

Revolutionary novel and low cost CMBD method for fabrication of CdTe absorber layer for use in thin film solar cells

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We developed revolutionary novel and low cost and non-vacuum chemical molecular beam deposition method for fabrication of thin film II–VI solar cells in the atmospheric pressure gas (He, Ar and H₂) flow. High quality polycrystalline CdTe films with different compositions (stoichiometric and Cd/Te ≤ 1.0 and Cd/Te ≥ 1.0) and thickness of 2–3 μm were fabricated on ceramic (SiO₂–Al₂O₃) substrates at a temperature of 600°C. Separate sources of Cd and Te with respective purities of 99.999% were used as precursors. The growth rate was varied in the range of 9–30 \AA s^{-1} . The effects of the composition and CdCl₂ treatment on the structure, intrinsic point defects and electrical properties of CdTe films were investigated by X-ray diffraction, atomic force microscopy, Raman spectra, photoluminescence and Hall methods.

Keywords: CdTe, Morphology, Photoluminescence, Resistivity, Chemical molecular beam deposition, Thin film

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Introduction

CdTe is the second material after Si in the world photovoltaic market.¹ The two key properties of this material are its near ideal band gap for photovoltaic conversion efficiency of 1.45 eV and its high optical absorption coefficient of 10^4 – 10^5 cm^{-1} . A thin film of CdTe with thickness of $\sim 2 \mu\text{m}$ will absorb $\sim 100\%$ of the incident solar radiation. II–VI thin film solar cells based on CdTe have been the subject of intense research and development in the past few years. Substantial progress has been made thus far in the area of materials research, device fabrication and technology development, and numerous applications based on CdTe have been deployed worldwide. Thin film CdS–CdTe solar cells with efficiency of 15.8–17.3% have been obtained.^{2–5}

Another advantage of the CdTe technology is the flexibility with regards to the method of manufacturing.^{6,7} Many methods (close spaced sublimation,^{2–5} electrodeposition,⁸ sputtering,⁹ close spaced vapour transport,¹⁰ spray pyrolysis,¹¹ metalorganic chemical vapour deposition,^{12,13} etc.) were used for fabrication of CdTe layer. Successful results of around 16–17% efficiency were obtained on

CdTe films deposited by close spaced sublimation.^{2–5} Thin film CdS–CdTe solar cells have a potential to increase the efficiency to 20–25%. Before this happens, it is necessary to develop a better understanding of the basic properties of the materials and processes involved in fabricating the photovoltaic device structure.

One of the critical stages in thin film solar cell fabrication is the deposition of CdTe layer with controllable composition and stoichiometry. It has been pointed out that highly efficient solar cells possess Te rich CdTe surfaces with smooth interfaces of p-CdTe/n-CdS.¹³ Another issue is the fabrication of low resistivity p-CdTe film. This can be solved by intrinsic or extrinsic point defects.^{1,14} Intrinsic point defects strongly depend on the composition of CdTe films. Therefore, fabrication of CdTe films with controllable composition in growth process is very important.

One of the key steps in fabrication of thin film CdS–CdTe solar cells is CdCl₂ treatment.¹⁵ The efficiency of thin film CdS–CdTe heterostructure increases drastically after CdCl₂ treatment. This process introduces several changes to the thin film CdS–CdTe solar cells. It modifies the structure¹⁶ and increases the grain size of CdTe films noticeably.¹⁷ The grain growth and recrystallisation reduce recombination losses and improve charge transport.¹⁸ The promotion of interdiffusion between CdS and CdTe¹⁹ helps reduce the lattice mismatch, which leads to reduction in microstress in CdTe.²⁰ Moreover, this treatment makes the CdTe film highly p type with significant improvement to the minority carrier lifetime.²¹

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Earlier, we had reported about revolutionary novel and low cost chemical molecular beam deposition (CMBD) method under atmospheric pressure gas flow for fabrication of binary, ternary and multinary II–VI films from separate metallic and chalcogenic precursors.²² Some characteristics of CdTe films with different compositions fabricated by this method were discussed in literature.^{23–26} The correlation between fabrication conditions in CMBD and morphology, photoluminescence (PL) and electrical properties of CdTe films will be presented in this paper. We have used atomic force microscopy (AFM), Raman, PL and Hall methods for this purpose.

Experimental

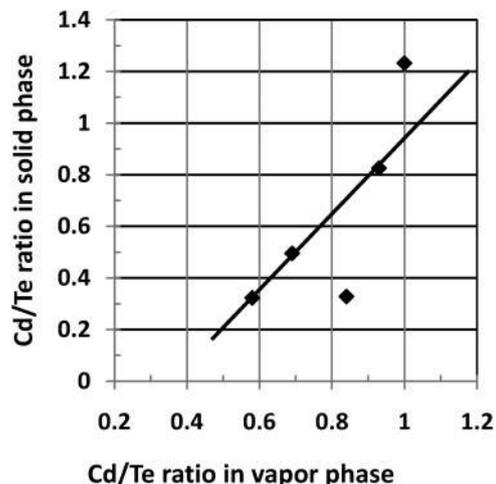
CdTe films with different compositions were fabricated by a novel and low cost CMBD method under atmospheric pressure hydrogen flow.²² Cd and Te granules of 99.999% purity were used as precursors. At the evaporation temperature, the metallic Cd and Te change into the vapour phase



Cd and Te atoms reach the surface of the substrate, and as a result of their interaction, the CdTe film is formed



The composition of CdTe films was controlled by changing the molecular beam intensity (MBI) ratio of Cd and Te (evaporated amount). Three samples fabricated at different MBIs (vapour phase mixture) had Cd/Te ratios of 0.86, 1.0 and 1.1. Samples were deposited at a substrate temperature of 600°C. All of the films were deposited on ceramic ($\text{SiO}_2\text{--Al}_2\text{O}_3$) substrates. Atomic force microscopy measurements were performed with a Digital Instrument, Dimension 3000 microscope. Hall measurements were carried out by four-probe method. Photoluminescence experiments were carried out with an Ar laser operating at $k=514$ nm with an output power of 0.5 W. The spectra were dispersed with a SPEX 500 spectrophotometer equipped with a Ge detector and InGaAs diode. The system settings were as follows: 1 mm slit, $T=20$ K, 655 nm interference filter in the range of 655–1300 nm (PL1) and 1050 nm filter in the range of 1300–1700 nm (PL2). Measurements were carried out at room temperature. The inelastic Raman scattering experiments were carried out at room temperature in a Labram Dilor–Horiba micro-Raman spectrometer using an excitation line of 514.5 nm in backscattering configuration. In order to avoid sample modification due to laser heating, appropriate neutral density filters were employed. The Raman system uses a holographic notch filter that produces a sharp cutoff close to 155 cm^{-1} . The signal was analysed with an 1800 g mm^{-1} grating and with the help of a thermoelectrically cooled charge coupled device detector. Photoluminescence experiments were performed at 20 K using 514 nm Ar laser line with 5 W cm^{-2} power density as an excitation source. Photoluminescence spectra were dispersed by a SPEX 500 and recorded with an N_2 cooled Ge detector. CdCl_2 treatment was carried out in atmospheric pressure He/O_2 (80:20%) at 390°C for 25 min.



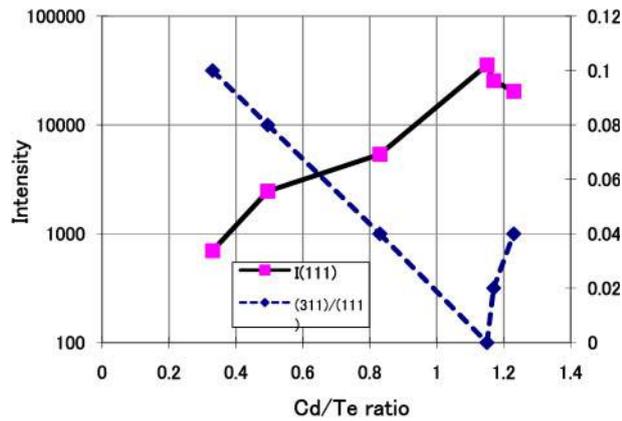
1 Dependence of composition of CdTe films on MBI ratio of Cd and Te

Results and discussion

The kinetics of the growth process of films by CMBD in a gas flow depended strongly on the MBI of the metal and the chalcogen. Therefore, the composition of CdTe films was controlled by changing the MBI ratio of Cd and Te. The growth rate can be varied in the wide range of $10^{-1}\text{--}10^2\text{ \AA s}^{-1}$. Cluster formation of Cd atoms was observed for $\text{Cd/Te} \gg 1$. Three samples fabricated at different MBIs to give Cd/Te of 0.86, 1.0 and 1.1 were studied.

As seen from Fig. 1, there is a good correlation between the solid phase composition Cd/Te and the MBI ratio for $\text{Cd/Te} \leq 1$. This fact allows us to control on molecular level the composition of CdTe films and consequently their physical properties, in particular, the intrinsic point defects. However, there was no good correlation between them for $\text{Cd/Te} \ll 1$ and $\text{Cd/Te} \gg 1$. In the case of $\text{Cd/Te} \gg 1$, it was probably due to the fact that the deposition temperature (600°C) used in our case is more than the evaporation temperature of Cd. Therefore, excess Cd atoms are reevaporated from the substrate. Moreover, change of Cd/Te flux ratio for $\text{Cd/Te} > 1$ does not necessarily change the composition of condensed films but strongly influences the density and the morphology of the samples.

X-ray diffraction (XRD) lines for samples fabricated at different MBI ratios have been characterised by the presence of sharply expressed peak at $2\Theta=23.7^\circ$. It corresponds to (111) crystallographic plane and evidences that all CdTe films were grown predominantly in (111) orientation of the sphalerite structure. We also observed (220), (311) and (400) peaks for CdTe films for different compositions (Table 1). Intensities of these peaks depend on the composition of samples. For nearly stoichiometric composition ($\text{Cd/Te} \approx 1$), the presence of (220), (311), (400) and (111) peaks shows that it has face centred cubic lattice with a coordination number of 12. According to these results, the stoichiometric samples are predominantly single phase CdTe with the lattice constant of $\bar{a}=6.487\text{ \AA}$ (accuracy is 0.093%). For all investigated CdTe films with different compositions, small peak at $2\Theta=21.40^\circ$ has been observed. This peak could not be assigned to pure Cd or Te films, so it can be explained by the polytypism of II–VI compounds.



2 Dependence of XRD intensities on composition of CdTe films

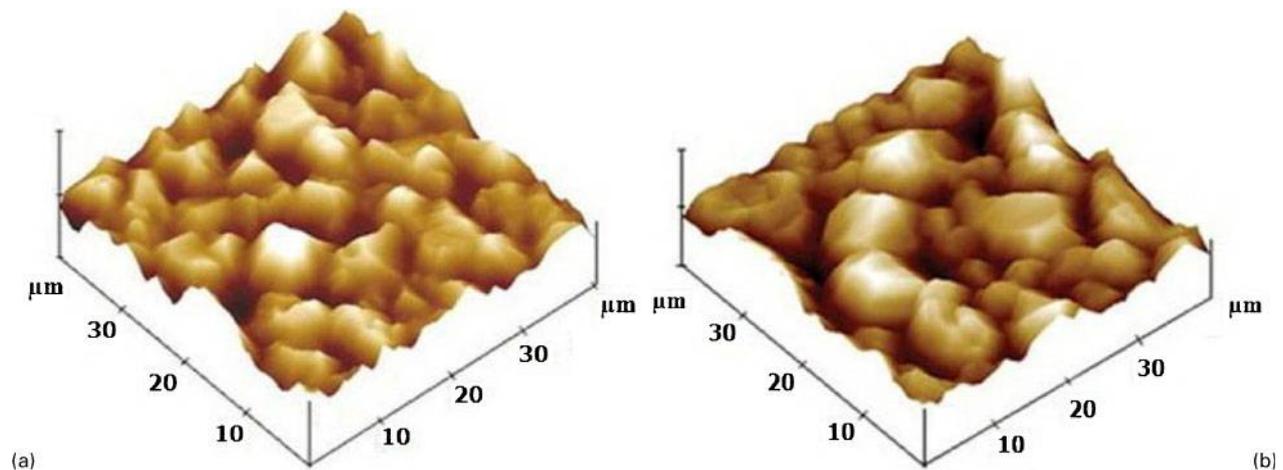
The dependences of the intensity of the (111) peak (solid line) and the intensity ratio of (311)/(111) peaks (dashed line) on Cd/Te ratio in the solid phase are shown in Fig. 2. As seen, the intensity of (111) peak increases with Cd/Te ratio. The reduction in the XRD intensity in non-stoichiometric films may be explained by the poor crystalline properties and appearance of other phases taking into account the morphology of films observed by an optical microscope.

Surface morphology of all three CdTe films was examined by AFM. All three samples had almost the same morphology. It is seen in Fig. 3a that the as deposited film contains high density and very well oriented cubic polycrystalline grains with an average grain size of 8–10 μm. After exposure to the CdCl₂ heat

treatment (Fig. 3b), the film structure remained essentially unchanged. This result indicates low stress in the structure of our samples.

Figure 4a shows the Raman spectrum of an untreated stoichiometric CdTe sample. The broad band (peak 1) has the contributions of the longitudinal optic (LO) mode of CdTe at 165 cm⁻¹ (Ref. 27) and the strong E_{TO}Te mode at 145 cm⁻¹.²⁸ It is known that Raman scattering is a powerful technique to detect Te micro-clusters or monolayers on CdTe²⁹ or on other semiconductors.³⁰ In addition, due to the high Cd vapour pressure, CdTe samples may exhibit Te segregation that is readily detected through Raman spectroscopy experiments.³¹ In the same figure, peak 2 may be associated to a sum mode of the strongest Te modes A₁ (120 cm⁻¹)+E_{TO} (145 cm⁻¹), while peak 3 corresponds to second harmony of the LO phonon. The Raman spectrum of the film treated with CdCl₂ (Fig. 4b) presents some changes. The modes associated with pure Te have disappeared, and a new mode (peak 4) has arisen. These modifications in the spectrum indicate that the treatment has favoured the redistribution of the formerly segregated Te atoms and that the overall crystalline quality has improved since the spectrum now extends up to the third LO overtone (peak 4). The appearance of phonon overtones is a measure of crystalline quality.

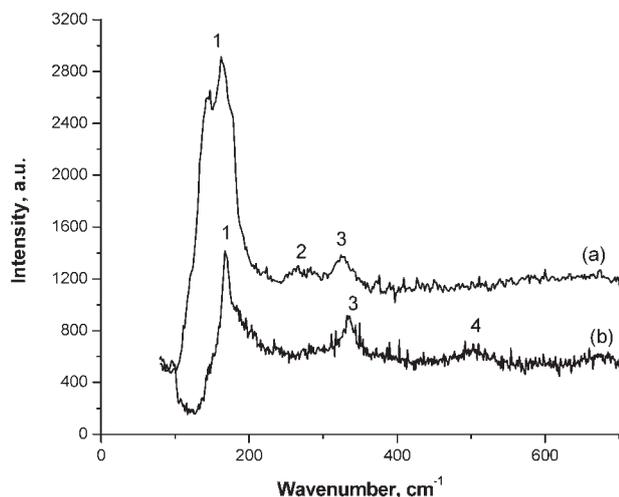
Photoluminescence spectra at 20 K of all samples exhibited a near band edge peak at 1.41 eV and a deep level related emission band with a maximum at 1.12 eV. A typical low temperature PL spectrum is shown in Fig. 5. Photoluminescence peak at 1.41 eV was also observed in literature.^{32,33} This peak is due to the



3 Images (AFM) of as a deposited and b CdCl₂ treated CdTe films fabricated at MBI ratios of Cd/Te=1.0

Table 1 Dependence of XRD parameters on composition of CdTe films

Sample	Cd/Te (analysis)	Relative intensity of XRD lines at 2 theta (hkl)					Unidentified line	XRD peak	Counts at peak [FWHM/°]	Thickness/μm
		23-72 (111)	39-24 (220)	46-36 (311)	56-70 (400)					
740	1.23	100	6	4	4	No	20380 [0.118]	3		
744	1.17	100	0	2	2	27.4 (2)	25563 [0.118]	1		
5-8	1.15	100	2	0	0	27.4 (2)	35599 [0.118]	1.3		
118	0.83	100	4	4	2	54.2 (2)	5371 [0.094]	0.8		
2	0.495	100	18	8	2	no	2463 [0.094]	1		
138	0.33	100	18	10	0	27.4 (10), 36.0 (8), 54.2 (6)	699 [0.118]	0.82		

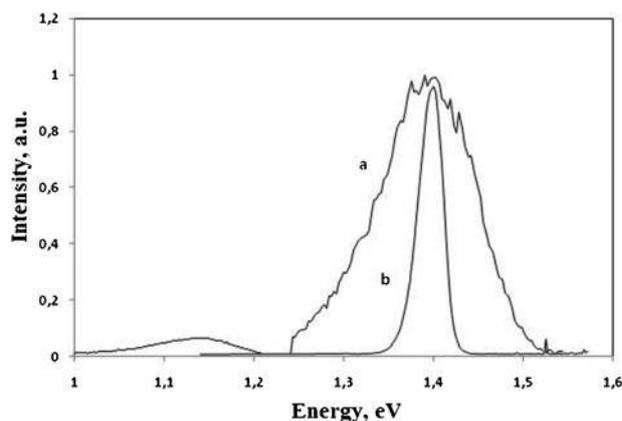


4 Raman spectra of *a* as deposited and *b* CdCl₂ treated CdTe films fabricated at MBI ratio of Cd/Te=1.0

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. Peak at 1.12 eV is possibly caused by the transition of electrons from the donor level $E_C-0.48$ eV to the valence band. This donor level may be attributed to the vacancy of tellurium V_{Te}^{++} related defect or the interstitial atoms of cadmium Cd_i^{++} .

After CdCl₂ treatment, the PL band located at 1.41 eV was still present; however, the 1.12 eV band was annealed out. A tentative conclusion is that the 1.12 eV peak was associated with a tellurium vacancy V_{Te}^{++} that was possibly substituted by oxygen following the CdCl₂ heat treatment. Oxygen is present during the heat treatment and most likely occupies the tellurium vacancy (as compared to chlorine), since oxygen and tellurium are the same group elements.

The values of the resistivity, carrier concentration, mobility and conductivity type before and after CdCl₂ treatment are presented in Table 2. The resistivity of the films was found to be in the range of 10^3-10^9 Ω cm, the carrier concentration is in the range of 10^7-10^{13} cm⁻³ and the mobility is in the range of $1.8-4.5 \times 10^2$ cm² V⁻¹ s⁻¹ depending on the composition. The CdCl₂ heat treatment did not lead to grain enhancement, in particular of large grain (8–10 μm) films, but it does have an effect on the point defects of CdTe films. The CdCl₂ heat treatment significantly decreased the resistivity of stoichiometric CdTe film from almost 10^9 to 10^3 Ω cm owing to the increase in carrier concentration from 10^7 to 10^{12} cm⁻³. This can be understood from the fact that oxygen occupies the tellurium vacancy V_{Te}^{++} . As a result, the donor level $E_C-0.48$ eV passivates and the acceptor level $E_V+0.15$ eV activates. The resistivity of Te rich p-CdTe films was not



5 Photoluminescence spectra at 20 K of *a* as deposited and *b* CdCl₂ treated CdTe films fabricated at MBI ratio of Cd/Te=1.0

essentially changed and remained at about 10^3-10^4 Ω cm as with before treatment. Therefore, low resistivity p-CdTe films can be fabricated by deviating the composition of samples from stoichiometry to Te rich.

CdTe films can be considered as compensated material containing the acceptor level $E_V+0.15$ eV and the donor level $E_C-0.48$ eV. Physical properties, in particular, the resistivity of CdTe films, are varied depending on the activity of these levels. The level $E_C-0.48$ eV predominates Cd rich films and samples had n type of conductivity. The level $E_V+0.15$ eV predominates Te rich films and samples had p type conductivity. We observed significant decrease in the resistivity of CdTe films under the CdCl₂ treatment. It is due to the priority of the level $E_V+0.15$ eV caused by oxygen occupying the tellurium vacancy V_{Te}^{++} . However, the decrease in the resistivity of CdTe films under CdCl₂ treatment is limited to a value of 10^3 Ω cm. The resistivity of CdTe films up to 10^3-10^4 Ω 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.

Thus, all the above mentioned one to three improvements and etching of CdTe in bromo methanol to form the Te rich layer at the rear surface can be done during the growth process of the CMBD method. Therefore, CdCl₂ heat treatment and the CdTe precursor are possibly not necessary for CdTe films fabricated by the CMBD method. This novel and low cost method can successfully be used for fabrication of high efficiency thin film CdS–CdTe solar cells.

Conclusions

By the precise control of the MBI, we were able to regulate the Cd/Te ratio in a revolutionary novel and

Table 2 Electrical parameters of CdTe films with different compositions before and after CdCl₂ treatment

Cd/Te	Resistivity/Ω cm		Mobility/cm ² V ⁻¹ s ⁻¹		Carrier concentration/cm ⁻³		Type of conductivity	
	Before	After	Before	After	Before	After	Before	After
0.86	1.4×10^3	1.1×10^3	185	210	2.4×10^{13}	2.7×10^{13}	p	p
1.0	7.1×10^8	3.2×10^3	390	410	2.3×10^7	4.7×10^{12}	p	p
1.1	2.2×10^4	4.3×10^4	450	520	6.3×10^{11}	2.8×10^{11}	n	n

low cost CMBD technique. Under atmospheric pressure hydrogen flow, we succeeded in obtaining stoichiometric (Cd/Te=1.0) CdTe films. It is also found that we can control the intrinsic point defects by deliberately changing the stoichiometry (Cd/Te=0.86 and 1.1). Atomic force microscopy studies have shown that CdTe films have high quality and the average grains size is 8–10 μm for films prepared at 600°C. CdTe films have been considered as compensated semiconductor containing acceptor level $E_V+0.15\text{ eV}$ (V_{Cd}^-) and donor level $E_C-0.48\text{ eV}$ (V_{Te}^{+}). $E_V+0.15\text{ eV}$ level dominates in the Te rich samples, and $E_C-0.48\text{ eV}$ level dominates in the Cd rich films. We have fabricated p- and n-CdTe films by the deviation of the composition from the stoichiometry in the growth process. The resistivity was varied between 10^3 and $10^9\ \Omega\text{ cm}$ depending on the composition of the samples. The CdCl_2 heat treatment significantly decreased the resistivity of stoichiometric CdTe film from almost 10^9 to $10^3\ \Omega\text{ cm}$. On the other hand, the resistivity of Cd and Te rich samples did not change much. Thus, we can control the resistivity by the deviation of the composition of CdTe films from stoichiometry. These results suggest that the CdCl_2 heat treatment does not always lead to grain enhancement, in particular, for large grain (8–10 μm) films, but it does have an effect on the point defects in CdTe films. These are explained by the fact that oxygen is present during the heat treatment and will most likely occupy the tellurium vacancy. Thus, all the above mentioned improvements in CdCl_2 treatment can also be carried out during CMBD growth. Therefore, CdCl_2 treatment is not necessary for CMBD method. This novel and low cost CMBD method can successfully be used for fabrication of high efficiency thin film CdS–CdTe solar cells.

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