XRD, AFM and UV-Vis Optical Studies of PbSe Thin Films Produced by Chemical Bath Deposition Method

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Abstract. PbSe thin films have been deposited on microscope glass substrates by chemical bath deposition technique. The chemical bath consisted of lead nitrate, sodium selenate and triethanolamine solutions. The influence of bath temperature on the properties of PbSe films was investigated. The X-ray diffraction, atomic force microscopy and UV/Vis Spectrophotometer were used to obtain the structural characterization, surface morphological and absorbance data, respectively. Based on the X-ray diffraction results, the thin films obtained were found to be polycrystalline in nature with cubic structure. The intensity of the (111) peak showed a significant increase as the bath temperature was increased from 40 to 80°C. The films deposited at 80°C indicated that the crystallinity was improved and more PbSe peaks were observed. On the other hand, the grain size, film thickness and surface roughness were increased while band gap energy decreased as could be observed in atomic force microscopy and UV-Vis optical studies, respectively.

Keywords: Lead selenide; X-ray diffraction; Optical properties; Chemical bath deposition; Thin films.

INTRODUCTION

Lead selenide is an important semiconductor material with interesting properties such as direct band gap and abundance in nature. Thus, lead selenide thin films have been widely used in a variety of fields such as solar cells, thermoelectric cooling, optical recording, light emitting diodes, sensors, laser and thin film transistors. Several techniques have been applied to obtain lead selenide thin films such as electrodeposition [1], chemical bath deposition [2], electrochemical atomic layer epitaxy [3], photochemical [4], molecular beam epitaxy [5], pulsed laser deposition method [6] and vacuum evaporation [7]. Basically, thin films prepared by chemical methods such as chemical bath deposition method and electrodeposition method are generally less expensive than those prepared by the capital-intensive physical technique. The chemical bath deposition method is an electroless method that is attractive as a simple, low cost instrumentation and potential for large-scale production. Up-to-date, chemical bath deposition method has been successfully used to deposit various thin films including NiSe [8], PbS [9], CdTe [10], AgIn₂S₃ [11] and Cu₄SnS₄ [12].

The present work reports the preparation and physical characterization of PbSe thin films onto microscope glass substrates using chemical bath deposition method. The chemical bath contains lead nitrate and sodium selenate which provides Pb²⁺ and Se²⁻ ions, respectively, while triethanolamine act as complexing agent. It is the first time, we report the influence of bath temperature ranging from 40 to 80°C on the PbSe thin films. The results of the investigation on structural, morphological and optical properties of thin films have been carried out using X-ray diffraction, atomic force microscopy and UV-Vis spectroscopy methods, respectively.

RESULTS AND DISCUSSION

Figure 1 shows the X-Ray Diffraction (XRD) patterns of lead selenide thin films deposited at different bath...
temperatures ranging from 40 to 80°C. All the samples are found to be polycrystalline in nature. For the films prepared at 40°C, two peaks at 2θ = 25.5° and 29.0° are observed. The corresponding interplanar distances are well in agreement with JCPDS data (Reference code: 00-065-1040) [13] of 3.55 and 3.06 Å which attributed to the (111) and (200) planes, respectively. As the bath temperature is increased to 60 and 80°C, the PbSe peaks increased to three and finally four, respectively. All these peaks are related to the compound of PbSe of cubic structure. The lattice parameter values are a = b = c = 6.128 Å. On the other hand, as the bath temperature is increased from 40 to 80°C, the intensities of the peaks attributable to PbSe improved. Diffraction along the (111) plane shows the highest intensity with well-defined sharp peak, indicating high crystallinity of the material prepared. Similar prominent plane has been reported for the chemical bath deposited PbSe thin films on GaAs substrate [2].

The presence of the silicone dioxide [14] (JCPDS reference No.: 01-074-0201) peaks in the XRD patterns are due to the microscope glass substrate used during deposition. Two peaks occurred at 2θ values of 43.3° and 53.8° corresponding to (211) and (213) planes are obtained. Based on the XRD patterns, the peaks marked with solid triangles are associated with reflections of the cubic structure of PbSe and those marked with open diamonds can be ascribed to the orthorhombic structure of silicon dioxide.

The PbSe thin films were morphologically characterized using Atomic Force Microscopy (AFM) technique. Figure 2 shows the three-dimensional representation of 20 × 20 µm area of the PbSe thin films deposited at different bath temperatures. The PbSe thin films prepared at lower bath temperature (40°C) indicate that the growth of small grains distributed across the surface of the substrate. The size of the grains is rather different from each other indicating irregular growth rate of the grains. The granules are made of different sizes varying from 0.3-0.5 µm. However, the sizes of the grains are noticed to increase as the bath temperature is increased to 60 (1.3-1.5 µm) and 80°C (2-3 µm), respectively. The films deposited at higher bath temperature (80°C) show compact morphology. Based on AFM image (Figure 2c), the grain density reduced indicating the smaller grains agglomerate together to form larger grains of PbSe.

On the other hand, the thickness and surface roughness of the films were measured using AFM.
技术。厚度值为322, 664和1994纳米的样品在40, 60和80°C下被观察到。同样地，表面粗糙度的相应值为24, 63和225纳米，分别。Root Mean Square (RMS)表面粗糙度定义为表面高度图的平均高度的方差，是表面粗糙度最常报告的测量值[15]。表面粗糙度是不可避免的，因为颗粒以不同的大小生长。它可以被检测到，表面粗糙度和厚度值随着提高的浴温度而增加，表明在粒径大小上的增加。我们可以得出结论，浴温度对PbSe薄膜的性质起着至关重要的作用。

基本的吸光度与电子激发从价带到导带带可以用来确定材料的性质和光学带隙[16]。吸光度(A)、带隙能量(εg)和光子能量(hν)之间的关系可以根据[17]等式1所示

$$A = \frac{k(\nu - \varepsilon_g)^{n/2}}{\nu},$$  

(1)

其中ν是频率，h是普朗克常数，k是一个常数，n是常数，表示值为1或4。n = 1对应于直接过渡[18]，而n = 4对应于间接过渡[19]，分别。吸收光谱的数据在350-800纳米的波长范围内被记录在UV-Vis光谱仪上。对于直接带隙半导体，(Ahν)²/n与hν图被预测为具有光子能量轴的直线，通过带隙的能量[20,21]。图3中(Ahν)²对光子能量(hν)的铅硫化物薄膜的图示在图3中。

带隙值被发现为1.3, 1.2和1.1 eV的薄膜在40, 60和80°C下沉积，分别。这是显而易见的，带隙随着浴温度的增加而降低。在更高浴温度下，结晶度的提高导致薄膜的厚度增加，导致带隙能量的材料的降低[22,23]。带隙值由铅硫化物薄膜在本研究中获得的薄膜与化学浴沉积PbSe薄膜在GaAs基板[24]。在射线衍射结果，薄膜在微晶结构中被发现，具有立方结构。PbSe薄膜在40到80°C之间被沉积。在80°C下，CuKα峰值的强度显著增加，光子能量在111峰中被观测到。在另一方面，薄膜的粒度，薄膜厚度和表面粗糙度增加，而带隙能量减少了，这可以通过原子力显微镜和UV-Vis光学研究可见。
EXPERIMENTAL SECTION

Materials and Sample Preparation

Lead selenide thin films were 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 15 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 [Na₂SO₄·Se] was supplied as selenate source and triethanolamine [(HOC₂H₆)₂N] acted as complexing agent. All these chemicals used for the deposition were analytical grade. All the solutions were prepared in deionised water (Alpha-Q Millipore). For deposition, 20 ml of 0.15 M lead nitrate was complexed with 10 ml of triethanolamine agent. To this, 20 ml of 0.15 M sodium selenate was added slowly to the reaction mixture. The pH was adjusted to 6 by addition of hydrochloric acid (0.5 M) with constant stirring. The clean glass substrate was vertically immersed into the chemical bath with the temperatures of 40, 60 and 80°C. After the deposition time of 60 min, the glass substrate was taken out of the bath, washed with distilled water and kept in desiccator for further characterization.

Characterization methods

X-Ray Diffraction (XRD) analysis was carried out using a Philips PM 11730 diffractometer for the 2θ ranging from 20° to 60° with CuKα (λ = 1.5418 Å) radiation. The surface morphology, thickness and roughness were examined by recording Atomic Force Microscopy (AFM) images with a Q-Scope 250 in contact mode with a commercial Si₃N₄ cantilever. Values of Root Mean Square (RMS) roughness were calculated from the height values in the atomic force microscopy images using the commercial software. The optical properties of the thin films were measured with a Perkin Elmer UV/Vis Lambda 20 Spectrophotometer in the wavelength range of 350 to 800 nm. The film-coated indium tin oxide glass was placed across the sample radiation pathway while the uncoated indium tin oxide glass was put across the reference path. From the analyses of absorption spectra, the band gap energy (E₉) was determined.

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REFERENCES


BIOGRAPHIES

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