Chapter IV
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Characterization of cadmium indium selenide (CdIn$_2$Se$_4$) thin films

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4.1 Introduction

Recently, ternary compounds of II-III-VI group attract many researchers due to their applications in optoelectronic devices and solar cells [1-3]. Among II-III-VI group ternary compounds, Cadmium indium selenide (CdIn$_2$Se$_4$) is one of the interesting semiconductors due to its optical absorption property with a narrow band gap and a low electrical resistance [4-5]. Because of its narrow band gap, CdIn$_2$Se$_4$ compound is also widely used in optoelectronic devices [2], non-linear optics [6], semiconducting devices, radiation detectors, laser materials, thermoelectric devices, solar energy converters etc [7-8]. CdIn$_2$Se$_4$ is a direct band gap semiconductor with an energy gap of 1.73 eV which make them interesting for solar cells through photoelectrochemical route [2, 9]. The basic requirements of good thin film photoelectrode for photoelectrochemical cells are low resistivity and large grain size. Large grain size leads to reduction of grain boundary area of thin films with important consequences for efficient energy conversion. The low resistivity of photoelectrode is required to minimize the series resistance of PEC cell. Due to its wide applications, this compound is always prepared as thin film via many methods such as electro-deposition [10], slurry pasting technique [11], vacuum evaporation [3] and spray pyrolysis [12-13]. The deposition of CdIn$_2$Se$_4$ thin films have been carried out using relatively inexpensive, simple and convenient for large area deposition, spray pyrolysis technique. Furthermore, synthesis of CdIn$_2$Se$_4$ thin film by spray pyrolysis technique obtained a good photovoltaic activity. An obtained thin film has high crystallinity with low electrical resistivity, which leads to high thermoelectric power [14].

The attempts have been made to prepare CdIn$_2$Se$_4$ thin films by simple and low cost chemical spray pyrolysis technique. The advantage of the technique is that just by varying the concentration of precursor and substrate temperature; it is possible to control stoichiometry of the deposits. The present study deals with the effect of substrate temperature and solution concentration on structural, morphological, optical, and electrical properties of spray deposited CdIn$_2$Se$_4$ thin films.
4.2 Experimental

4.2.1 Thin film deposition

4.2.1.1 Substrate cleaning

The micro slides and FTO glass substrates were used for CdIn$_2$Se$_4$ thin film deposition by spray pyrolysis technique. The substrates were cleaned using procedure discussed in section 3.2.1.2.

4.2.1.2 Preparation of solutions

Solution of CdIn$_2$Se$_4$ was prepared by dissolving appropriate amounts of ingredients into distilled water to get the required volume as discussed in section 3.2.1.3.

4.2.1.3 Deposition of CdIn$_2$Se$_4$ thin films

In order to find optimized conditions for deposition of CdIn$_2$Se$_4$ thin films, initially the deposition was carried out by varying one of the parameter as substrate temperature and keeping the others at fixed value and for optimization of solution concentration, deposition was carried out by varying solution concentration and keeping others at fixed value.

4.2.2 Characterization of CdIn$_2$Se$_4$ thin films

4.2.2.1 Effect of substrate temperature

1. Thickness measurement

The thickness of prepared films was determined by gravimetric method using the relation (1.1).

2. X - ray diffraction (XRD)

The structural characterization of CdIn$_2$Se$_4$ thin films was carried out by analyzing the X-ray diffraction patterns obtained using Philips X-diffractometer Model PW-1710 (λ= 1.05406 Å for Cu-Kα radiation).

3. Optical absorption

Optical absorption studies were carried out using a UV-VIS-NIR spectrophotometer in wavelength range of 350- 850 nm.

4. Electrical resistivity
Two point d.c. probe method was used to study the variation of resistivity with temperature as discussed in section 1.3.6 a.

5. Thermoelectric power (TEP)

The type of conductivity shown by the spray deposited CdIn$_2$Se$_4$ thin films was determined from TEP measurement as discussed in section 1.3.6 b.

4.2.2.2 Effect of solution concentration

Various characterizations viz. thickness measurement, XRD, optical absorption and electrical properties were carried out as discussed in section 4.2.2.

4.3 Results and discussion

4.3.1 Effect of substrate temperature

In the spray pyrolysis method, the starting materials required to form the desired compound are in the form of solutions, which are then sprayed onto the preheated substrates, resulting in the formation of thin films on the upper surface of the substrate. When droplets of above-sprayed solution reach on the hot substrate, owing to pyrolytic decomposition of the solution, well adherent and dark brown coloured films of CdIn$_2$Se$_4$ formed on the upper surface of the substrates. The film adherence was tested by scotch tape method [15].

![Graph showing variation of $I_{sc}$ and $V_{oc}$ with substrate temperature for CdIn$_2$Se$_4$ thin films](image)

Fig. 4.1 Variation of $I_{sc}$ and $V_{oc}$ with substrate temperature for CdIn$_2$Se$_4$ thin films
The variation of $I_{sc}$ and $V_{oc}$ with substrate temperature depicted in fig. 4.1 shows the $I_{sc}$ and $V_{oc}$ goes on increasing with substrate temperature; become maximum at substrate temperature of 280°C and then decrease with further increase in substrate temperature. This variation may be attributed to the microstructural and compositional changes taking place during the deposition at various substrate temperatures. The relatively higher values of $I_{sc}$ and $V_{oc}$ at 280°C substrate temperature may due to the formation of uniform & stoichiometric films [16].

1. Thickness variation

Variation of thickness versus substrate temperature is shown in Fig. 4.2. The thickness increases with increase in substrate temperature, attains the maximum value at 280°C and then decreases for further increase in substrate temperature.

![Fig. 4.2 Variation of film thickness of CdIn$_2$Se$_4$ thin films with substrate temperature](image)

The films were deposited at four different substrate temperatures viz. 240, 260, 280 and 300°C. From the plot, initially it is seen that the thickness increases with increase in temperature, attains a terminal thickness at 280°C and then
decreases for higher substrate temperatures. This can be explained as follows: initially, at lower substrate temperature (e.g. 240°C), the temperature may not be sufficient to decompose the sprayed droplets of solution and therefore, resulted into low thickness. At 280°C, the decomposition occurs at the optimum rate resulting in maximum thickness of about 1.24 µm. The decrease in film thickness at higher substrate temperatures may be due to higher evaporation rate of initial ingredients of the solution. Similar results have also been observed for spray deposited antimony selenide alloy films [17].

2. X-ray diffraction (XRD)

The as-grown CdIn$_2$Se$_4$ thin films deposited at various substrate temperatures were characterized by the XRD technique with Cu-Kα radiation with the help of Ni filter. The XRD patterns obtained for the films grown on bare micro slides glass plates were studied in 2θ range of 10–100°. The XRD patterns (figure 4.3) show that films are polycrystalline [12]. It is seen that the plane (202) of CdIn$_2$Se$_4$ appears with higher peak intensity in all diffractograms. Besides it, (111), (113), (224) planes are also observed in all diffractograms. However, the (111) peak of CdSe is also present in all films. The intensity of CdIn$_2$Se$_4$ peaks is comparatively higher for the 280°C substrate temperature. Table 4.1 compares the observed and standard ‘d’ values of the CdIn$_2$Se$_4$ thin films. A matching of observed and standard ‘d’ values confirm that the deposited films are of CdIn$_2$Se$_4$ having cubic crystal structure [18]. The calculated value of lattice constant 5.824Å agrees well with the standard values of single crystal of CdIn$_2$Se$_4$ [14].
Fig. 4.3 X-ray diffraction patterns for CdIn$_2$Se$_4$ films deposited at various substrates temperatures
Table 4.1 Observed and standard ‘d’ values for the CdIn$_2$Se$_4$ thin film deposited at various substrate temperatures

<table>
<thead>
<tr>
<th>Sr. No.</th>
<th>Standard ‘d’ values (Å)</th>
<th>Observed ‘d’ values (Å)</th>
<th>(hkl) planes</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>240°C</td>
<td>260°C</td>
</tr>
<tr>
<td>1</td>
<td>3.3700</td>
<td>3.3607</td>
<td>3.3514</td>
</tr>
<tr>
<td>2</td>
<td>2.9200</td>
<td>2.9313</td>
<td>-</td>
</tr>
<tr>
<td>3</td>
<td>2.0600</td>
<td>2.0505</td>
<td>2.0516</td>
</tr>
<tr>
<td>4</td>
<td>1.7510</td>
<td>1.7746</td>
<td>1.7766</td>
</tr>
<tr>
<td>5</td>
<td>1.4520</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>6</td>
<td>1.3340</td>
<td>1.3395</td>
<td>1.3425</td>
</tr>
<tr>
<td>7</td>
<td>1.1880</td>
<td>1.1822</td>
<td>1.1836</td>
</tr>
<tr>
<td>8</td>
<td>1.1200</td>
<td>1.1182</td>
<td>1.1189</td>
</tr>
<tr>
<td>9</td>
<td>1.0810</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>10</td>
<td>1.0280</td>
<td>-</td>
<td>1.0274</td>
</tr>
</tbody>
</table>

It is found that as the substrate temperature increases, crystallite size also increases and attains maximum (66 nm) for the optimized substrate temperature (Table 4.2). Increase in crystallinity with the substrate temperature is due to the sufficient supply of thermal energy for recrystallization and growth of the grains.
Further decrease in crystallite size after 300°C is due to non-uniformity and low thickness of the films.

<table>
<thead>
<tr>
<th>Sr. No.</th>
<th>Substrate temperature (°C)</th>
<th>Crystallite size (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>240</td>
<td>42</td>
</tr>
<tr>
<td>2</td>
<td>260</td>
<td>53</td>
</tr>
<tr>
<td>3</td>
<td>280</td>
<td>66</td>
</tr>
<tr>
<td>4</td>
<td>300</td>
<td>57</td>
</tr>
</tbody>
</table>

Table 4.2 Variation of crystallite size with substrate temperature

3. Electrical resistivity

The variation of log $\rho$ versus $(1000/T)$ for the films deposited at various substrate temperatures, shown in fig. 4.4. It reveals that the resistivity variation obeys the relation,

$$\rho = \rho_0 \exp\left(\frac{E_a}{kT}\right)$$

(4.1)

where $E_a$ is the activation energy and $k$ is the Boltzmann constant. It is well known that the resistivity of a semiconducting material strongly depends on the temperature, carrier concentration and mobility. In semiconductor, carrier concentration is rapidly increasing as a function of temperature. It increases due to thermal excitation of electrons, either from imperfections or across the bandgap. It is clear from fig. 4.5, the resistivity decreases with temperature [19], and showing semiconducting behaviour of the CdIn$_2$Se$_4$ thin films. A room temperature resistivity of the film deposited at 240°C is found to be of the order of $10^4$ Ωcm, which decreases to $10^2$ to $10^3$ Ωcm for the sample deposited at 280°C and it further increases at 300°C substrate temperature. Activation energy estimated for different substrate temperatures have been tabulated in Table 4.4. The activation energy increases with substrate temperature from 0.048 to 0.123 eV.
Table 4.4 Variation of activation energy with different substrate temperatures

<table>
<thead>
<tr>
<th>Substrate Temperature (°C)</th>
<th>Activation energy (eV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>240</td>
<td>0.048</td>
</tr>
<tr>
<td>260</td>
<td>0.083</td>
</tr>
<tr>
<td>280</td>
<td>0.098</td>
</tr>
<tr>
<td>300</td>
<td>0.123</td>
</tr>
</tbody>
</table>

Fig. 4.4 Plot of log $\rho$ against 1000/T for CdIn$_2$Se$_4$ thin films deposited at various substrate temperatures

4. Thermoelectric power (TEP)

The thermoelectric power (TEP) is defined as the ratio of thermally generated voltage to the temperature difference across a piece of semiconductor. The type of conductivity exhibited by the spray deposited CdIn$_2$Se$_4$ thin films is
determined from TEP measurement. The polarity of thermally generated voltage at the hot end is positive indicating that the films are of n-type. Fig. 4.5 shows the variation of thermo emf with temperature difference for the films deposited at various substrate temperatures. It is seen that the thermo emf varies linearly with substrate temperature [19].

![Graph showing variation of thermo-emf with temperature difference for CdIn$_2$Se$_4$ thin films deposited at various substrate temperatures](image)

**Fig. 4.5 Variation of thermo-emf with temperature difference for CdIn$_2$Se$_4$ thin films deposited at various substrate temperatures**

Relatively higher thermo emf is observed for the film prepared at 280$^\circ$C. The Seebeck coefficient or TEP depends upon the location of Fermi energy in the material, type of scattering mechanism & charge carries encounter. Increase in thermo-emf show Fermi energy shifts into the energy gap from the bottom edge of conduction band. It concludes the carrier concentration is inversely proportional to the Seebeck coefficient. Thus, relatively higher TEP for the sample prepared at 280$^\circ$C is due to higher crystallinity and crystallite size of the film, low carrier concentration and thus confirms the optimized substrate temperature of 280$^\circ$C.

5. **Optical absorption**

Optical absorption studies of CdIn$_2$Se$_4$ films deposited on the glass substrates were carried out in the wavelength ($\lambda$) range 350–850 nm at room
temperature. The variation of absorbance ($\alpha_t$) versus wavelength ($\lambda$) is shown in Fig. 4.6 (a). It is seen that the optical absorbance coefficient is a function of photon energy. It also shows higher absorption on the shorter wavelength side and the presence of an absorption edge.
The nature of transition involved can be determined on the basis of dependence of $\alpha$ on $h\nu$. For allowed direct band gap electronic transition the absorption coefficient $\alpha$ is given by [20],

$$\alpha = \left( \frac{A}{h\nu} \right) \left( h\nu - E_g \right)^{1/2} \quad (4.1)$$

where $\alpha$ is the absorption coefficient and it increases sharply at photon energies corresponding to the band gap for direct allowed transitions; A the constant depending upon the transition probability for direct transition, $\nu$ the incident photon frequency, $h$ the Plank’s constant and $E_g$ is the band gap energy. The plot of $(\alpha h\nu)^2$ versus $h\nu$ shown in Fig. 4.6 (b), is linear one indicating the direct nature of optical transition for the semiconducting CdIn$_2$Se$_4$. The intercept on the energy axis at $\alpha = 0$ yields $E_g$ to be 1.92 eV.
4.3.2 Effect of solution concentration

The depositions of CdIn$_2$Se$_4$ thin films were carried out for various solution concentrations 0.005 M, 0.01 M, 0.0125 M, 0.0150 M, 0.0175 M and 0.02 M at the optimized substrate temperature of 553K. The film formation does not observe below the concentration 0.005 M. This may be due to extra low concentration of the precursor solution. At high concentration the complete thermal decomposition of the solution does not take place. However, the CdIn$_2$Se$_4$ thin films deposited at intermediate (0.125) solution concentration (0.005M-0.02 M) are uniform and adherent to the glass substrates with dark brown in colour. The mechanism of formation of CdIn$_2$Se$_4$ is described by the following equation [19].

$$\text{Cd}^{2+} + 2\text{In}^{3+} + 4\text{Se}^{4+} + 9\text{e}^- \rightarrow \text{CdIn}_2\text{Se}_4$$  \hspace{1cm} (1)

Optimization of preparative parameters for deposition of good-quality and stoichiometric CdIn$_2$Se$_4$ thin films is most essential. Optimization of preparative parameters is carried out by noting the maximum values of $I_{sc}$ and $V_{oc}$ of the PEC cell. Fig. 4.7 shows the variation of $I_{sc}$ and $V_{oc}$ with solution concentration;
from the graph, it is observed that $I_{sc}$ and $V_{oc}$ increases with increase in solution concentration, attains maximum value for film deposited at 0.0125 M solution concentration indicating probably a better formation of stoichiometric semiconducting compound. The lower values of $I_{sc}$ and $V_{oc}$ may be originated due to increase in resistivity and non-stoichiometric growth of CdIn$_2$Se$_4$ thin films due to insufficient thermal energy provided during the deposition [21]. The PEC cell with configuration CdIn$_2$Se$_4$/0.1M polysulfide/graphite is used to check the type of conductivity exhibited by CdIn$_2$Se$_4$ thin films. The polarity of dark voltage is negative toward CdIn$_2$Se$_4$ photoelectrode and positive toward the graphite electrode for all films showing n-type semiconducting behavior.

1. Thickness measurement

![Graph](image)

**Fig. 4.8 Variation of film thickness of CdIn$_2$Se$_4$ thin films with solution concentration at optimized substrate temperature (280°C)**

A detailed kinetics study was carried out by changing the solution concentration. Fig. 4.8 shows the growth kinetics of CdIn$_2$Se$_4$ thin film deposited for various solution concentrations from 0.005 to 0.02 M. The film thickness
was slowly built up at the initial stages linearly and finally gets to maximum of about 1.24 µm at 0.0125 M concentration and then decreases for higher concentrations. The film deposited at 0.0125 M was found to be uniform, thick and well adherent to the substrate. At higher solution concentrations (>0.0150 M), film thickness decreases due to powdery nature of the film.

2. X-ray diffraction

![X-ray diffraction patterns for CdIn₂Se₄ films deposited at various solution concentrations at optimized substrate temperature (280°C)](image)

Fig. 4.9 X-ray diffraction patterns for CdIn₂Se₄ films deposited at various solution concentrations at optimized substrate temperature (280°C)

The XRD patterns for the films deposited for various solution concentrations are as shown in fig. 4.9. The films are polycrystalline with strong orientation along (202) plane having cubic crystal structure. Some weak reflections such as (111), (113), (313), (205), (404), (115) and (224) are also observed in all patterns. However CdSe phase having (111) orientation is also seen in patterns. Table 4.5 depicts the calculated and standard d values [18]; it is
seen that films are cubic. As the solution concentration increases, crystallinity of the films increases may be due to the sufficient increase in growth of the grains. Relatively higher peak intensity has been observed for the films deposited at 0.0125 M solution concentration. The peak intensity decreases for higher concentrations is attributed to the non-uniform and porous nature of films. The crystallite size for the film deposited at various solution concentrations at optimized substrate temperature of 553K varies from 52-66 nm.

Table 4.5 Comparison of observed and standard ‘d’ values for the CdIn$_2$Se$_4$ thin film deposited at various solution concentrations

<table>
<thead>
<tr>
<th>Sr. No</th>
<th>Standard ‘d’ values</th>
<th>Observed ‘d’ values (Å)</th>
<th>(h k l) planes</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0.005 M</td>
<td>0.01 M</td>
<td>0.0125 M</td>
</tr>
<tr>
<td>1</td>
<td>3.3640</td>
<td>3.3502</td>
<td>-</td>
</tr>
<tr>
<td>2</td>
<td>2.0600</td>
<td>2.0474</td>
<td>2.0485</td>
</tr>
<tr>
<td>3</td>
<td>1.7510</td>
<td>1.7549</td>
<td>1.7498</td>
</tr>
<tr>
<td>4</td>
<td>1.3340</td>
<td>1.3350</td>
<td>1.3312</td>
</tr>
<tr>
<td>5</td>
<td>1.1839</td>
<td>1.1815</td>
<td>1.1831</td>
</tr>
<tr>
<td>6</td>
<td>1.1200</td>
<td>1.1155</td>
<td>1.1185</td>
</tr>
</tbody>
</table>
Fig. 4.10 Variation of crystallite size for various solution concentrations for CdIn$_2$Se$_4$ thin films at optimized substrate temperature (280°C)

3. Scanning electron microscopy

Fig. 4.11 SEM of CdIn$_2$Se$_4$ thin film deposited at optimized solution concentration 0.0125M and substrate temperature 280°C at 10000x magnification
Fig. 4.11 shows the surface morphological structure of the films deposited at optimized 0.0125 M solution concentration and substrate temperature 280°C. At optimized concentration, periodically and continuously distributed well covered growth of film is observed. It shows smooth, uniform and pinhole free densely packed grains on the surface. The compositional analysis of the CdIn$_2$Se$_4$ thin films is carried out by EDAX technique for the films deposited at 0.001, 0.0125 and 0.0150M solution concentrations. At 0.0125M concentration films are found to be stoichiometric. The elemental analysis of CdIn$_2$Se$_4$ thin film deposited at various solution concentrations at optimized substrate temperature 280°C is tabulated in Table 4.6.

![Graph showing compositional analysis of CdIn$_2$Se$_4$ thin film deposited at 0.0125 M optimized solution concentration & optimized substrate temperature (280°C)](image)

**Fig. 4.12** Compositional analysis of CdIn$_2$Se$_4$ thin film deposited at 0.0125 M optimized solution concentration & optimized substrate temperature (280°C)
Table 4.6 Elemental analysis of CdIn$_2$Se$_4$ thin films deposited at various solution concentrations and 280°C substrate temperature

<table>
<thead>
<tr>
<th>Elements/Solution concentration</th>
<th>Se (at%)</th>
<th>Cd (at%)</th>
<th>In (at%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.01M</td>
<td>49.64</td>
<td>13.42</td>
<td>36.94</td>
</tr>
<tr>
<td>0.0125M</td>
<td>58.44</td>
<td>14.36</td>
<td>27.20</td>
</tr>
<tr>
<td>0.0150M</td>
<td>50.48</td>
<td>17.36</td>
<td>32.16</td>
</tr>
</tbody>
</table>

4. Electrical resistivity

![Graph showing log(ρ) against 1000/T for CdIn$_2$Se$_4$ thin films deposited at various solution concentrations at optimized substrate temperature (280°C)](image)

The variation of log $\rho$ versus 1000/T for the films deposited at various solution concentrations is shown in fig. 4.13. It is observed that the films deposited at 0.0125 M solution show the lower resistivity than other films. It shows that, as the solution concentration increases, the resistivity of the films
decreases up to 0.0125 M and then increases for higher solution concentrations, because carrier concentration is rapidly increasing with solution concentration. After 0.0125M solution concentration the substrate temperature 553K is insufficient to complete decomposition of the sprayed droplets of the solution results into non-uniform and porous films.

Room temperature resistivity (fig. 4.13) and activation energy (fig. 4.14) for the film deposited at 0.0125M concentration and substrate temperature 553K is found to be of the order of $10^2\,\Omega\text{cm}$ and 0.098 eV respectively. As the crystallinity increases, grain size increases thereby minimizing the grain boundary scattering losses and defects in the films. So the number of electron trap states reduces and hence the carrier concentration increases. For the films deposited at relatively low solution concentrations (< 0.0125 M) due to lower grain size of CdIn$_2$Se$_4$ particles, scattering at the grain boundaries result into higher resistivity of the films. As thickness increases grain boundary effect decreases leading to large mean free path, which in turn decreases, the scattering and thus the resistivity. The mean free path decreases up to 0.0125M and then increases with solution concentrations might be due to the increase in the conductivity and carrier concentration of the films.

![Fig. 4.14 Variation in activation energy w. r. t. solution concentrations at optimized substrate temperature (280°C)](image_url)
5. Thermoelectric power

Fig. 4.15 shows the variation of thermo emf with temperature difference for films deposited at various solution concentrations. It is seen that the thermo emf increases with solution concentration up to 0.0125 M and then decreases for higher concentrations and varies linearly with difference in temperature. The polarity of thermally generated voltage at the hot end is positive indicating that the films are of n-type. Relatively higher thermo-emf is observed for the film prepared at 0.0125 M solution concentration it may be due to higher crystallinity of the film.

Fig. 4.15 Variation of thermo-emf for different solution concentrations of CdIn$_2$Se$_4$ thin films deposited at optimized substrate temperature (280°C)
References
data file no. 17-356 (1996)