

## **Optical Properties of Chemical Bath Deposited Lead Selenide Thin Films**

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### **ABSTRACT**

*Lead selenide (PbSe) thin films were deposited on glass substrates using chemical bath deposition technique. The films growth was based on the decomposition of lead nitrate and selenium sulphate in the presence of ammonia and EDTA disodium salt acting as pH stabilizers and complexing agents respectively. Optical and morphological investigations were also performed. The deposited film (PbSe<sub>2</sub>) has the highest absorbance at wavelength range of 300nm – 500nm. The optical absorbance generally decreases with increase in wavelength. The optical band gap of the deposited film was found to be 1.18eV.*

**Keywords:** Lead selenide, chemical bath deposition and optical properties.

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### **INTRODUCTION**

A material is said to be a thin film when it is built up as a thin layer on a substrate by controlled condensation of the individual atomic, molecular or ionic species either directly by a physical process or through a chemical or electrochemical reaction. Otherwise, it is a thick film. [1]. Various processing routes, both physical and chemical deposition techniques have been utilized to prepare lead selenide thin films, including electrodeposition [2], chemical bath deposition [3], electrochemical atomic layer epitaxy[4], photochemical[5], molecular beam epitaxy[6] and pulsed laser deposition method[7]. Among these, the chemical bath deposition method was chosen as an ideal preparation method for preparing lead selenide thin films basically due to its low deposition temperature, simple, no requirement of sophisticated instruments and minimum material. Up-to-date, chemical bath deposition method has been successfully used to deposit many different thin films including FeS<sub>2</sub> [8], PbS[9] , Cu<sub>2</sub>S[10] , SnS[11], Cu<sub>4</sub>SnS<sub>4</sub>[12] and CuInS<sub>2</sub>[13].

PbSe is an important semiconducting material and has been extensively investigated for infrared detectors, photographic plates, photodetectors, photoresistors and photoemitters in the infrared (IR).It also has application in solar cell technology [3,6,11,13]. Lead selenide film is used as a target material in infrared sensor, grating, lenses and various optoelectronic devices.

The present work reports the optical characterization of PbSe thin films prepared using the chemical bath deposition method. The chemical bath contains lead nitrate and selenium sulphate which provide Pb<sup>2+</sup> and Se<sup>2-</sup> ions, respectively, while EDTA acted as a complexing agent. We report the influence of EDTA on the thickness of the fabricated PbSe thin films at 300K bath temperature.

### MATERIALS AND METHODS

PbSe thin films are prepared on glass substrate using the chemical bath deposition (CBD) method. The basic principle of the CBD technique 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. In the present case, the lead ion ( $\text{Pb}^{2+}$ ) is generated by the decomposition of  $[\text{Pb}(\text{EDTA})^{2+}]$  complex. The  $\text{Se}^{2-}$  ions are generated in alkaline medium by decomposition of the selenium precursor which is  $\text{SeSO}_4$ . The pH of the solution was adjusted by the use of 30% ammonia ( $\text{NH}_3$ ). The deposition bath was prepared as follows: 0.5 M of  $\text{Pb}(\text{NO}_3)_2$  solution was measured into 5 different 50ml beaker and to this was added 5ml of 0.5M of  $\text{SeSO}_4$ , various volumes of 1M EDTA disodium salt was then added (as indicated in table 1.1) in order to optimize the deposition parameters to obtain good quality thin films. The mixture was made up to the required volume with addition of various volumes of water.  $\text{NH}_3$  was used for pH adjustment. The resulting solution was stirred for a few seconds with a glass rod stirrer. A glass slide was inserted in the reaction bath and held vertically in a synthetic foam cover. The deposition process lasted 24 hours at a deposition temperature of 300K. After 24 hours, the slides were taken out, rinsed with distilled water and allowed to dry in air.

The Optical absorbance spectra of the deposited films were obtained by means of UV/VIS Janway 6405 spectrophotometer. Surface morphology of the thin films deposited on glass substrate were examined by a BHZ – UMA Olumpus optical microscope. From the spectrophotometer, the absorbance in arbitrary units was measured. Parameters such as transmittance, reflectance, refractive index and extinction coefficient were then calculated using the relationship explained below:

For weakly absorbing thin film on a non absorbing substrate, the transmittance (T) can be expressed as [14]

$$T = (1-R^2) \exp(-\alpha t)$$

$$t = 1/\alpha \{ \ln(1-R^2) / T \}$$

where R is the reflectance,  $\alpha$  is absorption coefficient, t is the thickness of the film. For semiconductors and insulators, where the extinction coefficient (k) and refractive index (n) are related as  $k^2 \ll n^2$ , the relationship between R and n is given by [15] as

$$R = (n-1)^2 / (n+1)^2$$

Also k and  $\alpha$  are related by

$$k = \alpha \lambda / 4\pi$$

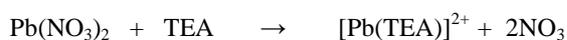
Where  $\lambda$ , is the wavelength of electromagnetic radiation.

In high absorption region under photon energy, the relation between absorption coefficient ( $\alpha$ ) and photon energy (hf) is given by [16]:

$$\alpha = (hf-E_g)^n$$

Where f is the frequency, h is the Planck's constant,  $E_g$  is the energy band gap and n is a number which characterizes the optical processes;  $n = 1/2$  is for direct allowed transition,  $n = 2$  is for indirect allowed transition and  $n = 3/2$  is for forbidden direct allowed transition. When the straight portion of the plot of  $\alpha^2$  against hf is extrapolated to  $\alpha^2 = 0$ , the intercept gives the value of the transition band energy ( $E_g$ ).

The equations governing the reaction and deposition of PbSe films are as follow:





The presence of  $\text{Pb}^{2+}$  and  $\text{Se}^{2-}$  ions lead to formation of PbSe.

TABLE: 1.1

SlideNo.	Dip time(hr)	0.5M SeSO <sub>4</sub> (ml)	EDTA	Ammonia solution(ml)
1. PbSe(1)	24	5.00	1.00	5.00
2. PbSe(2)	24	5.00	2.00	5.00
3. PbSe(3)	24	5.00	3.00	5.00
4. PbSe(4)	24	5.00	4.00	5.00
5. PbSe(5)	24	5.00	5.00	5.00

RESULTS AND DISCUSSION

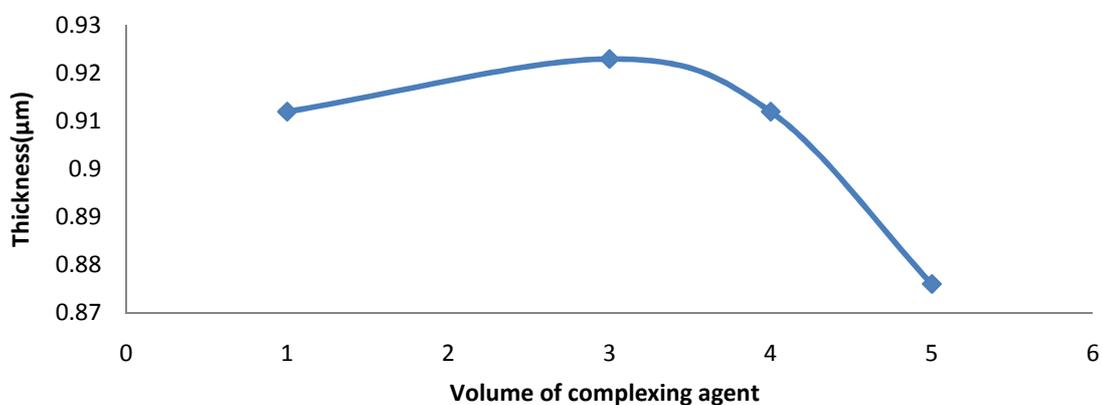


Fig. 1: Plot of thickness versus vol. of complexing agent for lead sulphide thin film

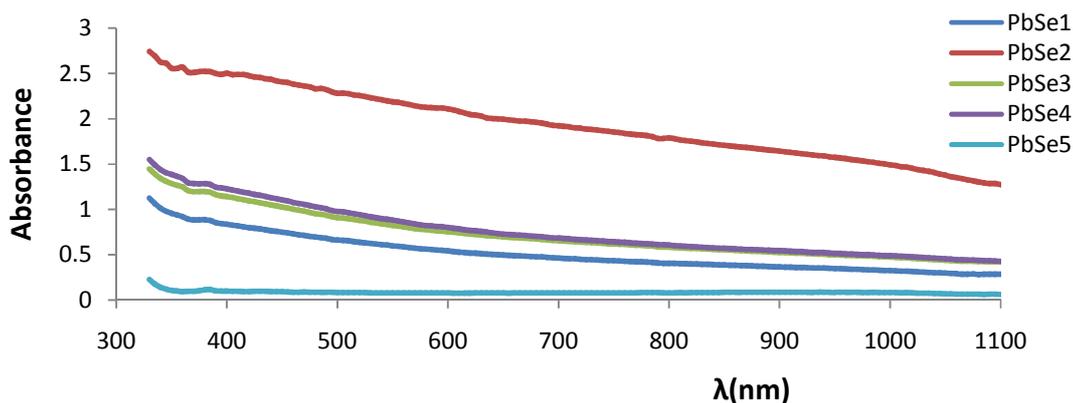


Fig. 2: Plot of absorbance versus wavelength for lead sulphide thin film

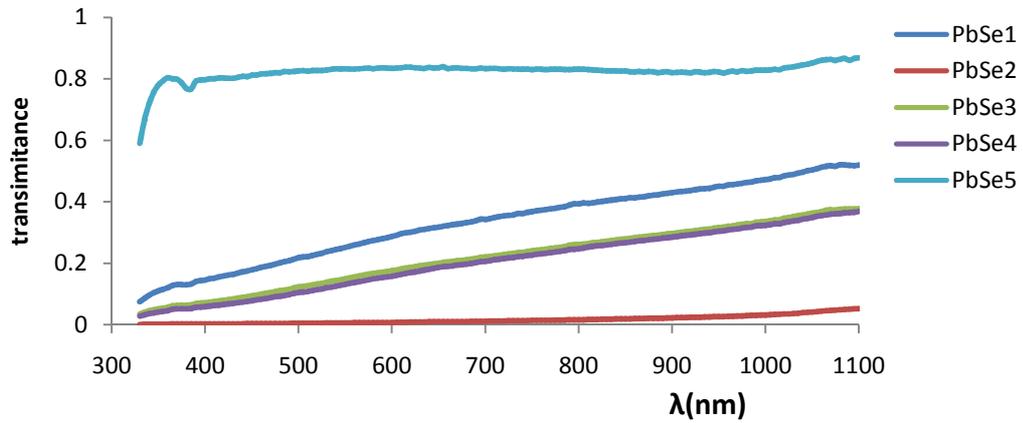


Fig. 3: Plot of transmittance versus wavelength for lead sulphide thin film

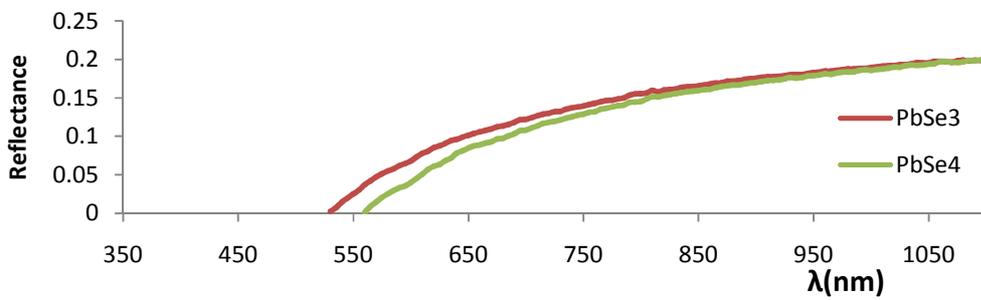


Fig. 4: Plot of reflectance versus wavelength for lead sulphide thin film

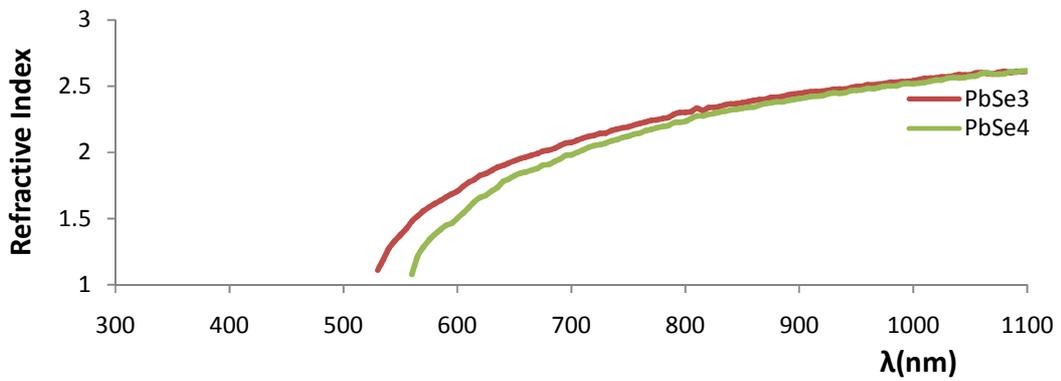


Fig. 5: Plot of refractive index versus wavelength for lead sulphide thin film

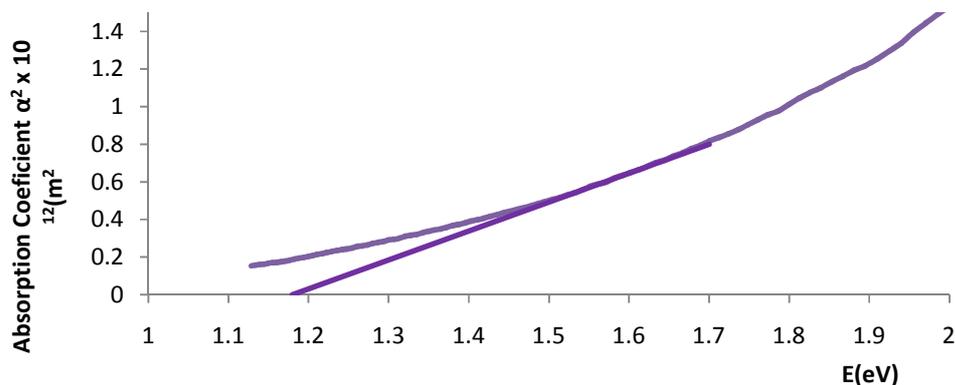


Fig. 6: Plot of absorption coefficient squared versus photon energy for lead sulphide thin film



Fig. 7: optical micrograph of lead sulphide thin film

Figure 1 shows the plot of thickness versus volume of complexing agent. Fig.1 indicates that thickness increased from about 0.912 $\mu\text{m}$  to about 0.923 $\mu\text{m}$  with 1mls to 3mls volume of complexing agent. At 4mls and above volume of complexing agent, the thickness of the film started to decrease. This indicates that as complexing agent increases, the thickness decreases.

Figure 2 and 3 shows the plots of absorbance and transmittance versus wavelength of PbSe thin films deposited in this work. The absorbance generally decreased with increase in wavelength and has relatively low values in the infrared region of the spectrum. A strong absorption was observed at wavelength range of 300nm – 500nm, hence the film has potential application in fabrication of solar cell.

The transmittance spectrum displayed in figure 3 show increase in transmittance as the wavelength increases, sample PbSe1 has about 80% transmittance through out the UV/ VIS/NIR regions. The very high transmittance in the visible region makes lead selenide films useful aesthetic window glaze materials. Also, the high transmittance of the film makes it suitable for solar energy collection because if coated on the surface of the collector, it will reduce reflection of solar radiation and transmits radiation to the collector fluid.

Fig. 4 shows a plot of reflectance index (R) versus wavelength of films PbSe3 and PbSe4. Generally all the films show a very low reflectance through out the UV/VIS/NIR region. This low reflectance value makes lead sulphide thin film an important material for anti-reflection coating.

Fig. 5 shows a plot of refractive index (n) versus photon energy (hv) of films PbSe3 and PbSe4. The refractive index (n) of the films increases with the photon energy revealing a refractive index of 2.5 at 900nm for the two samples. This result revealed that PbSe has high refractive index. The high refractive index possessed by PbSe films made it suitable for use as anti-reflection coatings.

Fig. 6 shows the optical micrograph of PbSe film. The surface morphology of PbSe thin film shows high density of grains. The high density of these grains implies that the nucleation has occurred on all sites. It shows uniformity in the distribution of the grains, the grains are small and regular.

Fig. 7 shows a plot of  $\alpha^2$  versus photon energy ( $h\nu$ ) of PbSe thin films. The energy gaps for these films are obtained by extrapolating the linear part of the curve to the energy axis. It is observed from the figure that PbSe thin film exhibits direct band transition and band gap of 1.18 eV is obtained. This is in close agreement with the finding [17], who reported a band gap of 1.30eV and [18], who reported a band gap range of 1.5eV–2.2 eV.

### CONCLUSION

The PbSe thin films have been prepared by the chemical bath deposition technique under optimized deposition conditions. Deposition was carried out from aqueous solutions using lead nitrate and selenium sulphate as  $Pb^{2+}$  and  $Se^{2-}$  ion source, respectively. The optical micrograph of PbSe film shows that the surfaces of the lead selenide films are dense. It shows uniformity in the distribution of the grains. It consisted of regular shaped grains. The PbSe thin films exhibited direct band gap transition with band gap energy of 1.18 eV.

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