



The effect of deposition time on the structural, morphological and optical band gap of lead selenide thin films synthesized by chemical bath deposition method

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ABSTRACT

Nanocrystalline lead selenide thin films have been deposited on silica glass substrates by chemical bath deposition method at a bath temperature of 90 °C and a pH of about 12. Three samples were prepared by employing different deposition times. The deposited thin films, which were uniform, well adherent and gray in color, were characterized by powder X-ray diffraction, scanning electron microscopy, energy dispersive X-ray analysis and optical absorption spectroscopy. The results showed that the deposition time had a strong influence on the morphology, preferred orientation of the crystallites as well as other structural parameters such as microstrain and dislocation density. The optical band gap also varied with increasing deposition time from 1.25 eV to 1.11 eV. The elemental compositions of the films were confirmed by energy dispersive X-ray spectroscopy.

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1. Introduction

Lead selenide (PbSe) is a IV–VI compound semiconductor, possessing excellent optoelectronic properties. It exhibits the cubic structure with face centered phase and space group $Fm\bar{3}m$ [1,2]. PbSe has a direct optical band gap of 0.27 eV at room temperature (RT) [2]. During the past two decades, PbSe has been the object of an inquiry into nanosized effects [3]. It exhibits strong quantum size effects below excitonic Bohr radius of 46 nm [4] and hence the energy band gap of its nanocrystals can be tuned to anywhere between 0.27 eV and 1.15 eV [3]. It is worth noting that the 1–1.5 eV range of the optical band gap of semiconductors is suitable for achieving high conversion efficiency when used as an absorber components [5]. The interest in lead selenide is due to its narrow band gap, which is employed to produce photoresistors, photo-detectors, solar cell and photoemitters in the IR range [2,3]. Another compound of Pb which has potential applications in nonlinear optical devices such as infrared sensor is PbS [6].

PbSe thin film can be obtained by several deposition techniques such as co-evaporated [7], pulse sonoelectrochemical [3], thermal evaporation [2], pulsed laser deposition [8] and chemical bath deposition [9,10]. Among these techniques chemical bath deposition

method is currently attracting considerable attention as it is relatively inexpensive, simple and convenient for large area deposition [11]. Properties of thin films such as film thickness, crystalline size and band gap can be controlled by varying the CBD growth parameters that include bath temperature, pH and deposition time [12]. In this work, we report the effect of deposition time on the morphology, structural and optical band gap of nanocrystalline PbSe thin films deposited by CBD technique using sodium hydroxide and ammonia as a complexing agent. The combination of sodium hydroxide and ammonia as a complexing agent has rarely been reported.

2. Deposition of PbSe thin film

The chemicals used were lead acetate trihydrate [(Pb(CH₃COO)₂ · 3H₂O)], sodium selenosulphate [Na₂SeSO₃], sodium hydroxide, (NaOH) and ammonia (NH₃). Sodium selenosulphate was prepared by refluxing about 9 gm of selenium powder with approximately 15 gm of anhydrous sodium sulfate (Na₂SO₃) in 250 ml of distilled water for 6 hours at 80 °C.

The mixture for the bath was prepared by mixing an appropriate amount of lead acetate and 10 ml (0.5 M) of NaOH in a 100 ml beaker. Initially, the solution looked milky turbid due to the formation of Pb(OH)₂ it later changed into a colorless solution after the addition of a sufficient amount of ammonia. Sodium selenosulphate and distilled water was added to obtain a final volume of

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70 ml. Chemically cleaned substrates were immersed vertically into the solution with the help of a substrate holder. The reaction mixture was continuously stirred with a magnetic stirrer and maintained at a temperature of 90 °C for the deposition. The prepared solution was initially clear and colorless but turned to dark gray after a few minutes and later, a mirror-like film began to deposit onto the sides of the beaker and the substrate confirming the formation of PbSe. The possible chemical reaction process for the formation of PbSe thin film has been described by [4]. Three different samples were prepared with deposition times of 30, 45 and 60 min respectively. After each deposition time, the samples were removed from the solution, rinsed ultrasonically with distilled water and dried under ambient conditions before film characterization. The crystal structure of PbSe thin films were analyzed by a PANalytical Empyrean X-ray diffractometer with a Cu-K α radiation ($\lambda_{\alpha 1} = 1.5406 \text{ \AA}$). The elemental composition and surface morphology of the samples were determined using energy dispersive X-ray analysis (EDX) attached with a high resolution JEOL JSM-7600F scanning electron microscope (SEM). Optical absorption spectra were measured using a Shimadzu UV/Vis mini-1240 Spectrophotometer within the wavelength range of 200–1100 nm.

3. Results and discussion

X-ray diffraction analysis: Fig. 1a–c shows the X-ray diffraction patterns of PbSe thin films for the different deposition times. The diffraction pattern was well matched with the standard JCPDS data file reference code: 01-077-0245. The diffractogram shows well defined peaks corresponding to reflections from the (111), (200), (220), (311), (222), (400), (420), (422) and (511) planes of the face centered cubic structure. The presence of well defined and sharp peaks in all the XRD patterns is attributed to a high degree of crystallinity in the thin films [11,13].

From the XRD patterns it can be observed that varying the deposition time has an effect on the peak intensities as well as the preferred orientations of the crystallites. A similar phenomena was observed in our previous report on PbS thin film [11]. For the deposition times of 30 and 60 min, the crystallites had a preferred orientation along the (111) plane, whilst for the deposition time of 45 min, the crystallites had a preferred orientation along the (200) plane. This indicates that the orientation of the grains growth along different directions depending on the deposition time. A similar observation was reported by [14]. The number of peaks appearing on the XRD spectrum increases with increasing deposition time, indicating an increase in crystal growth. Deposition times exceeding 60 min resulted in non adherent films which peeled off.

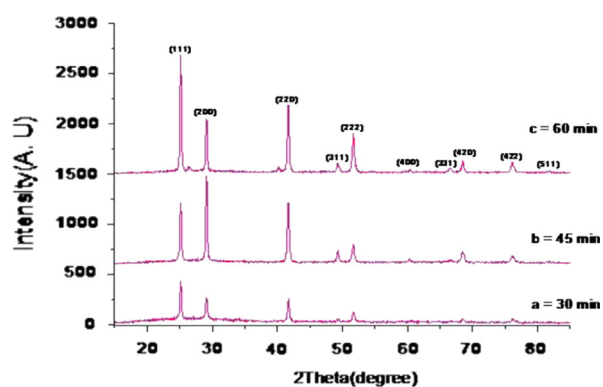


Fig. 1. XRD patterns of PbSe thin films prepared at different deposition times.

Lattice constant (a_{hkl}) for the cubic structure was calculated using Eq. (1) and the results are shown in Table 1.

$$a_{hkl} = d_{hkl} \sqrt{h^2 + k^2 + l^2} \quad (1)$$

The calculated value of the lattice constant ' a_{hkl} ' of the films shows a slight deviation from its standard value of 6.128 Å. Corrected values for the lattice constants are estimated from the Nelson-Riley plots, derived from Nelson-Riley function [6]. The corrected values of the lattice parameter for each deposition time and all the parameters obtained from the XRD data are presented in Table 1.

The average crystallite size was calculated from a full width at half maximum of the diffraction peaks using the Debye–Scherrer formula [11]. The average crystallite size of the films increased almost linearly from 17.84 nm to 21.57 nm as the deposition time increased from 30 to 60 min. A similar observation was reported by [15]. Similarly the average thickness of the films increased from 205 nm to 599 nm as the deposition time increased from 30 to 60 min. The film thickness was measured by weight difference method [16]. The deviation of the calculated lattice constant from the bulk sample value mentioned earlier (see Table 1), shows that the crystallites may be under some strain [17]. The microstrain value (ϵ) of the as-deposited PbSe thin films were calculated using the following mathematical relation [18]:

$$\epsilon = \frac{\beta \cos \theta}{4} \quad (2)$$

where θ is the diffraction angle and β is the full width at half maximum. The other structural parameter calculated is the dislocation density (δ) which is a measure of the defects in the film [17], and this was calculated using Williamson and Smallman's formula [19]. Strain and dislocation density were calculated from the (200) plane and the results are tabulated in Table 1. From Table 1, it can be observed that the dislocation density and strain decreased with increasing deposition time. This indicates the formation of high quality of thin films [18]. In addition the dislocation density decreased with increasing crystallite size. This may be due to a decrease in the occurrence of grain boundaries because of an increase in the crystallite size of the film with increasing deposition time.

Elemental analysis and morphological studies: The elemental analysis of PbSe thin films were carried out without elemental restriction. Figs. 2 and 3 show the EDX patterns of PbSe thin films deposited at 30 min and 60 min respectively. The EDX patterns of the PbSe thin films showed the presence of strong Pb and Se peaks. The average atomic ratio of Pb:Se was 60:40, which showed that the samples were rich in lead. Similar result was reported by [3]. The possible composition and structure of chemically deposited PbSe thin films can vary widely due to the variation of pH and possibly, the presence of reducing agents [20]. The presence of silicon, sodium and calcium in the EDX analysis of Fig. 2 is due to the composition of the silica glass substrate used for the deposition. Varying the deposition time did not have any significant effect on the atomic ratio of Pb:Se. However, elements such as silicon, sodium and calcium, observed in the spectrum for the deposition time of 30 min did not appear in the spectrum of the longer deposition time. This may be as a result of thicker films formed for the longer deposition time, thus, reducing the signals emanating from the substrate. The oxygen content found in the EDX spectrum could either emanate from the substrate [21] or due to the films getting oxidized on exposure to the atmosphere [22]. The carbon peak was due to the carbon coated grid [23].

The SEM micrographs (inset in Figs. 2 and 3) show that the surface morphology is composed of compact sharp edged cubic shaped grains of different sizes and uniformly distributed over a

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