

# Study of in situ $\text{CdCl}_2$ treatment on CSS deposited CdTe films and CdS/CdTe solar cells

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## Abstract

Effect of in situ  $\text{CdCl}_2$  treatment on the morphological, structural and electrical properties of CdTe films as well as on solar cell characteristics of CdS/CdTe junction has been investigated. XRD measurements show that the presence of  $\text{CdCl}_2$  vapours induces  $\langle 111 \rangle$  oriented growth in the CdTe films.  $\text{CdCl}_2$  concentration required for this oriented growth is found to be directly proportional to the substrate temperature. SEM measurements show enhanced grain growth in the presence of  $\text{CdCl}_2$ . Spectral response of the  $\text{CdCl}_2$  treated CdS/CdTe solar cells shows an enhanced CdS diffusion in to the CdTe, which results in an improved spectral response in UV range and a consequent reduction in the interface states density. A drastic reduction in the deep levels due to the  $\text{CdCl}_2$  treatment, as seen in the photo-capacitance studies, has results in CdS/CdTe solar cells having efficiency  $>8\%$ . © 2000 Elsevier Science S.A. All rights reserved.

**Keywords:** In situ  $\text{CdCl}_2$  treatment; Solar cells; CdS/CdTe; Close spaced sublimation

## 1. Introduction

CdTe/CdS solar cells require Cadmium Chloride ( $\text{CdCl}_2$ ) treatment to achieve higher efficiencies.  $\text{CdCl}_2$  treatment on CdTe is found to be beneficial in many ways. It helps the grain growth [1] and improves the electronic properties in CdTe films [2]. For screen printed-sintered CdTe films,  $\text{CdCl}_2$  acts as a flux, which helps to reduce the sintering temperature by promoting grain fusion at lower temperature [3]. It is also found to enhance the CdS diffusion in to CdTe, which results in the reduction of CdS thickness [4]. Cadmium chloride treatment has also been carried out on CdS films, which helps to reduce the Te diffusion in to CdS films [5].

Cadmium Chloride treatment can be carried out either as a post-deposition treatment or as an in situ treatment. However, post-deposition treatment is the most accepted method due to its inherent process flexibility. Conventionally,  $\text{CdCl}_2$  treatment has been carried out as solution treatment. In the solution treatment,  $\text{CdCl}_2$  is first dissolved in methanol and then applied on as-deposited CdTe films by dip or spray or spin coating techniques. In an alternative method, thin layer of  $\text{CdCl}_2$  is formed on CdTe by laser ablation [6]. After the  $\text{CdCl}_2$  application, CdTe film under-

goes air or vacuum heat treatment at 400 to 420°C for 10 to 30 min, during which the electrical and structural properties of CdTe films get suitably modified. The heat treatment is generally followed by a hot water wash to remove the excess  $\text{CdCl}_2$ . This technique appears to be simple to perform but has many inherent disadvantages. The major disadvantages are the non-uniformity in the device property and device degradation due to the humid processing ambient. It has also been reported that the residue left over by the  $\text{CdCl}_2$  treatment may not be completely removed by the washing, because of the formation of oxy-chlorides, which are insoluble in water [7]. There are alternative techniques, which are free from these disadvantages. They are generally classified in to vapour chloride treatments. In one of these techniques, CdTe films heated in the presence of  $\text{CdCl}_2$  vapours [8]. In a variant of this technique, CdTe films are also heat treated in the presence of Cl vapours generated from HCl. For this CdTe films are heat treated in the presence of nitrogen, which passes through HCl acid solution to generate HCl vapours [9]. The major problem with this technique is the poor control over the HCl concentration. These techniques are found to have less deteriorating effect on the devices. However, these techniques may not be acceptable for a fabrication process, where an increase in the process steps causes increase in the manufacturing cost. An in situ vapour  $\text{CdCl}_2$  treatment will be an ideal option for the manufacturing of CdTe/CdS solar cells. In this case the treatment

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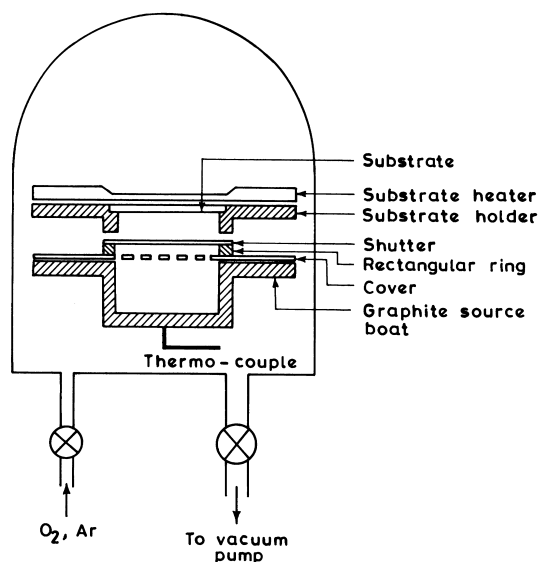


Fig. 1. Schematic diagram of Close Spaced Sublimation system.

will take place during the film growth itself and will result in better  $\text{CdCl}_2$  utilisation [10]. In this paper we report on our studies of the effect of in situ  $\text{CdCl}_2$  treatment on CdTe films and solar cells deposited by Close Spaced Sublimation (CSS) technique [11].

## 2. Experimental

We have designed and fabricated a CSS system for the deposition of in situ  $\text{CdCl}_2$  treated CdTe films. The experimental set up is as shown in the Fig. 1. It consists of a resistively heated graphite boat having a perforated graphite cover. The heating process required 100 to 120 A current at voltage of 3–4 V, which is supplied by a low-tension transformer (LT1). The source material, in the form of pellets, is made of CdTe with  $\text{CdCl}_2$  added to it in various concentrations. Since the molecular weight and diameter of  $\text{Te}_2$  are significantly greater than those of Cd, diffusion coefficient of Cd is 1.85 times of the diffusion coefficient of  $\text{Te}_2$  [12]. This may leads to non-uniform atomic flux near the substrate. However, the presence of perforated graphite cover,

which deflects back some of the vapours (Fig. 2), helps to achieve a near uniform mixture of Cd and Te flux. The

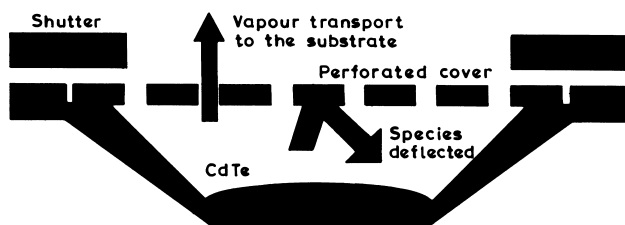


Fig. 2. Vapour deflection by the perforated graphite cover.

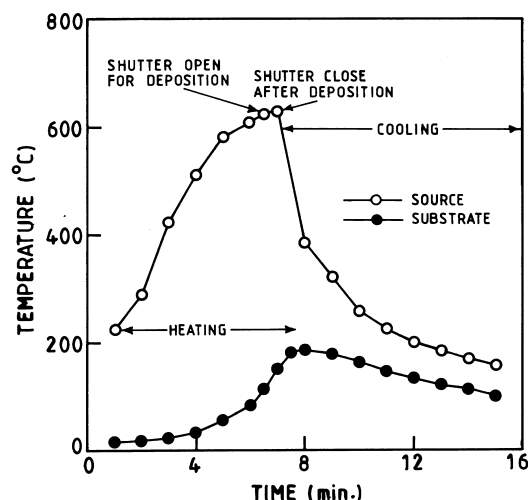


Fig. 3. Typical deposition cycle used for the deposition of in situ  $\text{CdCl}_2$  treated CdTe films.

substrate is kept at a distance of  $\approx 3$  mm using ceramic spacers. It can be heated by using a graphite heater, which is resistively heated with the help of another low-tension transformer (LT2). The temperatures of the substrate and the graphite boat can be controlled almost independently. This has been facilitated by the use of a stainless steel shutter between the substrate and source boat. A specially designed opening in the shutter allows deposition of uniformly thick CdTe films. The rise in temperature of the substrate due to the source boat during a typical deposition cycle has been given in Fig. 3. It shows that the heating of the source ( $675^\circ\text{C}$ ) takes 7 min and the film deposition lasts for 1 min. The rise in substrate temperature during source heating is below  $160^\circ\text{C}$  and the rise when shutter is opened is  $\approx 20^\circ\text{C}$ . To take care of this small rise in substrate temperature, deposition of CdTe films is carried out at substrate temperatures higher than  $200^\circ\text{C}$ . The heater assembly is placed in a vacuum chamber, which can be evacuated up to  $10^{-4}$  Pascal with the help of a diffusion pump. The deposition can be carried out in high vacuum or in the presence of carrier gases like oxygen or argon. For the deposition of in situ  $\text{CdCl}_2$  treated CdTe films, the substrate is heated to the required temperature, followed by the source heating in the graphite boat. Once the sublimation temperature reaches, deposition is carried out by opening shutter for required time. Since  $\text{CdCl}_2$  has very high vapour pressure at the CdTe sublimation temperature, the deposition takes place in the presence of  $\text{CdCl}_2$  vapours.

Deposition has been carried out with  $\text{CdCl}_2$  concentration varying from 1 to 5 wt.%. Source temperature is kept constant at  $675^\circ\text{C}$  and the substrate temperature has been varied from  $250$  to  $500^\circ\text{C}$ . Argon carrier gas pressure is kept constant at 1 Pascal. The growth rate is found to be independent of the  $\text{CdCl}_2$  concentration. The colour of the films is found to change from light to dark grey as the substrate temperature as well as the  $\text{CdCl}_2$  concentration reduces.

CdS/CdTe cells are fabricated by depositing this in situ CdCl<sub>2</sub> treated CdTe films on TCO/CdS substrate. CdTe films and cells hence obtained are then characterised for structural and electrical properties.

### 3. Results

#### 3.1. Structural properties of CdTe films

X-ray diffraction measurements are carried out to find out the impact of CdCl<sub>2</sub> treatment on the crystallinity of the CdTe films. Measurements were carried out using glancing angle X-ray diffractometer (Giegerflux-D/max-RB-RU2000) from Rigaku Corporation, Japan. It is observed that in XRD spectra the peaks at small  $2\theta$  values are shifted towards lower values of  $2\theta$ . This may be due to the high absorption coefficient of the CdTe material. However, at higher  $2\theta$  values, these shifts are very small. Fig. 4 shows the lattice constant 'a' derived from each diffraction peak plotted against  $\cos^2\theta/\sin\theta$ . The extrapolation of the least square fit of these points to  $\theta = \pi/2$  give the correct lattice constant of the CdTe films [13]. The lattice constant increases from 6.437 to 6.479 Å, respectively, for CdCl<sub>2</sub> concentration varying from 1 to 5 wt.%. For 5 wt.% CdCl<sub>2</sub>, the value of lattice constant 'a' is near to that for a powder sample. (6.481 Å) [14]. The reduction in the lattice constant value can be due to tensile stress in these films. The shift in the value of 'a' towards the powder sample value with increasing CdCl<sub>2</sub> concentration indicates that CdCl<sub>2</sub> reduces the stress in the CdTe films.

Fig. 5a shows the variation in the diffraction peak intensities with CdCl<sub>2</sub> concentration for films deposited at 250°C substrate temperatures. It shows that for films deposited without in situ CdCl<sub>2</sub> treatment most of the CdTe X-ray diffraction peaks are present. However, when CdCl<sub>2</sub> concen-

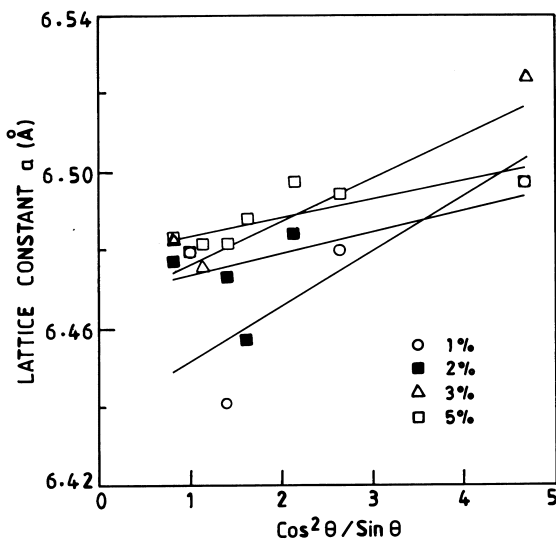


Fig. 4. Lattice constant of CdTe films deposited at 500°C substrate temperature.

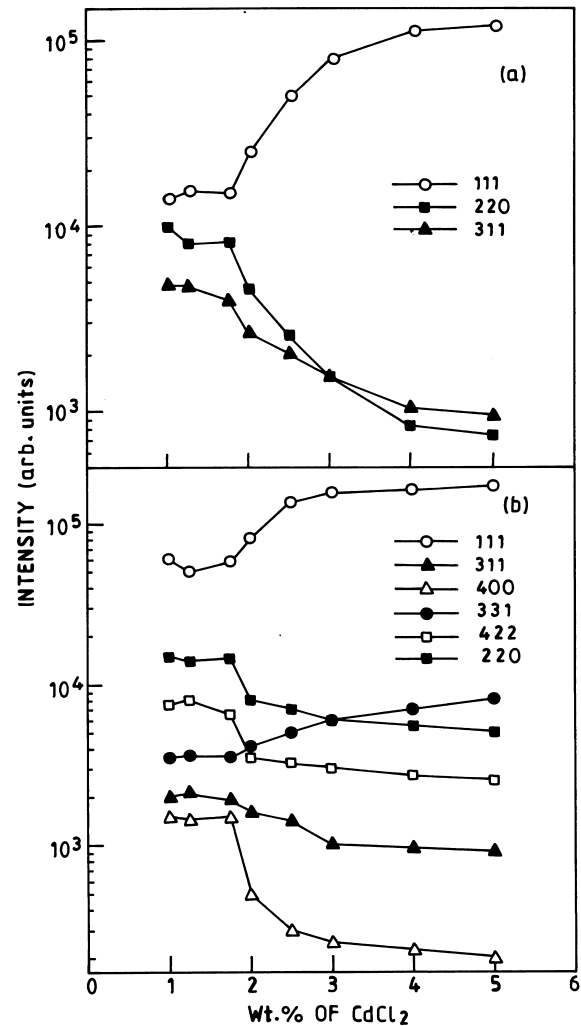


Fig. 5. Variation in the diffraction peak intensities with CdCl<sub>2</sub> concentration for CdTe films deposited at substrate temperature of (a) 250°C and (b) 500°C.

tration increases to greater than 1.5 wt.%, the X-ray reflection is predominantly from (111) plane and reflection from all other planes becomes negligibly small. This indicates that in the presence of sufficient CdCl<sub>2</sub> vapour pressure, CdTe grains preferentially grow with (111) orientation. The degree of preferred (111) orientation,  $p(111)$  is quantified from the X-ray diffraction scan using the following formula for polycrystalline fibre texture analysis.

$$p(111) = \frac{\frac{I(111)}{I_0(111)}}{\frac{1}{N} \sum_{hkl} \frac{I(hkl)}{I_0(hkl)}} \quad (1)$$

where  $N$  is the number of peaks in the region considered,  $I(hkl)$  is the measured intensity of peak  $hkl$  and  $I_0(hkl)$  is the intensity of the corresponding peak from a powder sample.

For CdCl<sub>2</sub> concentration lower than 1.5%, the value of  $p(111)$  is found to be equal to one, which indicate that the

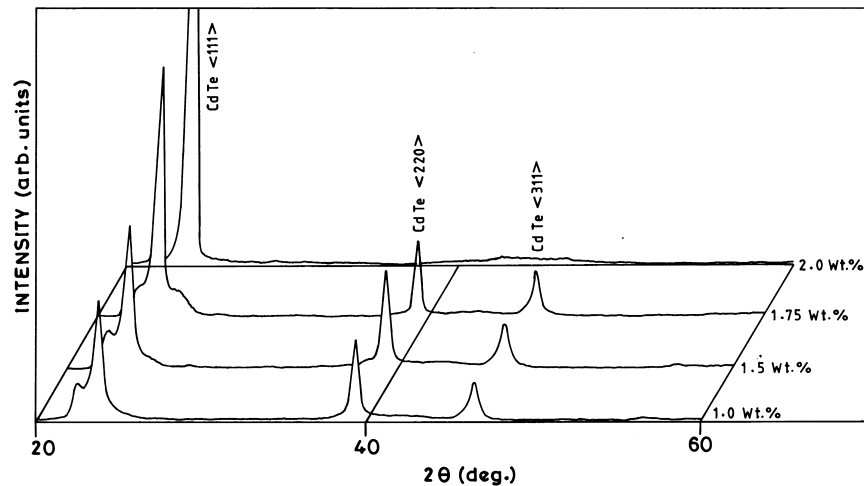


Fig. 6. Diffraction spectra for CdTe films deposited at 500°C substrate temperature.

grains are randomly oriented in the CdTe films. However for CdCl<sub>2</sub> concentrations greater than 1.5%, the value of P(111) is found to be greater than one, which is an indication of the (111) preferentially oriented growth in these films. It is known that the grain orientation in the film is influenced by the nature of the substrate on which it is deposited [15]. Hence the preferred oriented growth observed in the <111> direction can be attributed to the nature of the substrate. However the dependence on CdCl<sub>2</sub> concentration indicates that CdCl<sub>2</sub> vapours somehow facilitating this oriented growth. This may be due to the increase in the mobility of Cd and Te<sub>2</sub> atoms in the presence of CdCl<sub>2</sub>.

Fig. 5b shows the variation in the diffraction intensities with CdCl<sub>2</sub> concentration for films deposited at 500°C. In this case also CdTe film growth shows preferential growth in the presence of CdCl<sub>2</sub>. However, the oriented growth is observed at higher concentrations of CdCl<sub>2</sub> than for CdTe deposited at lower substrate temperature. Fig. 6 shows the XRD spectra for the same set of films. For 500°C substrate temperature, the number of peaks present in the diffraction spectra is larger than that for 250°C. The oriented growth has been observed for CdCl<sub>2</sub> concentration greater than 1.75 wt.%. This higher CdCl<sub>2</sub> concentration required for the oriented growth can be due to the change in the vapour transport of CdCl<sub>2</sub> with substrate temperature. It is known that CdCl<sub>2</sub> acts as a catalyst in the CdTe film growth and it increases the atomic mobility of Cd and Te. At high temperature CdTe dissociates into Cd and Te<sub>2</sub>. During the deposition of CdTe films, CdCl<sub>2</sub> along with Cd and Te<sub>2</sub> vapours get adsorbed on to the films. On the substrate Cd and Te<sub>2</sub> atoms react to form CdTe in the presence of CdCl<sub>2</sub>, which enhances their atomic mobility. As we reported earlier [16], the CdTe growth in our CSS system is diffusion limited. Since CdCl<sub>2</sub> preserves its linear molecular structure in the vapour phase, the diffusion transport of CdCl<sub>2</sub> towards the substrate can be expressed by the following equation [17].

$$J_{\text{CdCl}_2} = \frac{D_{\text{CdCl}_2}}{R\delta} \left[ \frac{\rho_{\text{CdCl}_2}(T_1)}{T_1} - \frac{\rho_{\text{CdCl}_2}(T_2)}{T_2} \right] \quad (2)$$

$$J_{\text{CdCl}_2} = \frac{D_{\text{CdCl}_2}}{R\delta} \rho_{\text{ratio}} \quad (3)$$

where  $J_{\text{CdCl}_2}$  is the diffusion flux of CdCl<sub>2</sub>,  $D_{\text{CdCl}_2}$  is the diffusion coefficient of CdCl<sub>2</sub>,  $\delta$  is the gap between the source and substrate,  $T_1$  and  $T_2$  are the source and substrate temperature, respectively,  $\rho_{\text{CdCl}_2}$  is the vapour pressure of CdCl<sub>2</sub>.

The change in the  $\rho_{\text{CdCl}_2}$  and  $\rho_{\text{ratio}}$  with temperature are given in Fig. 7. It shows that the  $\rho_{\text{ratio}}$  decreases as the substrate temperature reaches the source temperature. This causes the reduction in  $J_{\text{CdCl}_2}$  near to the substrate and less CdCl<sub>2</sub> vapours are present during the CdTe film growth. This reduction in the CdCl<sub>2</sub> vapour pressure near to the substrate

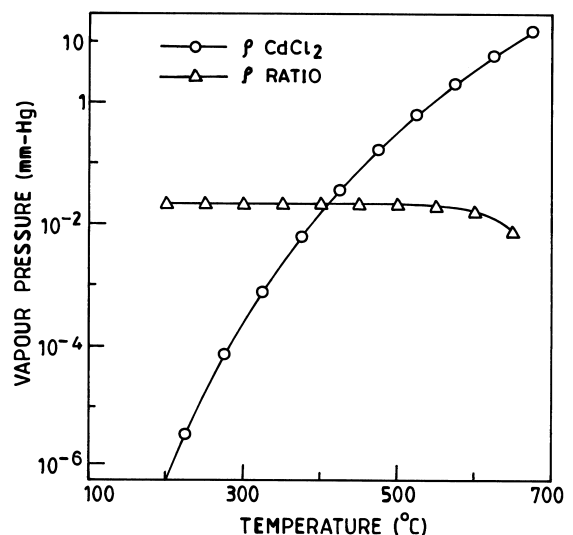


Fig. 7. Change in the  $\rho_{\text{CdCl}_2}$  and  $\rho_{\text{ratio}}$  with substrate temperature.

necessitates an increase in the  $\text{CdCl}_2$  concentration in the CdTe source material.

The preferentially oriented growth observed in our films at higher  $\text{CdCl}_2$  concentration is in contradiction to the growth reported for post-deposition  $\text{CdCl}_2$  treated CdTe films. There, the preferred  $\langle 111 \rangle$  orientation observed in the as-deposited CdTe films disappears during the  $\text{CdCl}_2$  solution treatment. However, (111) preferred orientation is retained when the treatment is carried out with  $\text{CdCl}_2$  vapour [18]. This difference may be due to the inherent difference in the  $\text{CdCl}_2$  treatment. For instance, in an in situ treatment, CdTe nucleation and grain growth are taking place in the presence of  $\text{CdCl}_2$  vapour. But in the case of post-deposition treatment, the grain growth is mainly superficial and new grains formed during the re-

crystallisation process is less influenced by nature of the substrate.

### 3.2. Morphology of CdTe films

SEM measurements are carried out to determine the surface morphology of the CdTe films. Fig. 8 shows the impact of  $\text{CdCl}_2$  on the morphology of the CdTe films. The grain size increases drastically with  $\text{CdCl}_2$  concentration. Grain size has been increased from 0.1 to  $>1 \mu\text{m}$  for a 5 wt.%  $\text{CdCl}_2$  treated film. This increase in grain size shows that for in situ treatment, similar to post-deposition treatment, the presence of  $\text{CdCl}_2$  facilitates the grain growth in the CdTe films.

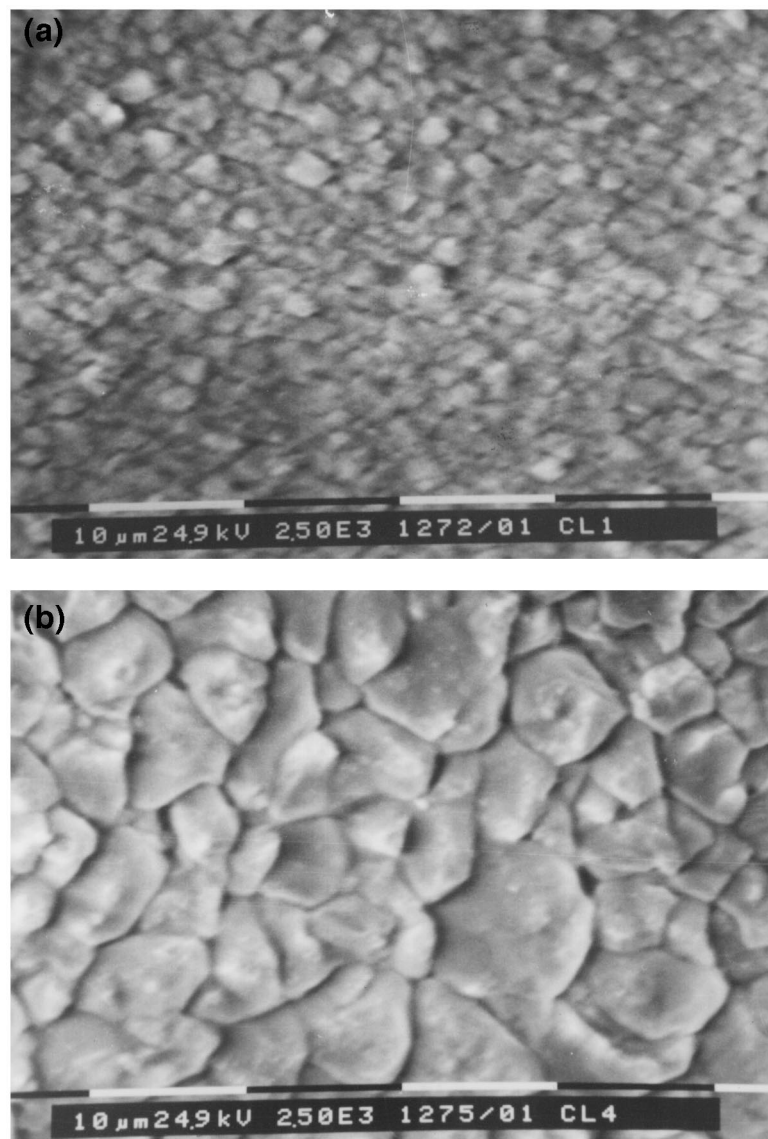


Fig. 8. SEM micrographs for CdTe films in situ treated with varying  $\text{CdCl}_2$  concentrations (a) 1 wt.% and (b) 4 wt.%.

### 3.3. Electrical properties

Fig. 9 shows the changes in the sheet resistance with  $\text{CdCl}_2$  concentration. As the  $\text{CdCl}_2$  concentration increases, the sheet resistance decreases initially, then starts increasing. The reduction in the resistivity can be attributed to the increased grain growths taking place in the  $\text{CdTe}$  films in the presence of  $\text{CdCl}_2$ . The increased grain size reduces the grain boundary potential and hence improves the inter-grain conduction. As the concentration of  $\text{CdCl}_2$  increases, Cl may also dope  $\text{CdTe}$  increasing the sheet resistance. Since Cl is an n-type impurity in  $\text{CdTe}$ , due to self-compensation effect it will increase the resistivity of p-type  $\text{CdTe}$  films. Hence there are two opposing phenomenon taking place in the  $\text{CdTe}$  films due to the  $\text{CdCl}_2$  treatment. Fig. 9 shows that at higher concentration of  $\text{CdCl}_2$ , the Cl doping dominates the conduction and hence results in higher sheet resistance. However, the Cl impurity concentration is still below the detection limit of the EDX. measurement.

### 3.4. Effect of cadmium chloride on the $\text{CdS}/\text{CdTe}$ solar cells

In situ cadmium chloride treatment has been utilised for the fabrication of  $\text{CdS}/\text{CdTe}$  solar cells. The cells have been deposited using  $\text{CdTe}$  source material with  $\text{CdCl}_2$  concentration varying from 1 to 5 wt.%. After the deposition of  $\text{CdTe}$  films, the bottom contact has been taken by depositing Au on  $\text{CdTe}$ . Noticeable change has been observed in the short circuit current density ( $J_{\text{sc}}$ ), which is found to increase drastically with  $\text{CdCl}_2$  concentration. However, there is no drastic change in the series resistance ( $R_s$ ). Open circuit voltage ( $V_{\text{oc}}$ ) and fill factor (FF) of the cells are found to increase with  $\text{CdCl}_2$  concentration up to 3 wt.% and later decrease. The efficiency of the solar cells is found to be consistently increasing with  $\text{CdCl}_2$  concentration. SR measurements are carried out on these samples to find out

reason for high current density associated with higher  $\text{CdCl}_2$  concentration,

Fig. 10 shows the result of SR measurement carried out on these samples. There is a gradual improvement in the spectral response of these samples at shorter wavelength (say QEB). The photo-electron collection from the wavelength ranging from 400 to 520 nm increases with increasing  $\text{CdCl}_2$  concentration. The photoelectron collection from wavelengths above the  $\text{CdS}$  bandgap (say QER) increases due to the  $\text{CdCl}_2$  treatment. The increment in the QEB suggests a reduced absorption in the  $\text{CdS}$  layer and this can be directly associated to the reduction in  $\text{CdS}$  thickness. Since deposition parameter and the thickness are same for  $\text{CdS}$  films on all these cells, this reduction in thickness can be due to  $\text{CdS}$  diffusion into  $\text{CdTe}$ . One has to believe that the  $\text{CdCl}_2$  enhances the  $\text{CdS}$  diffusion into  $\text{CdTe}$ , which then results in the gradual increase in the QEB with  $\text{CdCl}_2$  concentration. Similar result has been reported by Mavo et al. [13].

The panchromatic improvement in the QER can be associated with the reduction in the interface states. In order to calculate the recombination velocity at the interface, we have used the formula reported by Mitchell et al. for the quantum efficiency [19]. The quantum efficiency is given by

$$Q(\lambda) = h(V)g(V) \quad (4)$$

$$g(V) = 1 - [1 + \alpha L]^{-1} \exp(-\alpha \omega) \quad (5)$$

$$h(V) = [1 + S/\mu E_0]^{-1} \quad (6)$$

where  $S$  is the interface recombination velocity,  $\mu$  is the minority carrier mobility,  $E_0$  is the electric field in the junction,  $\alpha$  is the absorption coefficient,  $L$  the minority diffusion length and  $\omega$  is the depletion layer width.

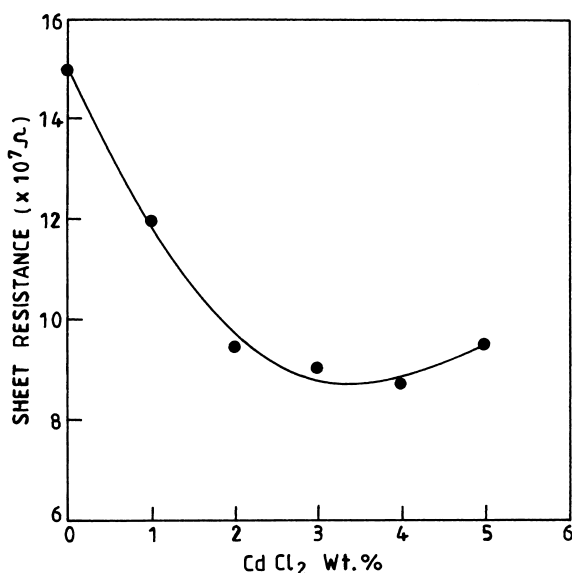


Fig. 9. Change in the sheet resistance with  $\text{CdCl}_2$  concentration.

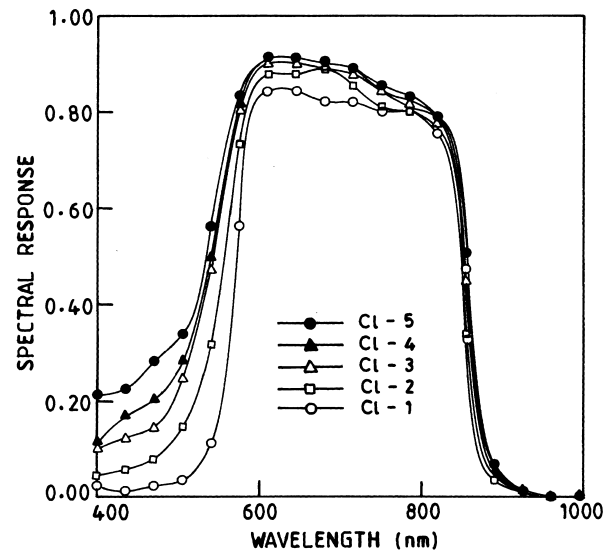


Fig. 10. Spectral response of  $\text{CdS}/\text{CdTe}$  solar cells in situ treated with varying  $\text{CdCl}_2$  concentrations.

In order to take care of the low diffusion length observed in these samples, we have taken  $L$  as  $0.4 \mu\text{m}$ . By fitting the theoretically calculated quantum efficiency with the measured value we are able to calculate the interface recombination velocity for these cells. The interface recombination velocity measured on these samples is found to be less than  $5 \times 10^5 \text{ cm/s}$  for 5 wt.%  $\text{CdCl}_2$  concentration. Which is found to be much lower than that for the cells without  $\text{CdCl}_2$  treatment ( $5 \times 10^6 \text{ cm/s}$ ). It shows that the recombination velocity reduces with increasing  $\text{CdCl}_2$  concentration. This can be due to the CdS diffusion, which reduces the lattice mismatch between CdS and CdTe by forming  $\text{CdTe}_{1-x}\text{S}_x$  buffer layer [20]. The formation of this buffer layer reduces the interface states density at the CdS/CdTe junction and hence the lower recombination velocity.

The improvement in the QER can also be due to the improvements in the CdTe bulk properties facilitated by the  $\text{CdCl}_2$  in situ treatment. Fig. 11a,b show the photo-capacitance spectra for samples deposited with 1 and 5 wt.% of  $\text{CdCl}_2$ , respectively. Cells deposited with 1 wt.% of  $\text{CdCl}_2$  show large photo-capacitance. The large photo-capacitance can be due to the large number of saturable deep levels presented in the cell. It has also been observed that, in the presence of light bias, SR of this cell shows reduction in the current collection. The reduced current collection in the presence of bias light is due to the decrease in the depletion layer width. These results confirm that cells contain a large number of deep levels. At the same time samples deposited with 5% show less photo-capacitance. It has also been observed that SR of this cell does not show any reduction in SR with bias light. This shows that cells deposited with 5 wt.% of  $\text{CdCl}_2$  have less number of defects states. A similar result showing a reduction in the deep level impurity density with the  $\text{CdCl}_2$  treatment, has been reported by Duke et al. [21]. They also report that it is possible to assign various peaks observed in the photo-capacitance spectra to the various deep impurity levels.

The improved collection from QEB as well as from QER reflects on the increased  $J_{\text{sc}}$ . However, the benefits associated with the increased CdS diffusion and formation of  $\text{CdS}_{1-x}\text{Te}_x$  alloy formation are offset by the reduction in the  $V_{\text{oc}}$  and FF at higher  $\text{CdCl}_2$  concentrations. The increase in the  $V_{\text{oc}}$  up to 3%  $\text{CdCl}_2$  can be due to the grain growth in the CdTe with increased  $\text{CdCl}_2$  concentration. But the reduction in  $V_{\text{oc}}$  for higher  $\text{CdCl}_2$  concentrations is unexpected as the  $V_{\text{oc}}$  is expected to increase with increase in the grain size. A detailed analysis of the above results reveals that there can be two possible reasons for the reduction in the  $V_{\text{oc}}$ . As we have seen earlier, the increase in  $\text{CdCl}_2$  can lead to CdTe films grow with a high degree of (111) preferred orientation and also help to grow large size grains. These growth conditions at higher  $\text{CdCl}_2$  concentration not only promote the larger grain growth but also generate larger voids or pinholes between the grains. In addition to that,

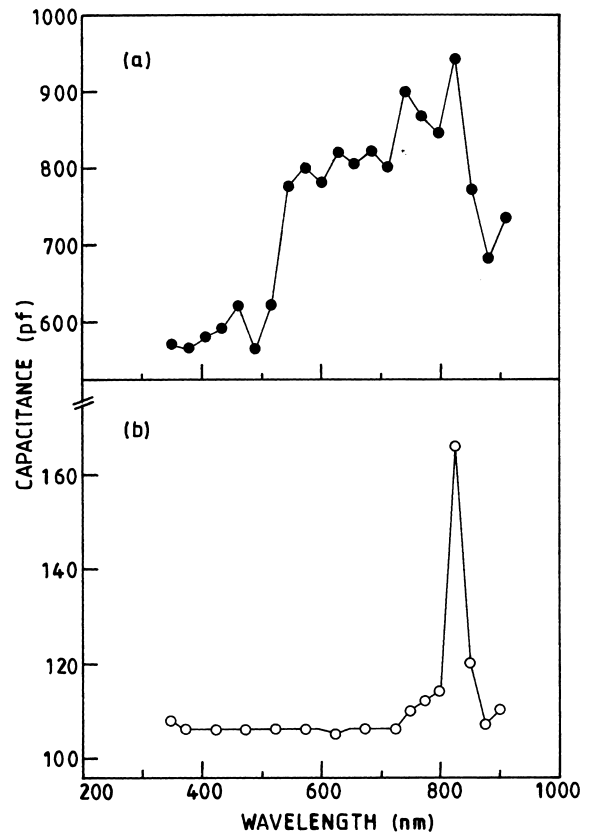


Fig. 11. Photocapacitance spectra for CdS/CdTe solar cells (a) without  $\text{CdCl}_2$  treatment and (b) with 5 wt.%  $\text{CdCl}_2$  treatment.

the reduction in the CdS thickness at higher  $\text{CdCl}_2$  concentration exposes any flaws in the CdS films. All these lead to generation of micro-shunting paths in the CdS/CdTe junction and result in poor  $V_{\text{oc}}$  and FF.

#### 4. Conclusion

An innovative in situ  $\text{CdCl}_2$  treatment is carried out on CdTe films and devices. This treatment improves the CdTe grain size and induces a (111) preferential oriented growth. For CdS/CdTe device, it annihilates deep levels in the CdTe and also reduces the interface state density by promoting CdS diffusion. However, the enhanced CdS diffusion and the preferential growth of larger CdTe grain found to generate shunting paths in the CdS/CdTe devices. Hence, to fabricate a high efficiency CdS/CdTe device, we need to optimise the  $\text{CdCl}_2$  concentration as well as the CdS thickness. However, there are reports saying that a thin layer of high resistive  $\text{SnO}_2$  layer in between the TCO and CdS can be used to support the junction. The solar cell structure TCO/ $\text{SnO}_2$ /CdS/CdTe/Metal is found to generate higher  $J_{\text{sc}}$  without drop in the  $V_{\text{oc}}$  and FF. Such a device with in situ  $\text{CdCl}_2$  treated CdTe can give an improved photovoltaic performance.

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