Structural, optical and electrical properties of (CdS)\textsubscript{1-x} (ZnTe)\textsubscript{x} solid solution thin films prepared by vacuum thermal evaporation method

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Abstract. The II–VI compounds and their solid solutions are promising source for various types of thin film devices such as thin film transistors, optoelectronic devices and solar cells. The formation of (CdS)\textsubscript{1-x}(ZnTe)\textsubscript{x} layer by interdiffusion of CdS and ZnTe during the fabrication of CdS/ZnTe heterojunction thin film was previous reported. This layer is thought to be important because it relieves strain at the CdS/ZnTe interface that would otherwise exist due to the excess 10 \% lattice mismatch between the two materials. Therefore, it is essential to have a full understanding of the physical properties of (CdS)\textsubscript{1-x}(ZnTe)\textsubscript{x} alloy thin films. In this work, (CdS)\textsubscript{1-x}(ZnTe)\textsubscript{x} thin films in the entire composition range (0≤ x≤ 1.0) were prepared by vacuum thermal evaporation on glass substrate using mixed powders of high purity of CdS and ZnTe compounds as the precursor. XRD revealed that the films exhibited a hexagonal structure with the preferred orientation of (002) plane when x ≤ 0.2. However, when x ≤ 0.8, they belonged to a cubic structure with the preferred orientation of (111) plane. For the composition 0.4 ≤ x ≤ 0.6, the hexagonal and cubic phases coexisted in the system and the films became less preferentially oriented. SEM and EDS were used to study the surface morphology and elemental composition of the samples. The crystallite size of the as-deposited films in the range 88–361 nm was observed by AFM image. The FTIR transmission spectra in the range 400–1,000 cm\textsuperscript{-1} revealed the characteristics of Cd–S and Zn–Te vibrational modes. Optical properties of the films were performed with UV-Vis spectrophotometer in the wavelength range 400–1,000 nm. The variation of direct energy gap with composition (x) was in good agreement with the quadratic form, giving an upward bowing parameter (b) of 1.23 eV. Electrical properties of the films were evaluated by resistivity and Hall effect measurements in the van der Pauw configuration. From transient photoconductivity measurement, the decay current data were better fitted with multiple exponential functions resulting in the several slow decay times. Density of trap states corresponding to its decay time was also evaluated from the decay current data.

1. Introduction

The II–VI compounds are very popular polycrystalline semiconducting materials studied for many years and a wide range of applications in photovoltaic cells, photodetectors and solid-state electronic devices like light emitting diodes, etc. [1]. Cadmium Sulfide (CdS) and Zinc Telluride (ZnTe) belongs to II–VI compounds family and crystalline structures for CdS and ZnTe are normally hexagonal wurtzite and cubic zincblende, respectively [2]. High absorption coefficients and closely matching band gaps with the visible region of the solar spectrum are the causes of the popularity of II–VI semiconductors. The energy gap of CdS and ZnTe are direct allowed transition type, and has leading to very strong light
absorption [3,4]. The formation of (CdS)\(_{1-x}\)(ZnTe)\(_x\) layer by interdiffusion of CdS and ZnTe during the fabrication of CdS/ZnTe heterojunction was previously observed [5-7]. The (CdS)\(_{1-x}\)(ZnTe)\(_x\) layer is thought to be important because it relieves strain at the CdS/ZnTe interface that would otherwise exist due to the excess 10% lattice mismatch between the two materials. Therefore, it is essential to have a full understanding of the physical properties of (CdS)\(_{1-x}\)(ZnTe)\(_x\) alloy thin films. CdS and ZnTe can be mixed so as to provide (CdS)\(_{1-x}\)(ZnTe)\(_x\) quaternary alloys which can lead to new semiconductor materials that may be suitable for accomplishing the twin tasks of enhanced resistance towards photocorrosion and increased absorption of solar spectrum [8-9].

Nowadays, CdS and ZnTe thin films can be prepared by a different film deposition techniques, such as vacuum thermal evaporation (TE), close-spaced sublimation (CSS), DC & RF magnetron sputtering deposition, plasma chemical vapour deposition (PCVD) and successive ionic layer adsorption and reaction (SILAR), etc. Generally, vacuum thermal evaporation technique is the most commonly used for the preparation of many thin films due to its reproducibility, simplicity and scalability to deposit onto many type substrates. In this paper is concerned the preparation of (CdS)\(_{1-x}\)(ZnTe)\(_x\) thin films with different x-values (0 ≤ x ≤ 1.0) by vacuum thermal evaporation on glass substrate. The crystal structure of the films was analyzed by X-ray diffraction (XRD) method. Surface morphology and elemental analysis were obtained by scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS). Crystallize size of the films was observed by atomic force microscopy (AFM). Energy gap value of the films was evaluated from the spectral transmission data in the range 400–1,000 nm. The FTIR transmission spectra revealed the lattice vibrational characteristics of the films were measured in the range 400–1,000 cm\(^{-1}\). Electrical properties of the films were evaluated by resistivity and Hall effect measurements in the van der Pauw configuration. The transient photoconductivity measurement was performed in order to investigate decay times and density of trap states from the decay current data.

2. Materials and Methods

(CdS)\(_{1-x}\)(ZnTe)\(_x\) thin films with the whole composition range (0 ≤ x ≤ 1.0) were prepared by a single source thermal evaporation in vacuum using high purity (99.99 %) of CdS and ZnTe compounds supplied by Sigma-Aldrich. CdS and ZnTe powders were weighed by taking the stoichiometric ratio in order to obtain different compositions of (CdS)\(_{1-x}\)(ZnTe)\(_x\) films. The starting powders were mixed and milled with ethanol and ionized water in the ultrasonic bath, respectively. Then, the slide glass substrate was cleaned with detergent, acetone, ethanol and de-ionized water in the ultrasonic bath, respectively. Then, the slide glass substrate was slide glass substrate to bake at 100 °C for 30 min, before used. In the film deposit process, we used a thermal evaporation apparatus at high vacuum about 10\(^{-5}\) mbar to prepare the (CdS)\(_{1-x}\)(ZnTe)\(_x\) thin films where x = 0, 0.2, 0.4, 0.6, 0.8 and 1.0. The distance between the substrate holder and the tungsten boat was 23 cm. The substrate temperature was kept at around 100 °C by using PID temperature controller. The thickness of the films of about 500 nm was controlled using a crystal thickness monitor (Edwards type FTM6). The crystal structure of these films was checked by XRD with a Brucker D 8 diffractometer using CuK\(_\alpha\) radiation. The surface structure and elemental composition were investigated using an energy dispersive X-ray spectroscopy (EDS) attached with the LEO 1455VP SEM. The grain size was examined by Park XE-100 AFM. FTIR spectra were performed by Thermo Scientific Nicollet 6700 in the range between 450 and 1000 cm\(^{-1}\). Optical transmission measurements were performed with thermo electron corporation, Heios model UV-VIS spectrophotometer in the wavelength range of 400–1,000 nm. The energy gap (E\(_g\)) of the films was determined by using the equation (d/hv)\(^2\) = A(hv-E\(_g\)) where \(\alpha\) is the absorption coefficient, A is a constant and hv is the photon energy. Electrical properties of the films were evaluated by resistivity and Hall effect measurements in the van der Pauw configuration. DC current supplied by Keithley 236 source measure unit in the range of 1–10 nA was applied on the films and recorded by Keithley 610R electrometer. The films were subjected to a uniform magnetic field of 0.46 T. The polarity was reversed.
for each measurement and the voltage was recorded by 4½ FLUKE digital multi meter. The transient photoconductivity of the films was measured using a two-probe method with ELH halogen lamp as the light source. Two silver electrodes were fabricated on the sample surface. The sample was connected to the experimental setup and maintained in darkness under a constant applied bias of 20 V to stabilize the current. The current was recorded in following sequence: 30 s in darkness, 100 s under illumination, and 200 s in the darkness.

3. Results and Discussions

3.1 Structural Characterization

3.1.1 XRD Analysis.

Figure 1 shows the XRD patterns of (CdS)\(_{1-x}\)(ZnTe)\(_x\) thin films with various compositions (x). The diffraction angle (2\(\Theta\)) varied from 20 to 70°. For \(x \leq 0.2\), XRD revealed that the films exhibited a hexagonal structure, and agreed well JCPDS file 77-2306, with the preferred orientation of (002) plane. However, when \(x \geq 0.8\), they existed in cubic structure, and agreed well JCPDS file 15-746, with the preferred orientation of (111) plane. For the composition \(0.4 \leq x \leq 0.6\), the hexagonal and cubic phases coexisted in the system and the films became less preferentially oriented. The coexistence of the hexagonal and cubic phases often occurring in II-VI alloy films has been previously reported [10,11]. For the composition \(x \leq 0.2\), the strongest peak intensity of (002) plane shifted to a lower 2\(\Theta\) value with increasing composition (x). In contrast, the strongest peak intensity of (111) plane shifted to a higher 2\(\Theta\) value with increasing composition (x) for the composition \(x \leq 0.8\). However, some small peaks corresponding to the excess Te element appeared in the films with \(x = 1.0\).

The lattice constant “a” and “c” for the hexagonal structure were calculated using the following equation [2]:

\[
\frac{1}{d_{hkl}^2} = \frac{4}{3} \left( \frac{h^2 + hk + k^2}{a^2} \right) + \frac{l^2}{c^2}
\]  

(1)

where \((h\ k\ l)\) is the Miller indices. However, the following relation was used to calculate the lattice constant “a” for cubic structure [2]:

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

(2)

Figure 2 shows the relation of the calculated lattice constants of (CdS)\(_{1-x}\)(ZnTe)\(_x\) thin films with composition (x). It is clear from figure that the phase changes from hexagonal to cubic when the composition \(x\) beyond 0.6. The lattice constant “a” and “c” of hexagonal phase decreased linearly with increasing composition (x) from 0 to 0.6. This behaviour in accordance with Vegard’s law corresponds to earlier reports [10,11]. This is mainly attributed to the partial replacement of Cd\(^{2+}\) ions (ionic radius \(r = 0.78\) Å) by Zn\(^{2+}\) ions \((r = 0.60\) Å), indicating a reduction in the lattice constant “a” and “c” [10]. However, the lattice constant “a” of cubic phase increased linearly with increasing composition (x) from 0.4 to 1.0. This is mainly due to the partial replacement of S\(^2-\) ions \((r = 1.84\) Å) by Te\(^{2-}\) ions \((r = 2.21\) Å) indicating an enhancement in the lattice constant “a”. The present lattice constant data clearly suggested that CdS and ZnTe are miscible in the intermediate range \(0.4 \leq x \leq 0.6\) because the incompatibility of ionic radii of S and Te generally excluded the formation of entirely miscible lattice in II-VI chalcogenide alloy in bulk except very close to the end compositions [10].
3.1.2 Surface Analysis

Figure 3 shows SEM images of (CdS)$_{1-x}$(ZnTe)$_x$ thin films with different compositions (x). The surface morphology of the films was uniform, smooth, homogeneous, without pinholes, cracks-free and well covered to the substrate. Figure 4 shows AFM images of (CdS)$_{1-x}$(ZnTe)$_x$ thin films with different compositions. The main difference among films is the distribution of grain size. The grain size values of the thin films were 361, 156, 117, 88, 205 and 264 nm for x = 0, 0.2, 0.4, 0.6, 0.8, 1.0, respectively. The corresponding roughness values were 33, 20, 7, 6, 1.6 and 1.8 nm, respectively. The change in morphology is possibly due to a change in the crystal structure from hexagonal to cubic system.

Figure 1. XRD patterns of (CdS)$_{1-x}$(ZnTe)$_x$ thin films.

Figure 2. Variation of lattice constant “a” and “c” of (CdS)$_{1-x}$(ZnTe)$_x$ thin films.

Figure 3. SEM micrographs of (CdS)$_{1-x}$(ZnTe)$_x$ thin films.
The EDS patterns of (CdS)$_{1-x}$(ZnTe)$_{x}$ thin films are shown in figure 5. The EDS analysis for all compositions both weight and atomic analysis is shown in Table 1. It clearly shows that the amount of Zn and Te increased as the x-value increased from 0 to 1.0. The EDS analysis confirmed the presence of Cd, Zn, S and Te elements in (CdS)$_{1-x}$(ZnTe)$_{x}$ thin films which slightly deviated from the nominal stoichiometry within the experimental error as tabulated in Table 1. This result confirms the solid solution in mixing of CdS and ZnTe phases. For $x = 1.0$ (ZnTe films), the average atomic percentage of Zn:Te was 49.35:50.65. It indicated that the sample exhibited a slightly incorporated Te element phase which agreed well with the XRD result.
Figure 5. EDS patterns of (CdS)\textsubscript{1-x}(ZnTe)\textsubscript{x} thin films.

Table 1. Elemental composition of (CdS)\textsubscript{1-x}(ZnTe)\textsubscript{x} thin films.

| Composition (x) | Cd  | S   | Zn  | Te  |
|-----------------|-----|-----|-----|-----|
|                 | wt.%| at.%| wt.%| at.%|
| 0               | 77.35 | 49.35 | 22.65 | 50.65 |
| 0.2             | 56.27 | 41.30 | 13.73 | 35.31 |
| 0.4             | 36.22 | 27.81 | 9.07  | 24.42 |
| 0.6             | 25.37 | 21.69 | 3.59  | 10.77 |
| 0.8             | 13.71 | 12.84 | 1.12  | 3.67  |
| 1.0             | -    | -    | -    | 33.30 |

\text{wt.\%} \text{at.\%} \text{wt.\%} \text{at.\%} \text{wt.\%} \text{at.\%} \text{wt.\%} \text{at.\%}
3.1.3 FTIR Analysis

Figure 6 shows the FTIR spectra of the (CdS)$_{1-x}$(ZnTe)$_x$ thin films in the wavenumber range of 400–1000 cm$^{-1}$. For $x = 0$ (CdS films), FTIR characteristics of Cd-S vibration peaks appeared around 594, 630 and 675 cm$^{-1}$, respectively. The result matches well with the values reported earlier [12–15]. The absorption peak around 594 cm$^{-1}$ slowly shifted to lower wavenumber with increasing composition (x) and reached 560 cm$^{-1}$ when $x = 1.0$. FTIR spectra belonging to ZnTe films indicated the existence of distinct characteristic Zn-Te vibration peaks at 560 and 760 cm$^{-1}$ [16–18]. The shift of wavenumber may be attributed to the difference of average bond length between Cd-S (2.52 Å) and Zn-Te (2.63 Å). The longer bond length yields a smaller wavenumber is indeed true in our case as well. However, for $x \leq 0.4$ characteristic peaks of Zn-O stretching vibrations occurring at 400–430 cm$^{-1}$ were observed [19,20]. The result confirms the substitution of Cd$^{2+}$ ions by Zn$^{2+}$ ions and of S$^{2-}$ ions by Te$^{2-}$ ions, respectively.

3.2 Optical Properties

Figure 7 presents the transmission spectra of the (CdS)$_{1-x}$(ZnTe)$_x$ thin films in the wavelength range 400–1,000 nm. It is seen from the transmission spectra that the absorption edge shifted towards the larger wavelength with increase in composition suggesting the decrease of energy gap. The interference fringes appearing in some of the transmission spectra implied that the films are good quality. Figure 8 shows the energy gap ($E_g$) of the films estimated by plotting $(\alpha h\nu)^2$ as a function of energy ($h\nu$). The energy gap value decreased from 2.43 eV (corresponding to the $E_g$ value of a CdS bulk material) with further increasing composition (x) passing through a minimum value of 1.85 eV, and then increased to 1.90 eV (corresponding to the $E_g$ value of a ZnTe bulk material). Figure 9 shows the variation of energy gap of (CdS)$_{1-x}$(ZnTe)$_x$ thin films as a function of composition (x). The composition (x) used in the figure was deduced from EDS spectra. The variation of the energy gap of the films estimated by using direct allowed transition model is expressed conventionally in the quadratic form [2,21, 22]:

$$E_g(x) = E_g(ZnTe) + (E_g(CdS)-E_g(ZnTe)-b)x+bx^2$$  \(3\)

where $b$ is bowing parameter, $E_g$ of CdS = 2.43 eV and $E_g$ of ZnTe = 1.90 eV corresponding to bulk material. The bowing parameter $b$=1.23 eV indicated an upward bowing of the energy gap, which agreed well with the values reported on various II-VI semiconductor compounds [2]. It is indicated that a small amount of ZnTe in CdS host lattice can drastically reduce its energy gap. This is because the impurity limit of Zn substitution on Cd site and/or Te substitution on S site leads to localized isovalent impurity levels. This behaviour was also observed in CdS$_x$Te$_{1-x}$ [23]. After that, we determine the band tail of thin films which were related to imperfection density located in the energy gap by studying the absorption edge which be increased exponentially. The band tail (or Urbach’s tail) can be found in the following expression [24]:

$$\alpha = \alpha_0 \exp\left(\frac{h\nu}{E_u}\right)$$  \(4\)

where $E_u$ is band tail energy and $\alpha_0$ is a constant. The variation of band tail as a function of composition (x) is shown in figure 10.
3.3 Electrical Properties

The electrical properties of the films were investigated by Hall effect and resistivity measurements in van der Pauw configuration. Variation of resistivity, carrier concentration and mobility against composition (x) is shown in figure 11. The resistivity of the (CdS)$_{1-x}$(ZnTe)$_x$ thin films with the different compositions has a wide distribution, and has value in the range from $10^{-1}$ to $10^7$Ω cm. The conductivity type of the films could be evaluated by considering the sign of Hall voltage. Normally, CdS exhibits n-type while ZnTe exhibits p-type [25]. The conductivity type started to convert from n-type to p-type when composition (x) beyond 0.4 due to auto-compensation of the charge carrier between the electrons and hole [11]. The highest resistivity of 1.18x$10^7$ Ω cm was observed for the films with x = 0.4. It indicated that the amount of charge carrier between the electrons and hole is approximately the same.

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Figure 6. FTIR spectra of (CdS)$_{1-x}$(ZnTe)$_x$ thin films.

Figure 7. Transmission spectra of (CdS)$_{1-x}$(ZnTe)$_x$ thin films.

Figure 8. (αhν)$^2$ plot as a function of energy (hν) of (CdS)$_{1-x}$(ZnTe)$_x$ thin films.

Figure 9. Variation of energy gap of (CdS)$_{1-x}$(ZnTe)$_x$ thin films.
order or refute. Moreover, CdS-rich phase (for \(x \leq 0.4\)) obviously manifested the higher mobility value than the one of ZnTe-rich phase (for \(x \geq 0.6\)).

3.4 Transient Photoconductivity

Transient photoconductivity of thin films was investigated by considering the change of current when light was applied on the samples. It found that thin films for \(x = 0.6\) has better photo response than other compositions. The photo response of the films for \(x = 0.6\) is shown in figure 12. Under illumination, the current increased immediately because a lot of carriers were excited by light illumination. After the illumination period, the persistent photoconductivity (PPC) effect on the films for \(x = 0.6\) occurred in darkness shown in the figure, which follows the illumination period, indicated the persistent of charge carrier traps in the films [26]. In figure 13, the dot line is the measured data and the solid line is the least squared fit data by the multiple exponential functions [27,28]. Method for calculation the trap density corresponding to the PPC decay time constant was reported in the literature [29]. The decay time and its corresponding density of trap state values of the films (for \(x = 0.6\)) are 4.84, 84.49, 1056.31, 1879.33, 2596.86 s and 5.25\(\times\)10^{12}, 4.09\(\times\)10^{12}, 3.35\(\times\)10^{12}, 2.61\(\times\)10^{12}, 2.47\(\times\)10^{12} cm^{-2}, respectively.

![Figure 10. Variation of band tail of (CdS)\(_{1-x}\)(ZnTe)\(_x\) thin films.](image1)

![Figure 11. Variation of resistivity, carrier concentration and mobility of (CdS)\(_{1-x}\)(ZnTe)\(_x\) thin films.](image2)

![Figure 12. Photo response of (CdS)\(_{1-x}\)(ZnTe)\(_x\) (x = 0.6) thin films.](image3)

![Figure 13. Normalized decay current of (CdS)\(_{1-x}\)(ZnTe)\(_x\) (x = 0.6) thin films.](image4)
4. Conclusion

(CdS)$_{1-x}$(ZnTe)$_x$ thin films with different compositions ($0 \leq x \leq 1.0$) were deposited by a single source vacuum thermal evaporation on glass substrate using mixed powders of CdS and ZnTe compounds in tungsten boat. From XRD results, CdS-like phase belonging to hexagonal structure was obtained when x ≤ 0.2. In contrast, ZnTe-like phase belonging to cubic structure was observed when x ≥ 0.8. However, samples with nominal composition different the above mentioned range (namely 0.4 ≤ x ≤ 0.6), the cubic and hexagonal phases coexisted in the system and the films became less preferentially oriented. The variation of lattice parameter values of both hexagonal and cubic structures is agreement with Vegard’s law as a consequence of the replacement of Cd$^{2+}$ ions by Zn$^{2+}$ ions and of S$^{2-}$ ions by Te$^{2-}$ ions. From SEM images, the surface morphology of the films was uniform, smooth, homogeneous, without holes, cracks-free and well covered to the substrate. The crystallite size in the range 88-361 nm was elucidated by AFM. The unique absorption peak shifted from 594 to 560 cm$^{-1}$ when the composition (x) increased from 0 to 1.0. The optical transition on (CdS)$_{1-x}$(ZnTe)$_x$ thin films was found to be direct allowed transition type. The variation of direct energy gap with composition (x) was in good agreement with the quadratic form, giving an upward bowing parameter (b) of 1.23 eV. The result confirms the substitution of Cd$^{2+}$ ions by Zn$^{2+}$ ions and of S$^{2-}$ ions by Te$^{2-}$ ions, respectively. From Hall effect measurement, the conductivity type started to change from n-type to p-type when composition (x) beyond x = 0.4 due to auto-compensation of the charge carrier between the electrons and hole. Persistent photoconductivity (PPC) effect was observed on the films for x = 0.6. The photo decay current data were better fitted with multiple exponential functions. Five slow decay times and five corresponding trap density values were obtained. Such slow decay time behaviour may be beneficial for enhancement of carrier lifetime in solar cell application. Finally, the fabrication and characterization of (CdS)$_{1-x}$(ZnTe)$_x$/CuSbS$_2$ heterojunction thin films are in progress.

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