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Effect of Deposition Period and pH on Chemical Bath Deposited PbSe Thin Films

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ABSTRACT

The PbSe thin films prepared through chemical bath deposition technique. The effect of deposition period and pH was studied to determine the optimum condition for deposition process. The structure and morphology of thin films investigated by X-ray diffraction and atomic force microscopy. The optical properties measured to determine transition type and band gap value. By the X-ray diffraction results it has been confirmed that polycrystalline nature with cubic in structure. X-ray diffraction data showed that the most intense peak at 2θ = 25.35° which belongs to (111) plane. As the deposition period increased up to 80 min, the film gradually grew thicker as shown by the AFM images. It is observed that the best crystalline of film is obtained at pH 1.5. Also, AFM images revealed that the grains distributed over the substrate surface. The band gap value found to be 2.6eV with direct transition.

Keywords: Metal chalcogenides, Optical properties, Semiconductor, X-ray diffraction

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INTRODUCTION

The thin films technology applications in the optoelectronic devices, solar cells, sensors, and laser materials. In the past few decades, several techniques such as chemical bath deposition, vacuum evaporation, electro deposition, molecular beamepitaxy, close spaced sublimation, thermal evaporation, spray pyrolysis, sputter deposition, metal organic chemical vapor deposition, and plasma-enhanced chemical vapor deposition is used in the deposition of thin films. The preparation of thin films by chemical bath deposition technique is currently attracting a great deal of attention as the technique is relatively simple and cost effective, has minimum material wastage, and can be applied in large area deposition at low temperature. The chemical bath deposition method uses a controlled chemical reaction to deposit a thin film. In the typical experimental approach, the substrates are immersed in solution containing the chalcogenide source, metal ion, and complexing agent. The preparation and characterization of thin films by chemical bath deposition is reported by many researchers. In this paper, we focus on the deposition of novel PbSe thin films using triethanolamine [(HOC2H4)3N] as a complexing agent by chemical bath deposition method. In order to get good quality of thin films, the preparation parameters such as deposition period and pH optimized. The thin films have been characterized by X-ray diffraction for structure determination, atomic force microscope analysis for surface morphology study, and UV-Visible Spectrophotometer for optical properties studies

MATERIALS AND METHODS

Deposition of Films

Lead Selenide thin films deposited on microscope glass slides using chemical bath deposition method. Prior to deposition, the substrate was degreased in ethanol for 10 min, followed by ultrasonically cleaned with distilled water for another 20 min, and finally dried in air. During deposition process, an aqueous solution of lead nitrate [Pb(NO3)2] was used as lead source; sodium selenate [Na2O4Se] was supplied as selenate source and triethanolamine [(HOC2H4)3N] acted as complexing agent. All these chemicals used for the deposition were analytical grade. All the solutions prepared in deionised water (Alpha- Q Millipore). For deposition, 30 ml of0.10 M lead nitrate was complexed with 10 ml of triethanolamine agent. To this, 30 mL of 0.10 M sodium selenate was added slowly to the reaction mixture by addition of hydrochloric acid (0.7M) with constant stirring. The ultrasonically cleaned glass substrate was immersed vertically into chemical bath. The film growth was carried out at 50°C. The films deposited in various deposition periods (40-80 min) and pH (pH 0.5-1.5). The deposited films tested for adhesion by subjecting it to a steady stream of distilled water. rinsed with de-ionized water. The films found to be homogenous, brownish and well adhered to the substrate. The films were annealed at 350ºC for 4 hours. After the annealing, films were kept in vacuum desiccators for further characterization.

Characterization of Films

X-Ray Diffractometer Bruker D8 focus with CuKα radiation (1.5406A°) was used for crystallographic analysis of the film in the 2θ range from 20º to 60°. Surface morphology studies were carried out using atomic force microscope (PARK SYSTEM XE 70). The optical properties of the PbSe thin films were studied using UV-VIS spectrophotometer (JASCO V630) at temperature in the wavelength range from 350 nm to 800 nm.

RESULTS AND DISCUSSION

Structural Analysis

Figure 1 shows the XRD patterns of thin films chemically deposited for a different deposition periods at different pH ranging form 0.5 to 1.5. The chemical bath deposited thin films are found to three dominant peaks corresponding to (111), (200) and (220) planes were observed. All these peaks corresponding to cubic phase of PbSe and matched with the standard JCPDS (Reference code: 00-065-1040) data The (111) peak is the preferred orientation. The films prepared at pH 0.5 produced two peaks at 2θ = 25.35° and 29.5° corresponding to *d*-spacing values of 3.53 and 3.07 Å, respectively. and are in good agreement with standard *d*-spacing values. As the pH value was increased to 1.5, the intensity of the peak corresponding to (111) plane increased. The structural parameters obtained are shown in Table 1.The average crystallite size was determined from peaks at 2θ=25.35º using the Debye Scherer formula.

$$D = \frac{0.94\lambda}{\beta cos\theta}$$

Where β is FWHM in radians and λ is a wavelength of X-rays. With increase in deposition time, the crystallite size increased from 13.9 nm. to 15.2 nm. The crystallite size and FWHM are shown in Table 2.

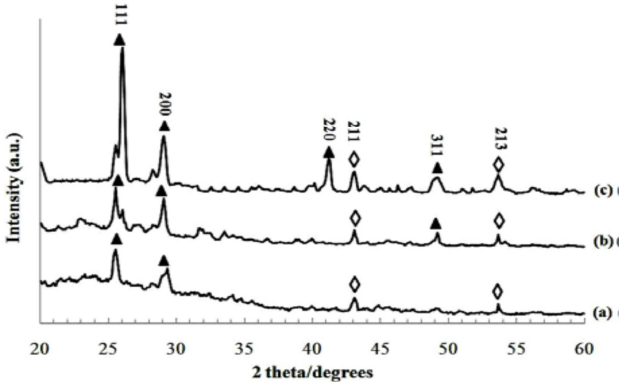

Figure 1: X-ray diffraction patterns of PbSe thin films chemically deposited for a different deposition periods at (a)pH 0.5 at 40 min (b) pH 1.0 at 60 min (c) pH 1.5 at 80 min

Table 1: Structural Parameters determined from XRD patterns

dst (Å) JCPDS	dobs (Å) Sample a	dobs (Å) Sample b	dobs (Å) Sample c	(hkl)
3.55	3.53	3.54	3.47	111
3.06	3.07	3.22	3.227	200
2.14	2.18	2.16	3.15	220

Table 2: FWHM, average crystallite size determined from XRD pattern and Optical band gap obtained From UV-VIS data

Film	FWHM (radians)	Average crystallite size(nm)	Band gap(E_g) (eV)
a	0.0308	13.9	2.06
b	0.0258	14.8	2.15
c	0.0242	15.2	1.76

Atomic Force Microscope Studies

Figure 2 shows three dimensional AFM images for an area of 20 µm X 20 µm for PbSe thin films. These thin films chemically deposited in different deposition periods at a different pH ranging from 0.5 to 1.5. The films prepared at 40 and 60 min showed incomplete coverage of material over the surface of substrates (Fig. a, b). As the deposition time was increased to 60 min, more surface coverage was noticed. When the deposition time was further extended to 80 min, there more materials deposited onto the substrate and thicker films .Table 3 shows the film thickness and root mean square roughness (RMS) observed by AFM. The thickness of the film increases as the deposition period is increased from 40 min (960 nm) to 80 min (2422 nm). This result concludes that an increase in deposition period allows more materials to be deposited onto substrates and thicker films to be formed. The RMS roughness is increased from 69 nm to 194 nm as the deposition period is increased from 40 min to 60 min. However, the decreases in RMS value from 160 nm can be observed for the films deposited at a longer period (80-40 min). It can be noted that the surface roughness decreases with decreasing films thickness.

Table 3. The film thickness and root mean square roughness values for PbSe thin films chemically deposited in different deposition periods at different range pH 0.5 to 1.5

Period (min)	Thickness (nm)	Thickness (nm) Root mean squarer roughness(nm)
40	960	69
60	1565	194
80	2422	160

Optical Studies :-

The fundamental absorption which corresponds to electron excitation from the valence band to conduction band can be used to determine the nature and value of optical band gap. The relation between the absorbance (A), band gap energy (Eg) and the photon energy (hν) can be written as shown below (Equation 1):

$$\alpha = \frac{A\left(h\nu - E_g\right)^n}{h\nu}$$

where ν is the frequency, h is the Planck's constant, k equals a constant, and n is a constant and carries the value of either 1 or 4. The n = 1 for the direct transition while n = 4 for indirect transition, respectively. The absorption spectra of the samples are recorded in the wavelength range 350-800 nm using a UV-Vis spectrophotometer. For the direct band gap semiconductor, the $(Ah\nu)^{2/n}$ versus hν graph is predicted to be a straight line with a photon energy axis intercept giving the value of band gap. The plots of $(Ah\nu)^2$ against the photon energy (hν) for the lead selenide thin films is presented and dependence of the optical gap on the deposition time for the as-deposited thin films is shown in Figure 3. It is obvious that the optical gap decreases with increasing deposition time and pH.The optical band gap increase form 1.7eV to 2.6eV as the partical size decreased form 15.2nm to 13.9nm

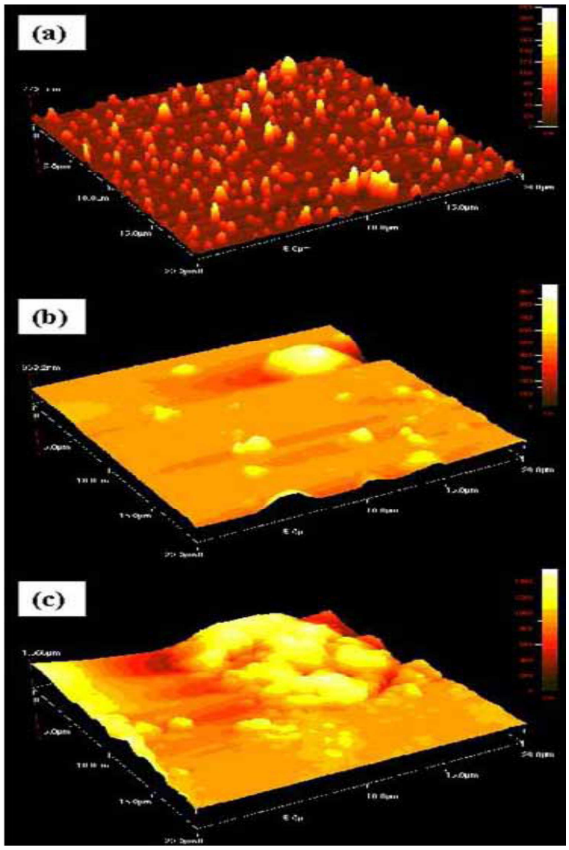

Figure 2: Atomic force microscopy images of PbSe thin films chemically deposited for a different deposition periods at different H(a) pH0.5 at 40 min (b) pH 1.0 at 60 min (c) pH 1.5 at 80 min

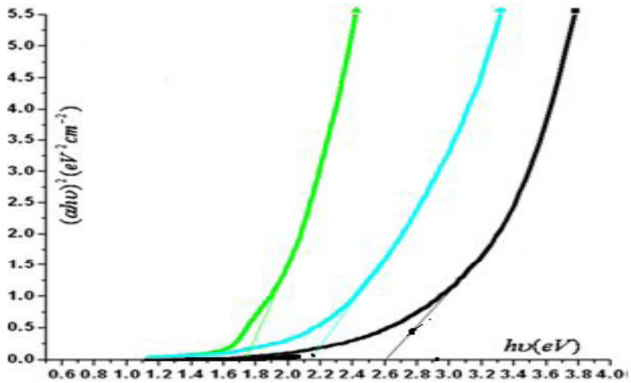

Figure 3:- Plot of $(\alpha h\upsilon)^2$ versus $(h\upsilon)$ for samples with different deposition times with different (a) pH0.5 at 40 min (b) pH 1.0 at 60 min (c) pH 1.5 at 80 min

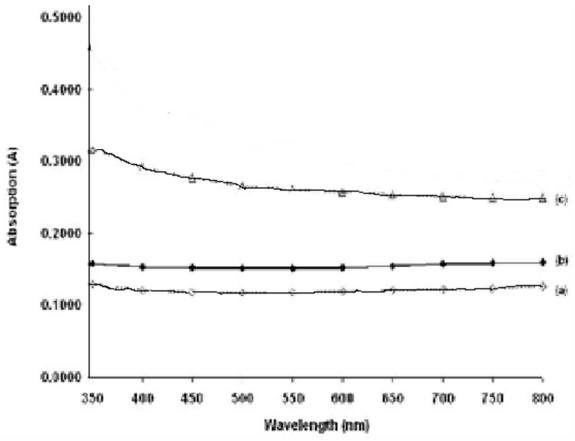

Figure 4. Optical absorbance versus wavelength of the PbSe thin films chemically deposited for a different deposition periods at a different PH

(a) pH0.5 at 40 min (b) pH 1.0 at 60 min (c) pH 1.5 at 80 min

CONCLUSION

The X-ray diffraction pattern showed that the most intense peak at $2\theta = 25.35°$ belonged to (111) plane of PbSe. As the deposition period was increased up to 80 min, the film starts to grow thicker as could be seen in the AFM images. It was observed that the best crystalline of the PbSe thin film is obtained at pH 1.5. The atomic force microscopy image showed that this film has uniform, smaller crystal size and covered the entire substrate surface completely. Therefore, deposition at pH 1.5 for 80 min and growth temperature at 50°C was found to be the optimum condition to prepare good quality thin films under the current condition. The band gap value found to be 2.6 eV with direct transition

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