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Effect of Annealed Temperature on Absorptivity of Chemically Deposited Lead Sulphide Thin Film

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Abstract: Thin films of Lead Sulphide have been grown by a chemical bath deposition method, using tri-ethanol amine as complexing agent. The films were deposited on both surfaces of the glass substrates at various deposition times of 3-5 h. Some samples were kept as prepared while some were annealed at different temperatures of 100, 150 and 200°C in an electric oven. Visible/Ultra-Violet Spectrophotometer, model 6405 was utilized to measure the absorbance of each sample at various wavelengths. The effect of annealed temperatures on absorptivity of chemically deposited Lead Sulphide thin film is then studied. It is observed that high values of absorptivity are obtained from annealed samples when compared with the as prepared samples. And also, the *critical* annealed temperature at which optimum absorptivity occurs shifted forward in trend from samples annealed at 100°C to samples annealed at 150 and then samples annealed at 200°C as deposition time increases from 3-5 h, respectively i.e., increasing the deposition time increases the critical annealed temperature at which maximum absorptivity occurs for chemically deposited Lead Sulphide thin film. These could be attributed to the modified grain boundaries and increase in grain size of Lead sulphide crystal after annealing. Hence, the study reveals that the annealed temperature has effect on the rate of absorptivity of lead Sulphide thin film and consequently influence the electrical and optical characterizations of chemically deposited Lead Sulphide.

Key words: Absorbance, annealed temperature, deposition time, wavelength, substrate, thin film

INTRODUCTION

Thin films have been noted for applications in the production of high quality camera lenses, multi-layer non-absorbing beam splitter, dichroic mirrors, photographic film, motion picture projector and astronauts' elements e.t.c.

It is a layer of material deposition whose thickness is of the order of a given electromagnetic radiation, which is coated on substrates that could be plastics, glasses or metals.

The importance of Lead Sulphide thin films as semiconductors has experienced a fast rising, mainly due to its application in piezoelectric transducers, laser materials and photovoltaic cells.

The commonly used methods for depositing thin films are vacuum evaporation, sputtering, spray pyrolisis, molecular beam epitaxy and electro-deposition.

The chemical bath deposition (CBD) is an electroless technique that is attractive as a simple and low cost method (Sasikala *et al*., 2000).

Bode (1996) explained the usage of Lead Sulphide in infra-red detectors since mid 1940s and it was for this application that the chemical bath deposition technique for Lead Sulphide thin films known since 1910, was initially developed in the late 1940s.

Nair *et al*. (1991) declared that the typical deposition process of Lead Sulphide thin film involved the immersion of glass substrates in the alkaline solution. Lead thiourea solution yielded Lead Sulphide (PbS) thin films of various thickness of 0.05 and 0.14 μm in about 2 h.

Scanning Electron Micrograph studies conducted by Fajinmi (2001) had shown that annealing of chemically deposited thin films modify its grain boundaries and consequently increase its grain size.

Grecu *et al*. (2004) reported that the films grown from the bath using ammonium chloride as chelating agent instead of citrate in Cadmium Sulphide formation are uniform and adherent, have a high transmittance in the visible region and are about 2 times thicker than the films deposited from a standard bath which contains citrate.

The aim of this study is to study the effect of temperature and specifically, the annealed temperature among many deposition conditions on absorptivity of Lead Sulphide thin film chemically deposited at varied time.

MATERIALS AND METHODS

Lead Sulphide films were prepared from Lead Acetate and thiourea by chemical bath deposition in alkaline solution.

The films were grown on optical glass slides as substrates that were cleaned thoroughly at the Physics Laboratory of Ladoke Akintola University of Technology, Ogbomoso, Nigeria in 2006.

The solution for deposition of Lead Sulphide thin film on both surfaces of glass substrates were constituted from aqueous solution of 1.0 mole of Lead Acetate, 1.0 mole of thiourea (TU) and 1.0 mole of Tri-Ethanol Amine (TEA) as complexing agent. Sodium Hydroxide (NaOH) was added to the solution to give a PH value between 9 and 10 to the chemical bath and temperature of 25°C were kept constant for all deposition time of 3-5 h.

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 50 mL beaker containing the solution.

$$SC(NH_2)_2 + OH \longrightarrow CH_2N_2 + H_2O + HS \quad (1)$$

$$2HS + 2OH \longrightarrow 2H_2O + S_2 \quad (2)$$

$$Pb^{2+} + S^{2+} \longrightarrow PbS \quad (3)$$

Some glass substrates were removed from a specific bath after 3 h deposition time, some were removed from second deposition bath after 4 h deposition time and others were removed from the third deposition bath after 5 h.

A batch of 3-5 h samples were put aside as as prepared. The second batch of 3-5 h samples was placed in electric oven for annealing at 100°C for 1 h. The third batch of 3-5 h samples was placed in electric oven for annealing at 150°C for 1 h. The fourth batch of 3-5 h was placed in electric oven for annealing at 200°C for 1 h.

Visible/Ultra-violet Spectrophotometer, model 6405 was then used to measure the absorbance of each sample under investigation at various wavelengths, between 400 and 1000 nm after annealing.

Absorbance of as prepared sample at deposition time of 3-5 h were then compared with the Absorbance of the annealed samples under the same ranges of wavelengths.

RESULTS AND DISCUSSION

Lead Sulphide films can be produced by decomposition of thiourea in an alkaline solution containing Lead Salt. The deposition is achieved from dilute solutions and Sulphide ions are released in the bath by the hydrolysis of thiourea in the presence of OH-ions.

Records of absorbance of all sample batches of the as prepared and annealed at different temperatures for 3-5 h deposition time are taken.

The graphs of absorbance against wavelength for a specific sample batch (i.e., as prepared and annealed) at different deposition time are plotted.

Figure 1 shows the variation of Absorbance with wavelength of Lead Sulphide samples at different temperatures of annealing but within the same deposition time of 3 h. The absorbance of the as prepared samples is then compared with the annealed samples. It is observed that highest absorbance values are obtained from the annealed samples and optimally from samples annealed at 100°C. The absorbance of as prepared samples and those annealed at 150 and 200°C are so low and gradually reduces to zero absorbance. Thus, the critical annealed temperature at which optimum absorbance occurs for each wavelength of 3 h deposition samples is 100°C.

Figure 2 reveals the variation of Absorbance with wavelength of Lead Sulphide samples at different temperatures of annealing but within the same deposition time of 4 h. Highest absorbance values are obtained from annealed samples when compared with as prepared samples and most significantly, with sample annealed at 150°C. The absorbance of as prepared samples and those annealed at 100 and 200°C are low compared with samples annealed at 150°C. Thus, critical annealed temperature for samples deposited for 4 h occurs at 150°C.

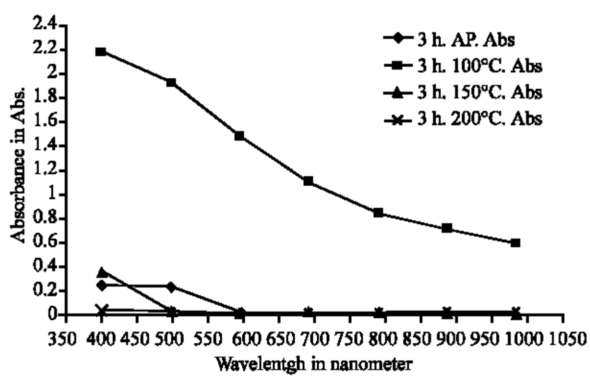

Fig. 1: Variation of absorbance with wavelength of PbS samples deposited for 3 h

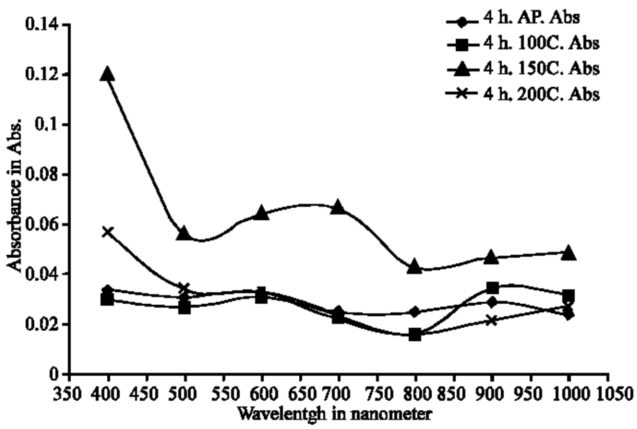

Fig. 2: Variation of absorbance with wavelength of PbS samples deposited for 4 h

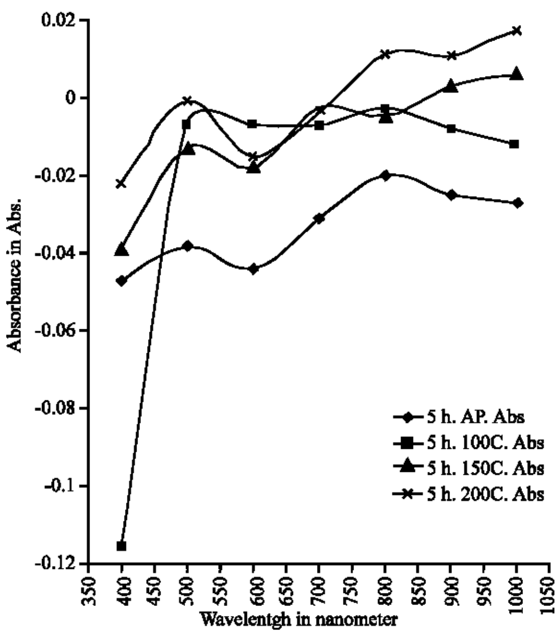

Fig. 3: Variation of absorbance with wavelength of PbS samples deposited for 5 h

Samples deposited for 5 h also produce highest absorbance values from samples annealed at 200°C between wavelength of 400 and 600 nm. Beyond these wavelengths, there are irregularities in the absorbance value which may be attributed to non-inform deposition of Lead Sulphide on those Substrates (Fig. 3). The critical annealed temperature for samples deposited for 5 h, thus, occurs at 200°C.

It is observed from these figures that annealed samples produce high absorptivity than the as prepared samples. This could be as a result of improved microstructure of Lead Sulphide after annealing as studied by Fajinmi (2001) who concluded that defects in PbS crystalline structure were minimized through annealing and that its grain boundaries were modified and consequently increase the grain size. Hence, the modify grain boundaries and grain growth are assumed to be responsible for high absorptivity obtained in annealed samples.

Also, it is observed that the critical annealed temperatures for maximum absorptivities at each wavelength shifted forward, that is, from 100-50 and then to 200°C as the deposition time increases from 3-5 h, respectively. So, it is assumed that the longer the deposition time of Lead Sulphide thin film, the higher the critical annealed temperature at which optimum absorptivity occurs.

This study, thus, confirm temperature and specifically, annealed temperature, as one of the conditions that strongly influence the film stoichiometry, microstruture and crystallinity and consequently the optical and electrical properties of PbS thin films. This is consistent with report from Greeu, 2004.

CONCLUSION

Lead Sulphide thin films have been grown on both surfaces of glass substrates. Some samples are as prepared at room temperature of 25°C and some are annealed at temperature of 100, 50 and 200°C. Absorbance of each samples are measured and compared at different deposition time.

Absorbance of annealed samples yielded high values of absorbance than the as prepared samples. This is attributed to the grain growth of PbS after annealing.

And also, up to a specific temperature, the higher the deposition time of PbS thin film, the higher the critical annealed temperature at which optimum absorptivity occurs.

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