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The role of Bath Temperature in aqueous Acidic Chemically PbSFilms

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ABSTRACT

Lead sulphide thin films have been deposited on glass slides from chemical baths constituted with sodium thiosulfate and lead nitrate. The PbS thin films were characterized by scanning electron microscope, profilometer, energy dispersive X-ray analysis and photoelectrochemical test. Photoelectrochemical test showed that the PbS films prepared at 35 °C indicated the lowest photo response activity. Meanwhile, a maximum thickness of 1313nm was achieved for the PbS films deposited at 75 °C.
KEYWORDS: profilometer, photo activity, thickness, deposition, scanning electron microscopy

INTRODUCTION

Thin films of semiconductor such as PbS, ZnS, CdS, CuS, MnS, NiS, SnS, etc. have attracted the attention of many researchers. This is because of their brilliant applications such as solar technology, optoelectronic devices, photo catalysis, infrared radiation detector, laser materials, piezoelectric transducer and so on. PbS can be prepared by spray pyrolysis[1], successive ionic layer adsorption and reaction[2], thermal evaporation[3],solid-vapor deposition[4], galvanic method[5], cyclic voltammetry method[6], electro deposition[7,8] and chemical bath deposition [9-15]. Chemical bath deposition method has been developed to produce thin films in consideration of its many benefits such as it does not require sophisticated instruments, the deposition parameters are easily controlled, low cost and low temperature processing.

Investigation and control of the film thickness are important in semiconductor technology. Thickness has a great influence on the band gap, surface roughness, optical and electrical properties of films. For these reasons, there have been many researches about an investigation of film thickness through various instruments such as profilometer [16-22], ellipsometer [23], auger electron spectroscopy[24], interferometric method[25-28], weight-difference method[29-30] and atomic force microscopy[31-34].In this paper, we describe for the first time an investigation of the film thickness using profilometer. There is no literature review related the measurement of thickness of PbS films using profilometer by other researchers.

MATERIALS AND METHODS

The substrate used for deposition of PbS thin films was optical microscope slide with the size of 25.4 mm x 76.2 mm and about 1.2 mm thick. The substrate was prepared and cleaned by the following method. First, it was immersed into the beaker that filled with hydrochloric acid for 24 hours. After that, the glass slide was immersed into the beaker that contained deionized water and then ultrasonically degreased for 20 minutes by using the sonicator. Later, it was immersed into the beaker that filled with ethanol and ultrasonically degreased for another 20 minutes. Then, the glass slide was taken out and rinsed with deionized water. Finally, it was ready to be preheated in the oven for about 10 minutes at 120^{o}C.

In this study, 20 mL of 0.1M lead nitrate [$Pb(NO_3)_2$was added to 20 mL of0.1M sodium thiosulphate in beaker. The mixed solutions were stirred well using glass rod. Next, sulfuric acid was added to change the pH of the bath into the pH 2.5using the pH meter. The preheated glass slide was mounted in the solution and the reaction vessels were put into the water bath at temperature required (35, 45, 65 and 75°C) for 2 hours. Then, the glass substrate was taken out and dried in the oven for about 10 minutes at 120 ^{o}C. Lastly, the sample was preserved in the desiccators for 2-3 days and kept for further analysis.

The surface morphology was observed by a scanning electron microscopy (JEOL, JSM-6400), at 20 kV with a 1000 X magnification. The elemental composition of the films was studied by scanning electron microscope attached with energy dispersive analysis of X-ray (EDAX) analyzer. High surface profilometer (which model was

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XP-200, Ambious Technology) was performed on each of the samples to measure the thickness for those samples. Lastly, photoelectrochemical (PEC) experiments were performed using a $[Fe(CN)_6]^{3-}/[Fe(CN)_6]^{4-}$ redox system, by performing linear sweep voltammetry between -500 to -900 mV (Ag/AgCl as reference electrode). The sequence of constant illumination, chopped illumination and dark period were performed on the PEC cell to study the effect on photo activity behavior. A halogen lamp (300 W, 120 V) was used for illuminating the electrode.

RESULTS AND DISCUSSION

The chemical bath deposition technique is based on a chemical reaction between dissolved precursors (lead ions and sulfur ions in this case) in aqueous solution. Bath temperature is an important parameter in the chemical bath deposition technique. The study on the effect of bath temperature indicates that the film thickness increases with bath temperature from 35 °C to 75 °C (Table 1). The thickness for the PbS films deposited at 75 °C is observed to be 1313 nm. This behavior can be attributed to the growth of PbS films onto the substrate during the deposition process. Therefore, the reaction that leads to the formation of thicker PbS is favored at bath temperature of 75 °C.

Table 1. The results of the film thickness of the samples deposited at various bath temperatures

Temperature (°C)	Thickness (nm)
35	815
45	928
65	1088
75	1313

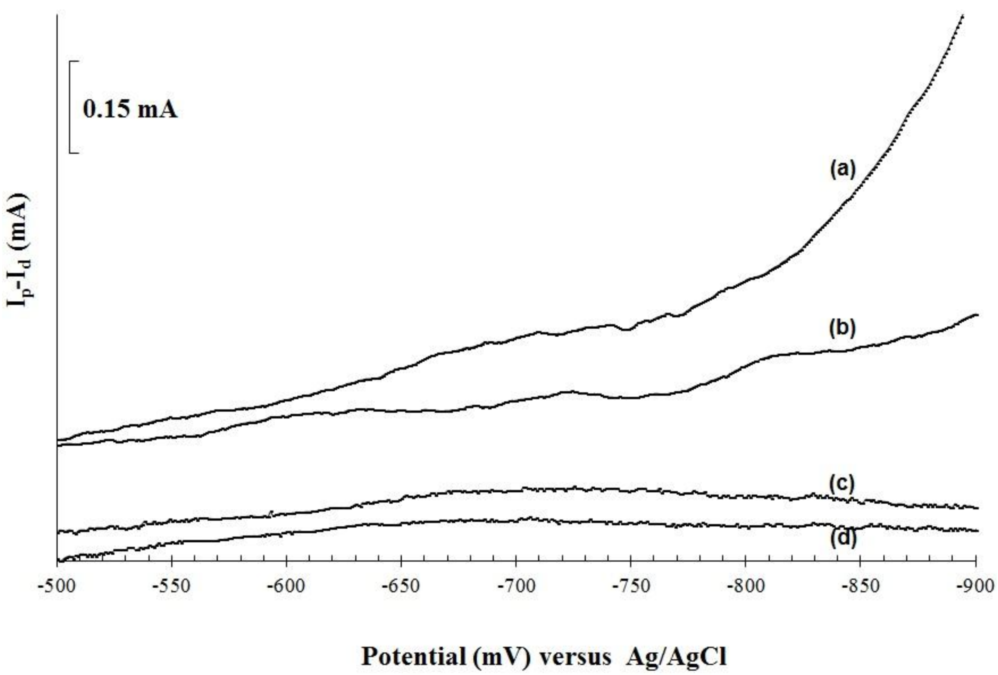

Figure 1: Comparison of photosensitivity of the PbS films deposited at different bath temperatures [a] 75 °C[b] 65 °C[c] 45 °C[d] 35 °C

Figure 1 indicates the comparison of photosensitivity of the PbS films prepared at various bath temperatures ranging from 35 to 75 °C. This figure represents the difference between the photocurrent (I_p) and dark current (I_d) versus potential (Ag/AgCl) for the PbS films in contact with 0.01M sodium thiosulfate in photoelectrochemical test. The graph shows the photocurrent occurs on the negative potential means that the films prepared are p-type. This is consistent with the report from P.K. Basu [13] and I. Pop[15].The films deposited at 35 °C shows the lowest photo response behaviors are characterized by the formation of the thinnest PbS films. The increase of the photosensitivity for the films deposited at 75 °C is explained through the formation of the thickest PbS films (Table 1). Therefore, the deposition parameters such as bath temperature have a great influence over the photo response behavior.

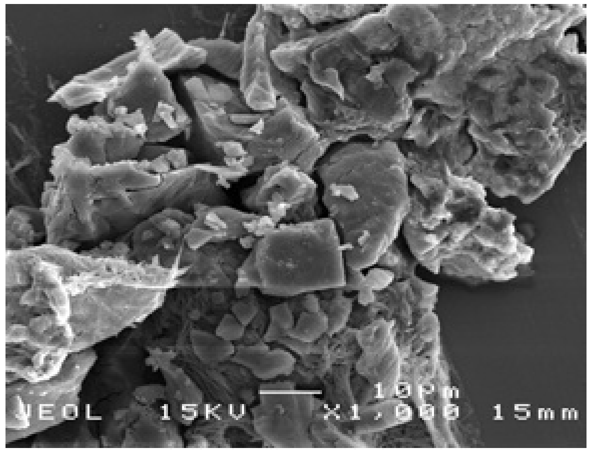

Figure 2: Scanning electron microscopy micrograph of the PbS films deposited at 75 °C

Scanning electron microscopy (SEM) micrograph allows us to obtain information about the morphology of sample surface. Figure 2 indicates the scanning electron microscopymicrograph of the PbS films deposited at 75 °C. This SEM micrograph reveals that the obtained films are rather dense and non-uniform. The size distribution of the grains seems to be broad, with the maximum size reaching 10 µm, whereas as small PbS nuclei as 1 µm can be observed on the glass slide substrate.

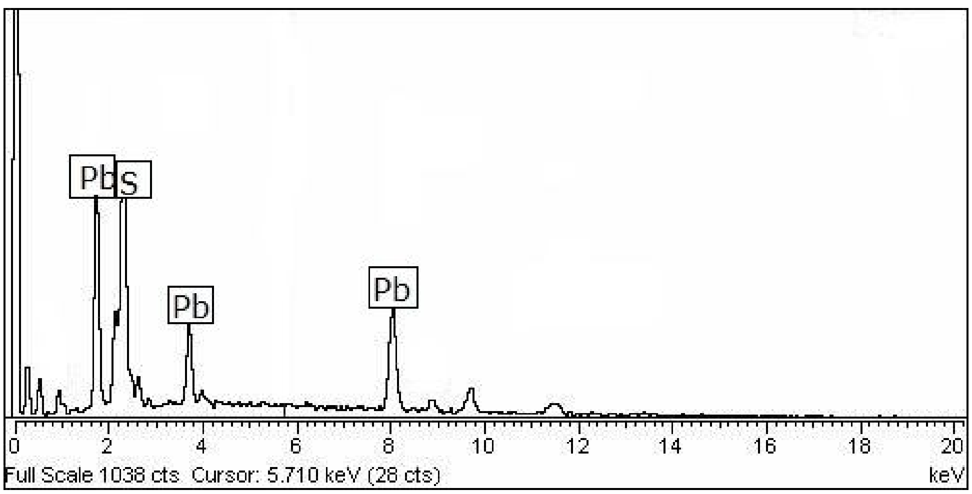

Figure 3: Energy dispersive X-ray analysis spectrum for the PbS films deposited at 75 °C

The quantitative analysis for energy dispersive X-ray analysis (EDAX) was performed for lead and sulfur. The atomic percentage of Pb:S is 49.18%:50.82%, showing that the sample is nearly stoichiometric. The corresponding EDAX spectrum of PbS films deposited at 75 °C is shown in Figure 3.

Conclusions

PbS films were prepared by chemical bath deposition technique on glass slide substrates at various deposition temperatures varying from 35 to 75 °C.The thinnest PbS films obtained at the bath temperature of 35 °C. However, as the bath temperature was increased, the film thickness increased.Photoelectrochemical test revealed that the films deposited at 75 °C showed the highest photo response behavior.

Acknowledgment

The authors declare that they have no conflicts of interest in this research.

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