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Surface Morphological Study of Pb0.6In0.4Se Thin Films Deposited by Thermal Evaporation technique.

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ABSTRACT :

Thin films having different thickness of $Pb_{0.6}In_{0.4}Se$ were deposited by Thermal evaporation techniques, onto precleaned amorphous glass substrate. The structural properties of films were evaluated by XRD, SEM and EDAX. The X-ray diffraction analysis confirms that films are polycrystalline having cubic structure. The Optical photo micrograph shows that all grains are aligned in one direction and uniform surface coverage with some voids on the substrate. SEM images have Spherical particles of different sizes are in the range of 35nm to 0.35µm. From EDAX the actual atomic % for Lead, Indium and Selenide are in the ratio of 30.22: 19.69:50.09. TEM micrograph shows the image obtained is a translucent. And SAED pattern furthermore indicates that the deposited films had a polycrystalline in nature. The particle size ranges from 28 to 39 nm.

Key words: XRD, SEM, EDAX, TEM.

INTRODUCTION

Indium Selenide is important material of III - VI group compounds, and lead selenide is important material in II – VI group. The energy gap of InSe at room temperature is 1.3 eV, which makes it an attractive material for solar energy conversion (1- 5), diodes (6), infrared devices, and lasers (4). It is also used as promising material for application in solid solution electrode (7), opto electronic devices (7), etc but little work has been reported concerning the thin film state (7). Due to its potential applications, thin films of indium selenide have been extensively studied by doping n or p – type, so that they may be used in various solid state devices [8, 9]. Research and development on thin films has led to the conclusion that different classes of materials are of particular interest for different applications. Indium selenide (InSe) thin films is of particular interest because the material has in recent a variety of applications in optoelectronic devices [10], solar cells [11], solid state batteries [12]. Literature reports indicate that InSe thin films have been prepared by a number of techniques by a number of researchers. These include the vacuum evaporation [13-15], flash evaporation [16], molecular beam deposition [17], electro deposition [18], sol-gel method [19],Although there have been several studies on the growth and characterization of PbSe and InSe thin films, deposited by different growth techniques, there is still a lack of understanding of the electrical and structural properties which strongly affect device performance [3]. Therefore we have made an attempt to synthesize Indium Selenide thin films by thermal evaporation technique. In present work, the effect of film thickness on the surface morphological properties of $Pb_{0.6}In_{0.4}Se$ films over the thickness range 1000 - 3000 A has been investigated. Majority of these compounds have been reported to be grown in the crystalline form. Thin films preparation with direct materials do not shows any contamination of impurities, therefore the pure form of powder of lead, indium and selenide was used for synthesis it.

EXPERIMENTAL

The compound ingot of $Pb_{0.6}In_{0.4}Se$ was obtained by mixing quantities of high-purity (99.999%) lead, indium and selenium powder in the atomic proportion 3:2:5. The mixture was sealed in an evacuated quartz tube at a pressure of 10^{-5} torr and heated at 1120 K for 36h and then quenched in ice cooled water. Polycrystalline $Pb_{0.6}In_{0.4}Se$ films have been deposited by thermal evaporation technique under pressure of about 10^{-5} torr. The substrate to source distance was kept 20cm. The samples of different thicknesses were deposited under similar conditions. The thickness of the films was controlled by quartz crystal thickness monitor model No. DTM-101 provided by Hind-HiVac. The deposition rate was maintained 5-10 Ǻ/sec throughout sample preparation. Before evaporation, the glass substrates were cleaned thoroughly using concentrated chromic acid, detergent, isopropyl alcohol and distilled water [20].

X – Ray diffractogram (Rigaku Miniflex, Japan) were obtained of these samples to find out structural information and to identify the film structure qualitatively. The scanning angle (2θ) range was from 20^0 - 80^0 (CuK_α line). Optical absorption was measured by UV-VIS spectrophotometer model no. Shimadzu -2450.

Results and Discussion

Materials in the nanometer scale, such as colloidal dispersions and thin films, have been studied over many years and many physical properties related to the nanometer size. The properties and behaviors observed and measured are typically group characteristics. A better fundamental understanding and various potential applications increasingly demand the ability and instrumentation to observe measure and manipulate the individual nano materials and nanostructures. Characterization and manipulation of individual nanostructures require not only extreme sensitivity and accuracy, but also atomic-level resolution. It therefore leads to various microscopies that will play a central role in characterization and measurements of nanostructured materials and nanostructures.

Optical Microscopy

Fig.1 and Fig 2 shows the photomicrograph of the $Pb_{0.6}In_{0.4}Se$ films of thickness 1500 and 2000 Å respectively. These photo micrograph shows that all grains are aligned in one direction and uniform surface coverage with some voids on the substrate.

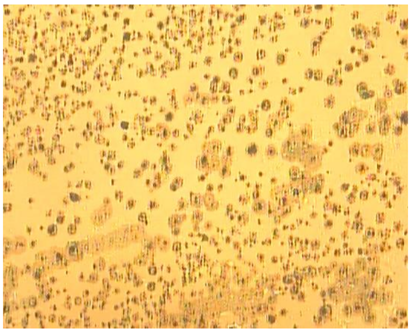


Fig. 1 Micrograph of $Pb_{0.6}In_{0.4}Se$ thin film of thickness 1500 Å

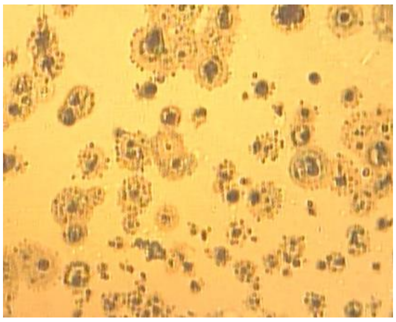


Fig. 2 Micrograph of $Pb_{0.6}In_{0.4}Se$ thin film of thickness 2000 Å

Further confirmation of the structure of the grown films was carried out using the x-ray diffraction pattern.

XRD of $Pb_{0.6}In_{0.4}Se$ Thin Films –

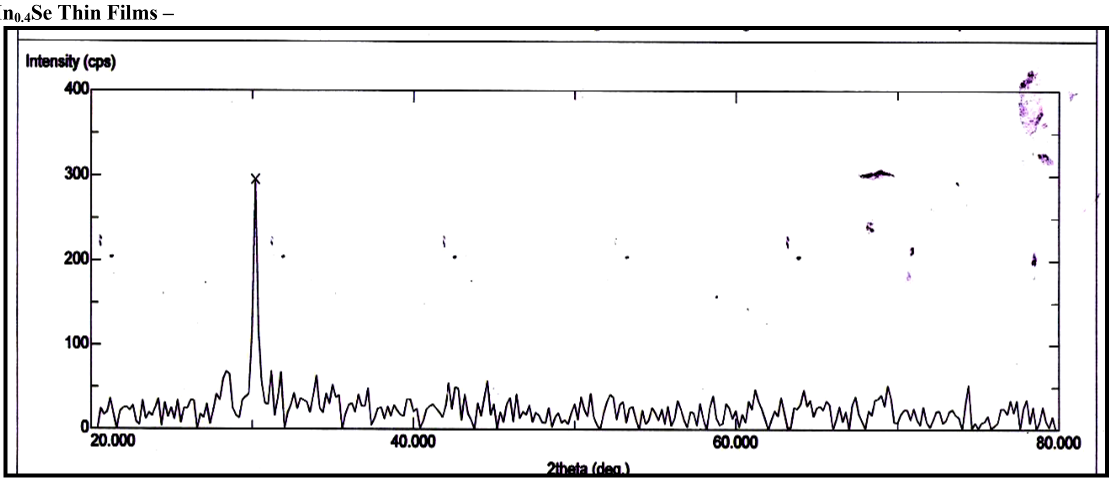


Fig. 3 XRD of $Pb_{0.6}In_{0.4}Se$ thin film of thickness 1500 Å

Fig. 3 shows the XRD pattern of $Pb_{0.6}In_{0.4}Se$ thin film prepared at substrate temperature of 303k. The 2θ peaks observed at 56^0 and 54.8^0 exhibit the formation of the orthorhombic phase of $Pb_{0.6}In_{0.4}Se$ which correspond to the (205) and (108) planes of reflections. The inter-planar distances as were indicated in the XRD result were found to be 1.36 Å and 1.72 Å. The presence of large number of peaks indicates that the films are polycrystalline in nature. The unit cell volume is 618 and lattice parameters are a = 15.285Ǻ, b = 12.105Ǻ and c = 4.140Ǻ.

Scanning electron microscopy (SEM)

Fig.4 shows SEM images of $Pb_{0.6}In_{0.4}Se$ thin film of thickness 2500 Ǻ. The surface of $Pb_{0.6}In_{0.4}Se$ deposits are covered with spherical and rough crystals. Spherical particles of different sizes are in the range of 30nm – 0.35µm. The surface roughness is greatly improved than that of $Pb_{0.6}In_{0.4}Se$**.** That may be due to increase in atomic percentage of lead.

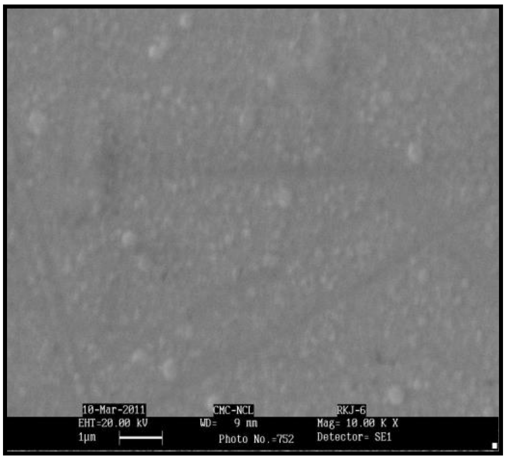
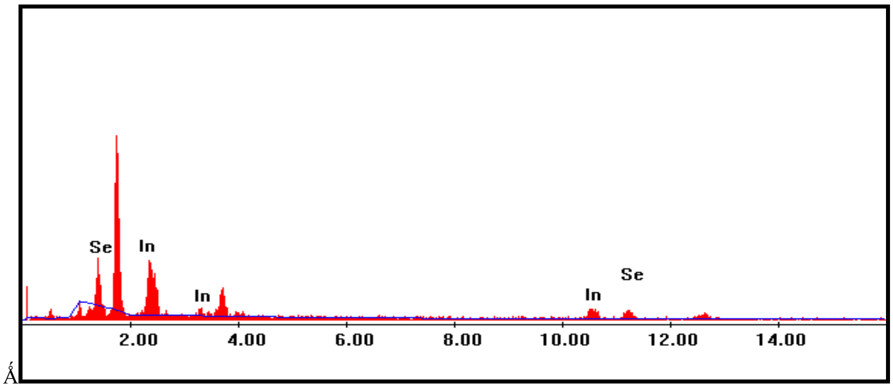


Fig. 4 SEM image of $Pb_{0.6}In_{0.4}Se$ thin film of 2500 Ǻ



Å

Fig. 5 EDS Spectra of $Pb_{0.6}In_{0.4}Se$ thin film

The EDX spectral analysis for the $Pb_{0.6}In_{0.4}Se$ thin film prepared by thermal evaporation technique is shown in Fig. 5. Fully quantitative analysis results were obtained from the spectrum by processing the data through a correction program. The obtained percentages of the constituent elements in all investigated films indicate that samples are nearly stoichiometric. The obtained results give support for the quality of the prepared $Pb_{0.6}In_{0.4}Se$ thin films by thermal evaporation technique. The actual atomic % for lead, indium and selenide are in the ratio of 30.22: 19.69:50.09.

Transmission electron microscopy (TEM)

Fig. 6(a) shows the TEM micrograph of as-prepared $Pb_{0.6}In_{0.4}Se$ Thin film of thickness 1500 Å. The image obtained is a translucent; it may be due to higher thickness. The selected area electron diffraction (SAED) pattern in fig. 6(b) furthermore indicates that the $Pb_{0.6}In_{0.4}Se$ had a polycrystalline in nature [20]. The particle size ranges from 28 – 39 nm. This result was a good agreement with XRD result.

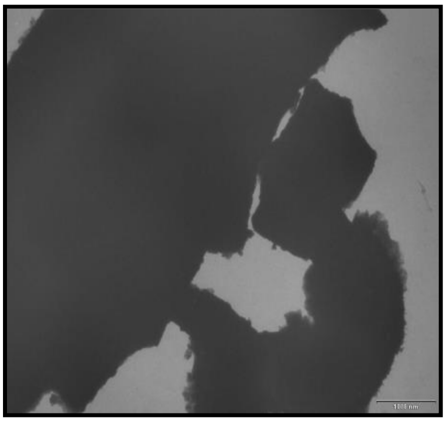


Fig. 6 (a) TEM micrograph of $Pb_{0.6}In_{0.4}Se$ thin film of thickness 1500 Ǻ

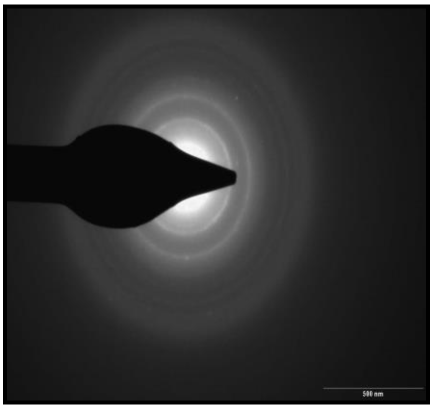


Fig.6 (b) SAED pattern of $Pb_{0.6}In_{0.4}Se$ thin film of thickness 1500 Ǻ

Conclusion

The X-ray diffraction analysis confirms that films are polycrystalline having cubic structure. The average grain size is found to be 37nm. The Optical photo micrograph shows that all grains are aligned in one direction and uniform surface coverage with some voids on the substrate. SEM images have Spherical particles of different sizes are in the range of 35nm - 0.35µm. The surface roughness is greatly improved due to increase in atomic percentage of lead. From EDAX the actual atomic % for $Pb_{0.6}In_{0.4}Se$ are in the ratio of 30.22: 19.69:50.09. TEM micrograph shows the image obtained is a translucent; it may be due to higher thickness. The selected area electron diffraction (SAED) pattern furthermore indicates that the $Pb_{0.6}In_{0.4}Se$ had a polycrystalline in nature. The particle size ranges from 28– 39 nm. This result was a good agreement with XRD result.

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