Effect of CdCl₂ heat treatment on ZnTe back electron reflector layer in thin film CdTe solar cells

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Abstract

The effect of CdCl₂ heat treatment on the ZnTe layer used as the back electron reflector layer in polycrystalline CdTe solar cells was investigated. A thin film of ZnTe was grown on top of polycrystalline CdTe layer using metalorganic vapor phase epitaxy, which was then subjected to CdCl₂ heat treatment. The effect was examined using various characterization techniques such as x-ray diffraction, energy dispersive x-ray spectroscopy and scanning electron microscope images and it was found that the ZnTe layer transformed into CdTe and ZnO due to the treatment. This was verified by performing CdCl₂ heat treatment on pure ZnTe thin layer directly grown on glass which also showed the conversion of ZnTe to CdTe and ZnO. To study the effect of this transformation on final device performance, thin film CdTe solar cell structures were fabricated with ZnTe electron reflector layer with and without the CdCl₂ heat treatment. The device without the ZnTe layer seemed to perform better than the devices with ZnTe layer and the CdCl₂ heat treatment.

Key words: CdTe solar cells, electron reflector layer, CdCl₂ heat treatment

1. Introduction

Near optimal bandgap (~ 1.5 eV), very high absorption coefficient and low cost manufacturing make CdTe highly suitable for solar cell applications. During the last few years, polycrystalline CdTe solar cell technology has witnessed a renewed interest with the highest efficiency reaching 22.1% (First Solar Press Release, 2016). But still the efficiency is well below the detailed balance limit of 29% and one of the major factors limiting the efficiency is the low open circuit voltage. There are two possible approaches to improve it, either by increasing carrier density and absorber lifetime or by reducing the back surface recombination.

Various strategies can be implemented to reduce the back surface recombination. One of the approaches is to create a conduction band offset that prevents electrons recombining with holes at the back contact. It can be achieved by using a high work function metal contact with the semiconductor and thereby creating a conduction band barrier for the flow of electrons. Alternatively, the back surface of the semiconductor can be heavily doped to create a back surface field (BSF) layer, which increases the charge collection efficiency by preventing the recombination. The third approach is by using an expanded band gap layer created either by introducing a layer of material with higher bandgap than the bulk or by reducing the bulk bandgap to create a conduction band offset for electrons. Simulation results suggest that the expanded bandgap layer strategy is more efficient than the other two (Hsiao, 2014). But electron reflector layer creates an extra interface, which may cause interfacial recombination. So the contamination to the back surface layer must be avoided while growing the electron reflector layer and the lattice mismatch must be kept to a minimum. ZnTe as electron reflector layer creates a conduction band barrier of 0.8 eV and a small valence band offset of 0.1 eV (Späth et al., 2005). Simulation studies on CdTe solar cells with ZnTe back electron reflector layer have

shown an enhancement in the open circuit voltage as well as the efficiency (Khosroabadi and Keshmiri, 2014). Our capability to grow ZnTe layer continuously after CdTe in the same chamber helps to avoid contamination of the CdTe: ZnTe interface and hence allows us to use it as an electron reflector layer.

CdCl₂ heat treatment plays a very important role in improving the efficiency of CdTe solar cells (Dharmadasa, 2014). Solar cells with efficiencies below 1% have been shown to improve their efficiencies beyond 10% after the treatment (Abbas et al., 2013). CdCl₂ heat treatment has been found to reduce stacking faults (Abbas et al., 2014), helps in grain growth and recrystallization (Kim et al., 2013; McCandless et al., 1997; Moutinho, 1998), passivates the grain boundaries and deep level defects (Komin et al., 2003; Li et al., 2014; Zhang et al., 2008), and also increases the carrier lifetime (Metzger et al., 2006; Ringel et al., 1991). CdCl₂ heat treatment usually involves treating CdTe with a layer of supersaturated solution of CdCl₂ in methanol or water and then evaporating the solvent by annealing the sample at 350-450 °C in air. Some studies also used evaporation, closed space sublimation, chemical vapor deposition, laser ablation, and electrodeposition (Cunningham et al., 2002; Mazzamuto et al., 2008; Romeo et al., 1999). It has been observed that the presence of oxygen during CdCl₂ heat treatment is very important to obtain higher efficiency (Duenow et al., 2012; Regalado-Pérez et al., 2015). This is done by annealing the sample in air, in oxygen ambient or in a mixture of halogen and oxygen ambient. Irrespective of the treatment method, all the CdCl₂ heat treatments result in improved device performance, making them an integral part of polycrystalline CdTe solar cell fabrication process.

In this work, we have used ZnTe electron reflector layer in CdTe solar cells grown continuously in metalorganic chemical vapor deposition chamber to improve the charge collection. Due to the significance of CdCl₂ heat treatment in improving the device performance, we have tried to incorporate it in the device structure and analyzed its effect on the device.

2. Experimental Details

The CdTe and ZnTe thin films were grown in a vertical cold wall metal-organic chemical vapor deposition (MOCVD) chamber with a 3-inch quartz substrate holder/heater. Dimethylcadmium (DMCd), diisopropyltelluride (DIPTe), diethylzinc (DEZn) were used as the metalorganic precursors of Cd, Te and Zn, respectively. AsH₃ was used as the precursor for *p*-type doping of both CdTe and ZnTe. The substrates used for the devices were *n*-CdS/FTO/glass obtained from First Solar. The *p*-type CdTe was grown at 400 °C and 100 Torr chamber pressure for 2 hours and the thickness obtained was 1.2 μm. The mole fraction of DMCd and DIPTe were 1.1×10^{-4} and 9.7×10^{-5} , respectively. AsH₃ flow was maintained at 2 standard cubic centimeters per minute (sccm) or a corresponding mole fraction of 4×10^{-7} . The hydrogen flowrate was 2200 sccm. Similarly, for the ZnTe electron reflector layer, the growth temperature was 400 °C and chamber pressure was 100 Torr. The growth time was 12 minutes and thickness obtained was 140 nm. The mole fraction of DEZn and DIPTe were 6.1×10^{-5} and 1.3×10^{-4} , respectively. AsH₃ flow rate was 10 sccm corresponding to a mole fraction of 2×10^{-6} . The H₂ flow rate was 2200 sccm.

For CdCl₂ heat treatment, CdCl₂ powder was dissolved in methanol to make a super saturated solution and a layer of it was spin coated on top of ZnTe layer. The substrates were

then annealed at 370 °C for 90 minutes in air. The excess CdCl₂ was then removed from the surface by rinsing with methanol.

To study the effect of ZnTe electron reflector layer and its heat treatment on solar cell performance, three different devices (D1, D2, D3) were fabricated on *n*-CdS (75 nm)/FTO/glass substrates provided by First solar. 1.2 µm thick As doped *p*-type CdTe layer was grown on top of these substrates by the MOCVD process. On samples D2 and D3, 140 nm *p*-ZnTe electron reflector layer was grown, after and before CdCl₂ heat treatment respectively. Then gold contacts were deposited by e-beam evaporation on all the devices with contact areas of 0.06 and 0.25 cm².

The X-ray diffraction (XRD) studies were done using a Bruker D8 discover powder diffractometer with 1.54 Å wavelength from a Cu target. The surface morphologies were studied using images from a Carl Zeiss Supra SEM 1550 scanning electron microscope (SEM). The energy dispersive x-ray spectroscopy (EDX) analysis of the sample was also done using the same instrument. The cross-section images of the samples were obtained from Carl Zeiss Ultra 1540 dual beam focused ion beam (FIB)/SEM system. The current density vs. voltage (*J*~*V*) characteristics of the devices were measured using a Keithley 2400 source meter. The light source used was a tungsten halogen lamp with adjustable source power along with a Newport solar simulator to provide AM 1.5 equivalent illumination.

3. Results and Discussion

To study the effect of CdCl_2 heat treatment on polycrystalline CdTe solar cell, two sets of samples were prepared. Both the samples had p -CdTe (1.2 μm) and p -ZnTe (140 nm) layers grown on top of n -CdS/FTO/glass substrates as shown in Fig. 1. ZnTe layer of sample B was treated with CdCl_2 heat treatment whereas sample A was not treated. The top surface of both samples were analysed with SEM to observe the effect of the treatment. As shown in Fig. 2, the surfaces of samples look different from each other. The surface morphology of sample B seems to have undergone a change after the CdCl_2 heat treatment, making it more uneven and rough.

The crystalline orientations of the samples were then studied through XRD analysis. Fig. 3 shows the XRD pattern of the samples with and without CdCl_2 heat treatment. The major CdTe and ZnTe peaks are indicated in the figure. As observed from Fig. 3(a), XRD spectrum of Sample A shows a (111) orientation preferred polycrystalline CdTe with the (111) ZnTe peak, indicating the top ZnTe layer. However, in XRD data of sample B as shown in Fig. 3(b), the ZnTe peak completely disappears while all the CdTe peaks remain unaltered. The elemental compositions of samples were studied by EDX analysis. Table 1 shows the results of the EDX analysis on the surface of both the samples with and without the treatment. Surface of sample A showed Zn and Cd weight percentages to be 6.2% and 40.1%. On the other hand, in sample B after the treatment, the Zn percentage was found to have decreased to 0.8% and Cd percentage has increased to 46.7%. EDX results suggest that due to the treatment, the surface Zn percentage decreases and Cd percentage increase.

The cross-sections of the samples were analyzed to verify the results obtained from EDX and XRD. The cross-section SEM images are shown in Figs. 4(a) and (b) for sample A and B,

respectively. A thick layer of Pt was deposited on top of the samples by FIB/SEM dual beam system to protect the top layer. The SEM image for sample A shows a thin ZnTe layer on top of CdTe layer with average thickness of ~140 nm. The cross-section of sample B was found to have CdTe and Pt layers present as they were in sample A, but the ZnTe layer was found to have disappeared from the interface. Hence, the CdCl₂ heat treatment to the sample B causes the disappearance of ZnTe from the CdTe surface, which increased the Cd percentage and decreased the Zn percentage.

3.1. CdCl₂ heat treatment on ZnTe on glass substrate

To study further the effect of CdCl₂ heat treatment on ZnTe electron reflector layer, 140 nm thick ZnTe layer was grown on glass substrate using the same recipe described earlier. The sample was analyzed before and after CdCl₂ heat treatment. First the surface morphology was studied using SEM images, as shown in Figs. 5(a) and (b). The surface before the CdCl₂ heat treatment appears to be smooth and uniform. However, after the treatment, the surface seems to have very different features with surface becoming more uneven. It looks similar to the surface of ZnTe layer deposited on CdTe after the CdCl₂ heat treatment.

To study the effect of CdCl₂ on ZnTe crystal quality, the sample was analysed with XRD. The XRD data in Figs. 6(a) and (b), show the XRD patterns obtained from the ZnTe films before and after the treatment. Before the treatment, the XRD pattern indicated the presence of polycrystalline ZnTe with a preferred orientation along the (111) and comparatively much weaker (311) and (222) oriented peaks. However, after treatment, there was significant change in the XRD pattern. The post-treatment XRD peaks indicated that the sample was polycrystalline

CdTe with (111) as preferred orientation. ZnTe peaks were completely absent from the XRD pattern, instead ZnO (002) and (101) peaks were present.

EDX analysis of the samples, shown in Table 2, suggest that the overall Zn wt. percentage has decreased significantly from ~33% to 4%, whereas Cd percentage increased from 0 to 44.9% in the sample. Also the data indicate the presence of oxygen (~1%) in the sample after treatment. It supports the XRD data, which indicated the presence of CdTe instead of ZnTe after the treatment and presence of Zn as ZnO in the sample. Hence, this experiment definitively shows the conversion of ZnTe to CdTe and ZnO after the CdCl₂ heat treatment.

The chemical reaction of ZnTe with CdCl₂ at high temperature and its conversion to CdTe can be explained by the electrochemical reactivity series. Zn has a higher electrode potential than Cd, making it easier to oxidize and more reactive. Hence, Zn easily reacts with CdCl₂ during the heat treatment and the resulting chemical reaction is given by:

The standard values of enthalpy of formation (H) and entropy (S) of ZnTe (-119.49 kJ/mol, 78.23 J/mol.K), CdCl₂ (-391.6 kJ/mol, 115.3 J/mol.K), CdTe (-92.5 kJ/mol, 100 J/mol.K) and ZnCl₂ (-415.05 kJ/mol, 111.46 J/mol.K) at STP were used to calculate the change in Gibbs free energy for the system by using the formula $\Delta G = \Delta H - T\Delta S$, at the temperature (T) of 370⁰ C (Brebrick, 2011; Lange, 1999). The ΔG was calculated to be -8 kJ/mol, suggesting a thermodynamically favourable reaction. Some of the ZnCl₂ formed during the reaction, were dissolved in methanol and were removed while removing the excess CdCl₂ from the surface. This possibly causes a loss of Zn at the back interface. Additionally, Zn has a high affinity

towards oxygen and hence gets readily oxidized during the annealing in oxygen atmosphere. This explains the ZnO peaks observed in XRD scans of post-CdCl₂ annealing of ZnTe layer. Zn possibly diffused towards CdTe:CdS interface during annealing, causing a further loss of Zn from the back contact as supported by the EDX results. Similar Zn diffusions to the front surface has been reported by Dhere *et. al.* during CdCl₂ heat treatment on CdZnTe and Su *et. al.* during annealing of homo-junction CdTe solar cell with ZnTe buffer layer (Dhere et al., 2003; Su et al., 2014).

3.2. Effect on solar cell performance

To test solar cell performance, three devices D1, D2 and D3 were fabricated as described earlier in the experimental details section. The current density-voltage (J-V) characteristics of these devices were measured with solar simulator at AM 1.5 illumination as shown in Fig. 7(b). The measured device parameters for device areas of 0.06 cm² are given in Table 3 and the results are discussed below.

The device D1 with CdCl₂ heat treatment on *p*-CdTe layer and without any ZnTe electron reflector layer, produced an open circuit voltage (V_{OC}) of 0.64 V and a short circuit current (J_{SC}) of 22 mA/cm². The *p*-CdTe layer makes an ohmic contact with Au and gives rise to a reasonable efficiency of ~7%. The difficulty to get a high *p-type* doping in CdTe is one of the main reasons for comparatively low efficiency. The standard practice of using ZnTe:Cu contact was not followed, since the goal was to use As as dopant. In case of device D2, all device parameters - V_{OC} , J_{SC} and fill factor are lower than that of device D1, e.g., lowering the efficiency to 3.4%. The device was CdCl₂ treated by taking the device out of the chamber after CdTe growth and

then it was returned to the chamber for ZnTe growth. The interface contamination during this process may create additional recombination centers causing a reduction in all device parameters. The additional *p*-ZnTe layer adds a small series resistance to the device, further lowering the device performance. Device D3, with CdCl₂ treated ZnTe layer on top of CdTe layer, shows an improvement in V_{OC} over D2 as ZnTe gets converted into CdTe. But the fill factor is lowered further. Decrease in the fill factor is possibly contributed by the band offset produced by ZnO (bandgap ~ 3.3 eV) formed due to the reaction between ZnTe and CdCl₂. Also, the ZnO formed is inherently *n*-type and creates a *p-n* junction with *p*-CdTe, which is in opposition with the *p*-CdTe and *n*-CdS junction (Dhere et al., 2003), further degrading the performance of the device.

4. Conclusion

In summary, we studied the effect of CdCl₂ heat treatment on the ZnTe electron reflector layer used in the MOCVD grown polycrystalline CdTe solar cells. It was found that ZnTe gets converted into CdTe and ZnO with subsequent loss of back surface electron reflection. Conversion of ZnTe to CdTe and ZnO was verified by CdCl₂ treating of a thin film of ZnTe directly grown on glass. Thin film polycrystalline CdTe solar cells were fabricated on CdS/FTO/glass substrates by growing As doped CdTe followed by ZnTe layer with and without CdCl₂ heat treatment. The open circuit voltage of the device without any ZnTe layer was the highest with 0.64 V as compared to the device with ZnTe layer followed by the CdCl₂ heat treatment. The lower V_{OC} can be attributed to the conversion of ZnTe into CdTe and ZnO. Alternatively, CdZnTe and CdMgTe can be studied as electron reflector layers owing to their small lattice mismatch with CdTe. But Zn and Mg in CdZnTe and CdMgTe have also been

found to react with CdCl_2 during heat treatment, causing problems similar to that of ZnTe (Dhere et al., 2003; Swanson et al., 2015). However, ZnCl_2 and MgCl_2 can be used as good alternatives to CdCl_2 heat treatment in devices to avoid any reaction with the electron reflector layers.

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Figure Captions

Fig. 1. Schematic view of device structures of (a) sample A (without any CdCl_2 heat treatment) and (b) sample B (with CdCl_2 heat treatment on ZnTe layer).

Fig. 2. SEM top view images showing surfaces of (a) sample A and (b) sample B with insets showing magnified regions. Changes in surface morphology occur due to the CdCl_2 heat treatment.

Fig. 3. XRD spectra of (a) sample A and (b) sample B. (hkl) peaks labeled in black represent CdTe peaks and the peak labeled in blue represents ZnTe peak. The ZnTe peak is missing in XRD spectrum of sample B after CdCl_2 heat treatment.

Fig. 4. SEM images showing cross-sections of (a) sample A and (b) sample B. The ZnTe layer of average thickness ~ 140 nm is clearly visible in sample A, but is missing from the image of sample B (indicated by an arrow).

Element	Wt.%	
	Before treatment	After treatment
C	1.1	0.8
Zn	6.2	0.8
Cd	40.1	46.7
Te	52.6	51.7
Total:	100.00	100.00

Table 1. Comparison of EDX data of samples A and B showing the weight percentage of different elements present in the sample. After a CdCl_2 heat treatment, there were a reduction in Zn percentage and an increase in Cd percentage.

Fig. 5. SEM top view images showing the ZnTe surface (a) before CdCl₂ heat treatment, and (b) after CdCl₂ heat treatment.

Fig. 6. XRD spectra obtained from ZnTe samples (a) before CdCl₂ heat treatment and (b) after CdCl₂ heat treatment. The ZnTe peaks (blue color) completely disappear and ZnO peaks (green color) and CdTe peaks (black color) appear in the data.

Element	Wt.%	
	Before treatment	After treatment
O	-	1.3
C	2.3	-
Si	0.5	0.2
Zn	33.2	4.6
Cd	-	44.9
Te	64.0	49.0
Total:	100.00	100.00

Table 2. Comparison of EDX data between ZnTe film before and after CdCl₂ heat treatment.

After treatment Zn percentage drops and Cd percentage increases significantly.

Fig. 7. (a) Cross-section view of fabricated thin film CdTe solar cells. (b) Current density vs. voltage characteristics for devices D1, D2 and D3.

Devices	Structure	CdCl₂ Treatment	Avg. V_{OC} (V)	J_{SC} (mA/cm²)	Fill Factor (%)	Efficiency (%)
D1	Au/p-CdTe/n-CdS/FTO/glass	Treatment on CdTe	0.64	22	50	7
D2	Au/p-ZnTe/p-CdTe/n-CdS/FTO/glass	Treatment on CdTe, then ZnTe growth	0.53	16	40	3.4
D3	Au/p-ZnTe/p-CdTe/n-CdS/FTO/glass	Treatment on ZnTe	0.55	16	30	2.6

Table 3. Comparison of extracted parameters from thin film CdTe solar cells D1, D2 and D3.