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Preparation of nano crystalline PbSe films by CBD Technique and its Characterization

Ms Sujit Anil Kadam

***Abstract*— Nanocrystaline thin film of PbSe have been prepared on glass substrate by chemical bath deposition method, using sodium selenosulfate , which acts both as a source of Se2- ions and complexing agent , in the chemical bath. The film were reddish brown, uniform , well adherent to substrate , well defined grained and nearly stoichiometric in nature . XRD the study confirm polycrystalline nature of cubic structure with preferred orientation along (220) the formation of uniform film with spherical grains. The optical absorption spectrum showed an exponential edge. Optical band gaps (Eg) in the nanocrystalline film is found to be about 1.5eV which value is larger than that of bulk PbSe. A blue shift was observed for the nanocytalline film which is expected to originate from quantum confinement in the nanograins.**

***Index Terms*— Nanocrystalline, thin film, optical band gap, blue shift, XRD.**

I. INTRODUCTION

Semiconductors based on selenium are important class of semiconducting systems which have been widely studied due to their fundamental electronic and optical properties. Intensive research has been performed in the past to study the fabrication and characterization of these compounds in the form of thin films[1]. A number of methods for the preparation of PbSe thin films have been reported, but chemical bath deposition is found to be attractive due to the low cost method of fabrication. Although chemical bath deposition has been used as a technique for preparing films since 1910 [2], utilization of CBD semiconductors in photovoltaic devices.

The large number of materials that can be prepared by CBD and their prospective applications in solar energy conversion, mainly in the area of thin-film solar cells, has increased the research interest in chemically deposited semiconductor thin films. It has been found that the microstructure and the electrical properties of thin films are different from the properties reported for the bulk material with the same composition. Semiconductor possessing excellent optoelectronic properties. Optical band gap of PbSe can be tuned (~1.5 eV) by decreasing the crystallite size upon changing the preparation conditions [3]. Eg range of 1-1.5 eV is suitable to achieve high energy conversion efficiency when used as absorber material in optoelectronic devices. In addition, PbSe nanocrystals have major industrial uses such as field effect transistors , infrared detectors, thermoelectric material, etc. due to their unique electronic, optical and physical properties. For these reasons, many research groups have shown a great interest in the development and study of this material by various deposition processes such as electro deposition [4], RF magnetron sputtering , Inert gas condensation, electron beam evaporation technique , Chemical bath deposition. CBD method is presently attracting considerable attention, as it does not require sophisticated instrumentation. It is relatively inexpensive, easy to handle, convenient for large area deposition and capable of yielding good quality thin films. The characteristics of chemically deposited PbSe thin film by CBD strongly depend on the growth conditions. In this paper, we report optical and surface morphological properties of PbSe thin films obtained by CBD method by optimizing different deposition parameters such as precursor concentration, bath pH and deposition time.

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II. MATERIAL AND METHODS :-

Although there are various techniques to prepare PbSe nanostructures, chemical bath deposition offers a simple and cost-effective route for fabrication of high quality chalcogenide films. Most of the workers had done a variety of fundamental research work and there by promoting the chemical route into an excellent synthesis technique in recent times 14-25. In the present work PbSe nanocrystals were grown chemically as thin films on glass substrates. Initially a 0.2M sodium sulphite (Na**2**SO**3**) solution was mixed with a 0.2M freshly prepared Se solution under constant stirring for 9 hours to form the sodium selenosulphate (Na**2**SeSO**3**) solution . A clear 3% PVA (Poly Vinyl Alcohol) solution was prepared with constant stirring prior to deposition as a capping agent. Lead acetate solution (0.2M) was mixed with PVA solution where the ammonia was added to form clear dispersed metallic complex.

The Na**2**SeSO**3** solution was then mixed with the lead ion complex to form the final matrix solution. The reaction was carried out in an alkaline medium keeping pH at 9. Commercial glass slides were first immersed into dilute HCl (hydrochloric acid) overnight which were washed throughly with constant rubbing by soft tissue papers. Those glass substrates were then put into freshly prepared chromic acid for a few minutes for further fine cleaning. After that the substrates were washed under running water and put into dilute NaOH (sodium hydroxide) solution to neutralize any acid tracing on the glasses. Finally the substrates were rinsed with distilled water several times and dried. The cleaned glass substrates were dipped into the final matrix solution with the temperatures of 359K. After the deposition time of 40 min, the glass slide was taken out of the bath, washed with distilled water and dried in desiccators for further characterization.The thin films were used for the X-ray diffraction (XRD) while the filtrated brownish colour solution for Scanning Electron Microscopy (SEM) as well as UV-VIS.

III. CHARACTERIZATION TECHNIQUES :-

As-deposited thin film of PbSe was characterized for structural, optical and electrical properties. Glancing incidence angle X-ray diffraction (GIXRD) pattern of the film was recorded on a Bruker AXS, Germany (D8 Advanced) diffractometer. The scanning range of diffractometer used is 20–70◦ (2θ), using Cu-K**α1** radiations with wavelength 1.5405 Å at 0.5° glancing angle. The scanning range of diffractometer used is 20–70º (2θ). The surface morphology was studied by scanning electron microscopy (SEM, JOEL-JSM-5600) Transmittance and absorbance spectra were recorded in the range 500–1200 nm by means of Jasco V630 spectrophotometer.

IV. RESULTS AND DISCUSSIONS RESULTS

Surface morphological and structural Studies:

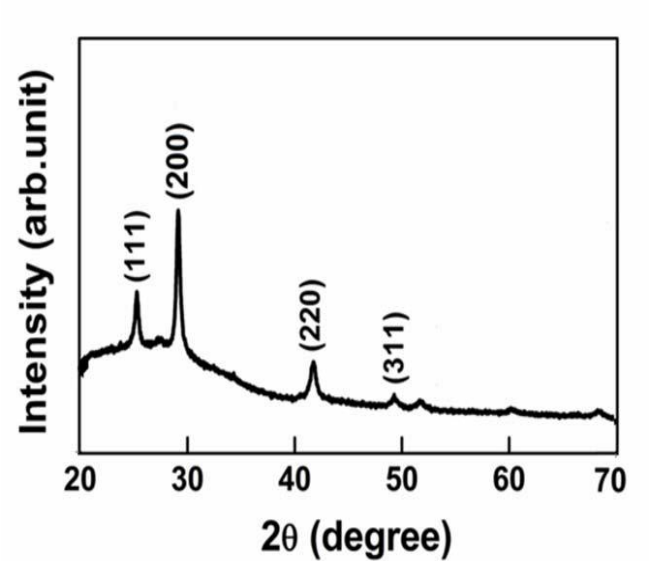

Fig1) X-ray diffraction pattern of PbSe thindeposited at 359K temperature.

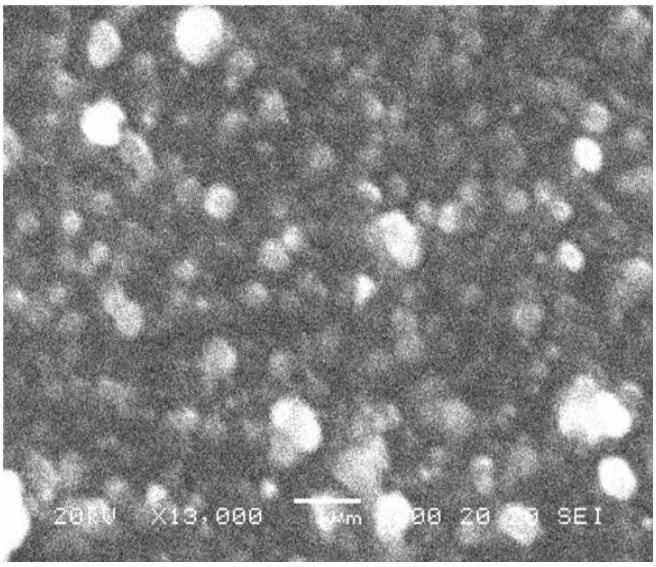

Fig.2 The SEM micrograph of PbSe thin films deposited at 359K temperature

Fig. 1 shows X-ray diffraction pattern of as-deposited PbSe nanoparticles. The XRD peaks indicate that the film is polycrystalline in nature. The 2θ peaks at 25.25°, 29.23°, and 41.81° correspond to reflections from (111), (200), and (220) planes, respectively. The (200) plane is the preferred orientation, and it is the close-packing direction of the zinc-blend structure of cubic PbSe phase (JCPDS Card No.78-1902). Crystallite size (D) of the film was calculated using Scherrer formula from the full width at half maximum (β) of the peaks expressed in radians,

$$D = \frac{K\lambda}{\beta \cos\theta} \qquad (1)$$

Where K is 0.9 which varies with (hkl) and crystallite shape. where λ is wavelength of x-ray, β is FWHM (full width half maximum), and 'θ' is angle between the incident and scattered X-ray. The average crystallite size (derived from Fig. 1) is found to be ~20 nm.

Fig.2 shows SEM image of as-deposited PbSe thin film. It is observed that the film is uniform and covers the entire substrate surface. The fine grains were well defined, spherical with different sizes and were uniformly distributed over a smooth homogeneous background corresponding to the nanocrystalline phase of PbSe. Some of the grains are seen to be united/fused forming agglomerates and the grain size obtained from SEM is about ~ 200 ± 20 nm.

OPTICAL STUDIES :-

Fig.3 shows transmittance and absorbance spectra obtained from as-deposited PbSe thin film. Band gap energy and transition type can be derived from mathematical treatment of data obtained from optical absorbance versus wavelength with following relationship for near -edge absorption (Equation 1) :

$$\alpha = \frac{A(h\nu - Eg)^{n/2}}{h\nu} \qquad (2)$$

where *v* is the frequency, *h* is the Planck's constant, *k* equals a constant while *n* carries the value of either 1 or 4. The value of *n* is 1 and 4 for the direct transition and indirect transition respectively.

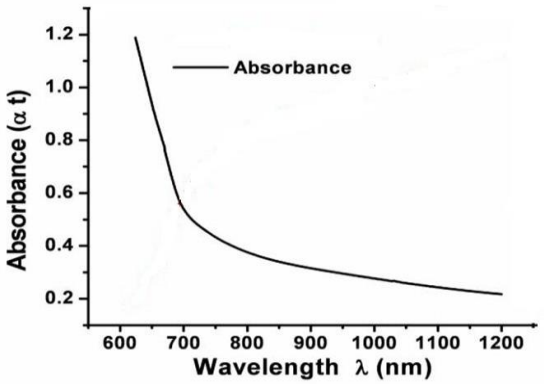

Fig.3. Plot of absorbance and transmittance versus wavelength

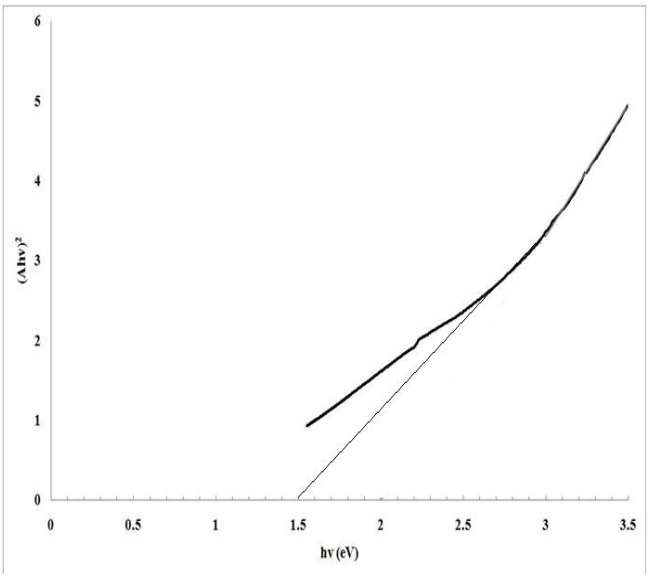

Fig.4. Plot of (αhν)² versus (hν) obtained from as-deposited PbSe thin film various bath. temperatures 359K.

Fig. 4 shows the plot of (αhν) ² against (hν) for PbSe thin film derived from the absorbance spectra. Extrapolating the straight-line portion of the plot of (αhν)² vs (hν) for zero absorption coefficient value gives the band gap, The band gap values are found to be 1.5 eV for the films deposited at 359K respectively. It is clear that the band gap decreases when the bath temperature is increased. At higher bath temperature, enhancement in crystallinity of the films leads to larger grains, and causes a reduction in the band gap energy of the material.

V. CONCLUSION:-

The PbSe thin films can be deposited by using simple and cost-effective chemical bath deposition method. Lead selenide thin films were successfully prepared varying pH by chemical bath deposition technique on glass substrates. The XRD pattern reveals that formation of cubic structure, with the strongest peaks attributed to (200) plane of PbSe. The X-ray diffraction studies revealed that all films had FCC crystal structure with a (200) preferred orientation .These films produced the highest absorption characteristic with band gap approximately to be 1.5 eV. Therefore, deposition at 359K was the best condition to prepare good quality PbSe thin films under the current conditions. The variations were found to affect thickness of the deposited films. The structural and electrical properties of the films were found to be thickness dependent.. Optical band gaps (***Eg***) in the nanocrystalline film is found to be about 1.5eV which value is larger than that of bulk PbSe. Optical studies indicated that the band gap decreased with increasing thickness of the films. The electrical conductivity decreases with thickness while the dielectric constant increases with thickness.

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