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GROWTH AND OPTICO-MICROSTRUCTURAL PROPERTIES OF PBS THIN FILMS PREPARED BY CHEMICAL BATH DEPOSITION METHOD

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ABSTRACT

Thin films of lead sulphide have been grown by chemical bath deposition method, using triethanolamine as complexing agent. The films were deposited on both surfaces of the glass substrate at deposition time of 4hours. One sample was kept unannealed while one was annealed at temperature of 150°C for 1 hour in an electric oven. Visible/Ultraviolet spectrophotometer, model 6405 was utilized to measure the transmittance and reflectance of each sample at various wavelengths. Also, the samples were studied with the use of optical microscope and Scanning Electron Microscope. The effect of annealing on transmittance and reflectance of chemically deposited lead sulphide thin film were then studied. It was observed that higher values of transmittance and lower values of reflectance were obtained from annealed sample when compared with the unannealed sample.The average grain size in the unannealed film was estimated to be 1.3µm while that of the annealed film was estimated to be 2.5µm. such increase is attributed to the grain growth during annealing. The high values of transmittance and low values of reflectance are also attributed to the absence of precipitate on PbS after annealing. Hence the study reveals that annealing has effect on grain size, transmittance and reflectance of the films.

KEYWORDS: Microstructural, Chemical Bath

INTRODUCTION

Lead sulphide is a chalcogenide with a direct band gap of 0.41 eV and has a cubic structure. Owing to their suitable bandgaps, PbS thin films are widely used in infrared detectors. Lead sulphide is suitable for the detection of the radiation between wavelengths 1 and 3 µm.The use of very thin chemically deposited PbS thin films as solar control application have been discussed by many researchers (Nair et al 1989). Chemical bath deposition (CBD) is one of the standard methods used for preparing PbS (Gadave et al 1994; Popa et al2006). This method is less expensive, easy to handle, allowing deposition of films on a large area. Although many investigations on PbS thin films deposited by Chemical Bath deposition techniques have been carried out. It was found out that not much results are reported on structural attributes of the films and their correlation toother deposition properties. Keeping in view all theseaspects, an experimental study on the structural characterization of chemically deposited PbS films has beenundertaken.

METHODOLOGY

The solution for deposition of lead sulphide thin film on both surfaces of glass substrate (microscopic slide) were constituted from aqueous solution of 1.0 mole of lead Acetate, 1.0 mole of Thiourea (TU) and 1.0 mole Tri-Ethanol Amino (TEA). Sodium hydroxide (NaOH) was added to the solution to give a PH value between 9 and 10 to the chemical bath. The concentration of the reagents, pH (between 9 and 10) and temperature of 25°c were considered for deposition time of 4hrs.There was slow release of Pb^{2+} and S^{2+} ions in solution, which condensed on the optical glass slides that were immersed vertically on the wall of 50ml beaker containing the solution. The deposition of PbS occurs when the ionic product of Pb^{2+} and S^{2+} exceeds the solubility product of PbS.

The formation of PbS thin film involves the following chemical reactions.

$SC(NH_2)_2 + OH\ CH_2N_2 + H_20 + HS$

$2HS + 2OH\ 2H_2O + S_2$

$Pb^{2+} + S^{2+}PBS$

The glass substrates were removed from a specific bath after 4 hours deposition time. One sample was annealed in electric oven at 150^oC for 1 hour. The transmittance and the reflectance of the samples were measured using a UV/VIS spectrophotometer model 6450 in the wavelength range 0.30 and 0.7 µm. The microstructures of the films were studied using optical microscope (model JOM6310) with accelerating voltage of about 15 kV. The surface morphology was analysed using Scanning Electron Microscope (model XL 20 SEM).

RESULTS

Observation shows that higher transmittance values are obtained from annealed sample when compared withas-prepared sample. This could be as a result of improved microstructure of lead sulphide after annealing. The transmittance increased with increasing wavelength. The films showed less transmittance in the UV region. The correlation between transmittance of annealed samples was done by using linear regression function in Excel Spreadsheet. The reading was plotted on the graph with the reading of transmittance of unannealed sample on horizontal x-axis and annealed samples on vertical y-axis. When trend line using linear least square was plotted, the correlation equation $y=9.87 + 1.1604x$ was obtained. The curve of variation of reflectance with wavelength for unannealed sample of PbS appears to be inverse of that of annealed sample.The correlation between reflectance of unannealed sample and annealed sample was also done by using linear regression function in excel spreadsheet. The reading of reflectance of unannealed sample was plotted on horizontal x-axis and annealed samples on vertical y-axis. The least square prediction line for reflectance of unannealed and annealed samples is $y=1.2183 + 0.2401x$.

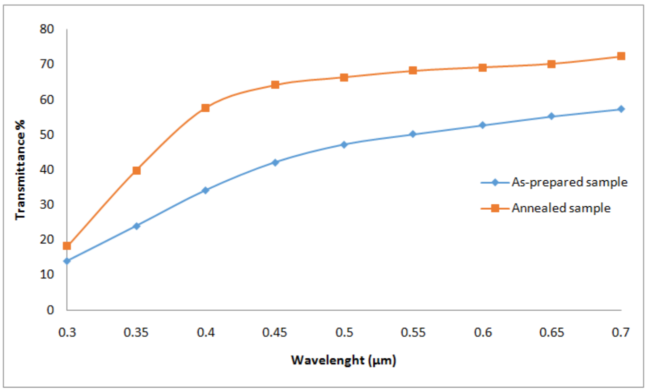

Figure 1: Variation of Transmittance with Wavelength of as Prepared and Annealed PBS Samples

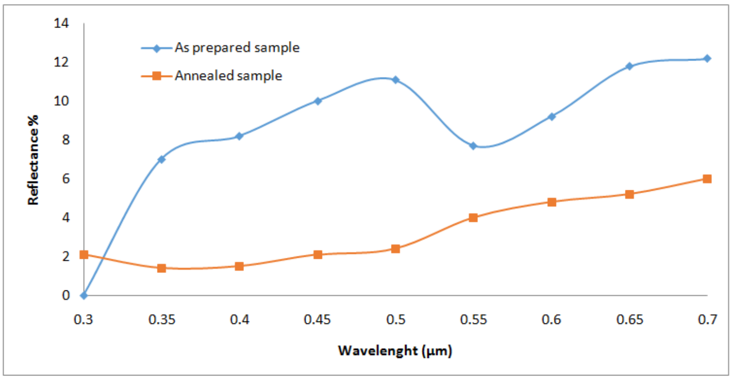

Figure 2: Variation of Reflectance with Wavelength of as Prepared and Annealed PBS Samples

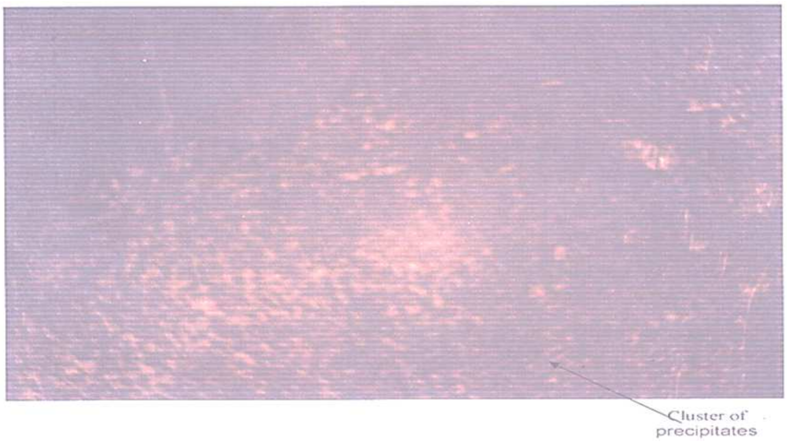

Figure 3: Optical Microscopy of Unannealed Sample

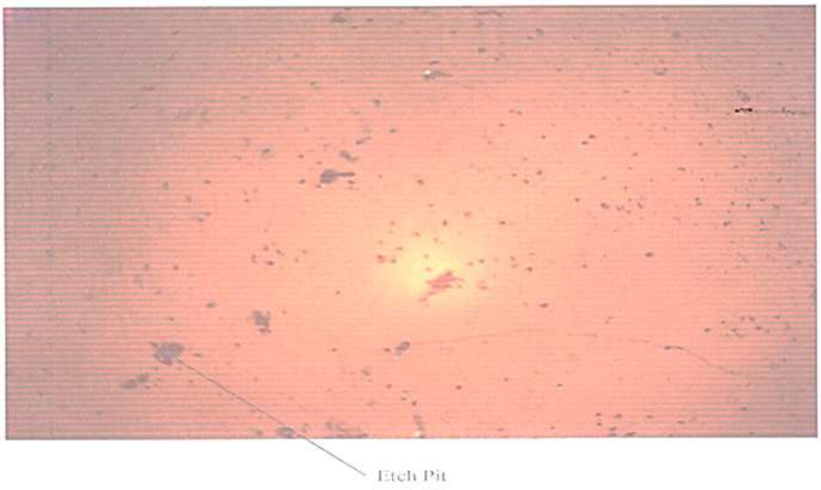

Figure 4: Optical Microscopy of Annealed Sample

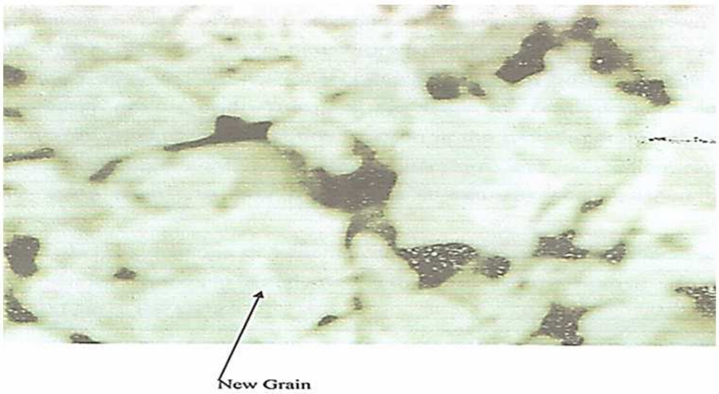

Figure 5: Surface Morphology of Unannealed Pbs Sample

Figure 6: Surface Morphology of Annealed Pbs Sample

Figure 3 show the optical micrograph of unannealed PbS sample. The micrograph showed that the clusters of precipitates cover the surface of the deposition. Figure 4 revealed the optical micrograph of annealed PbS films. From the micrograph, etch pits are obvious because there is no precipitate as a result of annealing.

Figure 5 illustrates the surface morphology of unannealed PbS films which show the effect of a poorly crystalline and adhesive film. Effects such as fracture and clusters of precipitates with other associated defects were observed. Figure

6 illustrate the surface morphology of PbS thin films. The micrography shows the formation of new grains in a recrystallized region. It shows less dislocation density. It is clear from this figure that the average grain size in the annealed sample is greater than as prepared sample. SEM reveals that the average grain size in the prepared film was estimated to be about 1.3µm while that of the annealed film was estimated to be about 2.5µm.The annealed PbS film is observed to compose of homogeneous crystalline arrangement. This observation supported the result obtained showing higher transmissivity and lower reflectivity values for unannealed one. Annealing the films results in equiaxial grains (grains of roughly equal size). There are gaps observed between the grains which could be as a result of incomplete grain growth. The gaps are areas full of dislocations and vacancies. Grain size is a strong function of the growth parameter like annealing temperature, substrate temperature and deposition rate. Increasing grain size after the growth is normally accomplished by annealing.Also, high values of transmittance and low values of reflectance of the annealed sample is attributed to the absence of the precipitate on the surface of the sample.

CONCLUSIONS

Conclusively, the high values of transmittance and low values of reflectance of the annealed sample were attributedto the absence of the precipitate on the surface of the sample. The SEMstudies indicated that there was grain growth during the annealing process. This resulted in polycrystalites with larger grain sizes compared with the grain size of the as prepared one.The optical microscopy and SEM micrographs revealed the microstructure of the films which support the results of transmittance and reflectance. These results have led to considering chemically deposited PbS films as potentially important for photo-conductive and photovoltaic application.This study also revealed that annealing has effect on grain size, transmittance and reflectance of the films and consequently influence the optical properties of chemically deposited lead sulphide thin films.

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