Synthesis and Characterization of Cadmium Selenide Thin Films by Vacuum Deposition Technique

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Abstract: Cadmium Selenide thin films were deposited on a cleaned glass substrate by a vacuum deposition technique. The compound of CdSe prepared by melt quench method kept in evacuated quartz ampoule at 10⁻⁵ torr pressure. The ampoule was heated at a temperature of about 1200°C for the required duration. Then the ampoule was quenched in ice cool water, in this process granules of core cadmium and powder of core selenium of Sigma Aldrich having purity 99.999% are used for ingot formation. In the present investigation uniform, thin films of CdSe of 2000Å thickness were prepared and characterized by X-ray diffraction (XRD) and scanning electron microscopy (SEM). It is found that the crystal structure of CdSe is polycrystalline in nature and cubic in structure. SEM results of the thin film showed that the grain sizes were from 300 nm to 5.0 μm.

Keywords: CdSe, thin film, a vacuum deposition technique, XRD, crystal structure, surface morphology, SEM

I. INTRODUCTION

CdSe is II-VI group material, because of its high absorption coefficient and nearly optimum band gap energy CdSe is a widely used photovoltaic material [1]. A direct band gap range of CdSe is between 1.65 eV to 1.84 eV [2, 4]. Cadmium Selenide (CdSe) is a solid binary compound of cadmium and selenium which have different crystal structures like hexagonal (wurtzite), cubic (zinc blende) and cubic rock-salt [3, 8]. Because of the low production cost of CdSe thin films, the use of polycrystalline semiconducting thin film has attracted much interest in various electric and optoelectronic devices in the last decade [5]. CdSe is an important material for the development of various modern technologies and having the number of applications like thin film transistors, light emitting diodes, photo-detectors, light amplifiers, lasers, gas sensors, large-screen liquid crystal display and photoluminescence response [2, 6]. Cadmium Selenide thin films were developed by the number of deposition methods and techniques such as thermal evaporation technique, molecular beam epitaxy, electrodeposition, spray pyrolysis, successive ionic layer adsorption, chemical bath deposition and reaction method, etc [18, 20]. Among these methods, a vacuum pump deposition technique is an attractive method which has been successfully used widely for the preparation of elemental, binary, intermetallic and ternary thin films [6]. Today semiconductor industry relies on vacuum deposited thin film technology. In this paper, we have discussed the synthesis and characterization of Cadmium Selenide (CdSe) thin films by using a vacuum deposition method. We have prepared CdSe thin films on a glass substrate by vacuum pump deposition technique at a 1200°C temperature and at the 10⁻⁵ torr pressure in Hind Hivac 12A4-D vacuum coater machine. The compound ground carefully to get fine powder of CdSe. All the films of CdSe system were deposited under the same experimental conditions [5]. To study crystal structure and surface morphology, deposited thin films of CdSe were characterized by X-ray diffraction and scanning electron microscopy (SEM). In recent years special attention has been given to the investigating more and more optoelectronic properties of CdSe thin films in order to improve the performance of devices and also for finding new applications of CdSe thin films [11, 13].

II. EXPERIMENTAL

A. Thin Film Deposition

The glass slides were first cleaned in a dilute solution of detergent to remove the impurities on the surface of the slides. Then rinsed with distilled water to remove the layer of detergent solution from the substrate surface. The glass slides were cleaned in an acetone solution for 10 minutes and left to dry [2, 10].

The CdSe powder was prepared from granules pure of Cd and pure Se powders of Sigma Aldrich having a purity of 99.999%. The materials were weighted according to their atomic percentage in equal ratio and send to ingot formation at Anand University, Gujarat. The mixture was heated inside evacuated closed quartz tube in an electronic furnace and melted at 1200°C to increase the homogeneity of the compound, then the melting compound of CdSe was cooled rapidly (Quenching) using water [5]. Then the compound ground carefully to get fine powder of CdSe. The powder of CdSe was placed in a molybdenum boat to prepare the thin films of different thickness. Then the heat was produced by passing an electrical current through the boat. The CdSe powder was evaporated from a molybdenum boat on a glass substrate with a deposition rate (2A/s). The CdSe film was prepared using a vacuum deposition method using the Hind Hivac 12A4-D vacuum machine. This system basically consists of a rotary pump, diffusion pump,
bell-jar vacuum chamber with gauges, deposition sources, substrate holder and other accessory equipment. The lowest attainable pressure in this system is around 10⁻⁶ Torr [9, 14, 19]. As soon as the pressure inside the vacuum chamber reached at 5x10⁻⁶ Torr, the source was heated up to 600°C, which is the starting temperature for the evaporation of the CdSe. The substrate temperature was kept at a lower value as possible by a continuous supply of cold water circulation [10]. The thickness and the growth rate of the films were measured by XTM/2 deposition monitor. The deposition rate was kept constant at 2 Å/sec through all of the growth cycles. To avoid oxidation of the films, the system was allowed to cool down to room temperature for 20 minutes after completing the deposition process, without disturbing the vacuum conditions [12]. The vacuum chamber has been cleaned and the grease has been applied to rubber gasket before each run to attain the desired vacuum in the chamber. The uniform, porous free and well adhered thin films with the glass plates have been obtained [13].

B. Characteristics
By using the X-rays of known wavelength and measuring the angle of diffraction of the most intense peak, the interplanar spacing ‘d’ in the crystal has been determined by using the formula given as,

\[ \lambda n = 2d \sin \theta \]

where ‘d’ is lattice spacing, ‘\( \lambda \)’ is a wavelength of monochromatic X-rays, ‘\( \theta \)’ is the angle between the incident beam and the planes (hkl), ‘n’ is the order of reflection (n = 1, 2, 3,……)[12, 22].

The lattice parameter (a) is determined for the cubic structure by using the following expression[7]

\[ \frac{1}{a^2} = \frac{h^2 + k^2 + l^2}{a^2} \]

Surface morphology of the films was studied by scanning electron microscope (SEM). SEM is a promising technique for the topography study of samples, which gives valuable information regarding the growth mechanism, shape and size of the particles and/or grains [5, 6].

III. RESULTS AND DISCUSSIONS
The X-ray diffraction (XRD) technique is the most promising analytical tool for the structural analysis of the thin film [15]. ‘d’ value calculated using Bragg’s equation for the known value of \( \theta \), \( \lambda \), and n. The x-ray diffraction data thus obtained is compared with the Joint Committee on Powder Diffraction Standard (JCPDS) data cards [16]. This x-ray diffraction data can also be used to determine the particle size, structural factors, residual stresses, Miller indices, etc [17]. The structural properties of the CdSe films were studied by the X-ray diffractometer (XRD) using Cu Kα radiation (\( l =1.54060 \) Å) [2]. Scan angle used 2\( \theta \) is from 5° to 80° at 40 kV voltage and 40 mA current.

The XRD patterns of crystalline CdSe thin film is shown in Figure 1

![CdSe 2000 (Coupled TwoTheta/Theta)](image)

Figure 1: XRD spectrum obtained for CdSe films of thickness 2000 Å
Table 1. X-ray diffractogram (XRD) data of bulk Cadmium Selenide sample of thickness 2000 Å

| [hkl] values from JCPDS data | d(A₀) values from JCPDS data | Observed values of d(A₀) | Observed (2θ)° values of peaks | intensity | Calculated values of a(A₀) |
|-----------------------------|-----------------------------|-------------------------|-------------------------------|----------|---------------------------|
| 100                         | 3.720                       | 3.724                   | 23.870                        | 584      | 3.724                     |
| 002                         | 3.516                       | 3.516                   | 25.310                        | 1195     | 7.032                     |
| 101                         | 3.290                       | 3.293                   | 27.050                        | 570      | 4.653                     |
| 102                         | 2.554                       | 2.555                   | 35.090                        | 301      | 5.713                     |
| 110                         | 2.151                       | 2.151                   | 41.960                        | 261      | 3.039                     |
| 103                         | 1.980                       | 1.980                   | 45.770                        | 307      | 6.261                     |
| 200                         | 1.861                       | 1.863                   | 48.830                        | 216      | 3.726                     |
| 112                         | 1.834                       | 1.834                   | 49.670                        | 221      | 4.492                     |
| 201                         | 1.800                       | 1.800                   | 50.660                        | 189      | 4.024                     |
| 202                         | 1.645                       | 1.645                   | 55.820                        | 159      | 4.652                     |
| 203                         | 1.456                       | 1.457                   | 63.830                        | 190      | 5.253                     |
| 210                         | 1.407                       | 1.406                   | 66.440                        | 215      | 3.143                     |
| 211                         | 1.380                       | 1.380                   | 67.820                        | 149      | 3.380                     |
| 105                         | 1.312                       | 1.311                   | 71.960                        | 176      | 3.211                     |
| 212                         | 1.305                       | 1.305                   | 72.350                        | 162      | 3.915                     |
| 300                         | 1.241                       | 1.239                   | 76.820                        | 137      | 3.717                     |
| 213                         | 1.205                       | 1.204                   | 79.520                        | 138      | 4.504                     |

The SEM pictures of CdSe films on glass substrates are shown in Figure 2. The oval and flower-like shape of the particles can be observed easily in high magnification micrographs. SEM observations show the crystalline growth for the films deposited [21]. in the present work, investigations are done by using a LEICA S440 SEM instrument at K.B.C.N.M.U., Jalgaon.
Figure 2: SEM pictures taken for CdSe films with different x content (a) 300 nm, x = 150k (b) 400 nm, x = 120k (c) 500 nm, x = 60k (d) 500 nm, x = 60k (e) 500 nm, x = 60k (f) 500 nm, x = 60k (g) 1 µm, x = 30k (h) 5µm, x = 60k

IV. CONCLUSION

It is found that the deposited CdSe films are highly oriented with cubic zinc blende structure and the preferred crystal orientation is (100) and (002) planes. The lattice parameters are almost matching with the JCPDS data of CdSe. The SEM result shows that the sizes of grain were from 300 nm to 5 µm. According to the surface morphology, the SEM image of the deposited film reveals the uniform surface in nature.

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