Influence of the $CdCl_2$ Solution Concentration on the Properties of CdTe Thin Films

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Abstract- This work presents the influence of the concentration of $CdCl_2$-treatment-solution on the microstructural, morphological, and optical properties of CdTe thin films. The CdTe films were deposited on CdS coated borosilicate glass by Close-Spaced Sublimation (CSS) technique using a standard recipe. The CdS films were grown by RF magnetron sputtering. The deposited CdTe films were treated with $CdCl_2$ solution of 0.20 M, 0.25 M, 0.30 M, 0.35 M, and 0.40 M concentration at 395 °C for 25 minutes. All the films exhibit polycrystalline nature with a predominant (111) Bragg peak of cubic CdTe. The highest crystallite size and smallest micro-strain and dislocation density are found for the treatment solution of 0.35M concentration. The results present that the treatment concentration enhances the S diffusion into the CdTe layer up to a certain limit of molar concentration. The highest S concentration (~2%) among the investigated samples is found for 0.35 M concentration. The SEM micrographs of the as-grown and treated films reveal that the grain size follows an increasing trend with the solution concentration up to 0.35 M. The optical bandgap of all samples is found to be in the range of 1.42 – 1.45 eV estimated from the UV-Vis-NIR transmission data. The lowest bandgap of 1.42 eV is found for the sample treated with 0.35 M solution due to the highest crystallinity among the samples investigated evident from both XRD and SEM results.

Keywords CdTe thin film, Close-spaced sublimation (CSS), $CdCl_2$ treatment, XRD, Solar cells.

1. Introduction

In the last few decades, thin film solar cells have gathered immense research attraction among researchers around the world due to their lower cost and minimal material usage compared to the silicon based solar cells [1]. In recent years, thin film solar cells have started to enter the solar photovoltaic (PV) market as the second generation solar cells and outperforming the Si-based solar cells in many applications, e.g., terrestrial applications where the net volume, weight, efficiency, and long-term stability of the solar panels are of major concern [2-4]. The polycrystalline Cadmium Telluride (CdTe) is one of the few most extensively studied thin film materials which is realized as an absorber layer in the corresponding thin film solar cells owing to its near optimum 1.45 eV direct bandgap [5] and high absorption coefficient around 1×10^5 cm^{-1} at 1.5 AM solar radiation [6]. Furthermore, it has the highest lattice constant [7], longer stability, and better conversion efficiency among the members of II-VI compound semiconductors. Moreover, during the last decade, while the efficiency of average wafer-based silicon PV modules has increased from 12% to about 17%, the efficiency of CdTe based PV modules has increased from 9% to 19% [2]. Meanwhile, the CdTe cell efficiency has crossed the benchmark of 22% efficiency level [8]. All the above features, together with rapid growing efficiency, have made CdTe the most successful contender to silicon in PV applications as well as a superior absorber material compared to its thin film counterparts.

A multitude of growth techniques such as screen printing [9], spray pyrolysis [10], molecular beam epitaxy [11], electrodeposition [12], MOCVD [13], thermal evaporation [14], electron beam evaporation [15], close-spaced sublimation (CSS) [16], and sputtering [17-18] have long been utilized to deposit ultra-thin films of CdTe for PV applications. Although these techniques have their pros and cons, each of them is capable of fabricating high quality CdTe films. But the literature suggests that the CSS technique offers the most efficient CdTe solar cells [19-20]. Researchers are trying to mitigate the gap between the theoretical efficiency limit (~30%) and the experimentally found efficiency of the CdTe solar cells by deploying some post deposition treatments of the CdTe films and incorporating some extra beneficial layers into the structure [17]. Among these two techniques, the former is used to enhance the quality of the films.

One such treatment commonly used by most researchers is the $CdCl_2$ treatment, followed by suitable thermal annealing. Literature also confirms that this $CdCl_2$ treatment improves electronic properties (carrier concentration and mobility, sheet resistivity, and minority carrier lifetime) [21-25], structural properties (crystallite size, stress, and dislocation density, S and Te inter-diffusion) [7, 22-24, 26-29], optical properties [22-23, 26-29], and surface morphology (grain size) [27-29] of the films by facilitating the recrystallization of the material inside the films. For this reason, $CdCl_2$ treatment of the CdTe films is given the utmost importance in the manufacturing process of high efficiency CdTe solar cells. Two different processes of $CdCl_2$ treatment that are dry process [29] and wet process [7, 23-28] are found in the literature. The dry process is complex and requires sophisticated equipment. On the other hand, the wet process is much simpler and easier. As a result, the wet process is more famous among researchers. This process requires the CdTe samples to be dipped into an aqueous solution of $CdCl_2$ salt of suitable concentration followed by subsequent thermal annealing at a high temperature of the range 350°C - 450°C for about 10 to 60 minutes [7, 22-28]. Different researchers have proposed different values of $CdCl_2$-solution-concentration starting from 0.1 M to 1.2 M [7, 23, 27, 28]. Moreover, no conclusion has drawn yet on how different properties of the CdTe films vary with the concentration of the $CdCl_2$ solution.

In this paper, a detailed and systematic investigation of the relation of structural, morphological, and optical properties of the CdTe thin films with the concentration of $CdCl_2$ treatment solution is presented. In addition, the effect of $CdCl_2$ concentration on the amount of S diffusion into CdTe film at the CdS/CdTe interface and hence, the formation of $CdTe_{1-x}S_x$ interfacial layer is investigated.

2. Experimental Details

2.1. Substrate Preparation

The CdTe thin films were deposited on borosilicate glass substrates (BGS) of 3 cm × 3 cm ×2 mm dimensions. These substrates have higher thermal stability and better optical transmittance than soda-lime glass substrates (SLG) [30]. At first, a tick mark was scratched on one side at the upper right corner of the substrates by a diamond cutter so as to distinguish two sides from each other and to make sure that, the films are deposited on the side opposite to the scratch marked side. Then, the substrates were made wax-free by scribing mechanically with a brush in acetone. To remove any grease type material and adhesive particles, the substrates were then cleaned ultrasonically in methanol, acetone, (second time) methanol, and finally, de-ionized, DI water environment respectively for 15 minutes which is briefly called the MAMD process [5, 30]. During the ultrasonic cleaning process in DI water the bath temperature was set at 50°C. After cleaning, the substrates were first dried by a dry N_2 blow and then baked for 10 minutes on a hot plate at 100 °C.

2.2. CdS/CdTe Film Deposition

An ultra-thin layer (~80 nm) of CdS was deposited on the substrates before depositing the CdTe thin films. A 2-gun RF magnetron sputter coater of NSC-3500 model from Nano Master Inc. was utilized to deposit the CdS films from a target of high (99.99%) purity. The CdS deposition was carried out at 30W RF power in Argon ambient. A deposited CdS film is presented in Fig.1.

Fig. 1. A CdS thin film deposited on borosilicate glass.

Afterwards, the CdTe thin films were deposited on pre-deposited CdS films using close-spaced sublimation technique from a compact solid CSS source. The CdTe source was prepared by sintering CdTe powder of 99.99% purity at 700°C temperature in 4 Torr Ar ambient [5]. The CSS equipment during the sintering operation is shown in Fig.2.

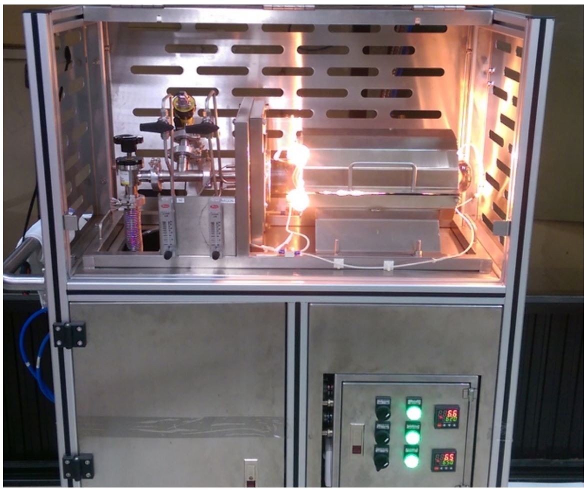

Fig. 2. CSS equipment used for CdTe film deposition.

Consecutively six CdTe thin films were deposited for 10 minutes at 650 °C source temperature and the temperature difference between source and substrate was kept 30°C. During the deposition, the CSS chamber pressure was maintained at 850 mTorr (Ar). The process parameters used during the deposition of CdS and CdTe films are summarized in Table 1. After deposition, all the samples were stored in a vacuum desiccator. A photograph of a deposited CdTe thin film is given in Fig.3.

Table 1. Process parameters used during the deposition of CdS and CdTe thin films.

CdS		CdTe	
Deposition Parameters	Value	Deposition Parameters	Value
RF Power	30 watt	Source temperature	650°C
Substrate Temperature	100°C	Substrate temperature	620°C
Ar Flow Rate	16 sccm	Chamber pressure	850-900 mTorr
Working Pressure	3.7×10^{-3} Torr	Process gas	Argon
Target-Substrate spacing	7 cm	Source-substrate spacing	2 mm
Deposition time	50 minutes	Deposition time	10 minutes

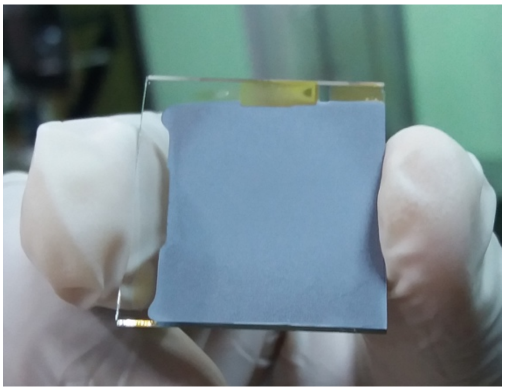

Fig. 3. A typical CdTe film deposited on CdS/BGS.

2.3. $CdCl_2$ Treatment

At the beginning of the wet $CdCl_2$ treatment process, $CdCl_2$ solutions of five different concentrations of 0.20 M, 0.25 M, 0.30 M, 0.35 M, and 0.40 M were prepared by mixing the proper amount of 99.99% pure $CdCl_2$ powder with DI water. The deposited CdTe samples were treated with the prepared $CdCl_2$ solutions to compare the film properties of treated samples with that of the as-deposited sample. The treatment was done by dipping the CdTe samples into a distinct $CdCl_2$ solution for 15 seconds. Then the samples were dried by a hand blower and baked on a hot plate for 5 minutes at 100°C. Thereafter, the samples were placed into MTI GSL-1100X-50-LVT-LD furnace for annealing. The annealing process was carried out for 25 minutes at 395°C [30]. During annealing, the vacuum pressure of the chamber was maintained at around 100 mTorr. After annealing, the samples underwent natural cooling to the room temperature inside the chamber. Finally, the samples were rinsed with warm DI water (60°C) for 60 seconds to remove $CdCl_2$ residues and subsequently dried by using a hand blower. Then the treated samples were baked on a hot plate to remove moisture from the films.

2.4. Characterization of the CdTe Films

The structural, surface morphological, and optical properties of the as-grown and $CdCl_2$ treated samples were investigated to observe the effects of $CdCl_2$ solution-concentration on these properties. To obtain a clear insight into the variation of structural properties with the concentration of $CdCl_2$-treatment-solution, the samples were analyzed by an X-ray diffractometer (XRD) from Pananalytical, which uses Cu-Kα (λ = 0.15408 nm) radiation source and records the XRD pattern in 2-Theta (degree) domain. The lattice type and lattice parameter, crystallite size, dislocation density, and micro-strain are determined from the XRD data. The surface morphology was recorded by using a JSM-7600F Schottky Field Emission Scanning Electron Microscope (FE-SEM). The films' optical transmittance was measured in the range of 220 nm to 1400 nm wavelengths using a SIMADZU UV-2600 plus UV-Vis-NIR spectrophotometer.

3. Results and Discussion

3.1. XRD Analysis

The effects of the concentration of $CdCl_2$ solution on the crystallographic properties of CdTe films are investigated from the XRD patterns shown in Fig.4. As seen in the figure, all the films show a prominent Bragg peak at around $2\theta \approx 23.7°$ which corresponds to the preferential growth orientation along the (111) plane of cubic CdTe material. The as-grown sample shows three other peaks corresponding to (220), (311), and (511) planes, which confirms the polycrystalline nature of the deposited films. For the samples treated with 0.20 M and 0.25 M $CdCl_2$-concentrations, the (111) peak intensity decreases, and a few more minor peaks appear corresponding to (400), (331), and (422) planes suggesting recrystallization and randomization of the grains [31] after treatment. As the concentration increases from 0.25 M to 0.35 M, most of the minor peaks disappear, and the peak intensity related to (111) plane starts to increase. This is due to the fact that increased $CdCl_2$ flux provides better recrystallization in the CdTe films. Remarkably, for 0.35 M solution concentration, all the minor peaks except the (511) peak disappear. As we further increased the concentration to 0.40 M, the smaller peaks start to reappear, and the prominent peak intensity is again reduced, indicating the randomization of the grains.

The $CdCl_2$ treatment causes diffusion of sulfur (S) from the CdS layer to the CdTe layer and thus creates a compositional variation by forming a beneficial $CdTe_{1-x}S_x$

layer at the CdS/CdTe interface. To study this compositional variation and strain relief, the (511) peak profile has been analyzed, which is shown in Fig.5. As seen in the figure, the (511) peak of the as-deposited sample occupies the largest lattice parameter side. The center of the (511) peak continues to shift to the lower lattice parameter region with the increase in $CdCl_2$-concentration up to 0.35 M. This is attributed to either stress relief from the films or a compositional variation at the CdS/CdTe interface by the formation of $CdTe_{1-x}S_x$ alloy as a result of S diffusion into the CdTe films [23, 32]. However, no additional peak shift is observed for concentration above 0.35 M, and the samples treated with 0.35 M and 0.40 M solutions show better S diffusion compared to the samples treated with lower concentrations.

The ascertainment of the lattice constant is of fundamental importance in order to realize the effects of $CdCl_2$ treatment on the texture of the CdTe films [23]. The Bragg's law and Vegard's law [33-34] are used to determine the interplanar distance, d_{hkl}, and the lattice parameter for the corresponding plane, a_{hkl} of the cubic CdTe thin films.

$$d_{hkl} = \frac{\lambda}{2} \times \cos ec(\theta) \tag{1}$$

$$a_{hkl} = d_{hkl} \times (h^2 + k^2 + l^2)^{0.5} \tag{2}$$

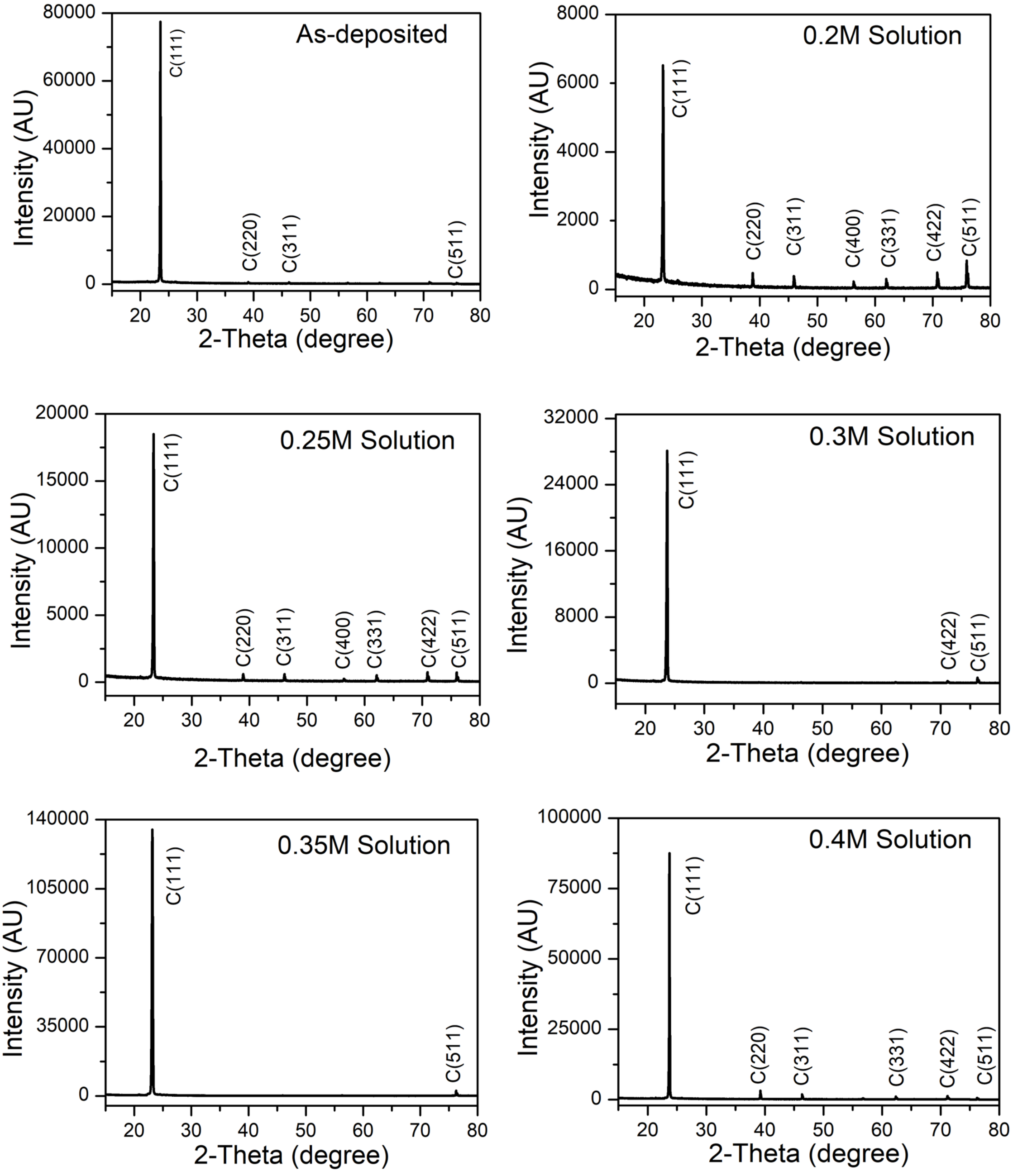

Fig. 4. XRD scans of as-grown and $CdCl_2$ treated CdTe samples grown on CdS/BGS.

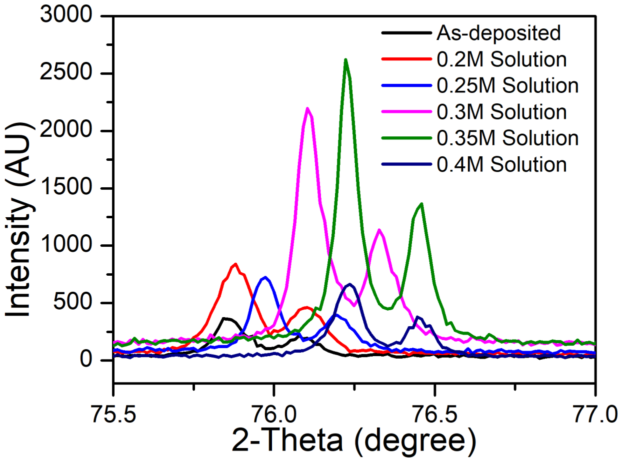

Fig. 5. (511) peak profile of the as-deposited and $CdCl_2$ treated CdTe films.

In Eq. (1) and (2), the λ stands for the wavelength, and the *θ* denotes the angle in radian formed between the crystallographic planes and the incident X-rays. The lattice constant, *a,* of each sample is estimated from the plot of the calculated value of a_{hkl} for each indexed peak in the XRD pattern with respect to the Nelson-Riley function by taking a linear regression of the points in the curves and determining the *y*-intercept where the function becomes zero. The intersection point on the *y*-axis provides an accurate value of the lattice constant [35]. The graphs for both the as-grown and treated samples with $CdCl_2$ are shown in Fig.6.

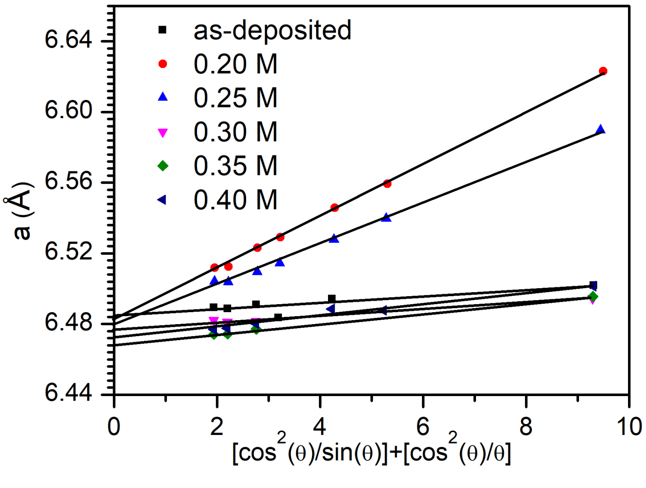

Fig. 6. Lattice constant of as-deposited and $CdCl_2$ treated CdTe films for different treatment-solution concentrations.

The lattice constant of the as-deposited film (a≅6.487 Å) is greater than that of powder CdTe (a≅6.481 Å), which indicates the existence of in-plane compressive stress acting perpendicular to the growth axis, i.e., in the growth plane of the films [32]. After heat treatment with a 0.20 M $CdCl_2$ solution, the lattice constant is reduced to 6.484 Å, suggesting that some of the in-plane stress is relieved, but it's still larger than the powder sample indicating to partial recrystallization. The in-plane stress is farther released for treatment with a 0.25 M $CdCl_2$ solution since the lattice constant reduces to 6.48 Å. An important peculiarity is observed in the XRD patterns of the CdTe films treated with 0.30 M, 0.35 M, and 0.40 M $CdCl_2$ solutions. The lattice constant of these films (6.476 Å, 6.469 Å, and 6.472 Å respectively) are lower than the one for powder CdTe sample which is attributed to the S diffusion into the CdTe lattice and hence the formation of the $CdTe_{1-x}S_x$ alloy at the CdS/CdTe interface [23]. The lowest lattice constant and hence, the maximum amount of S diffusion is obtained for the $CdCl_2$ solution of 0.35 M concentration. For this particular case, the S content, *x*, in the $CdTe_{1-x}S_x$ alloy is calculated according to the Vegard's law as shown in Eq. (3) [32], which is about 2%. The result is consistent with the previously reported result [23].

$$x = 1.508 \times (6.481 - a) \tag{3}$$

The crystallite size, micro-strain, and dislocation density are calculated from the XRD patterns to obtain a better understanding of the variation of the structural properties in terms of the $CdCl_2$ solution concentration. During the synthesis of any thin film, some atoms are displaced from their original lattice points. This wrong placement of atoms is called the lattice 'misfit.' A large number of lattice misfits can result in a lattice mismatch, which further develops a micro-strain in the crystal and deteriorates the long term performance stability of the CdTe absorber layer. Hence, a lower value of micro-strain is preferred for any thin film to be used in device fabrication. So, it is very important to study the micro-strain developed in the thin films during their deposition. Another important structural property is the dislocation density, which is considered as an imperfection in the crystal arising because of the density gradient of lattice points among the parts of a crystal [7]. Similar to the strain, the dislocation density should be small in a thin film. The Debye-Scherrer and Williamson-Smallman's formulas have been utilized to find numerically the crystallite size *D* and dislocation density, δ, respectively, using Eq. (4) [36] and (5) [37], whereas the micro-strain, *ε,* is calculated according to the relationship as in Eq. (6) [38].

$$D_{hkl} = \frac{0.9\lambda}{\beta cos(\theta)} \tag{4}$$

$$\delta = \frac{15\varepsilon}{\alpha D} \tag{5}$$

$$\varepsilon = \frac{\beta}{4tan\theta} \tag{6}$$

Where *β* refers to the Full-Width at Half Maxima (FWHM) of the corresponding diffraction peaks. The calculated values of these structural parameters are presented in Table 2. As observed in the table, the crystallite size is small for lower concentrations. As the concentration of the treatment solution increases, the crystallite size also increases, presumably due to improved recrystallization in the films caused by the increased amount of $CdCl_2$ flux. The largest value of crystallite size is obtained for 0.35 M $CdCl_2$-concentration beyond which the crystallite size reduces. The table also shows that the estimated values of micro-strain, as well as dislocation density, are higher in the films treated with $CdCl_2$ solution of lower concentrations. Both these properties show a decreasing behavior with the increasing concentration to a certain limit of 0.35 M.

Table 2. Structural parameters of the as-deposited and $CdCl_2$ treated CdTe films for various treatment-solution concentrations.

$CdCl_2$ solution concentration	Crystallite Size, D (nm)	Micro Strain, ε ($\times 10^3$)	Dislocation Density, δ ($\times 10^{11}$ cm^{-2})
As-deposited	35.28	4.82	6.52
0.20 M	48.67	3.53	1.64
0.25 M	49.00	3.49	1.62
0.30 M	56.77	2.97	1.21
0.35 M	75.72	2.23	0.68
0.40 M	41.83	4.12	2.22

3.2. Surface Morphology

It is reported that the $CdCl_2$ treatment enlarges the grains as well as eliminates the defect states, which consequently lessens the probability of electron-hole recombination and increases the lifetime of the minority carriers [39]. During annealing, the $CdCl_2$ sintering flux is decomposed into Cd and Cl and initiates the consolidation of smaller grains into larger grains according to the following chemical reactions [39]:

$$CdTe\ (s) + 2\ Cl_2\ (g) \longrightarrow CdCl_2\ (g) + TeCl_2\ (g) \qquad (7)$$

$$CdCl_2\ (g) + TeCl_2\ (g) \longrightarrow CdTe\ (s) + 2\ Cl_2\ (g) \qquad (8)$$

The CdTe product of the chemical reaction in Eq. (8) are supposed to have larger grains than the CdTe of the chemical reaction in Eq. (7) because of the Cl assisted recrystallization of the CdTe grains during the annealing process. The effect of $CdCl_2$ solution-concentration on CdTe grain size is studied from the FE-SEM micrographs shown in Fig.7. A compact and uniform surface morphology is observed throughout the investigated area of the substrates.

The average grain size of the samples is estimated by ImageJ software using the mean linear intercept method. The results are summarized in Table 3. As seen in the table, the grain size of the CdTe samples increases with treatment solution concentration. The largest grain size is obtained for 0.35 M concentration. Further increase in concentration causes a reduction in the grain size, which is accompanied by small voids in the film, presumably due to excessive chlorine diffusion into the CdTe lattice. The trend of variation of grain size of the treated CdTe films with the solution concentration corroborates the trend of crystallite size estimated from XRD analysis. This result is also in good agreement with the previously reported result [7].

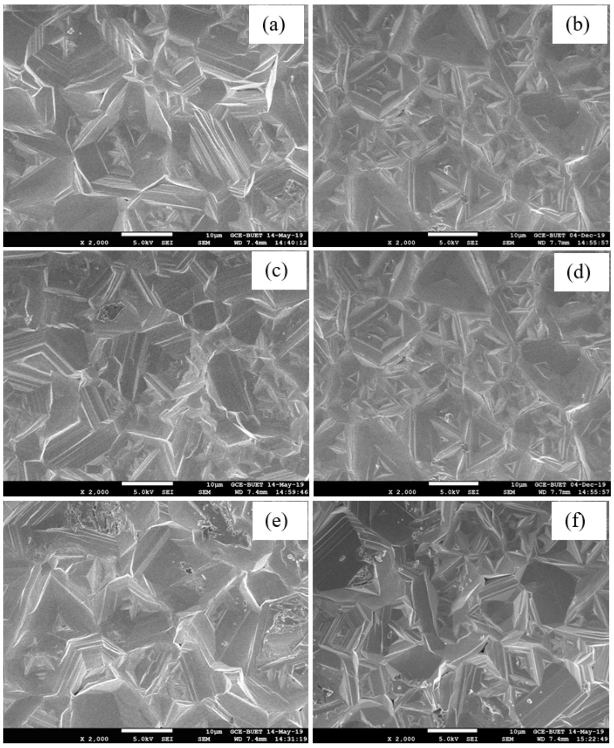

Fig. 7. SEM micrographs of (a) as-deposited and treated CdTe films for (b) 0.20 M, (c) 0.25 M, (d) 0.30 M, (e) 0.35 M, and (f) 0.40 M $CdCl_2$ concentrations.

Table 3. Grain size of the as-deposited and $CdCl_2$ treated CdTe thin films.

Sample	As-grown	Treated with $CdCl_2$ solution of molar concentration (M)				
		0.20	0.25	0.30	0.35	0.40
Grain Size (µm)	8±2	9±2	11±2	14±2	15±2	12±2

3.3. Optical Analysis

The optical transmittances of the as-deposited and treated samples are shown in Fig.8. The transmittance of both the untreated and treated samples approaches zero for the visible wavelengths, which is an indication of complete absorption of photon flux in that region. All the samples show a sharp transition edge, which is a sign of high crystalline property of the as-deposited and $CdCl_2$ treated films.

The bandgap of the CdTe films are calculated graphically from the Tauc plot shown in Fig.9. It is observed that the bandgap varies within a narrow margin of 1.42 eV to 1.45 eV with the variation of concentration of the $CdCl_2$ solution. The calculated bandgaps for as-deposited, 0.20 M, 0.25 M, 0.30 M, 0.35 M, and 0.40 M concentrations are of 1.45 eV, 1.43 eV, 1.43 eV, 1.44 eV, 1.42 eV, and 1.45 eV respectively. The minimum bandgap (1.42 eV) among the investigated samples is obtained for 0.35 M concentration, which upholds both the microstructural and surface morphological analysis. It is expected that the bandgap reduction will enhance the photon absorption efficiency of the CdTe absorber layer and hence, increase the photogenerated current density of solar cell devices.

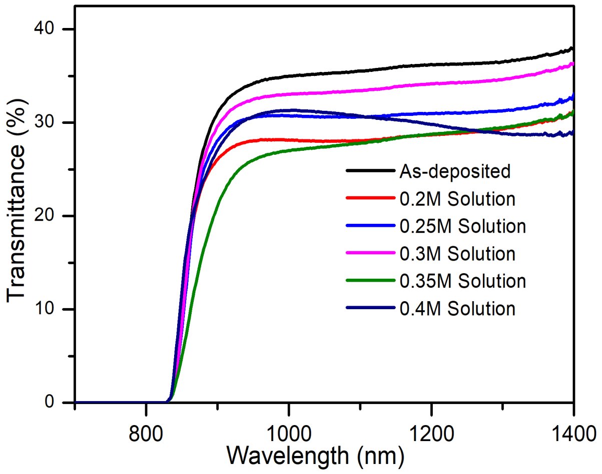

Fig. 8. Transmittance of the as- deposited and $CdCl_2$ treated CdTe films grown on CdS/BGS.

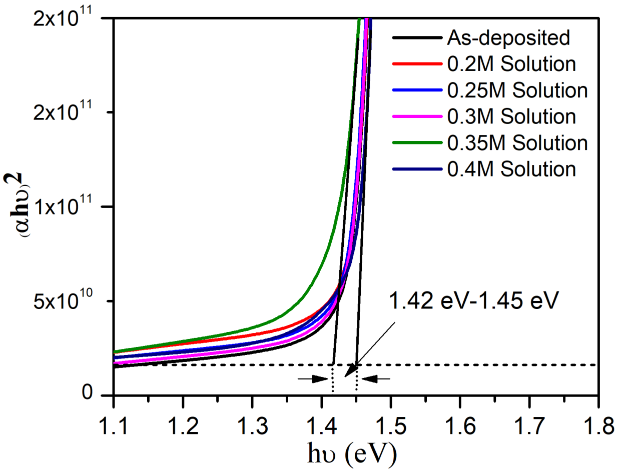

Fig. 9. Optical bandgap of the as- deposited and $CdCl_2$ treated CdTe films grown on CdS/BGS.

4. Conclusion

In this paper, the effects of the $CdCl_2$ solution concentration on the physical properties of CdTe thin films are studied. The XRD analysis reveals that the films were grown with a cubic structure of CdTe having a predominant orientation along the (111) plane. While the treatment doesn't affect the preferential orientation of the films, it promotes the recrystallization in the CdTe films and affects some structural properties such as crystallite size, lattice micro-strain, in-film stress, and dislocation. These crystallographic properties show respective improvements with increasing $CdCl_2$-concentration up to a certain limit of 0.35 M, whereas deterioration of these properties is observed beyond this molar concentration. The analysis of both the (511) peak and lattice constant clarify that the best S diffusion into the CdTe lattice occurs for 0.35 M concentration, which is estimated at about 2% ($CdTe_{0.98}S_{0.02}$). The FE-SEM micrographs present that the grain size increases with the increasing $CdCl_2$ concentration, where the largest grain size (15 μm) is found for 0.35 M concentration. The bandgaps of both the untreated and treated films are found in the vicinity of the 1.45 eV, which is the standard value for the CdTe powder sample whereas, 0.35 M treated CdTe film shows the lowest bandgap (1.42 eV) among all the samples. This can be attributed to the better crystallinity of the sample evident from both XRD and SEM results. The achieved microstructural, morphological and optical properties of CdTe thin films treated with $CdCl_2$ solution of 0.30 M and 0.35 M concentrations confirm their potentiality to be exploited as the absorber layer in high-performance CdTe solar cells.

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