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Studies on Nanocrystalline Cods Thin Films Prepared By Magnetron Sputtering Technique

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Abstract: Cadmium sulphide (CdS) thin films in nanocrystalline form were deposited on corning glass substrate at temperatures 240K and 260K by d. c. magnetron sputtering in argon plasma. The prepared films were characterized by XRD, optical and morphological studies. Polycrystalline nature of the films was confirmed with X-ray Diffraction data. The FESEM micrographs showed that the films contain well defined grains having size ~ 80 nm, which is well confirmed by the XRD study. The optical band gap of the prepared samples was found to be somewhat higher $\left(E_g \approx 2.45\, eV\right)$ ***compared to its bulk counterpart*** $\left(E_g \approx 2.42\ eV\right)$***, which is due to confinement of the carriers. Direct allowed transitions in the films were also found to be operative.******Keywords : Cadmium sulphide, Magnetron Sputtering, X-Ray Diffraction, Morphological studies, Carrier confinement.***

I. INTRODUCTION

During the last few decades, there has been an increasing interest in the field of nanoscience and technology because of some exotic properties exhibited by the nano-phase materials. The interest in this field stems from the point of basic research [1-3] as well as technological applications [4, 5]. Preparation of thin films of semiconductor nano-crystallites has opened the opportunity of observing the evolution of physical properties of materials with sizes. The low-dimensional systems are the most exciting class of materials because their properties lie between those of atomic/molecular clusters and their bulk counterparts. The reduction in crystallite size results in the changes in the electronic structure because of quantum confinement of the electrons. Also, there exists a close relationship between the electronic band structure and the optical response of such materials. Cadmium sulphide (CdS), a direct band gap semiconductor having bulk band gap value of 2.4 eV, has been widely recognized as a key component of the thin film-based solar photovoltaic cells technology due to its suitable optical, electrical and morphological properties. This material is the most suitable candidate to be used as buffer layer on the solar cell thin film heterostructures. CdS thin thins films are commonly used as the optical barriers for the solar radiation having wavelength below 515 nm [6, 7]. This work reports the preparation of CdS thin films in nanocrystalline form on corning glass substrates and characterization of the films by optical and optical and microstructural studies.

II. EXPERIMENTAL DETAILS

A. Cleaning of Substrates

The cleanliness of the substrate surface plays a decisive role on the nucleation and growth of the films and adhesion. The choice of cleaning technique depends on the nature of the substrate, the type of contaminants present on it and the degree of cleanliness required. Residues from manufacturing and packing, fingerprints oil and airborne particular matters are example of frequently encountered contaminants. Ordinary soda glass, corning glass substrate have been used for deposition of the CdS thin films in this work. When the glass substrate is polished during manufacturing, the surface develops an electrostatic charge, which firmly holds minute particles on the cleaned surface. In addition, a small amount of grease may also be present. To remove the contaminants, acid cleaners such as chromic acid was used to convert them to more soluble compounds. The effectiveness of solvents is probably more dependent on the ability to get the surface than their solvent action. The solvent to be used must be of high purity. Common solvents used in cleaning are methanol, acetone, trichloro- ethylene and distilled water. In this work substrates were initially cleaned in detergent solutions, dipped into chromic acid, then again in detergent solution and finally in running water. The preferred method for degreasing a substrate is to expose into solvent (methanol) vapours by keeping it above the boiling liquid in a vapour-degreasing chamber. The solvent vapour condenses on substrate and fall back into the liquid. In this way the substrate is cleaned by the action of solvent. By cleaning test it can be confirmed whether the substrate have been properly cleaned or not. A common such test is to slowly withdraw the clean substrate from a container filled with pure water, when a continuous film of water will be observed to remain on the substrate surface if the cleaning has been properly done. This method is known as *water-break* test.

Substrates cleaned by above method shows a remarkably high coefficient of friction for the liquid layer, which adheres strongly to the surface. For all type of substrates used, dipping in chromic acid, washing in soap solution and then distilled water followed by vapor degreasing was found to produce surface which were sufficiently clean for deposition of films.

B. Preparation of films

The Nano crystalline CdS thin films were prepared on glass substrates by high-pressure d.c. magnetron sputtering technique. Fused silica (area ~ 2 cm^2) were used as substrates for studying the optical and microstructural properties of the samples. The films were deposited in argon plasma at a gas pressure ~ 10 Pa. The sputtering of CdS (99.995%) targets were performed at ~ 1 kV and ~ 100 mA. The target to substrate distance was fixed at ~ 2.5 cm. Prior to actual deposition of the films, the deposition chamber was evacuated to ~ 10^{-6} torr using turbo-molecular pumping system and then flushed with argon gas several times so as to minimize the density of residual air particles in the deposition chamber and thereby reducing the impurity incorporation into the samples. Before starting the actual deposition, the targets were pre-sputtered for 10 minutes with a shutter covering the substrates. In this high-pressure sputtering regime, the particles ejected from the target suffer several collisions with the argon gas atoms and subsequently nucleate in form of ultra-fine particles on the substrate [8]. The substrate holder was a thick copper plate of thickness ~ 1 cm serving as a cold finger which could be cooled by pouring liquid nitrogen into a stainless steel dewier attached to the copper plate. Intermediate temperatures could be attained with the help of appropriate heater assembly attached to the cold finger. The temperature of the substrate could be measured and controlled (±1K) by a copper-constantan thermocouple acting as a probe of an electronic on/off temperature controller. Films were deposited at 240 K and 260 K temperatures. An increase of substrate temperature was noticed when the deposition was carried out at a stretch for a time longer than 1 minute resulting in an increase of grain size through the increased ad-atom mobility culminating in coalescence of the crystallites. To prevent coalescence, the deposition was carried out intermittently at regular intervals of time with the use of a shutter to allow the oncoming ad-atoms release their thermal energy before a second deposition was made. Two sets of samples were prepared on glass substrate with variation of temperature of the substrate – one at 240K and the other at 260K.

The structural analysis of the prepared samples was carried out by X-ray diffraction (XRD) (Philips PW 1710) with Cu-Kα radiation having wavelength $\lambda = 1.5405$ Å. The optical characteristics were studied using standard technique. The optical transmittance, reflectance and absorbance of the films were measured as a function of wavelength by a spectrometer (Hitachi U-3410). The morphological characteristics of the samples were studied using FESEM (LEO S-430i).

III. RESULTS AND DISCUSSION

A. Structural and micro-structural studies

The X-ray diffraction of the samples was studies with the help of X-ray diffractometer (Philips PW 1710). The typical diffractogram of a representative the CdS thin film deposited at 260K is shown in Fig. 1. XRD analysis clearly reveals that the film is polycrystalline in nature. The spectrum is observed to be asymmetric, which is indicative of the low dimensional structure of the sample. One can see the observed diffraction peak at the 2θ value of 26.84^0, which corresponds to the reflections from (111) plane of cubic CdS [9]. The peak height is not so much, which indicates that the preferred orientation in the (111) direction is not so predominant. The full width at half maxima (FWHM) of this peak was calculated to be 0.127^0. The grain size of the film was determined from the (111) diffraction line using Debye-Scherer formula [10].

$$D = \frac{0.9\lambda}{\beta \cos\theta} \tag{1}$$

where D is the grain size, $\lambda\left(=1.54058\,A^0\right)$ is the wavelength of the K_α radiation used for XRD study, β is the full width at half maxima (FWHM) of the peak and θ is the angle of diffraction. The value of grain size obtained from this study is ~ 75 nm.

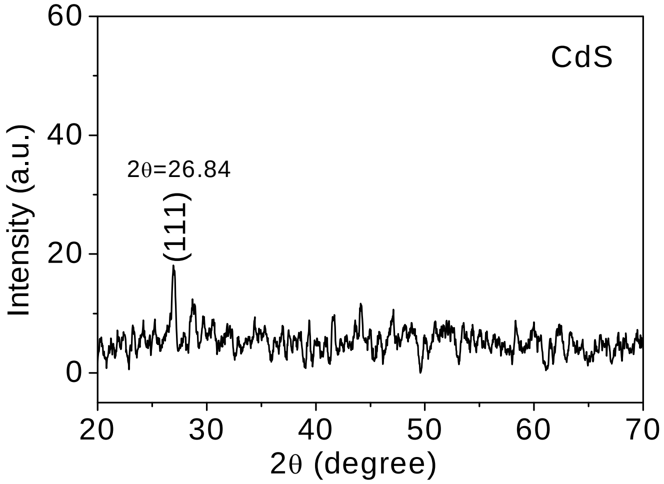

Fig.1: X-Ray Diffraction (XRD) pattern of a representative CdS thin film deposited at 260 K

Fig.2 shows the FESEM image of two representative CdS thin films deposited at 240 K and 260 K. It gives some important informations of the surface structure of the film. The images clearly reveal the formation of more or less densely packed CdS nanocrystals with average size ~ 80 nm. Thus we see that grain size obtained from FESEM micrograph is in close agreement with that obtained from the XRD analysis. From the FESEM images the agglomeration of the grains at higher deposition temperature (260K) to form relatively larger grains may be observed.

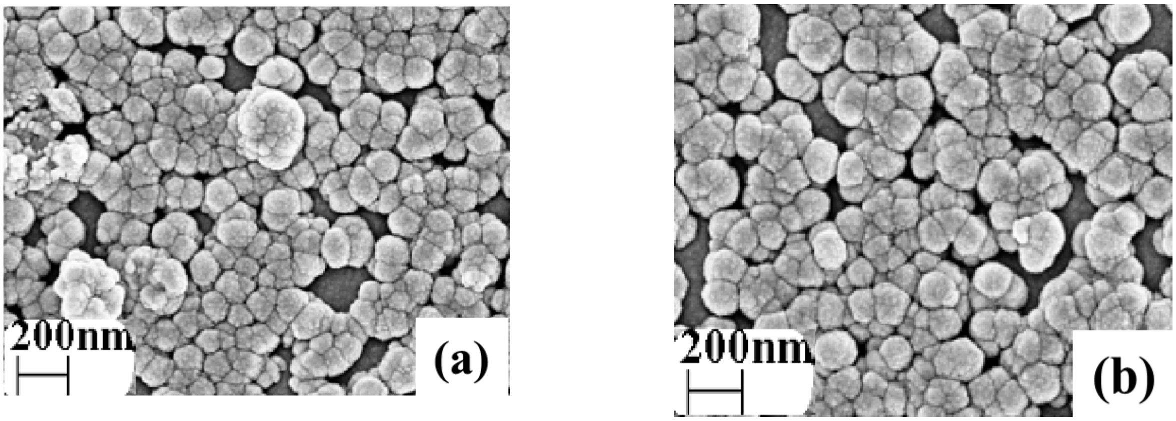

Fig.2: Field Emission Scanning Eelectron Micrograph of two representative CdS films – (a) deposited at 240K and (b) deposited at 260K

B. Optical studies

Optical transmittance, reflectance and absorbance of the films were recorded as a function of wavelength by a spectrometer (Hitachi U-3410). The transmittance versus wavelength trace for a representative CdS thin films deposited at 260K is shown in Figure 3. It may be clearly observed that spectrum is smooth with a fall around wavelength 510 nm. It is indicative of the fact that the band gap the sample is close to 2.4 eV.

In general, the absorption coefficient (α) may be written as a function of the incident photon energy ($h\nu$) so that [12]:

$$\alpha = (A/h\nu)(h\nu - E_g)^m \qquad (2)$$

where, A is constant, which is different for different transitions indicated by different values of m and E_g is the corresponding band gap. A plot of d[ln($\alpha h\nu$)]/d($h\nu$) vs. $h\nu$ indicated a divergence at a particular energy value (E_g) corresponding to an optical transition. Using this E_g value the nature of optical transition (i.e., m value) was obtained from the plots of $\ln(h\nu - E_g)$ vs. ln($\alpha h\nu$). The values of m obtained as above for different films varied within 0.48 – 0.52 which indicates a direct transition to be operative in these films. Thus, Eq. (2) may be represented as

$$\alpha = (A/h\nu)(h\nu - E_g)^{1/2} \tag{3}$$

The band gaps of the samples were then evaluated from the straight-line portion of the plots of $(\alpha h\nu)^2$ vs. $h\nu$ (shown in Figure 4). Band gap of the samples were found to vary within 2.43 – 2.46 eV. For the representative sample (shown in the figure) the band gap was found to be 2.44 eV. Band gap of bulk CdS is 2.42 eV. Here, the band gap is found to be somewhat higher than its bulk counterpart. It may be attributed to the reduction in grain size and thereby leading to confinement of the carriers.

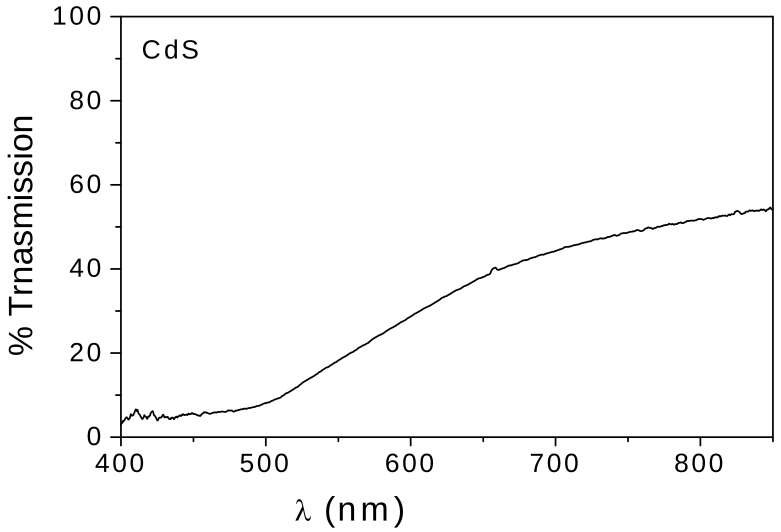

Fig.3: Transmittance versus wavelength trace of a representative CdS film prepared at 260K

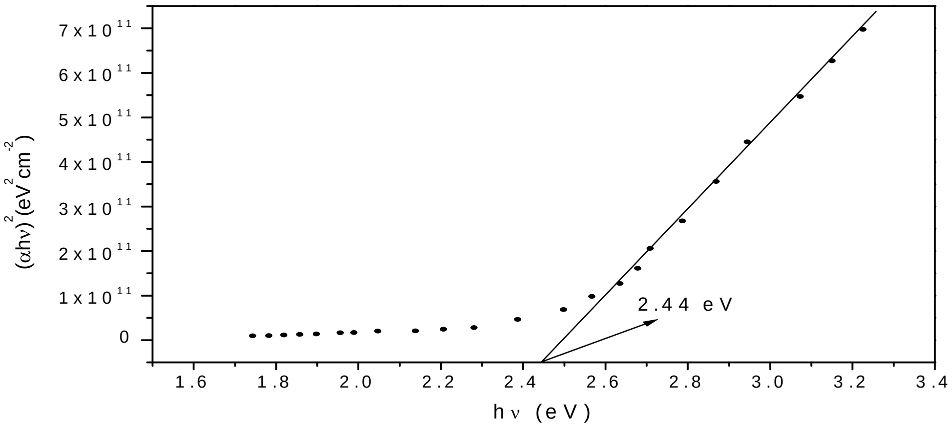

Fig.4: $(\alpha h\nu)^2$ vs. $h\nu$ graph for a representative CdS film deposited at 300 K

IV. CONCLUSIONS

A comprehensive study on structural, morphological and optical properties of the sputtered nanocrystalline CdS thin films was carried out and reported in this work. X-Ray Diffraction pattern shows that the films are of cubic structure of CdS, having orientation in the (111) direction. Measuring the FWHM of the (111) peak, grain size was calculated to be ~ 75 nm. FESEM micrographs indicate that films consist of well-defined grains with smoother surface. The grain size was found to be ~ 80 nm. Thus we see that the results of XRD study and morphological observations are in excellent agreement. Band gap the samples was determined from the optical transmittance studies and was found to vary within 2.43 - 2.46 eV. This increase in band gap is due to the reduction in grain size and consequent carrier confinement.

V. ACKNOWLEDGMENT

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