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RESEARCH ARTICLE

DEPOSITION AND CHARACTERISATION OF LEAD SELENIDE (PbSe) THIN FILMS

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ABSTRACT

Thin films of lead selenide (PbSe) were grown using the Chemical Bath Deposition (CBD) Technique. Optical and Electrical characterisation were carried out on the films with the view to determining their absorbance, reflectance and transmittance spectra which were used to calculate the optical constants: absorption coefficient and refractive index, while the conductivity (σ) of the film was determined by measuring the dark d.c. electrical resistivity using the dc two point probe method in the temperature range; 300k – 500k. The optical characterisations reveals that, the high absorbance and the low reflectance of the film indicates that the film is a good material for fabrication of solar cell and anti reflection coatings, while the electrical characteristics of the film showed that the films are semiconductors in nature and they exhibit n-type mechanism conduction.

Key words: Lead Selenide, CBD, Absorbance Transmittance, Reflectance and Conductivity.

INTRODUCTION

The IV-VI thin film semiconductor materials have attracted much interest in the field of material science due to their variety of applications in various electronic and opto-electronic devices.Several techniques, both physical and chemical, have been widely used to prepare thin film semiconductor materials. These include: chemical bath deposition (CBD) techniques (Barote *et al.*, 2012); electrodeposition (Molin and Dikusar, 1995); Pulsed laser deposition technique (Rumianowski *et al.*, 2003); chemical vapour deposition technique (Kashiwaba *et al.*, 2000); magnetron sputtering (Paragauy *et al.*, 1999); spray pryolysis sol-gel spin coating technique (Caglar *et al.*, 2006, Suping Huang *et al.*, 2009; Mohammedi *et al.*, 2010; Ganesh *et al.*, 2012). Among these, the chemical bath deposition (CBD) technique is the simplest, cost effective, has low deposition temperature and no sophisticated instrument is required.

Lead selenide (PbSe) thin films are important IV-VI semiconductor materials with cubic structures and have proved to be promising because of their unusual characteristics such as high carrier mobilities, high dielectric constants and narrow band gaps which make hem unique and have many important applications in the field of solid state basic research. PbSe thin films have been extensively investigated for IR detectors, photographic plates, selective and photovoltaic absorbers and lasers (Chaudhary *et al.*, 1981; Munoz *et al.*, 1998; Sustro, 2005; Dmitri and Murray, 2005). Also, they have been applied in long wave length imaging (Zogg *et al.*, 1994). The present work reports on the optical and electrical characterisation of PbSe films deposited using the chemical bath deposition (CBD) technique.

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MATERIALS AND METHOD

The chemical bath deposition (CBD) technique has been used in the deposition of PbSe thin films. The reagents used for the study are $Pb\ (NO_3)_2$ solution, $SeSO_4$ solution, NH_3 solution and EDTA with all chemicals being of analytic grade. The experimental solution of the desired concentration of the various reagents was prepared as follows (as demonstrated by Ezenwa, 2012): 0.5M of $Pb\ (NO_3)_2$ solution was measured into 5 different 50M beakers, then 0.5m of $SeSO_4$ was introduced to each of the 50Mℓ beakers, followed by different volumes of 1M EDTA, this was done with continuous stirring in other to optimise the deposition parameters for good quality thin films. 6mℓ of 5M NH_3 solution was introduced gradually to the beakers with stirring in other to adjust the pH. Distilled water was added to make up 50Mℓ of solution in each bath. The chemical bath composition gave a pH of 11.5$\pm$0.1. Glass slides were used as substrates; they were cleaned together with other equipments using the following procedure:

i. Scrubbing with liquid detergent.
ii. Rinsing several times with distilled water.
iii. Scrubbing again with detergent.
iv. Rinsing again with distilled water.
v. Scrubbing with trichloroethylene.
vi. Rinsing with distilled water.
vii. Scrubbing with acetone.
viii. Rinsing with distilled water.
ix. Scrubbing with alcohol.
x. Rinsing with distilled water.
xi. Drying using oven.

After cleaning, the substrates (glass slides) were inserted into reaction baths and suspended vertically from synthetic forms, which covered the beakers containing the baths. The deposition process took the duration between 60mins to 300mins at normal room temperature after which the substrates were withdrawn from the baths rinsed with distilled water and allowed to dry in air. The optical absorption spectra of the deposited films were recorded at room temperature using UV-VIS-IR spectrophotometer (Perkin Elmer) and the absorbance in arbitrary units was measured. Other optical parameters such as transmittance, reflectance, refractive index and extinction coefficient were calculated using the relationship given in section (3). The dark dc electrical resistivity of the PbSe film were measured using the dc two point probe method in the range 300k – 500k. The film thickness was measured by a weight difference method given by (Barote *et al.*, 2011): t = m/PA where t is the thickness of the film, m is the mass of the film deposited on the substrate, A is the area of the film deposited and P is the density of the bulk material.

Theoretical Considerations and Calculations

Optical

Transmittance (T) can be calculated from the relationship (Ezekoye, 2001 and Ajayi *et al*, 2002) as

$$A = \log \frac{1}{T} \qquad (1)$$

where A is absorbance and T is transmittance and is given by

$$T = \frac{1}{10^A} \qquad (2)$$

The reflectance is calculated from the relation

$$A + R + T = 1 \qquad (3)$$

or $$R = 1 - (A + T) \qquad (4)$$

The absorption coefficient can be calculated from the equation (Ballard *et al*, 1972, and Ezekoye *et al*, 2001).

$$I = I_o e^{-\alpha x} \qquad (5)$$

Where α is the absorption coefficient, I is the transmitted light, I_o is the incident light and x is the film thickness. Rearranging equation (5) above we have:

$$\frac{I}{I_O} = e^{-\alpha x}$$

This gives $$\frac{1}{x}\log\frac{I_o}{I} = \alpha \qquad (6)$$

However, $\frac{I_O}{I}$ is the transmission factor.

Also, the absorption coefficient (α) can be calculated (Islam and Podder, 2009) from the observed absorbance data using Beer Lambert's formula:

i.e. $$\alpha = 2.303\left(\frac{A}{d}\right) \qquad (7)$$

where A is the optical absorbance and d is the film thickness.

There photon energy E, is given (Ezekoye, 2001), by:

$$E = h\nu \qquad (8)$$

Where h is the plank constant ν is the frequency of the photon.

Similarly, $$E = \frac{hc}{\lambda} \qquad (9)$$

where c is the speed of light and λ is the wavelength.

Substituting for constants in equation (9) gives

$$E = \frac{12{,}400}{\lambda} eV \qquad (10)$$

For semiconductors (where $K^2 << n^2$) there exist a relationship between R and n (Ndukwe, 2004) given by

$$R = \frac{(n+1)^2}{(n-1)^2} \qquad (11)$$

where R is the reflectance and n is the refractive index.
There exist relationship between R and α (Ndukwe, 2004) as

$$K = \frac{\alpha\lambda}{4\pi} \qquad (12)$$

Where K is the extinction coefficient.

The relationship existing between n, R and K is given (Islam and Podder, 2009) by

$$n = \left(\frac{1+R}{1-R}\right) + \sqrt{\left(\frac{4R}{(1-R)^2} - K^2\right)} \qquad (13)$$

where n is the refractive index, K is the extinction coefficient and R is the optical reflectance

Electrical

The resistivity follows from the relation:

$$\rho = \rho_O \ell xp\left(-E_a/KT\right) \qquad (14)$$

where ρ is the resistivity at T temperature, ρ_o is constant, K is the Boltzman constant, E_a is the activation energy for conduction.

Similarly, the d.c. electrical conductivity of a semi-conductor at temperature T and is given by

$$\sigma_{dc} = \frac{-E_a}{KT} + \text{In}\sigma_O$$

i.e. $$In\sigma_{dc} = -\left(\frac{E_a}{1000K}\right)\left(\frac{1000}{T}\right) + In\sigma_o$$

RESULTS AND DISCUSSIONS

The PbSe thin films have been grown onto glass substrates using the chemical bath deposition (CBD) technique. EDTA was used as a complexing agent to control the precipitation and NH_3 solution was used for better adherence. The basic principle of the CBD consists of the controlled generation of the metal and chalcogenide ions in an alkaline medium and their precipitation on the substrate in order to form a film (Ezenwa, 2012). In this case EDTA was used as a complexing agent to control the precipitation and NH_3 solution was used for better adherence. The lead ion (Pb^{2+}) is generated by the decomposition of $[Pb(EDTA)^{2+}]$ complex. The Se^{2-} ions are generated in alkaline medium by decomposition of selenium precursor which is $SeSO_4$. The equations governing the reaction and deposition of PbSe films are given (Ezenwa, 2012);

$Pb(NO_3)_2 + TEA \rightarrow [Pb(TEA)]^{2+} + 2NO_3$
$[Pb(TEA)]^{2+} \rightarrow Pb^{2+} + TEA$
$S_e\,SO_4 + OH^- \rightarrow HSe^{2-} + SO_4^{2-}$
$Pb^{2+} + Se^{2-} \rightarrow PbSe$

Figure 1 shows the plot of absorbance of PbSe thin film versus the wavelength. The absorbance decreased with increase in wavelength, which relatively exhibits low values within the infrared region of the spectrum. The absorbance was high within the wavelength range of 200nm – 400nm which makes the film an important material in the fabrication of solar cell.

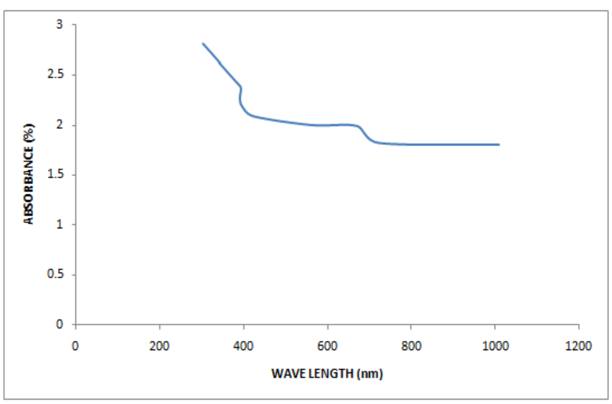

Figure 1. Absorbance of pbse thin film versus wavelength

Figure 2 shows the reflectance of PbSe versus the wavelength. The film shows very low reflectance throughout UV/VIS/NIR regions. This low reflectance makes the film good material for anti-reflection coating. Figure 3 shows the transmittance of PbSe versus the wavelength. The transmittance increases as the wavelength increases. The film shows about 80% transmittance throughout UV/VIS/NIR regions. This high transmittance in the VIS region makes the film a good material in window glazing and solar energy collection. Figure 4 shows the absorption coefficient of PbSe thin film versus the energy. When the energy gap of this film was obtained by extrapolating the linear part of the curve the energy axis, it was observed that the PbSe film exhibits direct band transition and the band gap of 1.26eV is obtained. This value is in close agreement with the results: 1.18eV (Ezenwa, 2012); 1.30eV (Okereke and Ekpunobi, 2010); 1.5eV – 2.2eV (Ishiwu and Nnabuchi, 2010).

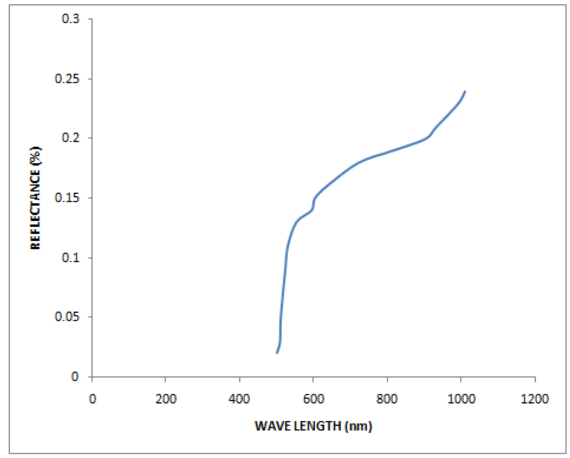

Figure 2. Reflectance of pbse thin film versus wavelength

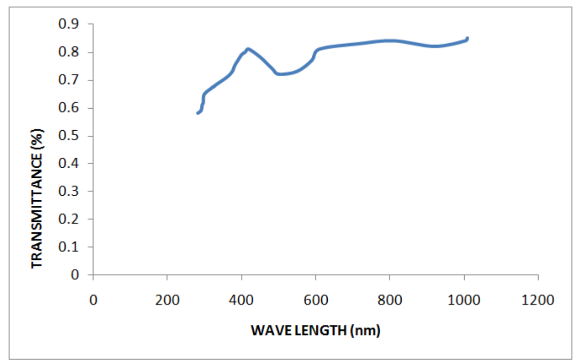

Figure 3. Transmittance of pbse versus wavelenght

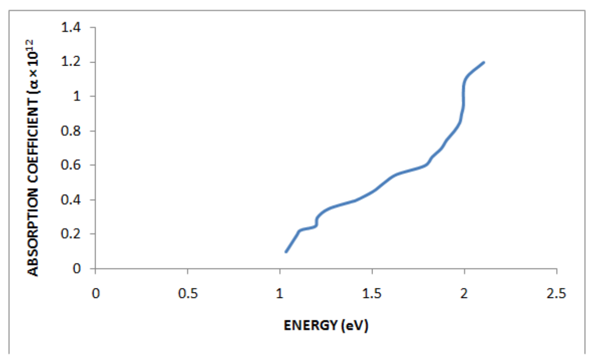

Figure 4. Absorption coefficient of pbse versus energy

Figure 5 shows the refractive index of PbSe versus wavelength. The refractive index of the film increases with the wavelength revealing high index of 2.45 at 950nm wavelength. This high refractive index makes the film a good material for anti-reflection coatings. Figure 6 depicts the variation of log of conductivity with the inverse of temperature of PbSe thin film. Temperature range of 300 – 500K was taken in measuring the dark d.c. electrical resistivity of the film. The electrical resistance was found to be of the order of 10^{-3}vc-cm which is in agreement with the work of Barote *et al.*, 2011. The variation shows two different zones of temperature. The first zone is identified with low temperature (i.e. from 300K – 375K), and is associated with the extrinsic conductivity of atoms (Barote *et al.*, 2011). The second zone is identified with high temperature (i.e. from 375K – 500K), and is associated with the transition to intrinsic conduction in semiconductor (Barote *et al.*, 2011).

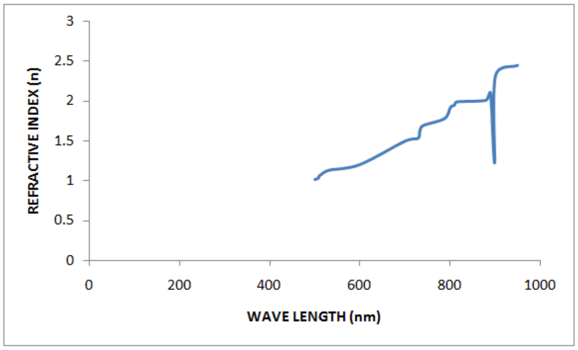

Figure 5. Refractive index of pbse versus wavlenght

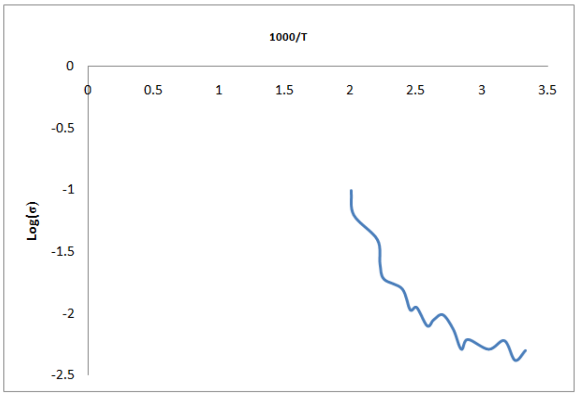

Figure 6: Variation of Log (conductivity) with inverse of temperature of PbSe thin film

Conclusion

This study has demonstrated the case of depositing good quality of PbSe thin films from chemical bath deposition (CBD) technique. The optical and electrical properties of the films were investigated. The optical characteristics of the films reveals that the PbSe films are good materials for the fabrication of solar cells, solar collectors, and anti-reflection coatings, while the electrical characteristics of the film showed that the films are semiconductors in nature and they exhibit n-type mechanism conduction.

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