Effect of Zinc concentration in CdS thin films deposited in complexing agent by chemical bath deposition: structural, optical, morphological and topographical studies

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Abstract
ZnCdS thin films were prepared by varying the concentration of Zinc (Zn) in the presence of complexing agent, tri-ethanol amine (TEA) and deposited on a glass substrate by chemical bath deposition (CBD) method. The influence of Zn on structural, optical and morphological properties of ZnCdS thin films was studied. From the x-ray diffraction pattern (XRD), the hexagonal structure of ZnCdS thin films was confirmed with an average size of the crystallite (30–36 nm). The optical properties of ZnCdS thin film were investigated using UV–visible absorption and photoluminescence spectral analysis (UV-Vis and PL). The optical studies of thin films indicated that the band gap increased from 1.85 eV to 2.81 eV with increasing the concentration of Zn. Transmission spectra showed a blue shift with increasing the concentration of Zn. The microstructure and morphology of ZnCdS thin films were studied by scanning electron microscope (SEM). The average (Ra) and RMS roughness was calculated by atomic force microscope (AFM). From the results, the nano ZnCdS thin films prepared using various concentrations of Zn could be useful for optoelectronic and device applications.

1. Introduction

Nowadays both binary Cadmium sulfide and Zinc Sulphide is the most important II-VI group semiconductor material used in many optoelectronic applications [1]. The material exists in Zn doped Cadmium sulfide thin film could be used for photoconductors, light emitting diodes solar cell, field effect transistors, sensor [2]. The ZnCdS thin films proved to be a promising material for solar cell applications [3]. The ZnCdS thin film has been deposited through various methods such as chemical bath deposition [4], spray pyrolysis [5], sol-gel [6], SILAR [7], sputtering [8], pulsed laser deposition and electron beam evaporation [9]. Among the various methods, chemical bath deposition is a simple one to prepare the thin films. This method does not require high temperature, low cost and fast method. The ZnCdS thin films have been used as photo luminescent materials [10]. The influence of different parameters such as concentration, pH, deposition cycle, deposition time and the mass of the materials of ZnCdS thin film have been investigated by many researchers [11].

Optical, electrical and morphological characteristics of CdS are suitable materials for energy harvesting in solar cell technology [12]. The bandgap energy 2.42 eV of CdS further strengthens the construction of solar cells. The wavelength below 515 nm, used as optical barriers for the solar radiation [13]. Performance of solar cells formed using CdS thin films have been studied by many researchers [14]. CdS thin films with film homogeneity, low electrical resistivity and high transparency produced by CBD method that makes the window layers for solar
The x-ray diffraction patterns of ZnCdS prepared in the presence of complexing agent, tri-ethanol amine are

Table 1. Composition of the ZnCdS thin film prepared in TEA at 72 °C.

| Samples | ZnCl₂ (g) | CdCl₂ (g) | CH₄N₂S (g) | NH₄OH (ml) | TEA* (ml) |
|---------|-----------|-----------|------------|------------|-----------|
| (a)     | 0.05      | 0.8       | 0.8        | 5          | 4         |
| (b)     | 0.10      | 0.8       | 0.8        | 5          | 4         |
| (c)     | 0.15      | 0.8       | 0.8        | 5          | 4         |
| (d)     | 0.20      | 0.8       | 0.8        | 5          | 4         |
| (e)     | 0.25      | 0.8       | 0.8        | 5          | 4         |

TEA*·tri-ethanol amine.

cell applications [15]. These properties of CdS thin films caused to apply in the field of transistors, photonics, near-infrared detectors, photocatalysis and gas sensing materials [16–20].

In this research work, the effect of Zn content on structural, optical and morphological properties of ZnCdS thin films is reported. Changes in band gap energy values (1.85–2.81 eV) are obtained in a broad range of Zn concentration (0.05–0.25 g) was compared. Further, crystallinity, shape, band gap and surface morphology of ZnCdS thin films were compared.

2. Experimental

2.1. Reagents

Cadmium Chloride (CdCl₂), Zinc Chloride (ZnCl₂) and Thiourea (NH₂CSNH₂) were used as Cd⁺², Zn⁺² and S⁻² ions, respectively. All the required chemicals, including NH₄OH and tri-ethanol amine were purchased from Labochemie, Mumbai, India. All analytical grade chemicals were used without further purification. Double distilled water was used to prepare the solutions.

2.2. Synthesis of ZnCdS thin film

The ZnCdS thin films were prepared using simple chemical bath deposition method of varying the concentration of Zn [21]. The composition of ZnCdS thin films is presented in table 1. The concentration of ZnCl₂ varied from 0.05 to 0.25 g while the concentrations of CdCl₂, CH₄N₂S, NH₄OH and tri-ethanol amine kept constant. Briefly, 20 ml of CdCl₂ and 20 ml of thiourea solution were mixed together and taken in a cleaned 100 ml beaker. To the above reaction mixture, 10 drops of NH₄OH and 4 drops of TEA were added. The reaction mixture in the beaker was kept in a temperature bath maintained at constant 72 °C. Glass substrates were cleaned with soap water, followed by double distilled water. Finally, the organic impurities on the glass substrates were removed by washing with acetone. The experimental glass substrates were mounted on substrate holder and immersed in the reaction beaker. The ZnCdS thin film prepared using 0.05 g of ZnCl₂ was named as (a).

Similarly, ZnCdS thin films were prepared using 0.10, 0.15, 0.20 and 0.25 g of ZnCl₂ was named as (b), (c), (d) and (e). All the ZnCdS thin films were prepared in optimizing the bath parameters (table 1).

2.3. Characterization techniques

2.3.1. XRD, SEM-EDS analysis

The structural studies were recorded by x-ray diffractometer using Cu–Kα radiation (λ = 1.5460 Å) at 30 KV and 40 mA from 20 to 70°. The surface morphology was analyzed by SEM (ZEISS). The composition materials were determined by energy dispersive x-ray analysis (EDX, HITACHI S2400).

2.3.2. PL, UV and FTIR studies

The photoluminescence spectra of the samples were recorded by Perkin–Elmer LS 45 fluorescence spectrophotometer. The optical absorption spectrum was recorded by Jasco V–700 series UV–visible spectrophotometer with a scanning range of 200 to 800 nm. The IR spectra were recorded using SHIMADZU, Japan FTIR 8400S spectrophotometer.

3. Results and discussion

3.1. XRD analysis

The x-ray diffraction patterns of ZnCdS prepared in the presence of complexing agent, tri-ethanol amine are shown in figures 1(a)–(e). The major diffraction peaks observed in the XRD pattern confirmed the formation of hexagonal phase (JCPDS: 010–780) [22]. Three crystalline phases observed were (100), (102) and (100). The average crystallite size of ZnCdS thin films was calculated from high intensity peak using Debye–Scherrer
The average size of the particles calculated were 36, 34 and 30 nm attributed to (100), (102) and (100) phases. Similarly, inter-planar distance (d) calculated was 3.4823 Å, found close to the JCPDS value (3.449 Å). Some unknown peaks observed in the XRD spectrum is due to the impurities. It is evidently noticed that the ZnCdS films prepared are polycrystalline in nature and the crystallinity is improved with increasing concentration of Zn. In addition, a peak shift towards higher angle was observed at lower concentrations of Zn (0.05 g) reveals the amorphous in nature. A broad peak obtained from 0.20 g of Zn could be due to smaller crystallite size. The characteristic of ZnCdS thin films, suggesting that the incorporation of Zn in the films does not simply changes in the crystalline phase of CdS. The crystallite sizes (D) of the ZnCdS films were estimated using Debye–Scherrer formula given equation.

\[
D = \frac{0.9X\lambda}{\beta \cos \theta}
\]  

(1)

Where \( \lambda \) is the wave length of x-rays, \( \beta \) is FWHM (full width at half maximum), ‘\( \theta \)’ is the diffraction angle and ‘D’ is crystallite size. The dislocation density (\( \rho \)) of the deposited film was calculated by the following equation (equation (2)), also the values are listed in table 2 [25]

\[
\delta = \frac{1}{D^2}
\]  

(2)

Here, D denoted as crystallite size.

The increases in concentration of Zn lead to increases in the dislocation density as it inversely proportional to the crystallite size. The value of micro strain (\( \varepsilon \)) was obtained using the following relation [26].

\[
\varepsilon = \frac{\beta \cos \theta}{4}
\]  

(3)

These observations indicate that, the micro strains of the films are found to increase with increasing the concentration of Zn. It is observed that the crystallite size decreased with the increasing the concentrations of Zn.

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**Figure 1.** (a)–(e). XRD patterns of TEA added ZnCdS thin films (a) 0.05 g Zn, (b) 0.10 g Zn, (c) 0.15 g Zn, (d) 0.20 g Zn (e) 0.20 g Zn.

**Table 2.** Structural parameters of TEA added ZnCdS thin film.

| Molar Concentration (g) | 2\( \theta \) (deg) | \( \beta \) (FWHM) (deg) | d -Spacing (nm) | Particle diameter (D) (nm) | Dislocation density (\( \rho \)) \( \times 10^{15} \) line m\(^{-2} \) | Micro strain (\( \varepsilon \)) \( \times 10^{-4} \) |
|------------------------|---------------------|------------------------|-----------------|------------------------|-------------------------------|------------------------|
| 0.05                   | 25.434              | 0.236                  | —               | —                      | —                             | —                      |
| 0.10                   | 25.434              | 0.236                  | 3.4905          | 36.07                  | 0.768                         | 1.018                  |
| 0.15                   | 25.434              | 0.236                  | 3.5071          | 36.07                  | 0.768                         | 1.018                  |
| 0.20                   | 25.637              | 0.244                  | 3.474           | 34.90                  | 0.82                          | 1.03                   |
| 0.25                   | 25.69               | 0.275                  | 3.474           | 30.97                  | 1.042                         | 1.153                  |

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formula [23]. The average size of the particles calculated were 36, 34 and 30 nm attributed to (100), (102) and (100) phases. Similarly, inter-planar distance (d) calculated was 3.4823 Å, found close to the JCPDS value (3.449 Å). Some unknown peaks observed in the XRD spectrum is due to the impurities.
3.2. SEM image analysis

The SEM images of the ZnCdS thin films are shown in figures 2(a)–(e). Two images (2 and 1 μm) were selected from each sample at different magnifications at 20.68 Kx and 10.63 Kx.

The films coated with Zn have a homogeneous appearance (figure 2(a)), the particles and clusters widely spread over the films. The ZnCdS film prepared using 0.10 g of Zn at 20.68 Kx and 40.24 Kx magnification shows uniform coating with spherical shaped particles (figure 2(b)). The figure 2(c) shows the film prepared using 0.15 g of Zn. Highly accumulated and homogeneously deposited particles observed at 17.51 Kx and 31.35 Kx magnifications. The figure 2(d) film (0.20 g Zn) shows the minimum spherical particle accumulation at 14.44 Kx and 40.47 Kx magnifications. The ZnCdS film prepared using 0.25 g of Zn (figure 2(e)), shows rod crystals.

![Figure 2](image_url)
vertically deposited at the magnification of 7.27Kx. The less population spherical particles noticed here. However, the surface of the films (b), (c) and (d) prepared with 0.10, 0.15 and 0.20 of Zn is highly occupied with film surface at figure 2(c) film. This observation confirms that the crystallinity ZnCdS increases with increasing concentration of Zinc. In the SEM image of figure 2(c) film concentration also shown like highly spherical particles, these particles were raised due to the influence of complexing agent TEA [27].

3.3. AFM analysis

The AFM images are utilized to evaluate the surface roughness of ZnCdS thin films. AFM microscope images show 3D topography of the deposited ZnCdS thin film is shown in figures 3(a)–(e). The average root mean square (Sq), maximum height (Sz) and arithmetic height (Sa) of ZnCdS thin films were 43.80, and 35.1 nm. AFM images were recorded in 5 μm of the thin films. The maximum peak height (Sp) and maximum pit height (Sv) of films found to be 121 and 208 nm, respectively. The thickness of the thin films found to be increased with increasing concentration of Zn. It can be noticed that, there are spherical shape particles in ZnCdS thin films spread over the surface (figures 3(a)–(d)). The AFM image reveals that the surface of the substrate is completely covered by ZnCdS. Minimum and maximum roughness were −0.154 and 2.91 nm, which assures the coating quality.

All the film reveals the maximum height of the particles, which confirms even surface morphology of the coating (figures 3(a)–(d)). The average size of the particles found in the range of 100–120 nm. Uniform
deposition of rod like particles found in figure 3(e) for 0.25 g of Zn. All the ZnCdS thin films reveal the maximum height of the particle which confirms the mostly even surfaces in figures 3(a)–(d). The films coated using a 0.10 and 0.15 g of Zinc is comparatively good as compared with the film prepared from 0.05 g of Zn. Thickness and morphology the ZnCdS thin films compared with the literature [28, 29].

3.4. Optical analysis

The optical properties of ZnCdS thin films determined from absorbance were recorded in the range of 200 to 900 nm using UV-visible spectrophotometer. The absorption spectra of the ZnCdS thin films prepared using 0.05, 0.10, 0.15, 0.20 and 0.25 g of Zn are shown in figures 4(a)–(e). As the concentration increases, the absorption shifted towards higher wavelength in the UV region. The absorption of thin films observed were from 580, 543, 533, 539 and 500 nm as shown in the figure indicates that the absorption increases with increasing the Zn concentration. The transmittance spectra of TEA added ZnCdS thin films with different concentration of Zn are shown in figures 5(a)–(e).

The transmittance is found to consent well with absorbance at 400 nm wavelength. The transmittance observed were 2.98, 2.76, 1.73, 1.07 and 0.28% for the corresponding 0.05, 0.10, 0.15, 0.20 and 0.25 g of Zn concentrations. From the figures 5(a)–(e), it is noted that, the transmission gradually decreases with increasing the concentration of Zn. Further, the thickness of the film increases with increasing concentration, which restrict the transmission of light. The ZnCdS thin film exhibits minimum transmittance films on glass substrate. Transmittance spectra primarily depend on various physical properties like roughness, microstructure, phase formation, defects and thickness of the ZnCdS thin films prepared.

The optical band gap energy of ZnCdS thin films (a)–(e) calculated were 1.85, 1.98, 2.03, 2.703 and 2.81 eV as shown in figure 6. It is well known that while increasing molar concentration, the ZnCdS promotes electrons from the valence band to the conduction band with the absorption of minimum energy [30]. It is observed that there is some variation in the band gap energy of the ZnCdS thin films. This variation could be due to the method of deposition, conditions and vary in the stoichiometry of the ZnCdS thin films.

3.5. Photoluminescence study

The PL spectra of ZnCdS thin films were prepared using various concentrations of Zn are shown in figures 7(a)–(e). It is seen that exhibit peak is found between 453 to 768 nm for different Zn concentrations [31].

Hence, the luminescence bands can be identified with transitions blue, bluish green, green, yellow and red emission band as presented in table 3. The PL spectrum of ZnCdS thin film exhibited a broad and intense emission peak in the visible region at 453 and 481 nm (2.51 eV) in agreement with the values found in literature [32]. The emission band observed in 516, 577 and 768 nm is due blue, yellow and red band. The green emission band noticed at 516 nm with the band gap energy of 2.17 eV is related to the band gap energy of semiconductor [33, 34]. The optical absorption of the deposited films increases with the increasing concentration. The optical transmittance and band gap energy of the deposited films decreases for the increasing concentration.
4. Conclusions

The ZnCdS thin films were prepared using different concentration of Zinc by chemical bath deposition technique. The effect of Zinc concentration on the crystallinity, microstructure, band gap, transmittance, absorbance and optical properties of ZnCdS thin films was studied. The crystalline nature of the ZnCdS films deposited at 72 °C was confirmed by x-ray diffraction study. Spherical shape particles produced at lower concentration, while at higher concentration, it produced rod shape crystallites. The influence of Zinc

![Graph showing transmittance and band gap for different concentrations of ZnCdS thin films.](image)

**Figure 5.** (a)–(e). Transmittance UV-Visible spectrum of TEA added ZnCdS thin films, (a) 0.05 g Zn, (b) 0.10 g Zn, (c) 0.15 g Zn, (d) 0.20 g Zn (e) 0.20 g Zn.

**Figure 6.** (a)–(e). Band gap UV-visible spectrum of TEA added ZnCdS thin films, (a) 0.05 g Zn, (b) 0.10 g Zn, (c) 0.15 g Zn, (d) 0.20 g Zn (e) 0.20 g Zn.
concentration of the structural properties of the ZnCdS films was noteworthy. Transmittance and band gap energy of ZnCdS thin films increased with increasing the Zn content. However, absorbance spectra showed some deviation from each other. The Zinc concentrations showed an insightful manipulate on the band gap energy with significant increase in the band gap energy significantly. The band gap increased 1.85 to 2.81 eV with increasing Zinc concentration. The prepared films showed good luminescent properties and hence it could suitable materials for solar cell applications.

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### Table 3. PL spectrum of TEA added ZnCdS thin films at 72 °C.

| Color   | Standard (nm) | Observed (nm) | Observed Emission Photon energy (eV) |
|---------|---------------|---------------|-------------------------------------|
| Blue    | 450–495       | 453, 481      | 2.50–2.75                           |
| Green   | 495–570       | 516           | 2.17–2.50                           |
| Yellow  | 570–590       | 577           | 2.10–2.17                           |
| Red     | 620–750       | 768           | 1.65–2.00                           |

TEA*—tri-ethanol amine.
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