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Full Length Research Paper

Fabrication and characterization of lead sulphide thin film by chemical bath deposition

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Semiconductor lead sulphide (PbS) thin films were deposited on glass substrate using chemical bath deposition method (CBD) at 300K temperature. The preparative conditions such as concentration of ions and deposition time were optimized. Surface morphology and optical characterization were carried out with the help of an Olumpus optical microscope and a Janway 6405 UV-VIS spectrophotometer. Optical investigation showed that, PbS thin film have bandgap energy of 1.90eV and refractive index range of 2.32–2.58. Our results reveals that the thickness of PbS films were dependent on time of growth and concentration of the ions.

Keywords: Band gap, chemical, bath, chemical bath.

INTRODUCTION

Lead sulphide (PbS) thin film have been projected as a material of interest to study quantum size effect in recent years (Wang et al.,1987;Gao et al., 2001 and Nanda et al., 2002), structural and optical properties of PbS thin films have been extensively studied (Joshi et al., 2004). Thin film of lead sulphide (PbS) have been found to have very important application in the manufacture of photoconductive infra-red detectors, transistors, contact rectifiers (Putley, 1967). They also have very important applications in areas of prisons, lenses, windows and other components of optical system (Ballard et al., 1972).

Chemical bath deposition (CBD) is a simple, reproducible and cost effective technique for fabricating high quality compound semiconductor thin films (Ramaiah, 2001). The method is well studied and produces films that have comparable structural and optoelectric properties to those produced using other sophisticated thin film deposition techniques (Osuji, 2003 Okujagu and Okeke, 1997; Campe et al., 1985). The technique has been applied in producing emerging material for solar cells, protective coating, solar thermal controls in buildings and is being adopted by some industries (Ezema and Okeke, 2002; Illenikhena and Okeke, 2002). A ligand acting as a catalyst is usually employed to control the reaction in a suitable medium as indicated by the pH to obtain crystal growth. Otherwise, spontaneous reaction and sedimentation of materials will be obtained. The condition for compounds to be deposited from a solution bearing it ions is that its ionic product (IP) should be greater than the solubility product (Ksp).

This work reports the successful deposition of Lead sulphide (PbS) thin films at 300K using chemical bath deposition The deposition of lead sulphide (PbS) thin film by CBD was based on the reaction between lead nitrate ($Pb(NO_3)_2$) and thiourea ($SC(NH_2)_2$) using EDTA di sodium salt as a complexing agent and ammonia solution as a pH adjuster at 300K.

MATERIALS AND METHOD

We synthesized all the films for this experiment using chemical bath deposition (CBD). The deposition of lead sulphide (PbS) thin film by CBD was based on the reaction between lead nitrate ($Pb(NO_3)_2$) and thiourea ($SC(NH_2)_2$), using EDTA disodium salt as a complexing agent and ammonia solution as a pH adjuster at 300K. Thiourea is used as our sulphide ion source and lead nitrate as our lead ion source. The deposition process is based on slow release of Pb^{2+} and S^{2-} ions in the solution which condensed on the substrate. The solution for deposition of lead sulphide thin film using a glass

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Table 1. Optimization of Lead Ion Source.

Slide NO.	Volume of complexing agent (EDTA) (mls)	Volume of thiourea (mls)	Volume of $Pb(NO_3)_2$ (mls)	Volume of ammonia (mls)	Time (hour)	Thickness (μm)
Pb_{11}	5.00	5.00	5.00	5.00	3.00	0.342
Pb_{12}	5.00	5.00	7.50	5.00	3.00	0.910
Pb_{13}	5.00	5.00	10.00	5.00	3.00	0.992
Pb_{14}	5.00	5.00	12.50	5.00	3.00	0.999
Pb_{15}	5.00	5.00	15.00	5.00	3.00	0.999

Table 2. Optimization of Deposition Time.

Slide NO.	Volume of complexing agent (EDTA) (mls)	Volume of thiourea (mls)	Volume of $Pb(NO_3)_2$ (mls)	Volume of ammonia (mls)	Time (hour)	Thickness (μm)
Pb_1	5.00	5.00	5.00	5.00	1.00	0.296
Pb_2	5.00	5.00	5.00	5.00	2.00	0.513
Pb_3	5.00	5.00	5.00	5.00	3.00	0.844
Pb_4	5.00	5.00	5.00	5.00	4.00	1.019
Pb_5	5.00	5.00	5.00	5.00	5.00	1.176

substrate (glass microscope slide) were constituted from solution of 1.0 mole of $Pb(No_3)_2)$, 1.0 mole of thiourea and 1.0 mole of EDTA disodium salt.

The reaction mechanism is of the form:

$Pb(NO_3)_2 + EDTA \rightleftarrows (Pb(EDTA)^{2+} + 2NO_3^{2-}$

$(Pb(EDTA]^{2+} \rightleftarrows Pb^{2+} + EDTA$

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

$HS^- + OH^- \rightleftarrows H_2O + S^{2-}$

$Pb^{2+} + S^{2-} \longrightarrow PbS$

Optimization of Deposition Time

For this experiment, the volumes of lead nitrate, thiourea, EDTA, ammonia solution and temperature (300K) were kept constant, while the deposition time was altered as shown in Table 1 above.

Surface morphology and optical characterization of the films were carried out using an Olumpus optical microscope and a Janway 6405 UV-VIS spectrophotometer respectively. From the spectrophotometer, the absorbance in arbitrary units was measured. Parameters such transmittance, reflectance, refractive index, extinction coefficient, dielectric constant and optical conductivity were then calculated.

Various parameters which include, volume of lead ion source and deposition time were optimized.

Optimization of Lead Ion Source

The concentration of thiourea, EDTA, ammonia solution, temperature (300K) and time (3hours) were kept constant, while that of lead nitrate was altered as shown in Table 2 above.

RESULTS AND DISCUSSION

Figures 1 and 2 are plots of thickness versus time and lead ion concentration for PbS thin film. Figure 1 shows an almost linear relationship with time of growth. At 1hour growth time, thickness was 0.296μm and at 5hours growth time, it was 1.176μm. Figure 2 indicates that the film thickness increased with increasing volume of lead nitrate, with 0.342μm thickness at 5.00mls of lead nitrate and 0.999μm at 15.00mls. Figure 3 is a plot of absorption coefficient squared (α^2) versus photon energy hυ for PbS thin film. The optical bandgap energy of PbS thin films were determined from this graph by extrapolating the straight portion of the graph to $\alpha^2=0$. This yielded bandgap of 1.90eV for PbS thin film. This value compares well with a range of 1.88-2.28eV by (Ubale and Kulkarni, 2006).

Figure 4 shows the plot of transmittance against wavelength for PbS thin film. The transmittance increased from 20% to 34% in the UV region and further increased from 37% to 64% in the visible region, with a peak value of 74% in the nir region for slide Pb_2 and a range of 52% to 66% at a wavelength range of (400-680) nm for slide Pb_1. This moderately high absorbance makes PbS thin film useful as buffer layers for CGS solar cells.

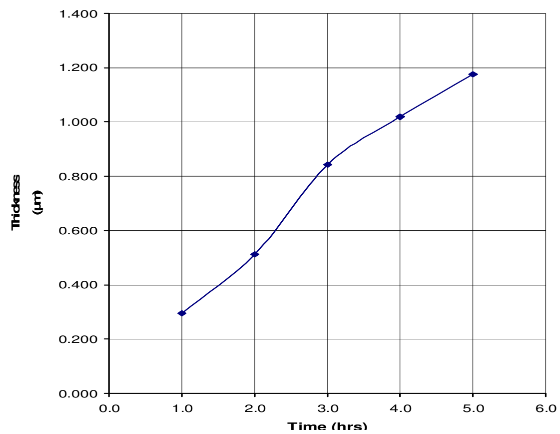

Figure 1. Plot of thickness versus time for PbS thin film

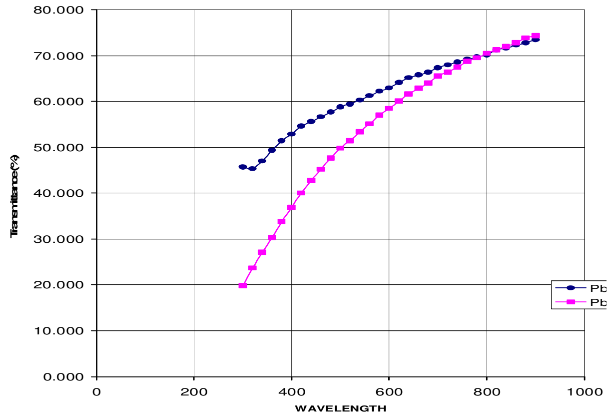

Figure 4. Plot of Transmittance versus wavelength for PbS thin film (slide Pb_1 and Pb_2)

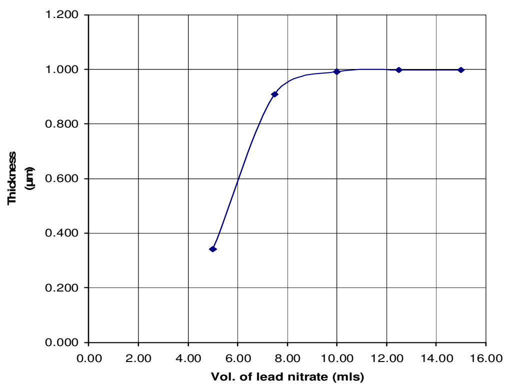

Figure 2. Plot of thickness versus lead ion concentration for PbS thin film

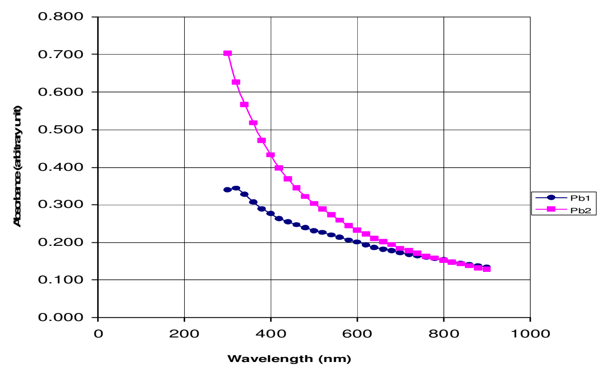

Figure 5. Plot of Absorbance versus wavelength for PbS thin film (slide Pb_1 and Pb_2)

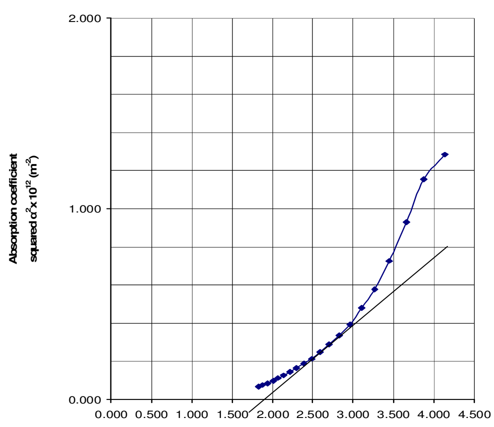

Figure 3. Plot of Absorption coefficient squared against Photon energy for PbS thin film

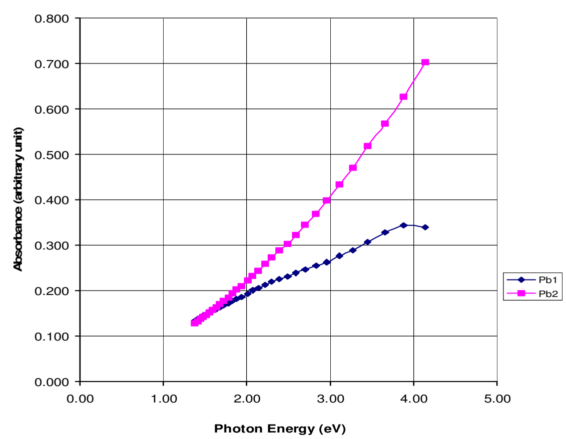

Figure 6. Plot of Absorbance versus Photon energy for PbS thin film (slide Pb_1 and Pb_2)

Figure 5 is a plot of absorbance against wavelength for PbS thin film. The optical absorption of PbS thin films was studied in the wavelength range of 300nm to 900nm. The results show higher absorption on the shorter wavelength side. This is consistent with the results of (Ubale and Kulkarni, 2006). The absorbance tends to be very high in the UV region with a peak value of 0.703 in the UV region, a range of (0.433 – 0.194) at a wavelength range of (400 – 680)nm and 0.129 in the nir region (for slide Pb_2), 0.340 in the UV region and 0.134 in the nir region (for slide Pb_1). Figure 6 is a plot of absorbance against photon energy hυ for PbS thin film. The absor-

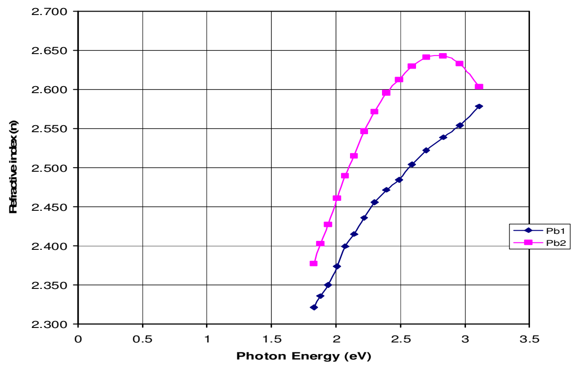

Figure 7. Plot of Refractive index versus Photon energy for PbS thin film (slide Pb_1 and Pb_2) vis-range

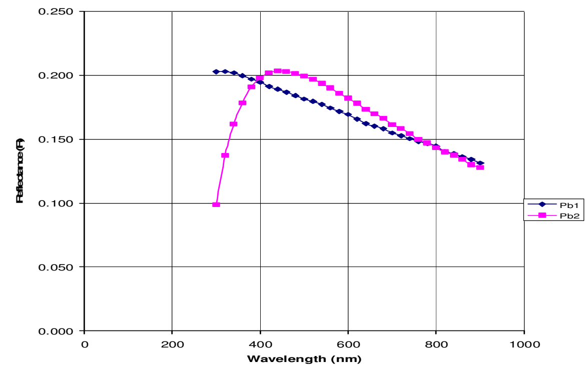

Figure 10. Plot of Reflectance versus wavelegth for PbS thin film (slide Pb_1 and Pb_2)

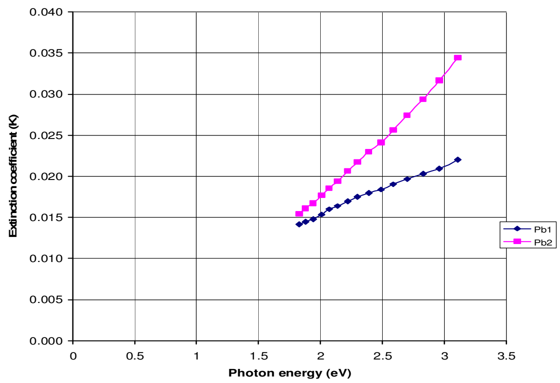

Figure 8. Plot of Extinction coefficient versus Photon energy for PbS thin film (slide Pb_1 and Pb_2) vis-range

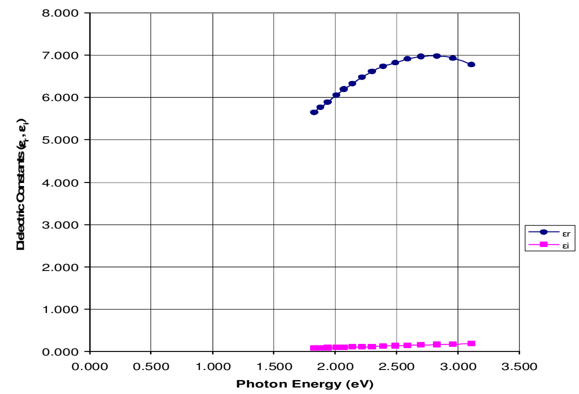

Figure 11. Plot of Dielectric constant versus Photon energy for PbS thin film (slide Pb_1 and Pb_2)

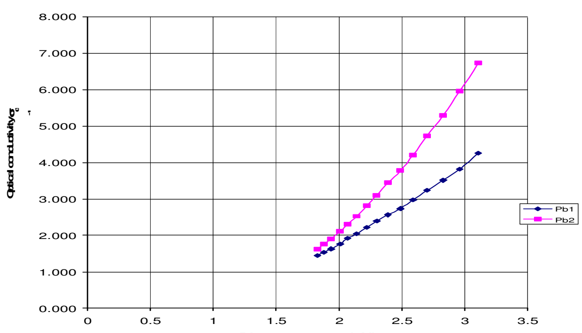

Figure 9. Plot of Optical conductivity versus Photon energy for PbS thin film (slide Pb_1 and Pb_2) vis-range

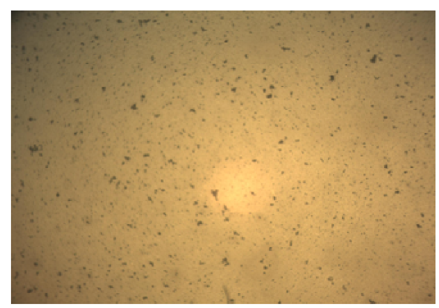

Figure 12. Micrographs of PbS thin film for slide Pb_2

bance decay as photon energy decreases. Figures 7, 8 and 9 show the refractive index (n), extinction coefficient (k) and optical conductivity (σ_o) as a function of photon energy for PbS thin film. The refractive index has a peak value of 2.64 at 2.70eV and a minimum value of 2.38 at 1.88eV (for slide Pb_2). For slide Pb_1 a value of 2.58 at 3.11eV and 2.32 at 1.83eV was obtained. This is in close agreement with the value of 2.62 reported by (Ndukwe, 1998). This high refractive index value makes PbS thin film a good material for photovoltaic application. A maximum extinction coefficient (k) value of 3.445 x 10^{-2} is obtained at photon energy of 3.11eV (for slide Pb_2) and 2.2 x 10^{-2} for slide Pb_1. For optical conductivity, a peak value of 6.73 x 10^{13} S^{-1} was obtained at photon energy of 3.11eV (for slide Pb_2) and 4.26 x 10^{13} S^{-1} (for slide Pb_1).Figure 10 shows the variation of reflectance with wavelength. All the samples reveal very low reflectance through out the spectrum. This will have a useful application in antireflection coating.

Figure 11 is a plot of dielectric constant (real and imaginary) for PbS thin film (using slide Pb_2). It has a peak real dielectric constant of 6.986 at 2.83eV and a minimum value of 5.650 at 1.83eV.

Figure 12 show the optical micrograph of PbS thin film. From the micrograph, it can be seen that the film exhibit the growth of small grains distributed across the surface of the substrate. It also shows uniform surface coverage.

CONCLUSION

Chemical bath deposition method has been successfully implemented to deposit PbS thin film. The optical absorption study reveals that PbS thin film have a bandgap energy of 1.90eV and a refractive index range of 2.32eV-2.58eV. our results also revealed that the thickness of the films were dependent on the time of growth and concentration of ion.

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