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12% efficient CdTe/CdS thin film solar cells deposited by low-temperature close space sublimation

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We report 12% efficient CdS/CdTe thin film solar cells prepared by low temperature close space sublimation (CSS). Both semiconductor films, CdS and CdTe, were deposited by high vacuum CSS in superstrate configuration on glass substrates with fluorine doped tin oxide (FTO) front contact. The CdTe deposition was carried out at a substrate temperature (T_{sub}) of $\leq 340^\circ\text{C}$, which is much lower than that used in conventional processes ($>500^\circ\text{C}$). The CdTe films were treated with the usual CdCl_2 activation process. Different optimal annealing times and temperatures were found for low-temperature cells ($T_{\text{sub}} \leq 340^\circ\text{C}$) compared to high-temperature cells ($T_{\text{sub}} = 520^\circ\text{C}$). The influence of the activation step on the morphology of high-temperature and low-temperature CdTe is determined by XRD, AFM, SEM top views, and SEM cross-sections. Grain growth, strong recrystallization, and a reduction of planar defects during the activation step are observed, especially for low-temperature CdTe. Further, the influence of CdS deposition parameters on the solar cell performance is investigated by using three different sets of parameters with different deposition rates and substrate temperatures for the CdS preparation. Efficiencies about 10.9% with a copper-free back contact and 12.0% with a copper-containing back contact were achieved using the low temperature CdTe process. © 2011 American Institute of Physics. [doi:10.1063/1.3639291]

I. INTRODUCTION

CdTe is one of the most promising absorber materials for thin film solar cells. Efficiencies as high as 16.7% have been reached.¹ The polycrystalline CdS and CdTe films can be prepared by a variety of different techniques, like sputter deposition,² chemical bath deposition (CBD),^{3,4} high vacuum evaporation,^{5,6} and close space sublimation (CSS).^{7,8} CSS is a very attractive method for large scale manufacturing, due to its high throughput. However, efficiencies of more than 10% could only be realized with CdTe substrate temperatures above 450°C for CSS.^{7,8} On the other hand, further improvements of the CdTe solar cells seem to be possible by changing the standard conditions of cell manufacturing.⁹ By reducing the deposition temperature, less input energy for solar cell production is needed. Furthermore, the end temperature can be reached in shorter time; thus, the heating and cooling lines can be shortened in production. An increased throughput and a cheaper production of solar cells are possible. In addition, the use of front contact materials with lower thermal stability, like aluminum-doped zinc oxide (AZO), becomes possible. Highly efficient CdTe thin film solar cells were already realized with low temperature methods, like sputter deposition² and high vacuum evaporation.^{5,10} However, these methods have low deposition rates

and, hence, a low throughput. Therefore, we investigated the preparation of highly efficient CdTe thin film solar cells with CSS at reduced substrate temperatures on fluorine-doped tin oxide substrates.

High efficiency CdS/CdTe solar cells have been obtained using copper or its compounds for the back contact.⁸ However, cells with a copper-containing back contact can degrade with time, due to the diffusion of copper toward the junction.¹¹ For this reason, we have investigated solar cells with and without copper for the back contact.

The most important production step for the improvement of CdTe thin film solar cells is the CdCl_2 activation. Grain growth,^{10,12} recrystallization,^{10,12,13} interdiffusion at the CdS/CdTe interface,^{12,14} and doping of the CdS and CdTe material¹⁵ during the activation step are reported. In the present work, the influence of the CdCl_2 activation on the CdTe morphology is investigated using X-ray diffraction (XRD), atomic force microscopy (AFM), and scanning electron microscopy (SEM) top views and cross-sections in dependency of the CdTe deposition temperature. Accordingly, different optimal activation parameters were found for CdTe deposited at 340°C and CdTe deposited at 520°C . The influence of CdS properties on the solar cell performance is reported for high-temperature CSS CdTe¹⁶ and for high vacuum-evaporated CdTe.¹⁷ Therefore, CdS films deposited with different sets of parameters were also tested for solar cells with low-temperature CSS CdTe.

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II. EXPERIMENTAL

A. Sample preparation

Both the CdS and CdTe films were deposited by CSS in superstrate configuration on commercial fluorine-doped tin oxide (FTO) substrates (Pilkington TEC15). The manufacturer states that the FTO film is covered by an additional undoped SnO₂ buffer layer. The substrates were cleaned sequentially with a commercial detergent solution, isopropyl alcohol, and de-ionized water in an ultrasonic bath. Both semiconductor films were deposited by CSS in the DArmstadt Integrated SYstem for SOLar cell research (DAISY-SOL). As shown before, CdS substrate temperature and deposition rate affect the morphology of the CdS film and the solar cell efficiency.¹⁶ Therefore, four different sets of parameters for the CdS deposition (Table I) were tested. The thickness of the CdS films was measured by white light interferometry. CdS film A2 is nearly similar to CdS film A1. The only differences are deposition time and film thickness. During pre-heating of the sample for the CdTe deposition, a re-evaporation of CdS can happen. The amount of re-evaporated CdS depends on the substrate temperature for the CdTe deposition. Therefore, the thinner A2 CdS film is used as substrate for CdTe films deposited at 340 °C and the thicker A1 CdS film for CdTe films deposited at 520 °C.

For all cells, an approximately 4-μm-thick CdTe layer was deposited by CSS with a substrate temperature of either ≤340 °C (low-temperature CdTe) or 520 °C (high-temperature CdTe) and a deposition time of 2 min 10 s. During the deposition of the low-temperature CdTe, the substrate temperature rises from 280 °C to 340 °C due to additional heating from the crucible. The CdTe source temperature was varied accordingly to the filling level of the crucible between 570 °C and 580 °C for the low-temperature CdTe films and between 590 °C and 600 °C for high-temperature CdTe films. For activation, the samples were covered with a saturated CdCl₂/methanol solution followed by an annealing step in air. Activation temperature and time were varied to find the optimal activation parameters. To form the primary Te back contact by selective etching, all cells were dipped into an aqueous solution of nitric (0.8% by weight) and phosphoric acid (70.0% by weight). The secondary back contact (~100 nm gold film) was deposited via sputtering. Finally, the back contact was separated from the front contact by mechanical scratching and the solar cells were divided into smaller cells, each of them having a size of 4 mm × 4 mm.

Additionally, some solar cells with a copper-containing back contact were prepared. For this, approximately 3 nm of copper was added by sputtering on the etched samples and

the gold deposition was followed by an annealing step at 150 °C for 40 min. IV curves were recorded under AM 1.5 G illumination and efficiency (η), fill factor (FF), short circuit current density (J_{sc}), and open circuit voltage (V_{oc}) were determined.

B. CdTe film characterization

To investigate how the activation step changes the morphology of the CdTe films deposited at low and high temperature, the samples were characterized by different methods before and after activation. For all samples, CdS film A (A2 for low-temperature CdTe samples and A1 for high-temperature CdTe samples) on FTO (TEC15) was used as substrate. Low-temperature CdTe was activated at 380 °C for 35 min and high-temperature CdTe was activated at the same temperature for 25 min. The surface morphology and roughness of the CdTe layer was studied by SEM and AFM. In addition, cross-sections of complete cells and non-activated samples were prepared at the Fraunhofer Institut for Electron Beam and Plasma Technology (FEP) in Dresden using a JEOL Cross-Section Polisher SM-09010. For imaging of the cross-sections, the channeling contrast caused by different crystallographic orientations and the atomic number contrast of backscattered electrons in a field emission scanning electron microscope (Hitachi SU 8000) was used. To probe changes in crystallographic properties, X-ray diffraction patterns of the CdTe films before and after activation were measured in θ : 2 θ -geometry in a Bruker D8 diffractometer. The intensities of the observed reflections (I_i) were quantified and texture coefficients (C_i) were calculated according to formula (1) by using relative intensities of a CdTe powder diffraction file¹⁸ (I_{0i}) and the number of considered reflections (N).

$$C_i = \frac{\frac{I_i}{I_{0i}}}{\left(\frac{1}{N}\right) \sum_{i=1}^N \frac{I_i}{I_{0i}}} \quad (1)$$

III. RESULTS AND DISCUSSION

A. Influence of CdCl₂ activation on CdTe morphology

We investigated the morphology of the CdTe films before and after activation by SEM, AFM, and XRD. Figure 1 shows the SEM micrographs of the CdTe film surfaces. For the CdTe deposition, two different substrate temperatures (≤340 °C and 520 °C) are compared. Smaller grain size and lower roughness are observed for CdTe films deposited with a low substrate temperature compared to samples deposited with a higher substrate temperature. Comparable results were published by Luschnitz *et al.*¹³ for low-temperature CSS deposition of CdTe using ITO substrates. In both cases, the activation step results in grain growth, but this is more pronounced for the smaller grains of the low-temperature CdTe. After activation, the grains are still smaller and the surface is still smoother for low-temperature CdTe than for high-temperature CdTe. The surfaces of both non-activated CdTe films are faceted;

TABLE I. Deposition parameters of the different used CdS films. CdS film A1 is used for high-temperature CdTe solar cells and the thinner CdS film A2 is used for low-temperature CdTe cells.

CdS film	T_{sub} [°C]	T_{source} [°C]	$t_{deposition}$	d [nm]
A1	470	710	20 s	~ 180
A2	470	710	15 s	~ 130
B	520	610	16 min 10 s	~ 200
C	380-420	630	2 min 10 s	~ 160

FIG. 1. SEM micrographs of the CdTe film surfaces. The CdTe is deposited at two different substrate temperatures, 340 °C (top) and 520 °C (bottom). For both, SEM measurements before (left) and after activation (right) are shown. The low-temperature CdTe films were activated at 380 °C for 35 min, the high-temperature CdTe film at 380 °C for 25 min.

during activation, the grain shape becomes more rounded in both cases. The roughness obtained from $10\ \mu\text{m} \times 10\ \mu\text{m}$ AFM images decreases from 64 nm to 58 nm in the case of CdTe deposited at 340 °C and from 156 nm to 109 nm in the case of CdTe deposited at 520 °C during activation.

In Fig. 2, SEM cross-sections of complete cells and non-activated CdTe samples are shown. The channelling contrast

of backscattered electrons (BSE) is used to differentiate between grains in different orientations and to visualize twin boundaries. For the non-activated CdTe films which were deposited at 340 °C, columnar grains with a lateral grain size of approximately $0.6\ \mu\text{m}$ are observed. At the grain boundaries, a high porosity is visible. The CdTe films which were deposited at 520 °C show large, more globular grains with a lateral average grain size of $1.4\ \mu\text{m}$. After activation, the low-

FIG. 2. SEM cross-sections in BSE channeling contrast of non-activated CdTe samples (left) and complete cells (right). The CdTe is deposited at two different substrate temperatures, 340 °C (top) and 520 °C (bottom). The solar cells were both activated at 380 °C; the low-temperature cell for 35 min, the high-temperature cell for 25 min.

temperature CdTe looks similar to the high-temperature CdTe, with large, globular grains. However, the surface is still smoother. The lateral mean grain size is increased to 1.8 μm . On average, the lateral grain size of the high-temperature CdTe after activation has the same value as measured for activated low-temperature CdTe. The grain shape of high-temperature CdTe does not change significantly with activation. The activated low-temperature CdTe no longer shows high porosity at the grain boundaries, but some pores inside the grains are visible. A very high density of planar defects, like twin boundaries, is observed for all samples. However, the highest density of these twins is found for as-deposited low-temperature CdTe. In contrast to high-temperature CdTe, the amount of planar defects seems to be reduced by CdCl_2 activation in the case of low-temperature CdTe. Loginov *et al.*¹⁹ similarly observed a reduction of stacking faults and dislocations in high-temperature CSS CdTe caused by the CdCl_2 activation step. However, Al-Jassim *et al.*²⁰ found an increased density of planar defects but decreased density of intra-grain dislocations in CSS CdTe using a higher activation temperature ($\geq 430^\circ\text{C}$).

In addition to the morphological investigations with AFM and SEM, the changes in crystallographic orientation were analyzed by XRD. For all samples, non-activated and activated, the cubic CdTe modification (space group F-43 m) was observed. The reflections 111, 220, 331, 400, 331, 422, 511/333, 440, and 531 were detected in all cases. However, the relative intensities of the reflections are different for the different samples. For as-deposited low-temperature CdTe, a strong preferential orientation in the $\langle 111 \rangle$ direction is determined. For better comparability of the preferential orientations, texture coefficients were calculated, comparing the measured intensities with relative intensities of a CdTe powder diffraction file.¹⁸ For this calculation, all observed peaks, except 511/333, were used, as it is not possible to distinguish between the 511 peak and the 333 peak. Figure 3 shows texture coefficients of all considered diffraction peaks

FIG. 3. Texture coefficients of low temperature CdTe films (triangles) and high temperature CdTe films (squares) in each case before (unfilled symbols) and after activation (filled symbols) for 8 different crystallographic orientations. For calculation of the texture coefficients, XRD measurements in 2:1 geometry and relative intensities of a powder diffraction file [Ref. 18] were used.

for activated and non-activated low-temperature and high-temperature CdTe.

Before activation, the low-temperature CdTe is strongly textured in the $\langle 111 \rangle$ direction. The high-temperature CdTe shows a preferential orientation in the $\langle 400 \rangle$ direction. For both samples, the texture is changed during activation. During activation, the strong $\langle 111 \rangle$ texture of low-temperature CdTe converts to a $\langle 400 \rangle$ texture. This texture is similar to the texture of non-activated high-temperature CdTe. For activated high-temperature CdTe, all texture coefficients are in the range of one; this is equivalent to a random crystal orientation of the grains, similar to a CdTe powder.

B. Solar cell characteristics

As shown above, different substrate temperatures lead to different CdTe morphologies. Therefore, it is expected to find different optimal activation parameters for low-temperature CdTe than for high-temperature CdTe. The optimal activation parameters for high-temperature CdTe are annealing at 380°C for 25 min. To find the best activation parameters for low-temperature CdTe, activation time and temperature were varied. For this optimization, only samples with CdS film A, working best for high-temperature solar cells,¹⁶ were used. Two annealing temperatures, 380°C and 400°C , were tested with different annealing times so far. Table II shows the solar cell characteristics for these cells. With an annealing temperature of 380°C and an annealing time of 35 min, the highest V_{oc} (0.8 V) was obtained. The best J_{sc} (22.1 mA/cm^2) and solar cell efficiency (10.8%) was reached for annealing at 400°C for 30 min.

To investigate the influence of the CdS deposition parameters on the solar cell characteristics, three different CdS films, listed in Table I, were used. For activation, all low-temperature CdTe samples were covered with CdCl_2 /methanol solution and annealed for 35 min at 380°C . IV curves and solar cell characteristics for the samples with the three different CdS films, compared to a high temperature CdTe cell activated at 380°C for 25 min, are shown in Fig. 4. We observed a higher V_{oc} , but lower J_{sc} by using a CdS film prepared with high deposition rate (CdS film A2) compared to the solar cell with a CdS film prepared with low deposition rate (CdS film B). This effect was already noticed for solar cells with high-temperature CdTe.¹⁶ The best efficiency (10.9%) was reached with CdS film C. During deposition of

TABLE II. Solar cell characteristics for different activation times and temperatures. In all solar cells, CdS film A2 (see TABLE I) and a copper-free back contact were used.

T_{sub} [$^\circ\text{C}$]	t [min]	η [%]	FF [%]	J_{sc} [mA/cm^2]	V_{oc} [V]
380	20	7.8	52.9	18.9	0.78
380	30	9.0	60.1	19.2	0.78
380	35	9.9	60.3	20.4	0.80
380	40	8.6	56.5	20.9	0.73
400	20	9.7	61.8	20.4	0.77
400	25	10.1	63.4	20.4	0.78
400	30	10.8	62.1	22.1	0.78

CdS film	CdTe T_{sub}	Efficiency [%]	FF [%]	J_{sc} [mA/cm²]	V_{oc} [V]
A2	340 °C	9.9	60.3	20.4	0.80
B	340 °C	10.3	59.7	21.9	0.78
C	340 °C	10.9	62.6	22.0	0.79
A1	520 °C	9.7	60.7	20.9	0.76

FIG. 4. IV curves under AM 1.5 illumination and solar cell characteristics of three low-temperature CdTe solar cells prepared with different sets of parameters for the CdS deposition (Table I) and one high-temperature CdTe cell prepared with CdS film A1. All solar cells have a copper-free back contact.

this film, the substrate temperature rises from 380 °C to 420 °C. This increase of sample temperature during deposition may lead to a more homogeneous and dense CdS film. Further investigations with varied activation parameters for samples with different CdS films are required.

Additionally, some solar cells with a copper-containing back contact were prepared. The copper was sputtered on the etched samples, and the whole solar cells were annealed at 150 °C for 40 min in air. The IV curve of a solar cell with low-temperature CdTe and a copper-containing back contact is shown in Fig. 5. This solar cell was activated at 390 °C for

FIG. 5. IV curve under AM 1.5G illumination and solar cell characteristics for the best solar cell with low-temperature CdTe and a copper-containing back contact. For the CdS deposition parameter set, A2 was used.

35 min. By using this copper-containing back contact, we reached a relatively high short circuit current density of $J_{sc} = 23.9$ mA/cm² and fill factor with $FF = 64.9\%$. The best-so-far obtained efficiency with low-temperature CdTe and a copper-containing back contact is 12.0%. This value, obtained with CSS, is comparable to efficiencies obtained by Perrenoud *et al.*⁵ using high vacuum evaporation for the CdTe deposition and the same Pilkington TEC15 substrate. By using CSS instead of high vacuum evaporation, an increased deposition rate is possible.

IV. SUMMARY AND CONCLUSION

It is shown that the CdCl₂ activation on CSS CdTe results in grain growth and recrystallization. Further, the concentration of planar defects and the porosity at grain boundaries in low-temperature CdTe is reduced by CdCl₂ activation. The surface of the low-temperature CdTe after activation is still smoother than the surface of high-temperature CdTe. This may enable the use of thinner CdTe films in low-temperature solar cells without risking pinholes and short circuits.

The optimal activation for high-temperature CdTe is annealing at 380 °C for 25 min. Optimized activation for low-temperature CdTe needs a higher annealing temperature of 400 °C and an annealing time of 30 min. Best efficiency (10.9%) with low-temperature CdTe, excluding copper in the back contact, is reached using CdS film C, with rising substrate temperature during CdS deposition. The CdS film preparation is found to be very important for solar cell performance.

The possibility to achieve high solar cell efficiencies (12.0%) with reduced CdTe deposition temperature is shown. The use of lower sample temperatures during CdS and CdTe deposition can reduce the production costs and enable the use of front contact materials with lower thermal stability, like aluminum-doped zinc oxide (AZO). Further investigations on AZO as alternative front contact material for CdTe thin film solar cells are planned.

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