



Effect of annealing temperature and CdCl₂ treatment on the photo-conversion efficiency of CdTe/Zn_{0.1}Cd_{0.9}S thin film solar cells

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Abstract. We report the effects of annealing in conjunction with CdCl₂ treatment on the photovoltaic properties of CdTe/Zn_{0.1}Cd_{0.9}S thin film solar cells. CdTe layer is subjected to dry CdCl₂ treatment by thermal evaporation method and subsequently, heat treated in air using a tube furnace from 400 to 500°C. AFM and XRD results show improved grain size and crystallographic properties of the CdTe film with dry CdCl₂ treatment. This recrystallization and grain growth of the CdTe layer upon CdCl₂ treatment translates into improved photo-conversion efficiencies of CdTe/Zn_{0.1}Cd_{0.9}S cell. The results of dry CdCl₂ treatment were compared with conventional wet CdCl₂ treatment. Photo-conversion efficiency of 5.2% is achieved for dry CdCl₂-treated cells in comparison with 2.4% of wet-treated cell at heat treatment temperature of 425°C.

Keywords. CdCl₂ treatment; thin film solar cells; photo-conversion efficiency.

1. Introduction

CdS/CdTe photovoltaic (PV) cell is the potential candidate for extensive commercial success in solar energy conversion. CdTe has the advantage of a nearly ideal band gap for solar terrestrial photo conversion ($E_g = 1.45$ eV) [1]. Cadmium sulphide (CdS) is popularly used as a window material as an *n*-type heterojunction partner to CdTe. CdS has the energy band gap of 2.4 eV [2]. Thus, photons with energies within this range can reach CdTe layer, where they contribute to the cell's photocurrent. If CdS is replaced by a higher energy band gap material, it will allow more photons to pass, and result in higher efficiencies [3–6]. In this work, CdS is therefore, replaced by Zn_{0.1}Cd_{0.9}S thin film.

Fabrication of high efficiency CdTe-based PV cell requires the CdTe grain sizes $>1\ \mu\text{m}$ [7]. The higher grain size avoids significant bulk recombination, larger interface state density and high resistance. However, CdTe films have typically small grains, and short minority carrier lifetime and high resistivity due to many grain boundaries [8]. Kampmann and Lincot [9] suggested a method known as CdCl₂ treatment (also referred as activation step), which they utilized to enhance the grain size and density of the CdTe film. Although the exact role of this treatment is not clear, various research groups have suggested that it affects the cell in several ways: Alamri [10] suggested that the conversion type, such as *p*-doping in the material takes place (from intrinsic to *p*-type). It is also suggested that the CdCl₂ treatment induces the Cl⁻ related acceptor

levels in CdTe, which leads to the *p*-type behaviour in CdTe [11]. Romeo *et al* [12] reported that CdCl₂ treatment enhances the CdTe recrystallization, leading to better efficiencies. Compann *et al* [13] and McCandless *et al* [14] showed that CdCl₂ treatment not only helps in recrystallization, it also leads to the CdTe/CdS intermixing (interdiffusion). Paulson and Dutta [15] suggested that interdiffusion reduces interface state density at CdTe/CdS interface. Marquez *et al* [16] have applied CdCl₂ along with HCl treatment to improve the grain size of CdS.

This treatment is undertaken by applying CdCl₂ on the back surface of the CdTe layer. The application of CdCl₂ is done mostly via covering the CdTe sample with a saturated CdCl₂-methanol solution [15,17]. The aim of CdCl₂ treatment is to diffuse chlorine into the CdTe and its effectiveness depends on the annealing conditions (e.g., time, temperature and ambient) [18]. Moreover, these conditions must be optimized for CdTe deposited by different methods, e.g., different grain size can be obtained via different deposition techniques. Gupta *et al* [19] optimized the CdCl₂ treatment for sputtered CdTe/CdS cells with various CdTe thicknesses. Major *et al* [20] have shown that insufficient treatment of CdCl₂ creates a buried junction, whereas excess treatment may delaminate the films from the substrate. Hence, optimizing the CdCl₂ treatment conditions for CdTe cells is necessary to achieve good performance.

Purpose of this work is to present the application, role and optimization of CdCl₂ treatment on CdTe layer. In this work, a novel 'dry CdCl₂ treatment' is carried out,

wherein CdCl_2 is deposited by the evaporation and subsequent annealing is carried out in air using a tube furnace. Effect of dry CdCl_2 treatment on the performance of PV cell and $\text{CdTe}/\text{Zn}_{0.1}\text{Cd}_{0.9}\text{S}/\text{ITO}$ cells is investigated. Results were also compared with wet CdCl_2 treatment.

2. Experimental

In the present work, $\text{Zn}_{0.1}\text{Cd}_{0.9}\text{S}$ films were deposited on ITO-coated glass substrates by ablating stoichiometric $\text{Zn}_{0.1}\text{Cd}_{0.9}\text{S}$ targets [21] using pulse laser deposition technique. The detailed film deposition parameters are reported elsewhere [22]. The CdTe layer of thickness $\sim 4\ \mu\text{m}$ is deposited by vacuum evaporator (HINDHIVAC 12 Vacuum Coating Unit, Model: 12A4DM). $\text{Zn}_{0.1}\text{Cd}_{0.9}\text{S}$ -coated ITO glass was used as the substrate and was held at 300°C . Evaporation was carried out by commercially procured CdTe powder (99.99% purity, Sigma Aldrich, UK) in a vacuum of 2×10^{-5} torr.

The stack is further subjected to two different types of CdCl_2 treatment, wet and dry. In wet treatment, the $\text{CdTe}/\text{Zn}_{0.1}\text{Cd}_{0.9}\text{S}/\text{ITO}/\text{glass}$ stack were immersed in the solution (0.1 mol% CdCl_2 in methanol) for 5 min and then dried in flowing nitrogen. In dry treatment, 100 nm thick CdCl_2 layer is deposited by the evaporation of CdCl_2 powder (Sigma Aldrich, 99.9%). After CdCl_2 treatment, both wet and dry samples were heat-treated (400 to 500°C in steps of 25°C) in tube furnace. Samples were then washed by hot de-ionized (DI) water to remove any excess amount of CdCl_2 . Heat-treated wet CdCl_2 samples were named as Cell#1, Cell#2, Cell#3 and Cell#4, while dry CdCl_2 heat-treated samples were Cell#5, Cell#6, Cell#7 and Cell#8.

For the measurement of device performance, CdTe surface was etched in a bromine/methanol solution ($250\ \mu\text{l}/50\ \text{ml}$) for 5 s. Samples were rinsed thoroughly using DI water and then dried with nitrogen gas. A conductive graphite paste (Agar Scientific) was applied on the etched surface of CdTe for the back contact. Front contact was taken from ITO. Cell area is taken as $2\ \text{mm}^2$. The samples were characterized for the structural properties by using X-ray diffractometer (Bruker D8 Advance). The surface morphology of films was studied using atomic force microscope (NT-MDT Ntegra). I - V measurements were carried out in this work using a Keithley 2400 source meter. I - V measurements were done in both dark and light environments. For light measurement, AM 1.5 solar simulator with 1 SUN is used. From I - V characteristics, PV working parameters, such as photo-conversion efficiency (η), fill factor (FF), open circuit voltage (V_{oc}) and short circuit current density (J_{sc}) were retrieved.

3. Result and discussion

Figure 1 shows the X-ray diffraction (XRD) patterns of nanocrystalline thin films of ZnS , $\text{Zn}_{0.1}\text{Cd}_{0.9}\text{S}$ and CdS corresponding to cadmium mole fractions (x) = 0, 0.9 and 1. This

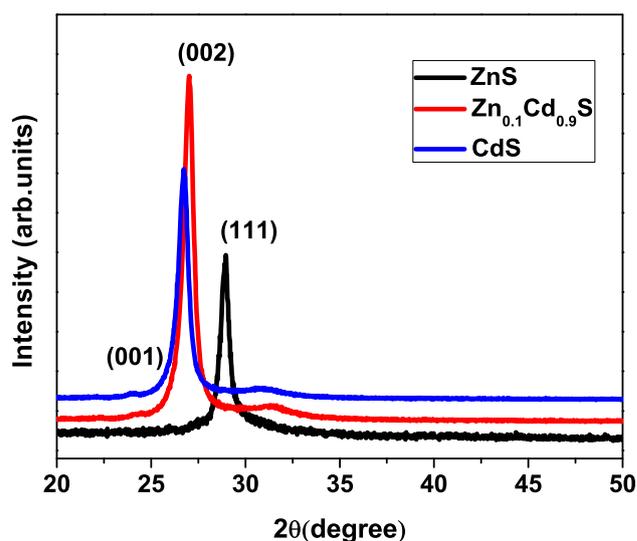


Figure 1. XRD patterns of ZnS , $\text{Zn}_{0.1}\text{Cd}_{0.9}\text{S}$ and CdS nanocrystalline thin films.

figure shows the shift in the XRD peaks towards lower angles with increase in x . XRD peak (002) is positioned at $2\theta = 26.9^\circ$ for $\text{Zn}_{0.1}\text{Cd}_{0.9}\text{S}$ and at 26.68° for CdS film. This shift towards lower angle with increase in x is believed to result from the incorporation of Cd ions into the ZnS lattice and suggests a successful formation of $\text{Zn}_{0.1}\text{Cd}_{0.9}\text{S}$ thin film.

Semiconductor alloys (solid solutions) were proposed to obey Vegard's law [22], revealing the linear relationship between the lattice constant and composition as follows:

$$a_{\text{A}_{1-x}\text{B}_x\text{C}}^0(x) = (1-x)a_{\text{AC}}^0 + xa_{\text{BC}}^0,$$

where $a_{\text{A}_{1-x}\text{B}_x\text{C}}^0$ is the natural constant of the ternary form $\text{A}_{1-x}\text{B}_x\text{C}$ and a_{AC}^0 and a_{BC}^0 are the natural constants of the binaries AC and BC, respectively, and x is the mole fraction of binary BC. In the case of hexagonal structure, $a_{\text{ZnS}}^0 = 3.777\ \text{\AA}$, $c_{\text{ZnS}}^0 = 6.188\ \text{\AA}$ (PDF # 80-0007) and $a_{\text{CdS}}^0 = 4.142\ \text{\AA}$, $c_{\text{CdS}}^0 = 6.724\ \text{\AA}$ (PDF # 01-0780). The deposited $\text{Zn}_{0.1}\text{Cd}_{0.9}\text{S}$ nanocrystalline thin films ($x = 0.9$) shows a close agreement between the experimental ($a = 4.157\ \text{\AA}$ and $c = 6.621\ \text{\AA}$) and the theoretical lattice constant values ($a = 4.105\ \text{\AA}$ and $c = 6.670\ \text{\AA}$). This indicates that the composition of the ternary compound $\text{Zn}_{0.1}\text{Cd}_{0.9}\text{S}$ was very close to the mixing molar ratio of the starting materials of ZnS and CdS .

3.1 CdCl_2 treatment of CdTe layer

Durose *et al* [23] and Sites *et al* [24] have shown in their studies of CdS/CdTe solar cells that the CdTe grain boundaries act as barriers to current transport. Yan *et al* [25] suggested that the grain boundaries are strong electrical recombination centres. It is therefore, suggested that for improved device performance, grain boundaries should be minimized. This can be achieved by increasing the grain size in CdTe films. With the application of CdCl_2 during the heat treatment, the

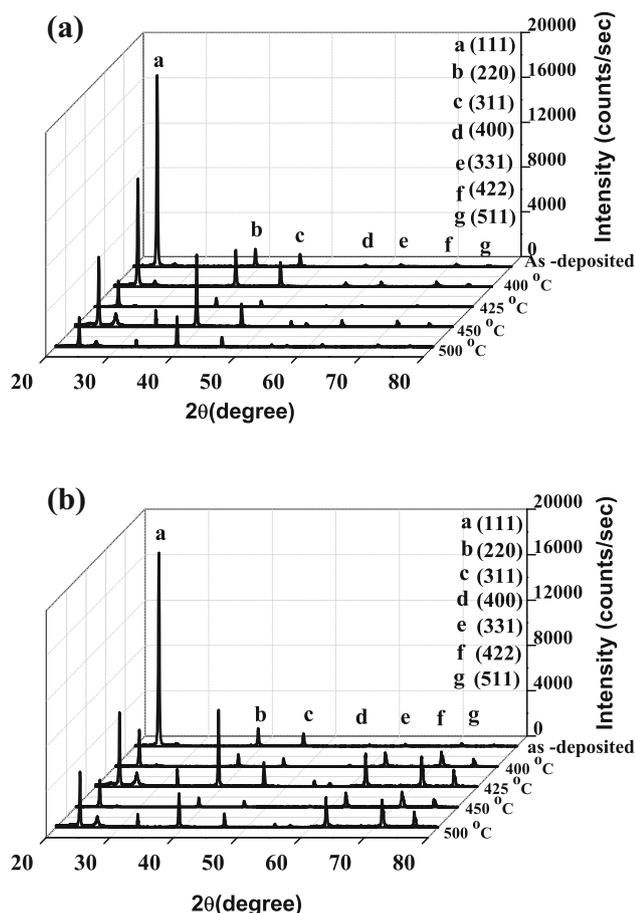


Figure 2. XRD patterns of CdTe films annealed with (a) wet CdCl₂ and (b) dry CdCl₂.

crystallographic rearrangements take place in the CdTe layer of the cell.

3.1a Structural properties: recrystallization: Figure 2a shows the XRD pattern of as-deposited CdTe film in Zinc blende with preferential orientation of (111) positioned at $2\theta = 23.7^\circ$. XRD pattern of Zn_{0.1}Cd_{0.9}S film (figure 1) shows the wurtzite structure preferentially oriented in (002) plane. Generally, it is difficult for materials of different crystal systems to form a heterojunction. Nakamura *et al* [26] suggested that the *c*-plane of the wurtzite structure and the (111) plane of the zinc blende structure readily form the heterojunction. In present case, CdTe and Zn_{0.1}Cd_{0.9}S may form the heterojunction.

Figure 2a and b shows the CdTe films annealed at 400, 425, 450 and 500°C with wet and dry CdCl₂ treatments, respectively. In both the treatments along with (111) peak, other peaks corresponding to (220), (311), (400), (331) and (422)

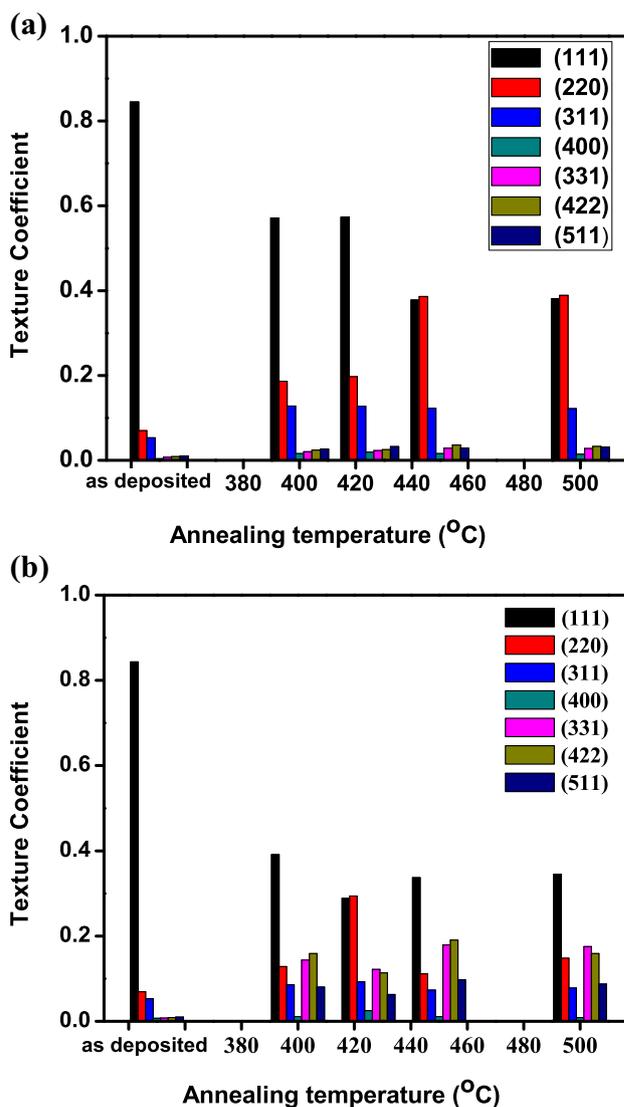


Figure 3. Texture coefficients of CdTe films with (a) wet CdCl₂ treatment and (b) dry CdCl₂ treatment with variation in annealing temperature.

planes of zinc blende structure, began to appear. Emergences of these peaks show the loss of preferential orientation. This loss of preferential orientation suggests the recrystallization in CdTe film. The magnitude of recrystallization in CdTe films is found as a function of annealing temperature. With increase in annealing temperature, intensity of the (111) peak decreases and intensity of other orientations increases.

To gauge the recrystallization in CdTe films, the texture coefficients (TC) were calculated using the below equation [27]:

$$TC = \frac{I(hkl)}{I(111) + I(220) + I(311) + I(400) + I(331) + I(422) + I(511)}$$

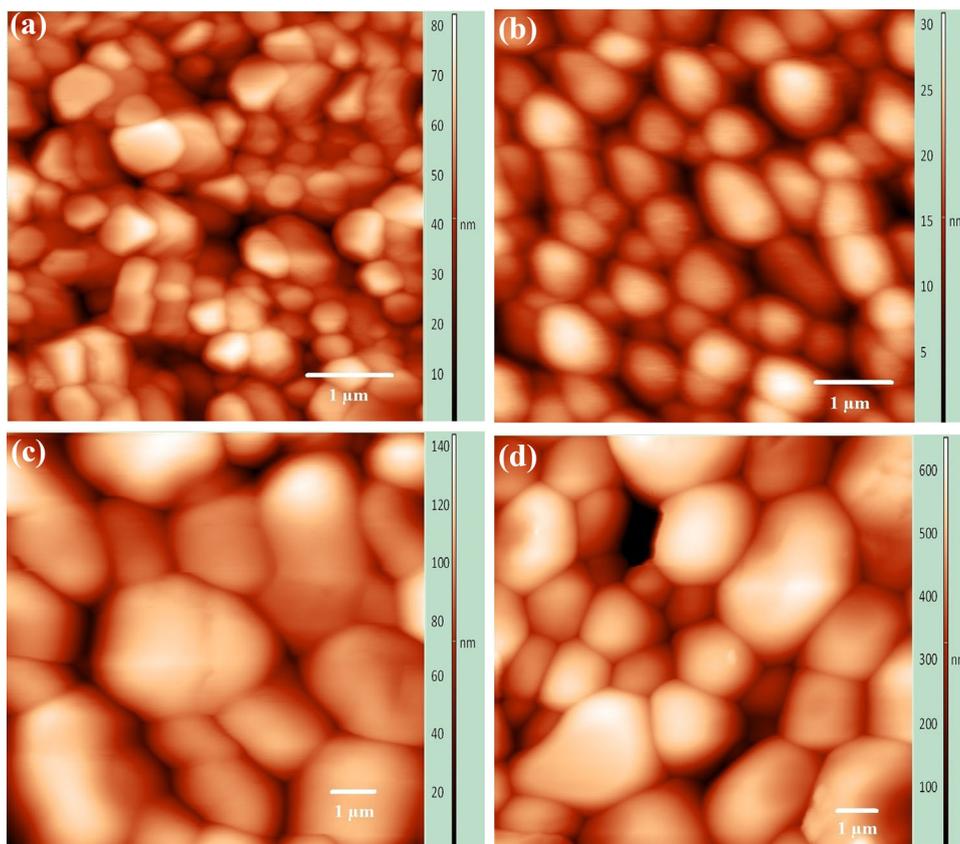


Figure 4. AFM images of CdTe films: (a) as-deposited, (b) wet CdCl₂ treated at 450°C, (c) dry CdCl₂ treated at 425°C and (d) dry CdCl₂ treated at 500°C.

Figure 3a shows the TC of as-deposited and wet-treated CdTe films annealed at 400, 425, 450 and 500°C. It is observed that as-deposited CdTe film is preferentially oriented to $\langle 111 \rangle$ plane. With increase in annealing temperature up to 450°C, TC of (111) orientation decreases and of other orientations increase. It suggests the random orientation of crystal planes. Further increase in annealing temperature does not produce any noticeable change in TC. Figure 3b shows the TC as-deposited and dry-treated CdTe films annealed at 400, 425, 450 and 500°C. Figure 3b shows that the TC of (111) plane is the lowest at 425°C. It suggests that the recrystallization is achieved at lower temperature in comparison to wet CdCl₂-treated cell. Also, the degree of random orientation as gauged by TC value is higher for dry CdCl₂-treated cell.

XRD measurement can also be used as measure for intermixing and stress in the CdTe layer [12]. Lattice constant of as-deposited CdTe film is calculated from XRD pattern and is obtained as 6.500 Å. The as-deposited film consists of compressive stresses due to lattice and thermal mismatch between the CdTe and Zn_{0.1}Cd_{0.9}S layers. It is observed that the lattice constant of CdTe film decreases when annealed. Lattice constant of dry CdCl₂-treated CdTe film annealed at 425°C becomes 6.478 Å (close to the CdTe bulk lattice constant $a_{\text{CdTe}}(\text{bulk}) = 6.468 \text{ \AA}$ [28]). Decrease in the lattice

constant is an indication of decrease of stress in the films. Lattice constant may also decrease because of the intermixing of Zn_{0.1}Cd_{0.9}S and CdTe films on annealing. Thus, the XRD measurements corroborate that the annealed CdCl₂ treatment improves the Zn_{0.1}Cd_{0.9}S/CdTe junction by enhancing the inter-diffusion.

3.2 Surface morphology–grain enhancement

Figure 4a shows AFM image of as-deposited CdTe films with a grain size of $\sim 0.3 \mu\text{m}$. Figure 4b shows AFM image of the wet-treated CdTe films annealed at 450°C with a grain size of $\sim 0.8 \mu\text{m}$. Figure 4c shows AFM images of the surface of dry CdCl₂-treated CdTe films annealed at 425°C. Significant grain growth was observed with dry CdCl₂ treatment when compared to as-deposited and wet-treated cells. Increase in grain size on annealing is attributed to recrystallization. These results are consistent with the study carried by Mendoza-Pérez *et al* [29,30]. Their group suggested that the heat treatment induces a sintering mechanism within the CdTe films that acts to decrease inter-grain pore size and increases the average grain size. Romeo *et al* [12] suggested that the creation of new grains are the results of disintegration of some larger grains. They attributed this to the relaxation of excessive strain in the

lattice. Their group further suggested that the coalescence of small grains into bigger ones is caused by the CdCl_2 sintering flux and by the high temperature [12]. Further increase in annealing temperature (both wet and dry CdCl_2 -treated) does not produce any significant effect on grain size (figure 4d). It is due to the fact that CdCl_2 acts as sintering agent, which was vapourized at temperature $> 490^\circ\text{C}$ [31]. This temperature is the eutectic temperature between CdCl_2 and CdTe .

3.3 Solar cell performance with CdCl_2 treatment

The CdCl_2 treatment improves the structural and morphological properties of the CdTe layer such as recrystallization and grain growth as discussed in previous section. It eventually results in a significant increase in photo-conversion efficiency (η). The average of three cells was taken for identical processing conditions. The photo-conversion efficiencies of cells fabricated with as-deposited CdTe layer had very poor efficiencies ($\eta < 1\%$).

3.3a Wet CdCl_2 treatment: Figure 5a and b shows the effect of annealing temperature on wet CdCl_2 -treated cells on photo-conversion efficiency (η), fill factor (FF), short circuit current density (J_{sc}) and open circuit voltage (V_{oc}). The photo-conversion efficiency η remains almost constant at $\sim 2.4\%$ with increase in annealing temperature from 400 to 425°C . Higher photo-conversion efficiency of 3.1% is obtained at 450°C . An increase in η at 450°C is attributed to the increase in the grain size of CdTe film. It however, rapidly reduces to 1.65% at 500°C . Figure 5a shows that the FF follows the similar trend (as of η) with variation in annealing temperature. The maximum FF $\sim 38\%$ is obtained at 450°C . Figure 5b shows that the short circuit density (J_{sc}) is $\sim 11.5 \text{ mA cm}^{-2}$ for annealing temperatures $\leq 425^\circ\text{C}$. It increases to 13 mA cm^{-2} with increase in annealing temperature from 425 to 450°C . Further increase in annealing temperature results in decrease of J_{sc} . Figure 5b shows that the open circuit voltage (V_{oc}) is maximum at $\sim 617 \text{ mV}$ for cells annealed at 450°C . V_{oc} decreases substantially to 535 mV at annealing temperature of 500°C . The reason for the relatively low efficiency for annealing temperatures $< 450^\circ\text{C}$ is probably due to the small grain size and consequent increase in grain boundaries. At elevated temperatures close to 500°C , diffusion of impurities such as sodium from soda lime glass substrate to ITO can be responsible for the deterioration of cell performance [32]. The best performing cell with wet CdCl_2 treatment was obtained at 450°C with an efficiency of 3.1% .

3.3b Dry CdCl_2 treatment: Figure 6a and b shows the effect of annealing temperature of dry CdCl_2 treatment on photo-conversion efficiency (η), fill factor (FF), short circuit current density (J_{sc}) and open circuit voltage (V_{oc}) of PV cells. V_{oc} and J_{sc} improve with increase in annealing temperature from 400 to 425°C , this in turn increases the η . The efficiency increase is attributed to both, the

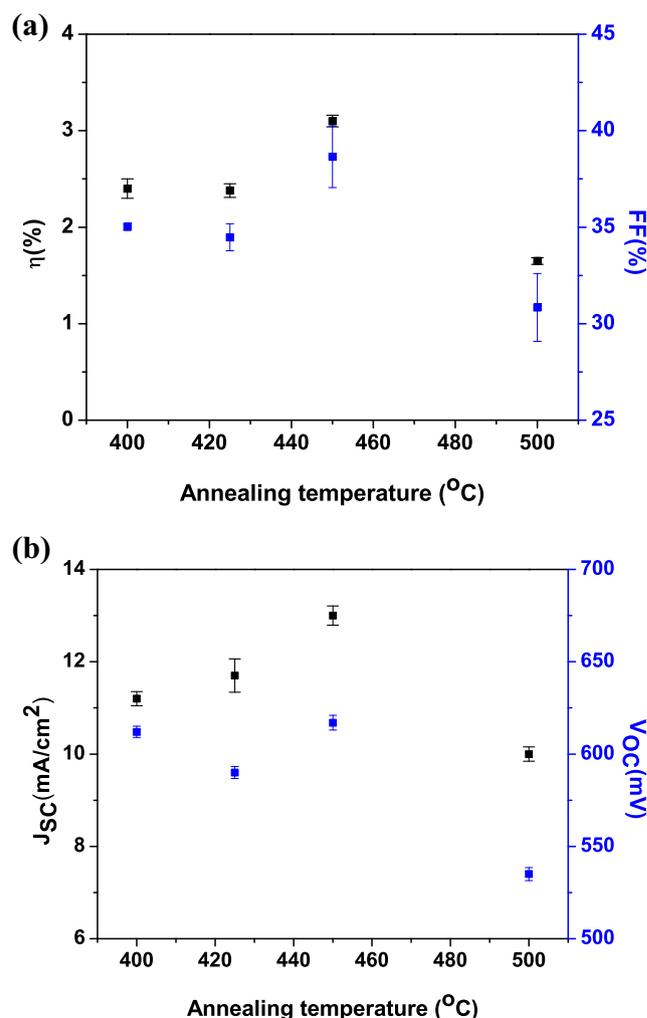


Figure 5. Cell parameters of the $\text{CdTe}/\text{Zn}_{0.1}\text{Cd}_{0.9}\text{S}/\text{ITO}/\text{glass}$ device with wet CdCl_2 treatment: (a) photo-conversion efficiency, η (%) and fill factor, FF (%) and (b) short circuit density, J_{sc} (mA cm^{-2}) and open circuit voltage, V_{oc} (mV).

enhanced grain size and increased recrystallization. Grain size of $\sim 3.5 \mu\text{m}$ for CdTe film is obtained at 425°C (figure 4c). The larger grains result in lesser grain boundaries, which reduce the carrier trapping significantly [32]. TC corroborates that the annealed CdCl_2 treatment enhances the inter-diffusion at $\text{Zn}_{0.1}\text{Cd}_{0.9}\text{S}/\text{CdTe}$ junction. Both factors result in the improved η at 425°C . A photo-conversion efficiency of 5.2% is achieved at annealing temperature of 425°C . For annealing temperature $> 425^\circ\text{C}$, photo-conversion efficiency decreases. Atomic force microscopy (AFM) results (figure 4d) suggest that annealing temperatures above 425°C have negligible effect on the grain size. Hence, grain size does not contribute towards influencing the photo-conversion efficiencies for annealing temperatures $> 425^\circ\text{C}$. On annealing, the sulphur diffuses from $\text{Zn}_{0.1}\text{Cd}_{0.9}\text{S}$ layer to CdTe film. The rate of sulphur diffusion increases with increase in annealing temperature. This can lead to situation, where the

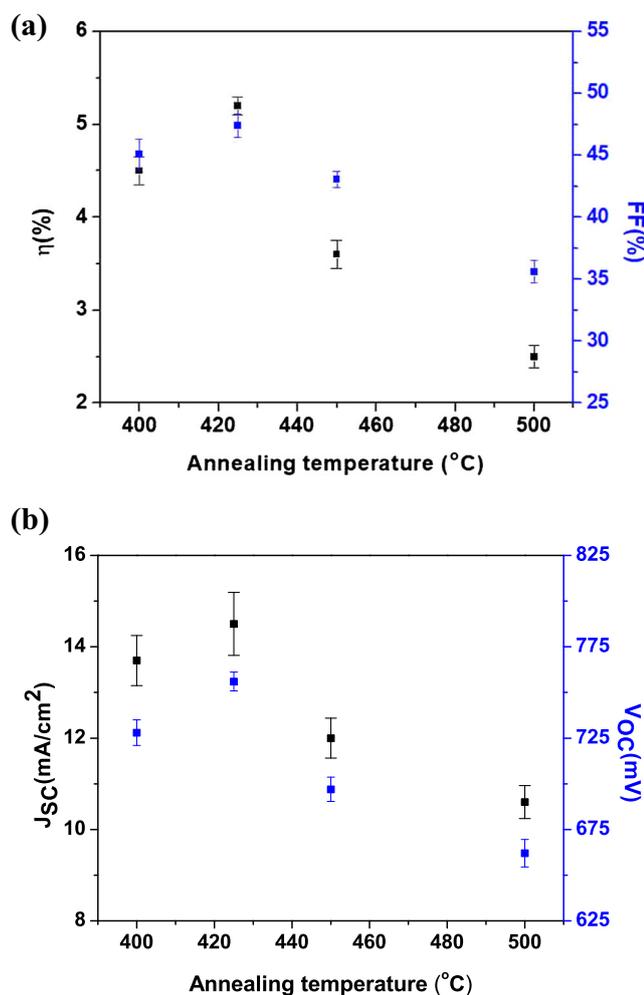


Figure 6. Cell parameters of the CdTe/Zn_{0.1}Cd_{0.9}S/ITO/glass device with dry CdCl₂ treatment: (a) photo-conversion efficiency, η (%) and fill factor, FF (%) and (b) short circuit density, J_{sc} (mA cm⁻²) and open circuit voltage, V_{oc} (mV).

consumption of Zn_{0.1}Cd_{0.9}S by CdTe film can take place. It reduces the lower cutoff wavelength region and can be responsible for the reduced efficiencies at higher annealing temperatures. In addition to sulphur, sodium diffusion from glass substrate to ITO also takes place at higher annealing temperatures [33], thereby results in the overall reduction in the cell performance.

3.4 Series resistances (R_s) of wet- and dry-treated cells

Figure 7 shows the series resistances of annealed wet and dry CdCl₂-treated cells. Series resistance of PV cell was calculated by taking the inverse of the slope of J - V characteristic at $V = V_{oc}$ point [34]. For all the annealing temperatures, the value of series resistance for wet CdCl₂-treated cell is high in comparison to those of dry CdCl₂-treated cell. For wet CdCl₂ treatment, series resistance does not vary appreciably and remains at $\sim 24 \Omega\text{-cm}^2$ for annealing temperatures $\leq 450^\circ\text{C}$.

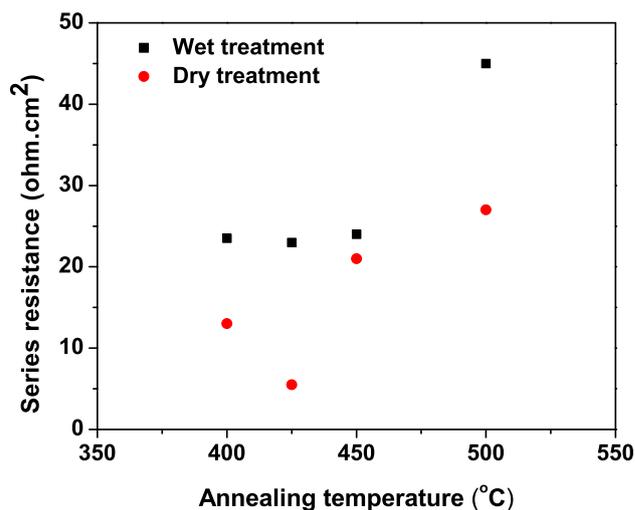


Figure 7. Series resistances of the CdTe/Zn_{0.1}Cd_{0.9}S/ITO/glass device structure with wet and dry CdCl₂ treatments.

However, it shoots to $45 \Omega\text{-cm}^2$ at 500°C and is responsible for the degradation in cell efficiency. For dry CdCl₂ treatment, series resistance decreases from 13 to $5 \Omega\text{-cm}^2$, while increasing the annealing temperature from 400 to 425°C . Reduction in series resistance suggests the decrease in recombination current of the cell and results in the photo-conversion efficiency of 5.2%. This improvement in photo-conversion efficiency can be attributed to two factors: (i) reduced carrier trapping in CdTe films resulting from larger grain size and reduction in defect concentration and (ii) higher open circuit voltage of the cell (lesser shunting probability). Increase in annealing temperature $> 425^\circ\text{C}$ results in higher series resistances and thereby degradation in overall efficiency. Lee *et al* [35] have observed similar behaviour of series resistance for methanol CdCl₂-dipped CdS/CdTe cells with variation in annealing temperature. Low values of FF (for both dry and wet treatments) shown in figures 5 and 6 indicate that the series resistances of these cells are contributing to reduction in overall cell performance.

4. Conclusion

This work presents the fabrication and characterization of CdTe/Zn_{0.1}Cd_{0.9}S/ITO/glass solar cell. In the present study, the effect of two types of cell activation steps namely wet and dry CdCl₂ treatment with annealing on cell parameters is analysed. Cell-activation step i.e., CdCl₂ treatment was found to play a dominant role in governing the photo-conversion efficiency. XRD studies showed that the dry treatment is more effective in recrystallization as compared to wet CdCl₂ treatment. The optimal value of TC of (111) orientation, grain size and annealing temperature (0.29, $\sim 3.5 \mu\text{m}$, 425°C) and (0.4, $\sim 0.8 \mu\text{m}$, 450°C) are obtained respectively, for dry and wet CdCl₂-treated CdTe films. The photo-conversion efficiency

of 5.2% is achieved at 425°C in dry CdCl₂ treatment. For wet CdCl₂ treatment, maximum efficiency of 3.1% is obtained at 450°C. In addition, for dry treatment, cells are subjected to heat treatment for 5 min, while for wet treatment, the heat treatments were carried out for 30 min. Thus, most of the layer is expected to evaporate away from the film in wet treatment as it is exposed for longer time and recrystallization was not effective for wet-treated samples. Thus, the use of dry CdCl₂ treatment provides two fold benefits: (i) higher efficiencies and (ii) lower processing temperature.

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