# SURFACE MORPHOLOGICAL AND OPTICAL PROPERTIES OF CdSe THIN FILMS BY CLOSED SPACE SUBLIMATION TECHNIQUE

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Thin films having different thickness of CdSe were deposited by closed space sublimation techniques, onto precleaned amorphous glass substrate at room temperature. The structural properties of films were evaluated by XRD, Scanning Electron Microscopy and Atomic Force Microscopy. The quantitative analysis was done by Energy Dispersive Analysis for X – Ray to determine atomic % of the material used. The optical band gap of the films was measured by using optical absorption spectra. It is found that CdSe is direct band gap material having value of 2.3 eV. The X-ray diffraction (XRD) patterns of these CdSe samples were recorded by X-ray diffractometer. The X-ray diffraction analysis confirms that films are polycrystalline in nature having hexagonal (wurtzite) structure with a preferential orientation along the (002) plane. The degree of such a preferred orientation was found to increase with film thickness. The lattice parameters (a = 4.30 and c = 7.02) and crystallite size (D) were calculated and found to be 85.9 nm. Unit cell volume is found to be 112.41. SEM investigation confirms that films were uniformly deposited over the surface and particles were circular in nature. The particle size were determined by using SEM and found to be 12.60 – 19.23 nm.

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*Keywords:* Optical band gap, closed space sublimation, XRD, SEM, AFM, EDAX.

## 1. Introduction

CdSe, a direct band gap material II-VI semiconductor with band gap energy of 1.74 eV, has long been found as promising material for optoelectronic devices such as solar cells, high efficiency thin-film transistors, electron-beam pumped lasers, LED, electroluminescent devices, etc [1-5]. In addition, CdSe has large absorption coefficient to visible light. This property is useful for good theoretical conversion efficiency and has led to the investigations for obtaining efficient solar cells. There are a number of reports on the different structural, optical and electrical properties of CdSe polycrystalline thin films prepared by various techniques such as vacuum evaporation [6-8], quasi-closed volume technique [9], electrode position [10-13], chemical bath deposition (CBD) [14-15], spray pyrolysis [16], sputtering [17] etc. It is seen that different parameters of a film are structural dependent which is also depends on the method of preparation, its thickness and other factors. In this work we have studied the effect of thickness on the structural and optical properties of CdSe thin films of different thicknesses prepared by closed space sublimation technique. Structural parameters such as grain size measured from XRD spectra are found to depend on the film thickness. The details have been reported in this paper.

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## 2. Experimental

**Material Preparation**

The CdSe compound ingots were obtained by taking appropriate amount of 99.999% pure Cd and Se in an evacuated quartz ampoule. The ampoule with the charge was then sealed under a pressure of $10^{-5}$ torr and was placed in rotating furnace. The temperature of the furnace was raised gradually to 970 K and left at this temperature for about 48 h. Well mixed charges were then quenched in an ice bath. The CdSe ingot was taken out from the ampoule and made into fine powder and used for film preparation.

**Synthesis and Characterization of sample -**

Polycrystalline CdSe films have been deposited by closed space sublimation technique under vacuum of about $10^{-5}$torr. The substrate to source distance was kept 8 cm. 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-Hi Vac. Further confirmation of thickness was estimated by Tolansky's method [13] using multiple beam Fizeau fringes. The deposition rate was maintained 5-10 Å/sec throughout sample preparation. Before evaporation, the glass substrates were cleaned thoroughly using concentrated chromic acid, detergent, isopropyl alcohol and distilled water.

X – Ray diffractogram (Bruker, Germany) were obtained of these samples to find out structural information and to identify the film structure qualitatively. The scanning angle (2θ) range was from $20^0$ - $80^0$ ($CuK_\alpha$ line). Surface morphological studies of the thermally deposited CdSe thin films were done using the Scanning Electron Microscope (Zeiss) operating with an accelerating voltage 15 kV and Atomic Force Microscopy (AFM). The quantitative compositional analysis of the CdSe films were carried out by EDAX (Energy dispersive X-ray Analyzer) technique attached with the SEM. Optical absorption was measured by UV-VIS spectrophotometer model no. Shimadzu -2450.

## 3. Results and discussions

### 3.1 Structural characterization-

Fig. 1 (a) shows the XRD pattern of CdSe thin film prepared at substrate temperature of 303K. The X-ray diffraction (XRD) analysis revealed that the films are polycrystalline in nature possessing hexagonal (wurtzite) structure. It can be seen that the film thickness strongly affects the XRD pattern. For lower thickness, the films have random particle orientation, identified by the presence of various peaks at (002), (101), etc. As the film thickness increases, the (0 0 2) diffraction peak becomes more and more dominant. This means that, at the initial stage of film formation i.e., during the atomistic condensation of the film formation, the deposited atoms are at random orientation. As the thickness of the film increases the polycrystalline grains begin to orient mainly along (0 0 2) direction which is evident from the Figs. 1(b). The value of the lattice parameters obtained from the analysis of x-ray diffraction pattern were a = 4.30 and c = 7.02. Unit cell volume is found to be 112.41. The average grain size is found to be 18.76 nm.

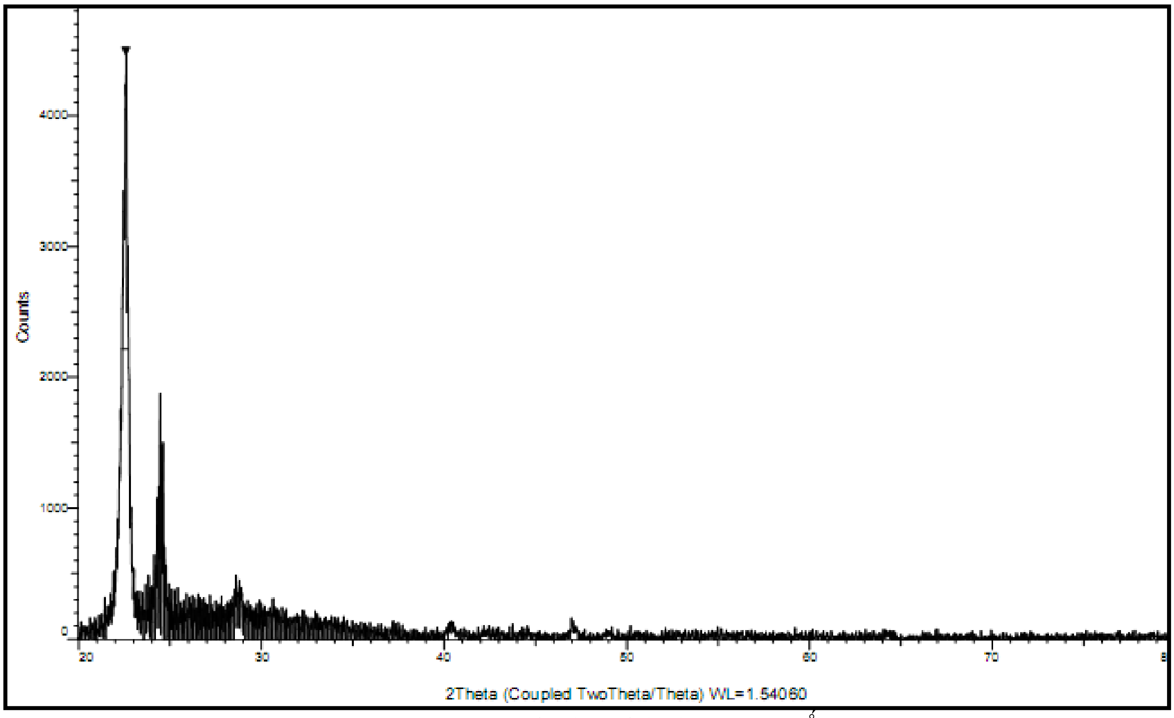


*Fig. 1(a) XRD of CdSe of thickness 1000 Ǻ*

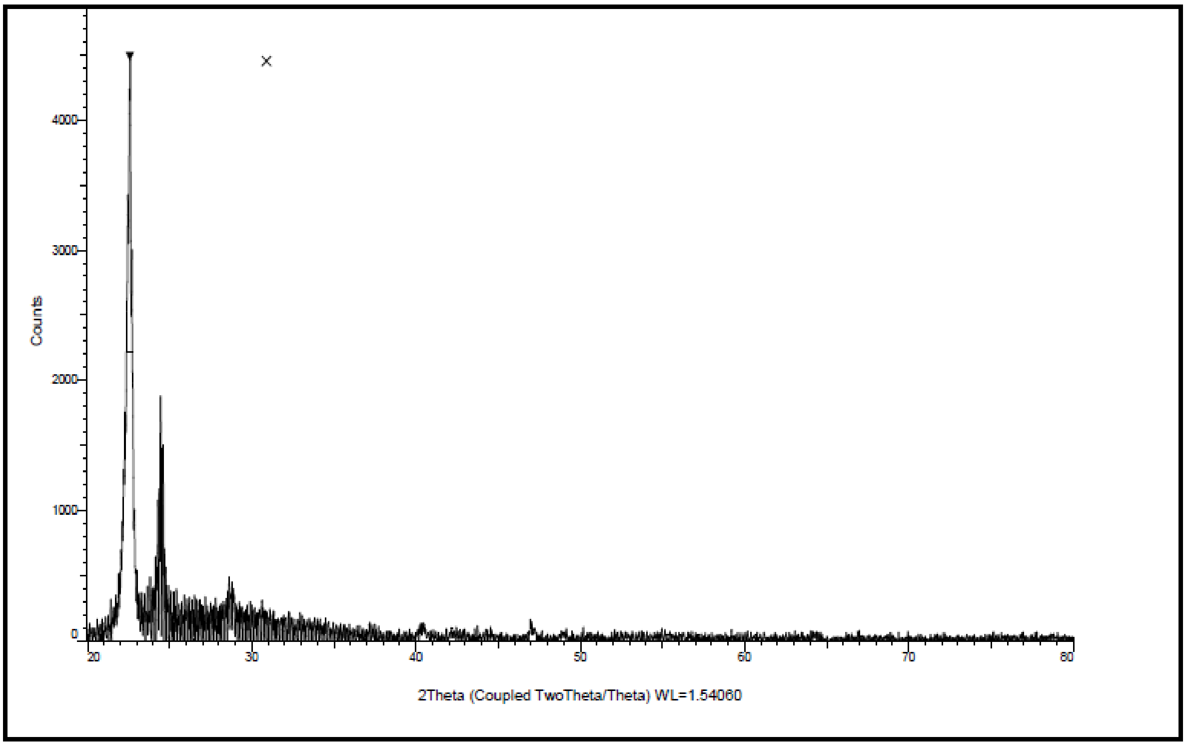


*Fig. 1(b) XRD of CdSe of thickness 2000 Ǻ*

The SEM image of CdSe thin film show that the film is uniform, polycrystalline, well cover on glass slide and free from microscopy defect like cracks or peeling. Nano size grains were uniformly distributed over smooth homogeneous background. The particle size were found to be 12 -20 nm. The cross sectional micro structure was observed by high resolution scanning electron microscope with compositional contrast detectors.

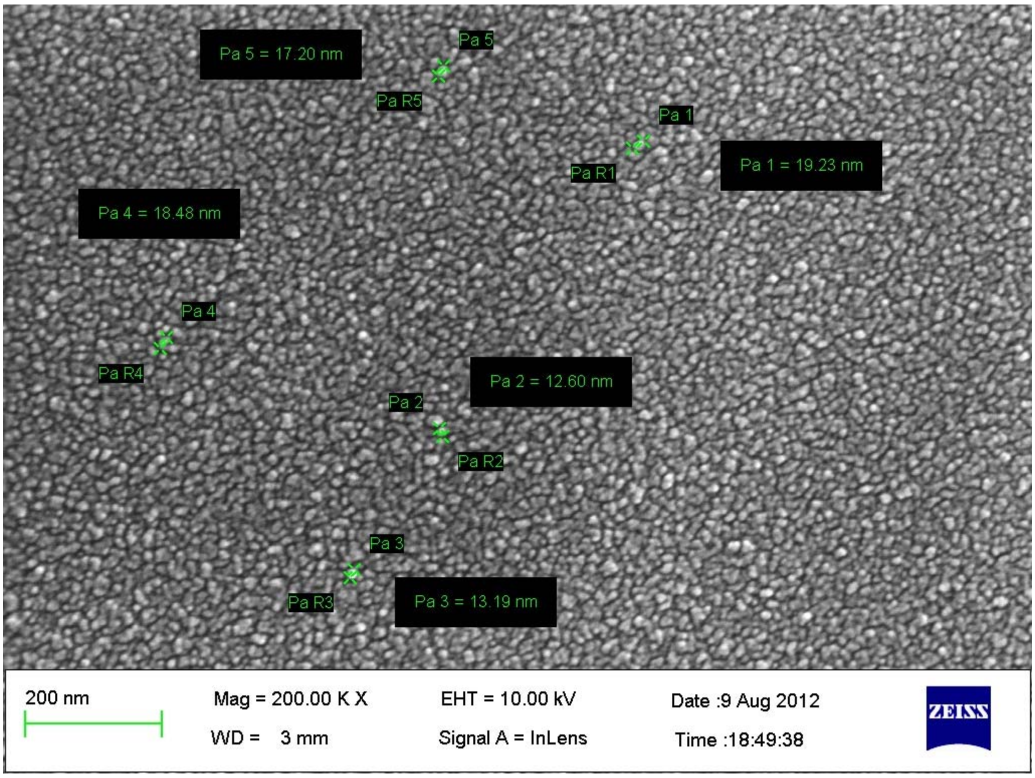


*Fig: 2(a) SEM image of CdSe thin film*

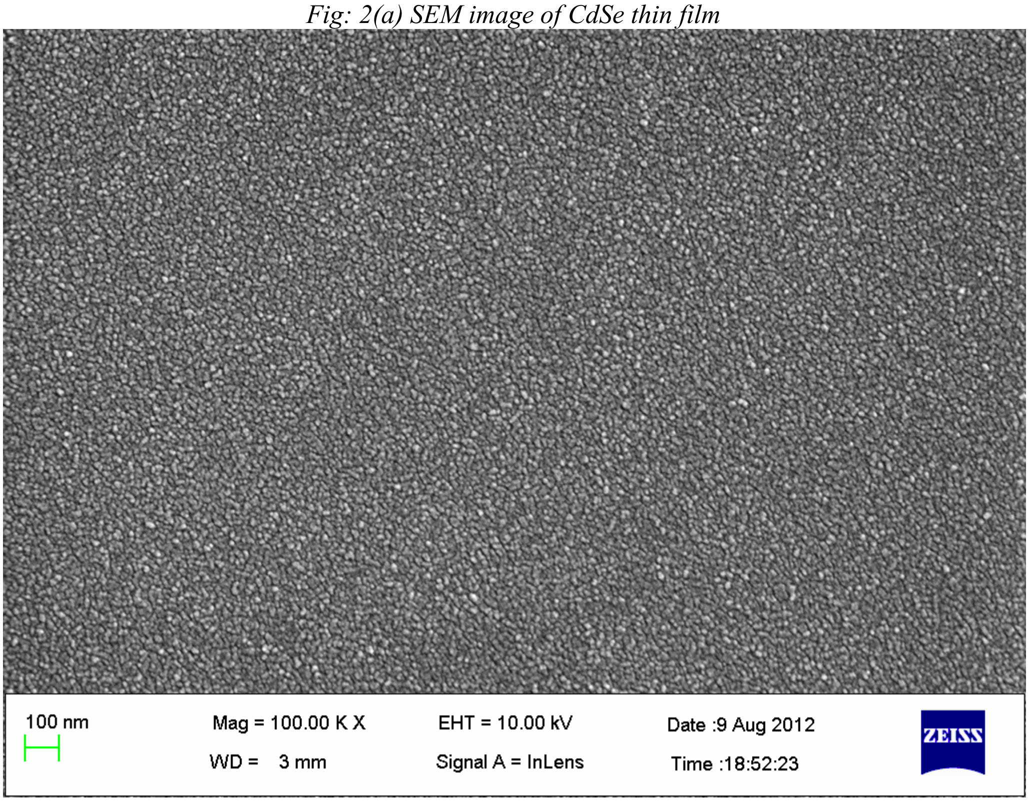


*Fig: 2(b) SEM image of CdSe thin film*

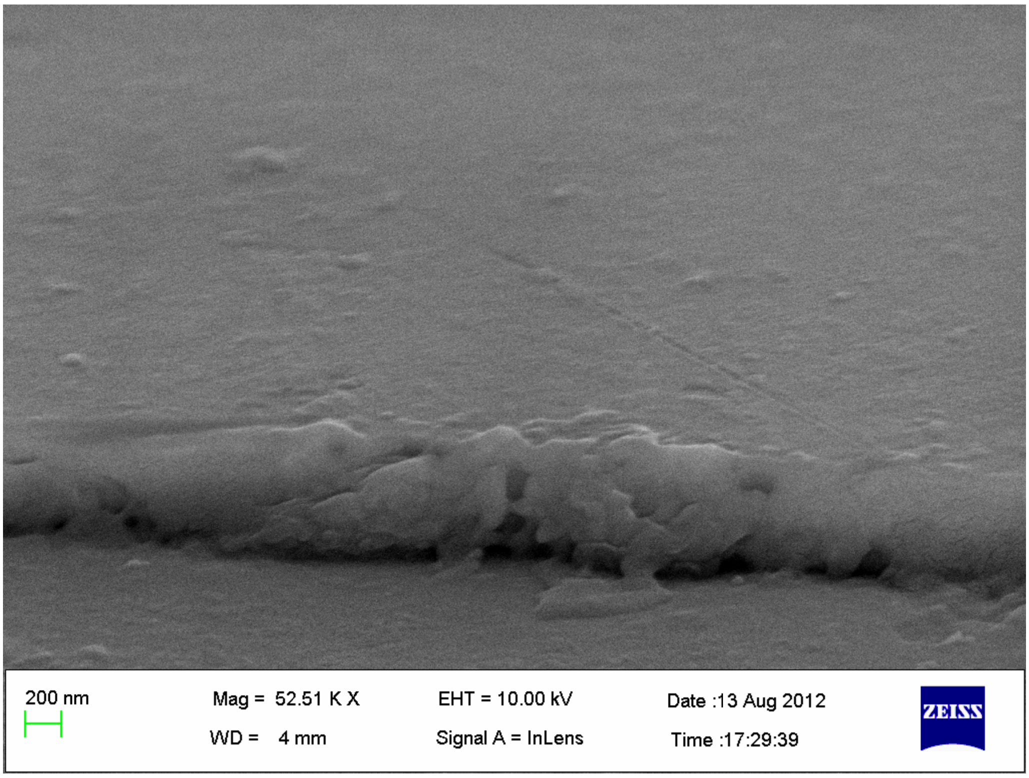


*Fig: 2(c) Cross sectional view of CdSe thin film*

The EDAX spectral analysis for the CdSe thin film prepared by closed space sublimation technique is shown in Fig.3.The obtained percentages of the constituent elements in all investigated films indicate that samples are nearly stoichiometric. The obtained result give support for the quality of the prepared CdSe films by thermal evaporation technique. It is found that the prepared films are selenium rich due to difference in vapour pressure of Se and Cd.

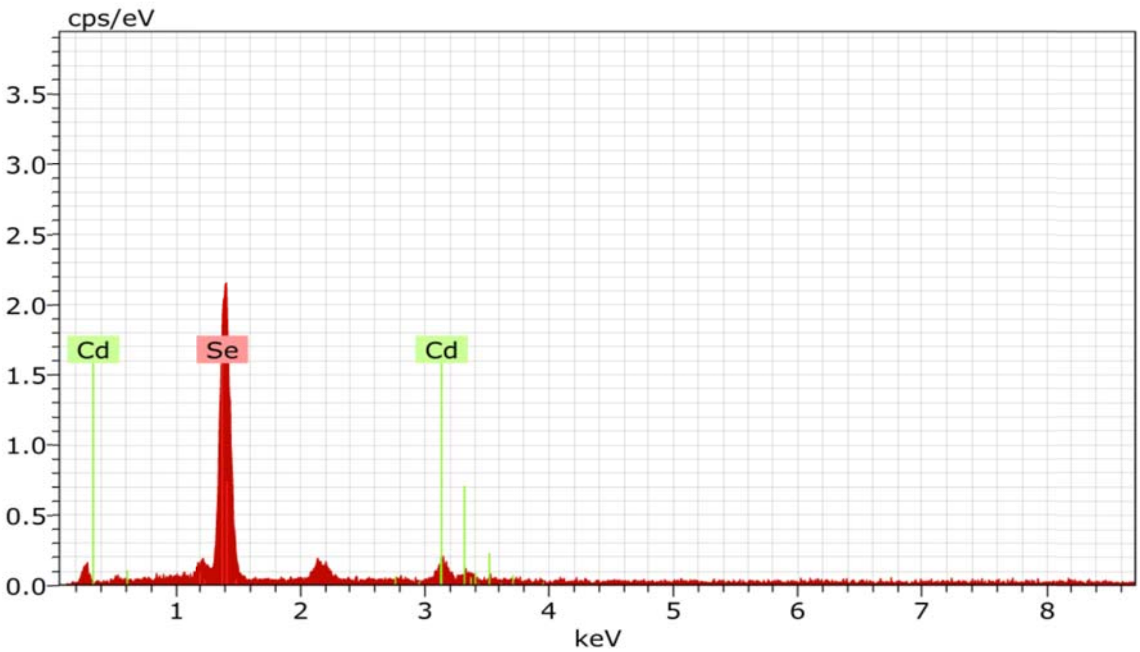


*Fig: 3 EDX spectrum of nanocrystalline CdSe.*

Fig. – 4 and Fig. - 5 represent the two-dimensional (2D) and three dimensional (3D) topographic images of CdSe films prepared by thermal evaporation technique onto glass substrates. The film matrix was found to have some spherical particles embedded into the background fine grained matrix. These granual particles may be due to the CdSe agglomerates deposited over the uniformly spread CdSe nano particles. Our investigations showed that the grain size determined by means of AFM ranged between 40 - 100 nm in the matrix. The roughness of the film surface is small. This will provide valuable information on the height deviation of the roughness profile and on its lateral distribution. From the line profile analysis, the average roughness values calculated are 0.36, 0.22, 0.21, and 0.23 nm for the CdSe films deposited at RT. These observations show that CdSe films deposited at RT have the device quality surface which will be suitable for developing photo-electrochemical devices.

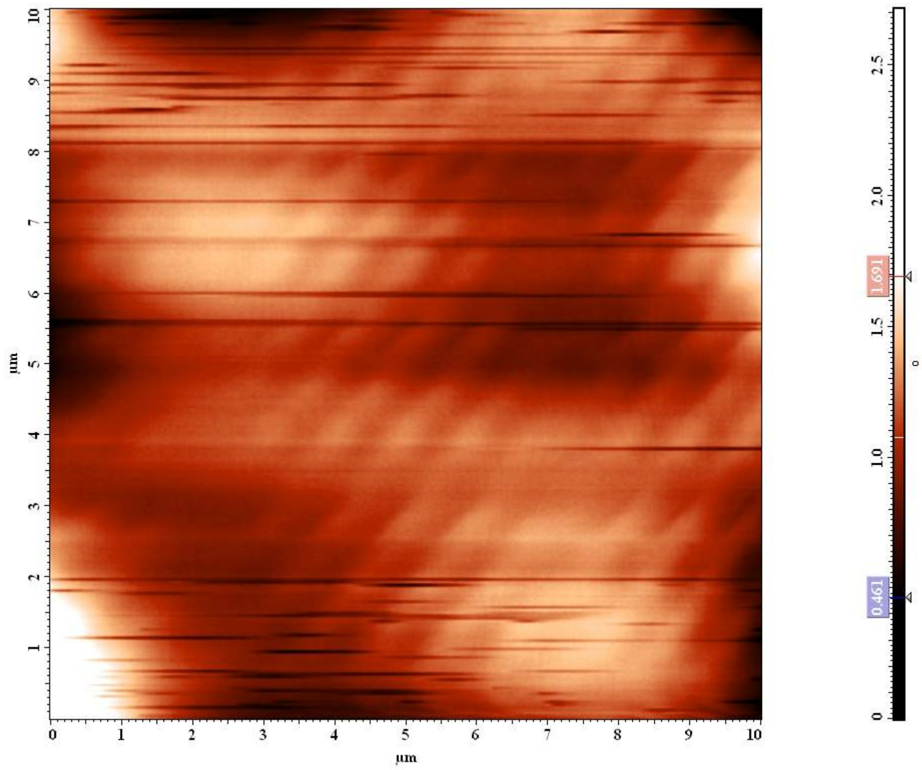


*Fig. 4: 2D AFM image of CdSe film*

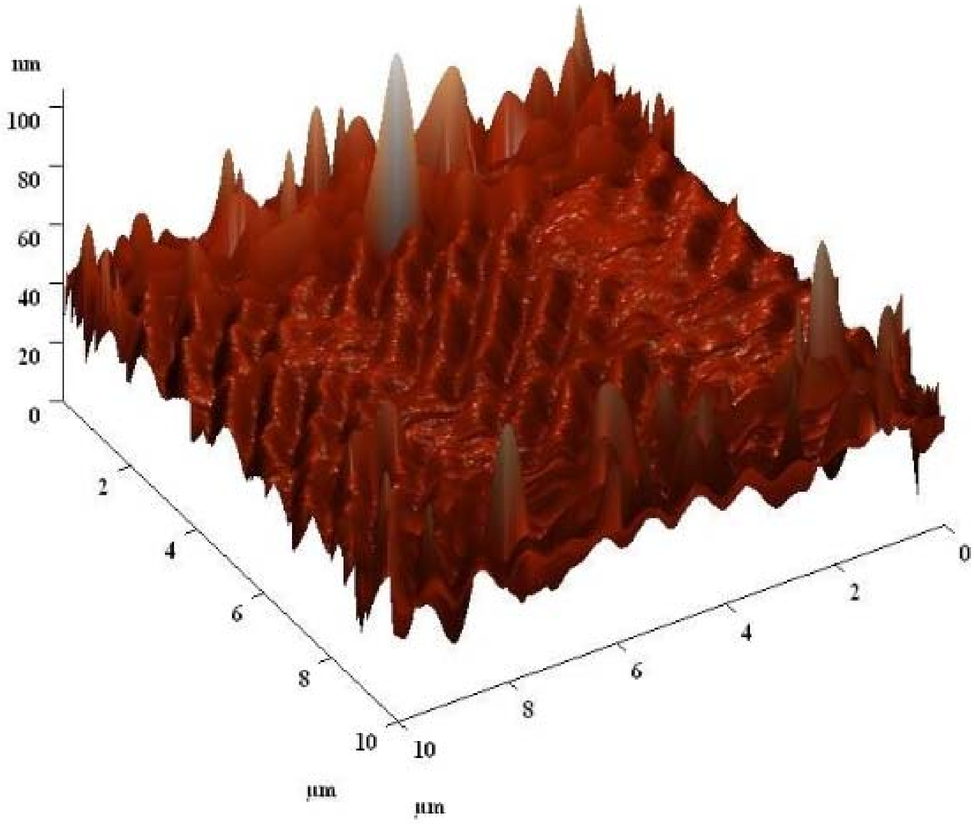


*Fig. 5: 2D AFM image of CdSe film*

### 3.2 Optical properties of CdSe thin films -

The optical band gap is measured by integral method. The spectra displayed in Fig. – 6 shows the transmittance spectra. The plot of $(\alpha h\upsilon)^2$ versus hυ for these CdSe films is presented in figure 7. This figure clearly shows the linear dependence for the value of P=1/2. This is attributed to an allowed and direct transition with direct band gap energies. The observed trend at absorption edge towards lower photon energies for the increasing film thickness could be attributed to the change in the grain size and the stoichiometric. The straight line portion is extrapolated to cut the x-axis, which gives the energy gap. All graphs show straight line portions supporting the interpretation of direct band gap for all the films.

The evaluated band gap energies are given in table - 1 which clearly indicates the dependence of band gap on thickness of the films. The estimated band gap values were in good agreement with those published in the literature for CdSe. It is observed that the band gap decreases with increase of film thickness which is in good agreement with the earlier investigations on CdSe films [18].

*Table -1 Variation in band gap with thickness.*

| Thickness (Å) | Band gap energy (Eg) eV |
|---|---|
| 1000 | 2.12 |
| 1500 | 1.86 |
| 2000 | 1.84 |
| 2500 | 1.78 |
| 3000 | 1.72 |

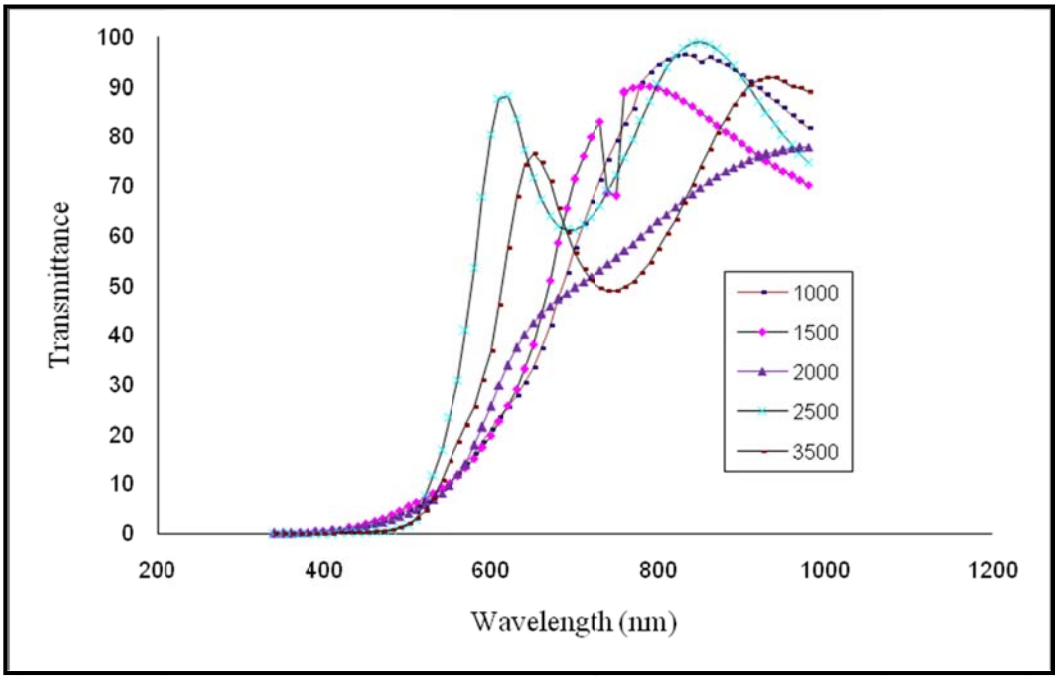


*Fig. 6: Plot of Transmittance vs. wavelength*

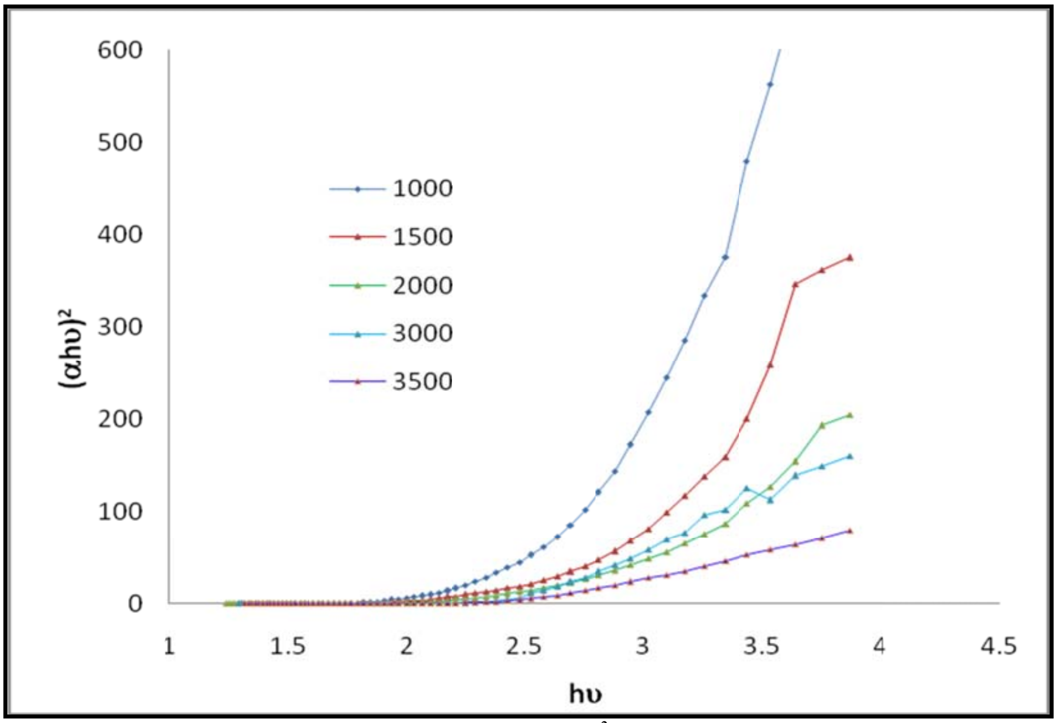


*Fig. 7: Plot of $(\alpha h\upsilon)^2$ vs. hυ*

## 4. Conclusion

CdSe thin films of different thickness have been deposited successfully on glass substrate with different thicknesses. XRD confirms that the structure of the film is polycrystalline in nature possessing hexagonal (wurtzite) structure. From SEM study it is observed that in CdSe films nano size grains were uniformly distributed over smooth homogeneous background. The particle size were found to be 12 -20 nm. From AFM study it is observed that surface image is homogeneous and well connected grains. It is observed that the band gap decreases with increase of film thickness.

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