ranging from 40 to 80 °C. The structural and surface morphological studies of lead selenide thin films have been studied using X-ray diffraction and scanning electron microscopy technique, respectively.

2. Methods

Lead selenide thin films were deposited on microscope glass slide using chemical bath deposition method. Prior to deposition, the glass slide was degreased in ethanol for 10 min, followed by ultrasonically cleaned with distilled water for another 15 min and finally dried in air. An aqueous solution of lead nitrate [$Pb(NO_3)_2$], was used as lead source, sodium selenate [Na_2O_4Se] as sulfide source and sodium tartrate [$C_4H_4Na_2O_6$] as complexing agent for depositing PbSe thin films. All these chemicals were analytical grade and all the solutions were prepared in deionised water (Alpha-Q Millipore). For deposition, 20 ml of 0.2 M lead nitrate was complexed with 10 ml of 0.2 M sodium tartrate solution. Then, 20 mL of 0.2 M sodium selenate was added slowly to the reaction mixture. The pH was adjusted to 5 by addition of hydrochloric acid with constant stirring using pH meter. The clean glass slide was vertically immersed into the chemical bath solution with the temperatures of 40, 60 and 80 °C, respectively. After the deposition time of 90 min, the glass slide was taken out of the bath, washed with distilled water and dried in desiccators for further characterization.

The X-ray diffraction analysis was carried out, using a Philips PM 11730 diffractometer for the 2θ ranging from 20° to 60° with CuKα (λ = 0.15418 nm) radiation. Surface morphology was studied by JEOL (JSM-6400) scanning electron microscopy operating at an accelerating voltage of 20 kV under 500 X magnification. The optical absorption study was carried out using the Perkin Elmer UV/Vis Lambda 20 Spectrophotometer in the wavelength ranging from 350 to 800 nm. The film-coated microscope glass slide was placed across the sample radiation pathway while the uncoated microscope glass slide was put across the reference path. The absorption data were used for the determination of the band gap energy (E_g).

3. Results and Discussion

The X-ray diffraction (XRD) patterns in Figure 1a-1c show the crystallinity of the films deposited at various bath temperatures ranging from 40 to 80 °C. At lower bath temperature, only two peaks of PbSe are observed. As the bath temperature is increased further to 60 and 80 °C, the number of peaks increased to three and four, respectively indicating more favorable conditions for the formation of cubic structure of PbSe thin films. The d-spacing values obtained for PbSe thin films prepared at theses bath temperatures are found to match the standard Joint Committee on Powder Diffraction Standards (JCPDS) (Reference code: 00-065-1040) [17] data (Table 1). The lattice parameter values are a=b=c=6.128Å.

On the other hand, as the bath temperature increases from 40 to 80 °C, the degree of crystallinity of the PbSe thin films increases. The diffraction peaks in the XRD patterns of the films deposited at 80 °C are very sharp with the high intensity indicating the significant increase in grain size. The diffraction peak at 2Θ = 25.5° shows the preferential orientation along the (111) plane. Based on the XRD patterns, the peaks correspond to (211) and (213) planes are observed for all samples. These diffraction peaks can be indexed in terms of the structure of silicon dioxide [18] (JCPDS reference No.: 01-074-0201) that comes from the substrate during deposition process.

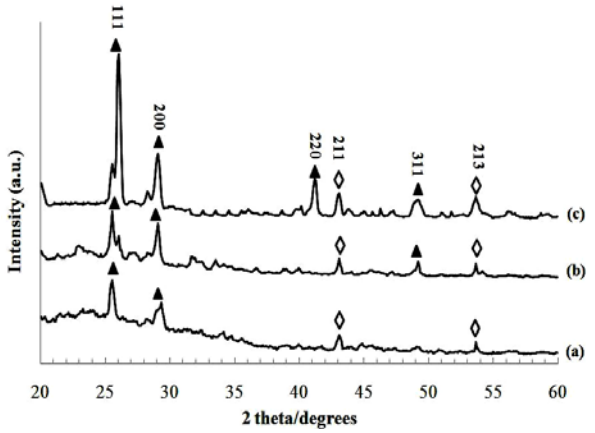

Figure 1. X-ray Diffraction Patterns of PbSe Thin Films Deposited at Various Bath Temperatures (a) 40 °C, (b) 60 °C, (c) 80 °C, PbSe (▲), SiO_2 (◊)

The surface morphology of the lead selenide thin films obtained by chemical bath deposition has been investigated by scanning electron microscopy (SEM). The SEM micrographs of the thin films deposited under different bath temperatures are presented in Figure 2. The films deposited at 40 and 60 °C contain randomly distributed spots on their surfaces. That is, all films have different islands with various sizes, shapes and their distributions on the surface are not homogeneous. These can be the results of the bath temperature that influence the chemical reaction during the deposition. The films obtained at 80 °C which have the most homogeneous surface as compared with other bath temperatures can be seen in Figure 2c. It is also observed that with the increase in bath temperature, the grain size increases while the density of the grains decreases. The increase in grain size with bath temperature indicates that the increase in the crystallinity of the films. These results also corroborate the results obtained from the XRD data. From the SEM micrographs obtained, we can conclude that the chemical bath deposition method is very much dependent on the bath temperature.

Figure 3 shows the absorption spectra of PbSe thin films deposited at different bath temperatures. All the films show a gradually increasing absorbance throughout the visible region, which makes it possible for these materials to be used in a photoelectrochemical (PEC) cell. From the figure, it is indicates that the films prepared at higher bath temperature (80 °C) have higher absorption values as compared with other bath temperatures. This is due to these films have the most homogeneous surface and better crystallinity films compared with other samples.

Table 1. Comparison of the JCPDS d-spacing Data for Lead Selenide Thin Films to Experimentally Observed Values for the Sample Deposited at Various Bath Temperatures

Bath temperature (°C)	2Θ (°)	*hkl*	d-spacing (Å) Observed value	JCPDS value
40	25.1	111	3.53	3.54
	29.0	200	3.07	3.06
60	25.1	111	3.53	3.54
	29.0	200	3.07	3.06
	49.1	311	1.84	1.85
80	25.1	111	3.53	3.54
	29.0	200	3.07	3.06
	41.5	220	2.15	2.17
	49.1	311	1.84	1.85

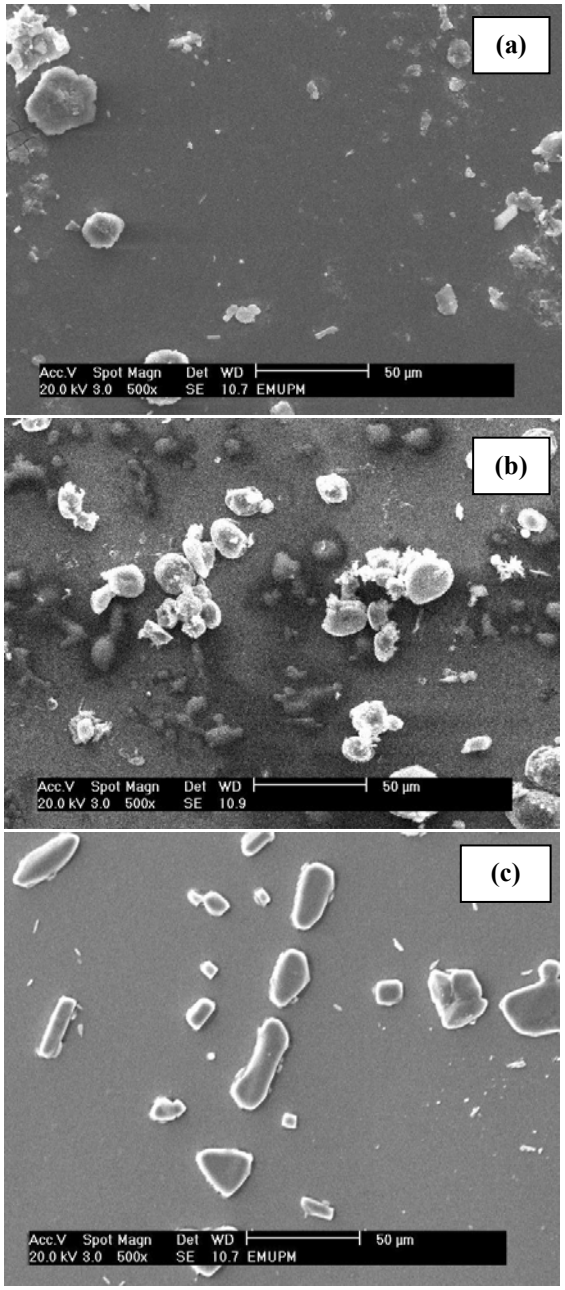

Figure 2. The SEM Micrograph of PbSe Thin Films Deposited at Various Bath Temperatures (a) 40 °C, (b) 60 °C, (c) 80 °C

In order to determine the band gap of thin films, the equation of Stern [19] was used.

$$A = \frac{[k(h\nu - E_g)]^{n/2}}{h\nu} \quad (1)$$

where ν is the frequency, h is the Planck's constant, k equals a constant while n carries the value of either 1 or 4. The n value is 1 for a direct gap material and 4 for indirect gap material. The plot of $(Ah\nu)^2$ against $h\nu$ is shown in Figure 4. Extrapolation of the linear portion of the curve to $(Ah\nu)^2 = 0$ gives the band gap energy. The

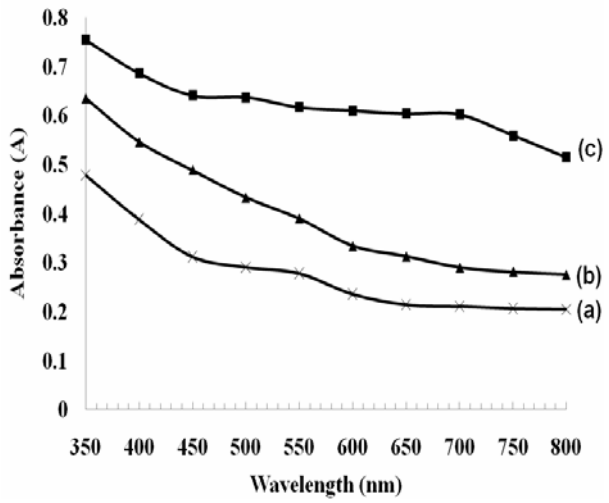

Figure 3. Absorbance Versus Wavelength of PbSe Thin Films Deposited at Various Bath Temperatures (a) 40 °C, (b) 60 °C, (c) 80 °C

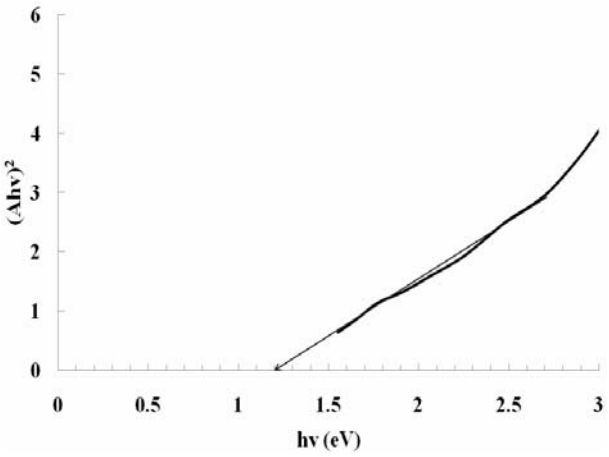

Figure 4. The Plot of $(Ah\nu)^2$ Versus $h\nu$ for the PbSe Thin Films Deposited at 80 °C

straight line behaviour establishes that all the films have a direct band gap. Based on the figure, the band gap energy of the lead selenide thin films deposited at 80 °C is approximately to be 1.2 eV.

4. Conclusion

The results obtained indicated that we successfully to produce PbSe thin films using a low cost chemical bath deposition method. The chemical bath consisted of lead nitrate, sodium selenate and sodium tartrate solutions. The XRD results confirmed the polycrystalline cubic structure of PbSe thin films. The intensity of major peak at $2\Theta = 25.1°$ which belonged to (111) plane of PbSe, increased with bath temperature from 40 to 80 °C. The SEM micrographs showed that the most homogeneous surface and larger grain sizes could be seen for the films

deposited at 80 °C as compared with other bath temperatures. These films produced the highest absorption characteristic with band gap approximately to be 1.2 eV. Therefore, deposition at 80 °C was the best condition to prepare good quality PbSe thin films under the current conditions.

Acknowledgement

The authors would like to thank the Department of Chemistry, Universiti Putra Malaysia for the provision of laboratory facilities and MOSTI for the National Science Fellowship (NSF).

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