



Characterization of CdTe thin films with different compositions obtained by CMBD for thin film solar cells



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ABSTRACT

CdTe thin films with different compositions, i.e. Cd/Te ratio of ~0.79, 0.88, 0.94, 1.01, 1.05, 1.08, were fabricated by a novel and low cost chemical molecular beam deposition method (CMBD) under hydrogen flow in atmospheric pressure. For this purpose, Cd and Te granules were used as precursors. Thin films were deposited on glass and metal substrates at 600 °C. The growth rate was varied in the range of (10–30) Å/s. The composition ratio in the samples was controlled by varying the molecular beam intensity (MBI) ratio. The morphological, structural, electrical and optical properties of the CdTe thin films were investigated by SEM, XRD, Hall-effect and photoluminescence (PL) measurements, respectively.

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1. Introduction

Cadmium telluride (CdTe) is one of the most suitable materials for thin film solar cells mainly due to two physical properties. Namely, they are its optimal bandgap ($E_g = 1.5$ eV) and high absorption coefficient (5.1×10^5 cm⁻¹), which give possibility to harvest photons effectively in the visible region of the solar radiation (Horodyský and Hlídek, 2006; Fonthal et al., 2000).

Table 1 summarizes the latest advances on the efficiency of the CdTe based thin film solar cells. As show, from year to year, the efficiency is monotonically increasing. Recently, First Solar has reported the world record conversion efficiency, i.e. 21.5% in laboratory conditions and 16.1% in modules (<http://investor.firstsolar.com/>). As depicted in Table 1, the short circuit current and fill factor have reached their practical maximum. Unlike, the improvement on the open-circuit voltage has been only in the range of 50 mV over the last two decades, which can be addressed to further increase the conversion efficiency of the devices. Solution of this problem requires deep studies on the

physical properties of the CdTe absorber layer. From this point of view, many scientific works have devoted to the study the physical properties of the CdTe thin films prepared by various methods. For example, Aramoto and coworkers studied the morphological and the structural properties of the CdTe thin films, obtained by the low-temperature method of electrodeposition (ED) and physical vapor deposition (PVD) (Aramoto et al., 1997). In general, the efficiency of the CdTe thin film solar cells, i.e. obtained by low-temperature methods, is found to be lower than 15%, whereas thin films prepared by high-temperature growth methods have yielded higher efficiencies, which are above 15% (Ferekides and Britt, 1993). Hence, all CdTe thin films investigated in this work were prepared at high growth temperature of 600 °C.

Another important step to obtain CdTe thin film solar cells with high efficiency is a post deposition heat treatment of the CdTe layer with CdCl₂ (Girish Kumar and Koteswara Rao, 2014). In the past several decades, many research works have been done to determine the mechanisms of the CdCl₂ treatment and to find out the suitable ways to carry out the treatment (McCandless and Birkmire, 1990. Mahathongdy et al., 1998).

The effects of the CdCl₂ treatment may vary depending on the initial CdTe deposition techniques as well as on applied method of the heat treatment. In general, the treatment involves to deposit a thin CdCl₂ layer, which follows by annealing step. For instance,

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Table 1
Reported conversion efficiencies of the CdS/CdTe based thin film solar cells, obtained by various methods and research groups (Razykov et al., 2011; <http://investor.firstsolar.com/>).

Year	Type	CdS and CdTe/process	Activation process	V _{xx} (mV)	I _{sc} (mA/cm ²)	FF (%)	η (%)	Area (cm ²)	Group
1991	Glass/SnO ₂ :F/CdS/CdTe/Au	PM/CSS	CdCl ₂ heat treatment	840	21.9	72.6	13.4	1.20	USF
1992	Glass/SnO ₂ :F/CdS/CdTe/Au	PM/CSS	CdCl ₂ heat treatment	850	24.4	70.5	14.6	1.05	USF
1993	Glass/SnO ₂ :F/CdS/dTe	CBD/CSS	CdCl ₂ heat treatment	843	25.1	74.5	15.8	1.05	USF
2001	Glass/CTO/ZTO/CdS/CdTe	CBD/CSS	Thermal evaporation of CdCl ₂	845	25.9	75.5	16.5	1.03	NREL
2013	Glass/CTO/ZTO/CdS/CdTe	CBD/CSS	Thermal evaporation of CdCl ₂	875	28.9	78.0	19.7	1	First Solar
2014	Glass/CTO/ZTO/CdS/CdTe	CBD/CSS	Thermal evaporation of CdCl ₂	872	29.5	79.5	20.4	1	First Solar
2014	Glass/CTO/ZTO/CdS/CdTe	CBD/CSS	Thermal evaporation of CdCl ₂	903	30	79.8	21.5	1	First Solar

PM, CSS, CBD, USF and NREL refer to the Printing method, closed space sublimation, chemical bath deposition, University of South Florida and the National Renewable Energy Laboratory, respectively.

precipitation of CdCl₂ layer on the CdTe surface by dipping the CdTe layer in a heated CdCl₂ solution and methanol (CH₃OH) followed by drying is more common. However, in this method it is difficult to control the concentration of the chemicals (Moutinho et al., 2000). In order to control the chemical composition of CdCl₂ layer, vapor CdCl₂ treatment is often used as an alternative. In this method, CdTe layer is exposed to flux of CdCl₂ vapor, followed by an annealing step to form a thin layer. Many researchers have applied this method to obtain CdTe devices with high efficiency. Being in line with literature data, this technique was also used in this study. In addition to CdCl₂ treatment, other treatments have also been reported, namely, vapor HCl, vapor Cl₂, MnCl₂:C₅H₅N solution, CdCl₂ mixed with CdTe screen print paste, and in-situ CdCl₂ treatments (Zhou et al., 1994. McCandless et al., 1996).

One of the main effects of the CdCl₂ treatment on electrical properties is that the introduced Cl forms an acceptor complex with Cd vacancies (McCandless and Sites, 2004). At the same time, the CdCl₂ treatment is also believed to passivate grain boundary defects which act as recombination sites within the CdTe bandgap (Moutinho et al., 1998). This is evident by an increase in minority carrier lifetime after treatment (Metzger et al., 2006).

Furthermore, it has also been shown that the CdCl₂ treatment promotes intermixing of the CdS and CdTe layers (Al-Douri et al., 2010) and diffusion of the sulfur from the CdS into the CdTe layer (Metzger et al., 2006. Al-Douri et al., 2015). The formation of CdTe_{1-x}S_x and CdS_{1-y}Te_y alloys at the CdS/CdTe interface leads to consumption of the already thin CdS layer and can significantly affect device performance (Moutinho et al., 2000. Potter et al., 2000). Significant sulfur diffusion will only occur in the presence of a CdS layer and CdCl₂ treatment. Only heat treatment, i.e. without CdCl₂ treatment, will not prompt any major sulfur diffusion. It has been shown that the high temperature annealing (T_{an} = 450 °C) of the CdTe layer prior to the CdCl₂ will greatly reduce the amount of sulfur diffusion from the CdS layer (McCandless et al., 1999). It has been argued that the high temperature annealing prior to the CdCl₂ treatment promotes further recrystallization of the CdTe layer which reduces the volume of grain boundaries which act as diffusion paths.

Depending on the method of deposition of the CdTe layer, the CdCl₂ can promote grain growth, recrystallization, and change the degree of preferred orientation (McCandless and Sites, 2004). It has been proposed that the chlorine diffusion into the CdTe lattice in the early stages of the treatment creates additional defects at grain boundaries which creates lattice strain and initiates the recrystallization process (Moutinho et al., 1998). As known, some deposition methods (PVD, ED, sputtering, metalorganic chemical vapor deposition (MOVCD)) yield a substantial increase in grain size and change in preferred orientation while others (CSS, spray, vapor transport deposition (VTD)) do not show any considerable grain growth or changes in preferred orientation. For example, the study by Moutinho, it was highlighted that the differences between CdTe layers prepared by PVD and CSS (McCandless

et al., 1999). The major factors that prevent secondary grain growth during the CdCl₂ treatment are high temperature deposition of the CdTe layer, large initial grain size, and annealing before the CdCl₂ treatment (McCandless and Sites, 2004. Moutinho et al., 1997).

In the article, the morphological, structural, electrical and photoluminescence (PL) properties of the CdTe thin films were studied before and after CdCl₂ heat treatment. For this purpose, The CdTe thin films were prepared by chemical molecular beam deposition (CMBD) by controlling the ratio of the metal and chalcogenide components during the vapor phase of the growth process. This method has several advantages compared to CVD or molecular beam epitaxy (MBE) techniques, such as: (1) deposition process is carried out in the atmospheric pressure gas flow, which does not require additional (expensive) vacuum equipments, (2) deposition process can be controlled on molecular level that allows to control the film composition and other physical properties precisely, (3) deposition rate can be varied in the wide range, e.g. 10⁻¹–10⁴ Å/s, (4) thin films can be deposited on a large area (50 cm² on existing equipment), (5) extrinsic dopants can be introduced during the growth process, (6) fabrication of thin Te rich layer on CdTe films is possible during the growth process, which is necessary to avoid additional chemical treatment.

2. Experimental

CdTe thin films were obtained by CMBD at atmospheric pressure in a stream of hydrogen. Before the deposition, the substrates, i.e. borosilicate glass and molybdenum foil with purity of 99.96%, were cleaned by ultrasonic bath and dried in nitrogen. The elements Cd and Te (with purity of 99.999%) were used as starting substances. The composition of the films was controlled by varying the intensity of molecular beams (IMBs) of both metal and chalcogenide in the vapor phase (Razykov, 1991). The samples investigated in this work have the Cd/Te IMBs ratios of about 0.8, 0.88, 0.94, 1.01, 1.05 and 1.08. The thin film deposition was carried out at a substrate temperature of 600 °C.

The sign of the thermoelectric power of defined-type conductivity was showed that all samples have *p*-type conductivity. The thickness of the films was measured by micro interferometer (MII-4), and double weight methods by measuring the mass of the samples before and after deposition to calculate their thickness. The thickness of the samples was found to be (2–5) μm, where the thickness variation mainly depends on the deposition time.

The heat treatment with the presence of CdCl₂ was carried out in a closed space (in pure argon) to improve the electrical and structural properties of the CdTe thin films. The process consists of the following steps: (a) CdCl₂ thin film with thickness of 200–300 nm is deposited on CdTe thin films in a high vacuum; (b) then thermal annealing was carried out in argon environment at 400 °C

for 40 min. After annealing the films were cleaned with deionized water and dried by nitrogen flow in order to remove CdCl_2 precipitates.

Nickel (Ni) and silver paste, i.e. prepared on the annealed CdTe thin films by vacuum deposition, were used as ohmic contact to investigate the electrical transport properties of the samples.

The morphological, structural and photoluminescence properties of samples were investigated by scanning electron microscope (SEM), X-ray diffraction (XRD) and photoluminescence (PL) measurements, respectively.

3. Results and discussion

Fig. 1 shows SEM images as-grown CdTe thin films for different Cd/Te composition ratios. As shown, the microstructure (grain size and shape) of the samples with Cd/Te ratios of 0.94, 1.01, 1.05 and 1.08, i.e. close to a stoichiometric composition, have similar appearance. These samples have well oriented polycrystalline structure and grain size of 2–3 μm , but the structure (grains) of the films is not closely packed. On the other hand, the films with higher tellurium, i.e. Cd/Te of 0.79 and 0.88, also have well oriented polycrystalline structure with the grain size of 3–5 μm , but the texture (grains) of these films is more denser compared to those of other samples.

Fig. 2 depicts the SEM images of the samples with a near-stoichiometric compositions after heat treatment. The samples were obtained on glass and molybdenum substrates. As shown, the morphology of the films changed and the presence of a uniform surface was observed after CdCl_2 heat treatment. The results shows the following: (a) the films have well-oriented polycrystalline structure with a grain size of (2–5) μm ; (b) after CdCl_2 heat treatment, morphology of the samples changed. This is due to the fact that during the heat treatment, the recrystallization of the film structure and realignment of the grain size take place. At the same time, the disappearance of small grains is observed. Furthermore, large grains break down into smaller ones, thus the film structure becomes more closely packed.

Diffraction patterns for the CdTe films grown in a hydrogen stream with different Cd/Te ratios are shown in Fig. 3. XRD spectra for all samples have an intense peak at $2\theta = 23.7^\circ$, which corresponds to the (1 1 1) crystallographic axis. This shows that all CdTe films have a sphalerite structure with (1 1 1) orientation. In addition, weak peaks were also observed at $2\theta = 39.2^\circ$, 46.4° , 56.7° ,

62.4° and 71.2° , corresponding to the (2 2 0), (3 1 1), (4 0 0), (3 3 1) and (4 2 2) crystallographic orientations, respectively (cf. Fig. 3).

The film having the composition of Cd/Te of 0.94 the intensities of the X-ray peaks corresponding to the crystallographic orientation of (2 2 0), (3 1 1) and (4 2 2) slightly increased compared to other samples. This may be due to the different thickness of the CdTe films, as this behavior was also observed in (Salavei et al., 2013). In all films, (1 1 1) orientation was predominant (cf. Fig. 4). All these samples had the lattice constant of $a = 6.487 \text{ \AA}$ before and after heat treatment.

Fig. 4 shows the XRD spectra of samples prepared on glass substrates after heat treatment and on metal substrates before and after heat treatment. As depicted, all of the samples have a maximum peak at about $2\theta = 23.7^\circ$, which corresponds to the (1 1 1) crystallographic axis. After heat treatment, the intensity of (1 1 1) peak of all samples, i.e. except Cd/Te – 0.94, decreased and the intensity of (2 2 0), (3 1 1), (4 0 0), (3 3 1) and (4 2 2) peaks slightly increased. At the same time, the weak peak corresponding to the (4 0 0) orientation for film with Cd/Te – 1.08 increased considerably. This situation is also visible for film with Cd/Te – 1.0, which was grown on metal substrate. In addition, similar results with weak peaks were observed on spectra, which are related to molybdenum substrate (Dhar et al., 2015). On the other hand, for the sample having a composition of Cd/Te = 0.94, the intensity of (1 1 1) peak increased and almost all weak peaks decreased.

Furthermore, the results obtained by XRD were analyzed to determine the preferred orientation of the grains in CdTe films using the following formula (Loginov et al., 1996):

$$C_i = \frac{I_i/I_{oi}}{\left(\frac{1}{N}\right)\sum_{i=1}^N I_i/I_{oi}} \quad (1)$$

where C_i is the texture coefficient; I_i is the total peak intensity in the spectrum; I_{oi} is the intensity of a randomly selected total peak; and N is the number of reflections considered in the analysis. The texture coefficient determines the orientation of each reflection in a plane. When the value of C_i is less than or equal to 1, then the film grains grow in a random orientation, and if the value of $C_i > 1$, then the grains have a preferred orientation in this direction. As can be seen from Table 2 and Fig. 4, all the films obtained by chemical molecular beam deposition exhibit the preferred (1 1 1) orientation. The same results were also obtained in (Ferekides et al., 2013), when the films were obtained by elemental vapor transmission.

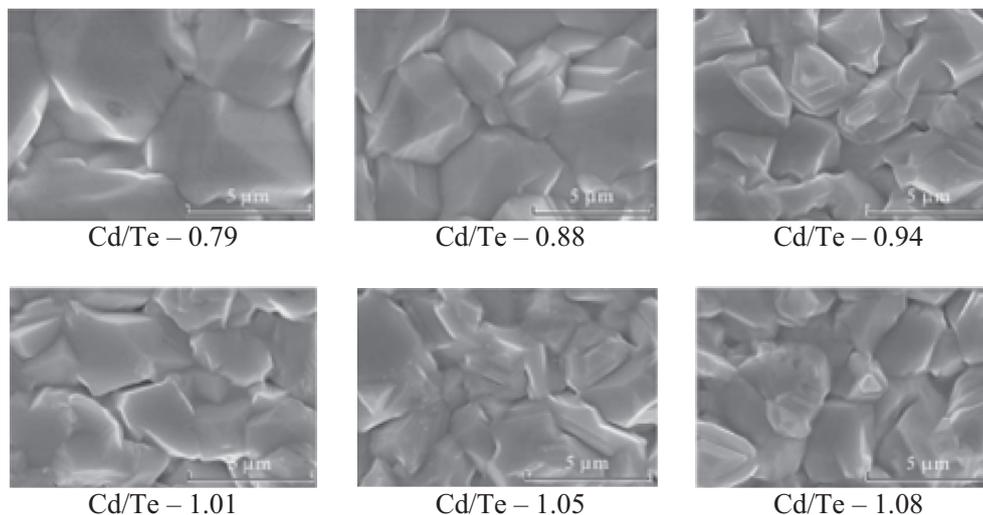


Fig. 1. SEM images of as-grown CdTe thin films for different composition.

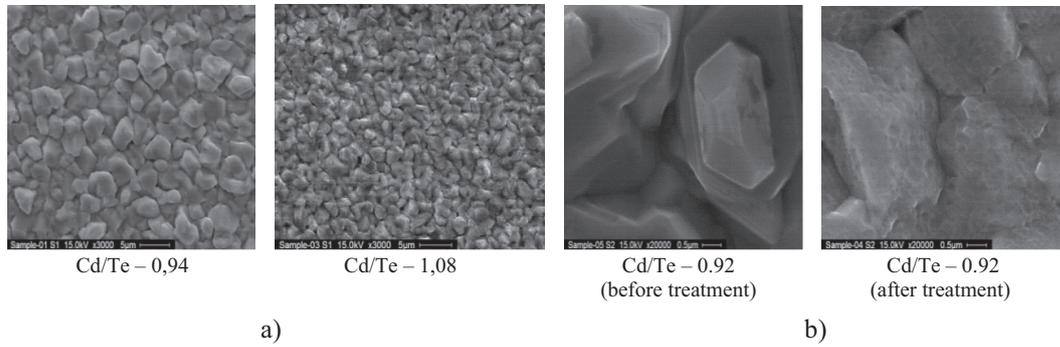


Fig. 2. The SEM images of the film structures after CdCl₂ heat treatment on (a) glass substrate and (b) Mo substrates.

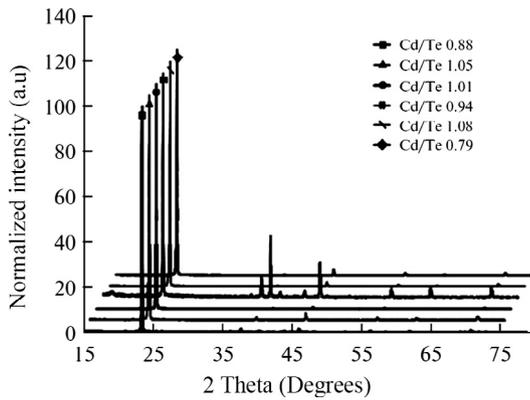


Fig. 3. X-ray diffraction spectra of as-grown CdTe thin films on glass substrates as a function of different composition.

To analyze the preferential orientation of each sample as a whole, the standard deviation of all the C_i values were used compared to randomly oriented samples:

$$\delta_i = \sqrt{\frac{\sum_{i=1}^N (C_i - 1)^2}{N}}$$

Values of δ can be used to compare the degree of orientation between samples. A value of 0 indicates a completely random sample. However, all samples showed that >0 , which corresponds to the cubic structure of CdTe, i.e. (111) orientation. The analysis results of the data given in Figs. 3–5 are presented in Table 2.

Furthermore, current–voltage (CV) characteristics of CdTe films were also measured, i.e. under dark and illuminated conditions, before and after heat treatment. The dark CV characteristics of the films showed (not given here) “ohmic” behavior, i.e. no rectification in the range of measured voltages of between (0.01 and 2.0) V. The electrical transport properties, e.g. resistivity, carrier concentration, mobility and the type of the conductivity, were obtained by Hall-effect measurement using van-der-Pauw method (cf. Table 3). The resistivity of the films were found to be in the range of 10^3 – 10^8 Ω cm, the carrier concentration is in the range of 10^7 – 10^{12} cm^{-3} and the mobility is in the range of $(1.5$ – $3.6) \times 10^2$ $\text{cm}^2/\text{V s}$ depending on the composition and the heat treatment. CdCl₂ heat treatment significantly reduced the resistivity of stoichiometric CdTe film from 10^8 to 10^6 Ω cm owing to the increase in carrier concentration from 10^7 to 10^9 cm^{-3} . This can be under-

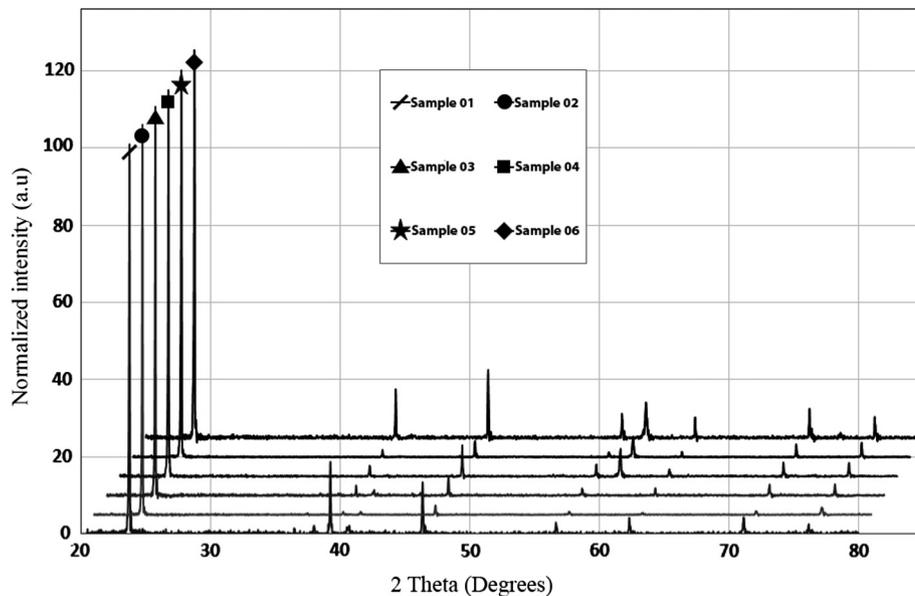


Fig. 4. X-ray diffraction spectra of CdTe films for different composition ratios: sample 01 (on glass) - Cd/Te 0.88, after treatment; sample 02 (on glass) - Cd/Te of 0.94, after treatment; sample 03 (on glass) - Cd/Te 1.08, after treatment; sample 04 (on Mo) - Cd/Te 0.92, after treatment; sample 05 (on Mo) - Cd/Te 0.92, before treatment; sample 06 (on Mo) - Cd/Te 1, after treatment.

Table 2
The texture coefficient (C_i) values for CdTe films of various compositions before and after CdCl₂ treatment.

Cd/Te (analysis)			C_i						δ
Substrate	Treatment	Cd/Te	(111)	(220)	(311)	(400)	(331)	(422)	
Glass	No	0.79	4.7	0.02	0.31	0.5	0.11	0.35	1.66
Glass	No	0.88	5.23	0.016	0.18	0.25	0.11	0.2	1.9
Glass	No	0.94	2.0	0.85	0.85	0.9	0.73	0.66	0.46
Glass	No	1.01	5.38	0.017	0.11	0.22	0.09	0.18	1.96
Glass	No	1.05	4.12	0.1	0.36	0.55	0.52	0.54	1.4
Glass	No	1.08	4.73	0.025	0.27	0.46	0.17	0.35	1.67
Glass	Yes	0.79	2.43	0.7	0.87	0.77	0.78	0.7	0.6
Glass	Yes	0.94	4.73	0.056	0.3	0.4	0.18	0.26	1.55
Glass	Yes	1.08	2.99	0.11	0.36	1.54	0.43	0.52	1.05
Mo	Yes	0.92	2.96	0.12	0.63	0.97	0.41	0.67	0.87
Mo	No	0.92	3.47	0.096	0.38	0.46	0.35	0.71	1.1
Mo	Yes	1.0	1.83	0.35	0.85	1.21	0.74	0.85	0.43

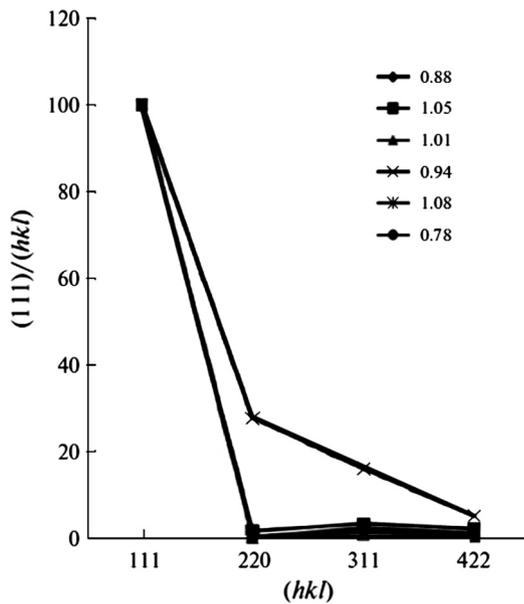


Fig. 5. Intensity ratios of X-rays peaks for CdTe films with different Cd/Te composition ratios.

stood from the fact that oxygen occupies the tellurium vacancy V_{Te}^{++} (Gessert et al., 2013). Hence, the donor level at $E_C - 0.48$ eV was passivated and the acceptor level at $E_V + 0.15$ eV was activated (Gessert et al., 2013). The resistivity of Te-rich films did not change significantly and remained in the range of $10^3 - 10^4 \Omega \text{ cm}$ before and after heat treatment. So, low resistivity p-CdTe films can be fabricated by deviating the composition of samples from stoichiometry to Te-rich. However, the decrease in the resistivity of CdTe films under CdCl₂ treatment is limited to a value of $10^5 - 10^6 \Omega \text{ cm}$. The resistivity of CdTe films up to $10^5 - 10^6 \Omega \text{ cm}$ can be reached by deviating the composition from stoichiometry to Te-rich for p-CdTe films and Cd-rich for n-CdTe films grown by CMBD. So, CdCl₂ heat treatment and the CdTe precursor are possi-

bly not necessary for CdTe films fabricated by CMBD method. This novel and low cost method can successfully be used for fabrication of high efficiency thin film CdS–CdTe solar cells.

PL measurements were also performed on CdTe thin films after CdCl₂ heat treatment. The PL spectra of the samples measured at low temperatures, i.e. 10 K, were shown in Fig. 6. The composition (ratio of Cd/Te) of these samples was 0.93, 0.94 and 1.1. Experimental results showed that all the samples have a high peak at 1.40–1.41 eV and the low (blurred) peaks in the region of 0.9–1.15 eV. PL peak at 1.41 eV was also observed in Ferekides et al. (2007) and Armani et al. (2007). This peak is caused by transition of electrons from the conduction band to the acceptor level $E_V + 0.15$ eV and may be attributed to the vacancy of cadmium V_{Cd} -related defect or the interstitial atoms of tellurium Te_i in CdTe films. A blurred peak in the 0.9–1.15 eV requires future investigation.

4. Conclusion

CdTe thin films with different composition were prepared on glass and metal substrates by CMBD method. All samples have p-type conductivity. The CdCl₂ heat treatment were carried out at a temperature of 400 °C for 40 min in a closed space (in pure argon).

The analysis of SEM images showed that the overall surface morphology of the CdTe thin films with different compositions did not change significantly after the CdCl₂ heat treatment. However, some definite trend in terms of the changes in the shape and size of the CdTe grains were observed.

The analysis of XRD results showed that the films of different compositions obtained on both glass and molybdenum (Mo) substrates have a sphalerite structure with a strong preferential (111) orientation before and after CdCl₂ heat treatment.

The CdCl₂ heat treatment significantly decreased the resistivity of stoichiometric CdTe films from 10^8 to $10^6 \Omega \text{ cm}$ owing to the increase in carrier concentration from 10^7 to 10^9 cm^{-3} .

PL spectra of CdTe films with various compositions showed that all samples have a high peak at 1.40–1.41 eV and low (blurred) peaks in the region of 0.9–1.15 eV.

Table 3
Electrical parameters of CdTe films with different compositions before and after CdCl₂ treatment.

Cd/Te	Resistivity ($\Omega \text{ cm}$)		Mobility ($\text{cm}^2/\text{V s}$)		Carriers concentration (cm^{-3})		Type of conductivity	
	Before	After	Before	After	Before	After	Before	After
0.88	2.4×10^3	1.4×10^3	185	263	1.4×10^{12}	1.7×10^{13}	p	p
0.94	3.4×10^4	2.5×10^4	153	178	1.2×10^{12}	1.4×10^{12}	p	p
1.01	7.4×10^8	6.4×10^6	367	208	2.3×10^7	4.7×10^9	p	p
1.05	6.4×10^8	5.4×10^6	297	231	3.3×10^7	5.0×10^9	p	p

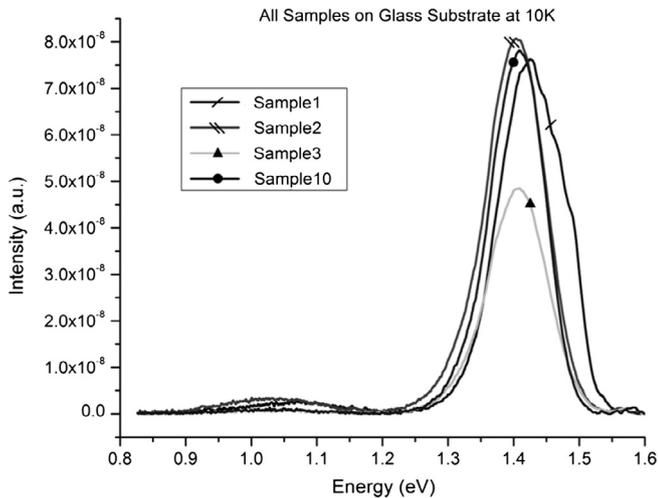


Fig. 6. PL spectra of CdTe of various composition after heat treatment with CdCl₂. Sample 1: Cd/Te = 0.93. Sample 2: Cd/Te = 0.94. Sample 3: Cd/Te = 1.1. Sample 10: Cd/Te = 0.94 without treatment.

The results can be implemented for producing high efficiency CdTe thin film solar cells.

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