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Cu-doped CdS and its application in CdTe thin film solar cell

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INTRODUCTION

CdTe thin film solar cell is based on a polycrystalline CdTe absorber which can efficiently absorb the solar irradiation photons with energy higher than the band gap.1 In a CdTe solar cell the CdTe film acts as the p-type semiconductor and CdS is generally employed as the n-type part for the cell diode. CdS is also called the window layer in the CdTe solar cell structure because of its relatively large band gap energy (∼ 2.42 eV).2 In the last several years the highest cell efficiency for small-area CdTe solar cell has been increased sharply from 16.5% to 21.5%.3 Also the highest efficiency of the large-area commercial modules fabricated by the First Solar Company was increased to 18.6%, approaching the efficiency of single-crystalline Si module.4 The Si-based solar cell is made of Si single or polycrystalline wafer with a thickness of ∼ 180 µm, while that for a thin film CdTe solar cell, the CdTe absorber layer thickness is only 2-6 µm.5,6 Due to both the enhanced solar cell efficiency and the much less material needed for CdTe thin film solar cell fabrication, CdTe solar cell is believed to be one of the promising candidates in the photovoltaic market. However, even though much progress in the CdTe solar cell fabrication has been achieved in the last decade, some critical issues still remain to be solved or understood. The critical issues regarding the fabrication of a high efficiency CdTe solar cell include efficient doping of the CdTe...
absorber, controllable CdS/CdTe heterojunction formation during the device fabrication process, reduction of pinhole density and shunting path formation at the CdS/CdTe junction, and fabrication of low resistive and stable back contact.\textsuperscript{7,8} Among these critical problems encountered in our laboratory, we found that the formation of a low resistive contact on the CdTe surface was one of the most critical issues for the fabrication of a high efficient CdTe thin film solar cell. This is due to the CdTe high work function with a value of 5.7 eV. A relatively high Schottky energy barrier, usually higher than ~ 0.4 eV, is formed at the CdTe/metal back contact interface.\textsuperscript{9,10} Theoretically this Schottky barrier can be efficiently lowered by forming a degenerated or high-carrier-concentration semiconductor thin layer on the CdTe surface. This is generally done by doping the CdTe surface using a thin layer of Cu.\textsuperscript{6,11} However, Cu is easy to diffuse from the back contact into the CdTe absorber layer and even into the CdS window layer.\textsuperscript{15} Cu in CdTe can exist as an interstitial as Cu\textsuperscript{i}, which is a shallow donor. Cu can also substitute Cd or occupy a Cd vacancy to form a deep acceptor state Cu\textsubscript{Cd}. Cu has been reported to have a diffusion coefficient of ~ 10\textsuperscript{-12} cm\textsuperscript{2}/s at room temperature, which is rather large. The long-term device stability experiments carried out at room and higher temperature in our laboratory demonstrated that the presence of Cu in a CdTe solar cell indeed was the main factor inducing cell performance deterioration.\textsuperscript{13,14} Cu\textsuperscript{i} was suggested to be mobile at room temperature, and this is consistent with the cell stressing tests carried out at room temperature in our laboratory.\textsuperscript{12–15} It was reported that Cu doping of CdTe absorber layer could lead to Cu diffusion to the CdTe/CdS interface.\textsuperscript{16} In a solar cell structure, accumulation of Cu near the CdS/CdTe junction interface would have profound influence on the cell performance. Recombination centers and electric shunting paths may be introduced near the junction area, which is the most essential part in a cell device. The effect of impurities in CdS on CdTe thin film solar cell device has been far less investigated.\textsuperscript{17} CdS window layer in a CdS/CdTe heterostructural solar cell is usually very thin, ~ 80–150 nm, and it acts as the n-type part for cell p-n junction. In such a thin layer, it is supposed that any defects in CdS would have large effect on the cell device performance. In this study we intentionally doped Cu into CdS to study its effect on the microstructural, the optical, and the electrical properties of the CdS material. CdTe solar cells employing Cu-doped CdS film as window layers were fabricated to investigate the effect of the impurity Cu, which located in CdS and near the CdS/CdTe junction, on the cell performance and device stability.

**EXPERIMENTAL SECTION**

In this study, CdS thin films were deposited on glass/SnO\textsubscript{2}:F(FTO) substrates by chemical bath deposition (CBD) technique. The aqueous solution was composed of cadmium acetate, ammonium acetate, thiourea and deionized water. A certain amount of ammonia was added to modify the solution to a pH value of 9.0. The Cu-doped CdS films were prepared by adding additional copper acetate to the aqueous solution. By controlling the concentrations of the copper acetate in the reaction solution (1 × 10\textsuperscript{-6}, 3 × 10\textsuperscript{-6} and 1 × 10\textsuperscript{-5} mol/L), CdS films with different Cu concentrations were fabricated. The atomic ratio of Cu/Cd in the Cu-doped CdS thin films were 0.5%, 1.5% and 5% respectively, which were measured by inductively coupled plasma-atomic emission spectroscopy (ICP-AES). The film deposition temperature was maintained at 85 °C and the film growth time was 30 minutes. The CdS precursor films were separated into two groups, simples of Group 1 were heat treated at 400 °C in the air atmosphere, and simples of Group 2 were dip coated with a saturated CdCl\textsubscript{2} – methanol solution followed by heat treatment at 400 °C in the air atmosphere.\textsuperscript{18} The CdTe solar cells fabricated in this study had a structure of glass/FTO/n-CdS/p-CdTe/Cu-Au. The CdTe layers were deposited on the glass/FTO/CdS templates in a homemade close-spaced sublimation (CSS) system.\textsuperscript{19} The thickness of the CdTe films was ~ 5 μm. The glass/FTO/CdS/CdTe templates were heat treated in a CdCl\textsubscript{2} atmosphere and then the CdTe surface was etched in a bromine-methanol solution.\textsuperscript{20} The Cu-Au back electrodes were fabricated by successive thermal evaporation of Cu and Au in a vacuum chamber.

The film morphological microstructures were characterized by a field emission scanning electron microscope (SEM, Sirion 200). The crystalline structures of the films were analyzed by X-ray diffraction (XRD, TTR-III) with a CuKα line as the radiation. The optical absorption/transmittance
spectra were measured by UV–Visible and near–Infrared spectrometry (U-4100). Photoluminescence (PL) measurements of CdS films were carried out by using a spectrofluorometer (FLUOROLOG-3-TAU) at room temperature. The solar cell current-voltage (J–V) curves were measured under the standard AM 1.5 illumination (1 kW/m², 25 °C) using a solar simulator (Oriel Sol 3A, USA).

EXPERIMENTAL RESULTS AND DISCUSSIONS

The X-ray diffraction spectra for the as-prepared and CdCl₂-annealed CdS films with Cu doping concentrations of 0.5, 1.5 and 5.0% are shown in Fig. 1. The peaks located at $2\theta = 25.1^\circ$ and $26.7^\circ$ correspond to the scattering from the hexagonal H(100) and cubic C(111) or H(002) atomic planes. The XRD scattering of the cubic C(111) and the hexagonal H(002) could not be identified due to the same lattice spacing for the two atomic planes in the cubic and the hexagonal phases. Therefore the peak at the angle $2\theta = 26.7^\circ$ is usually denoted as H(002)/C(111). It can be seen that with increased Cu doping concentration, the XRD intensity of the H(002)/C(111) peak of the as-prepared CdS films, compared to the undoped sample, was quickly decreased, see Fig. 1(a). The as-deposited films consisted of clusters of up to 200 nm, which were formed through gathering of nano-sized grains of about 30 nm. The peak widths at half maximum (FWHM) were also widened with increased Cu doping concentration. The FWHM values are 0.25, 0.28, 0.32 and 0.38 degree for the undoped, 0.5, 1.5, and 5.0% Cu-doped films, respectively. This was caused by the deteriorated crystalline structure, shown in Fig. 3, and this will be discussed in the following paragraphs. With increased Cu doping concentration, the peak H(002)/C(111) position was gradually increased from $26.70^\circ$ to $26.86^\circ$, see Figs. 1(a), 1(c) and 2(a). The atom size of Cu is smaller than that of Cd atom, the increased $2\theta$ values indicated that Cu, at least part of the nominally doped Cu, substituted the Cd atom in the CdS lattice. The nano-crystalline grain sizes of the as-prepared films were estimated

![Image of X-ray diffraction spectra](image-url)

**FIG. 1.** X-ray diffraction spectra for (a) the as-prepared and (b) the CdCl₂-annealed CdS films with Cu doping concentrations of 0.5, 1.5 and 5.0%; (c) and (d) are the XRD spectra in enlarged scale near the scattering peak for (a) and (b), respectively.
FIG. 2. (a) The peak H(002)/C(111) positions and (b) the nano-crystalline sizes of the as-prepared films doped with different Cu concentrations.

by using the Scherrer formula and are shown in Fig. 2(b). It can be seen that the nano-crystalline size was decreased from 31.5 nm for the undoped CdS to a value of 27.4 nm for the 5% Cu doped CdS film. After the CdCl₂-annealing, the characteristics hexagonal H(103) peak was observed for all the samples, see Fig. 1(b), indicating that the nano-crystalline grain coalescence occurred and the crystalline quality was improved. Fig. 1(c) and 1(d) show the XRD spectra in enlarged scale to show the spectrum details near the scattering peaks. After the CdCl₂-annealing for the 5% Cu doped film a small peak corresponding to the Cu₂S (102) was observed, indicating that at this relatively high doping level, only part of the nominally doped Cu was incorporated into the CdS lattice, and the rest reacted with sulfur to form Cu₂S. From the XRD data we calculated the Cu concentration incorporated in the Cd₁₋ₓCuₓS. The nominal Cu doping concentrations of 0.5, 1.5, and 5% corresponded to real Cu concentrations in CdS of 0.45, 1.2 and 2%, respectively.

The doping of Cu in the CdS precursor films dramatically influenced the recrystallization during the annealing process. The SEM images of the undoped, 0.5 and 5% doped precursor films and that after the CdCl₂-annealing are shown in Fig. 3. Fig. 3(a), 3(c), and 3(e) show the surface microstructures of the precursor layers. The morphologies are similar for the three samples with nanocrystalline grain size of ∼30 nm. After the CdCl₂-annealing treatment, the undoped film showed well crystallized grains with grain size of 50–150 nm, while that for the 0.5 and 5% doped samples, the grains showed much smaller size of only 30–50 nm. This clearly demonstrates that the doping of Cu hindered the coalescence of the nanocrystalline CdS gains during the CdCl₂ annealing process.

The observed SEM microstructures are consistent to the XRD data discussed above. Cu substituted the Cd to form CuCd or existed as an interstitial in CdS, and the rest undoped Cu formed Cu₂S with sulfur. These point defects and the formation of Cu₂S hindered the coalescence of the nanocrystalline CdS gains during the CdCl₂ annealing process.

The possible lattice position of the doped Cu atoms can be detected or back tracked by using the photoluminescence measurements (PL). Impurity Cu can either substitute Cd to form a single acceptor state, or situate as an interstitial Cuᵢ in CdS generating a single donor state. The PL spectrum of a material depends critically on the defect types and their complex configurations. In order to have a good baseline for the analysis of the measured PL spectra to be discussed in the following paragraphs, we first characterized the PL spectrum of a CdCl₂-annealed undoped CdS film, which is shown in Fig. 4. In our lab we have developed a CdCl₂-annealing technology for CdS precursor films, which enables us to obtain CdS thin films having well-defined crystalline facets as shown in Fig. 3(b). Employing CdS films with high crystalline quality, we could easily obtain PL spectrum with two well recognized peaks at room temperature, one is a defect-related emission and the other is the band-edge emission around 2.35 eV, as shown in Fig. 4(a). The observation of a relatively strong band-edge peak at room temperature clearly demonstrated that the CdCl₂-annealed undoped CdS film in this study has a high crystalline quality. The broad defect-related band located around 1.5–2.0 eV is ascribed to the emission between the energy levels of the defects, such as, Cd vacancy (V Cd), surface states, Cl at the lattice of S (Cl S), S vacancy (V S), interstitial Cd (Cdi) and the the valence band state (VB), impurities and acceptor. The detailed peak components, which
are ascribed to the specific emissions, are shown in Fig. 4(b). In CdS the sulfur vacancy $V_S$ has a relatively low formation energy, therefore it is expected that the concentration of $V_S$, whether it is interior in a CdS grain or at the grain surface, is relatively high. This is manifested by the intense peak at 1.76 eV, which is ascribed to the emission involved the defect complex Cl$_2$-$V_S$. The peaks at 1.52, 1.57 and 1.64 eV are related to the emission between Cd vacancy $V_{Cd}$ and impurity levels or between surface states to the valence band.$^{24}$
Upon Cu doping we found that the PL intensity of the defect-related emission was dramatically decreased or quenched, both for the as-deposited, the air- and the CdCl$_2$-annealed samples, as shown in Fig. 5. In order to study the effect of Cu doping on the variation of defect and/or defect configuration formation, we measured the PL spectra for the CdS films with three Cu doping concentrations, namely, 0.1, 0.3, and 0.5%. It was found that the Cu doping had relatively large influence on the defect-related emissions with energy between 1.5 and 1.7 eV, as shown in Fig. 6(a). With increased Cu doping, the $V_{\text{Cd}}$-related emission at 1.53 eV was dramatically decreased. The peak at 1.53 eV came from a donor-acceptor pair (DAP) transition, which was associated with Cd vacancies. This observation demonstrates that the doped Cu had formed Cu$^+$ – $V_{\text{Cd}}$ complexes and/or Cu$_{\text{Cd}}$ states, indicating that the doped Cu, or at least part of the doped Cu in CdS, had taken the lattice positions of Cd vacancies.\textsuperscript{16} The decreased $V_{\text{Cd}}$-related emission intensity with increased Cu doping, indicated by the arrow in Fig. 6(a), showed that during the CBD deposition Cu atoms/ions adopted the $V_{\text{Cd}}$ lattice sites. The higher Cu doping concentrations led to lower $V_{\text{Cd}}$-related emission intensity, see Fig. 6(a) and 6(b). From Fig. 6(a) we can also see that the peak widths of the band-edge-related emission were slightly enlarged with increased Cu doping, indicating an increased crystalline lattice distortion in the CdS:Cu films. It is noted that with increased Cu doping, the band-edge emission shifted from 2.35 to 2.39 eV for the samples doped with a Cu concentration of 0.3 and 0.5%, respectively. The band gap increase after Cu doping could be attributed to the decreased CdS crystalline size, see the SEM images of Fig. 3, which induced quantum confinement effect. The experimental results observed in this study are consistent to the reported data in the publications.\textsuperscript{27,28} The formation of Cu$_2$S and/or Cu-S bonding increased the density of crystalline nucleation during the CdS deposition leading to decreased CdS nano-grain size.

Cu in CdS usually has two possible lattice positions, one is a donor state as an interstitial Cu$_i$, and the other is an acceptor substituting Cd as Cu$_{\text{Cd}}$. The formation energy of Cu$_{\text{Cd}}$ is lower than that of Cu$_i$. The radii of Cu$^+$ is 0.96 Å and that of Cd$^{2+}$ is 0.97 Å, these two values are very close, therefore, Cu$_{\text{Cd}}$ is more easily to be formed in CdS than Cu$_i$.\textsuperscript{23,29,30} The Cu$_{\text{Cd}}$ acceptor level is about 0.6 eV above the valence band maximum. For the air-annealed CdS films, the much decreased peak

![Image](image1.png)

**FIG. 5.** Comparative PL spectra of (a) an as-prepared CdS, (b) the air-annealed CdS, and (c) the CdCl$_2$-annealed CdS films, with that of the corresponding 0.5%-Cu-doped films.

![Image](image2.png)

**FIG. 6.** PL spectra for (a) the as-deposited, and (b) the CdCl$_2$-annealed CdS films with different Cu-doping concentrations.
intensity of the defect-related emission was mainly induced by two factors, namely, the formation of CuCd defects, which were formed by Cu taking up the VCd lattice site leading to decreased VCd defect concentration, and the oxygen incorporation into CdS forming O$_3$ by oxygen taking over the sulfur vacancy V$_{S}$.$^{22,31}$

For the three CdS samples doped with different Cu doping concentrations, after the CdCl$_2$-annealing, see Fig. 6(b), the V$_{Cd}$-related defect emission intensity for the 0.1%-Cu-doped sample was much larger than that of the 0.3% and 0.5%-Cu-doped samples. This was due to the better crystalline quality for the 0.1%-Cu-doped sample compared to the other two samples. The band edge emission peak for the 0.1%-Cu-doped sample shifted to a lower energy, because the CdCl$_2$-annealing promoted the nano-sized grains to coalesce, producing larger grains compared to the as-prepared and the higher concentration Cu-doped samples, see Fig. 3. The increased band gap energy upon Cu doping evidenced in the PL spectra was directly confirmed by the light absorption measurement, shown in Fig. 7. The optical absorption coefficients $\alpha$ were derived from the optical transmission and reflection data. The absorption edge corresponding to the band gap energy was determined by extrapolation of the linear portion of $\alpha^2$ versus $h\nu$ plots shown in Fig. 7. It can be seen that with increased Cu-doping concentration, the band gap energy was increased.

For a thin film CdTe solar cell, the Cu material employed in the back contact fabrication may diffuse into CdTe and even to the window layer CdS.$^{32}$ The presence of Cu in CdS and at the CdS/CdTe junction would have profound influence on solar cell performance and device stability. In order to study the effect of the impurity Cu in the window layer CdS on a CdTe solar cell, we fabricated CdTe solar cells with CdS films doped with different Cu-doping concentrations as discussed above. The CdTe solar cells had a structure of glass/FTO/Cd$_x$S$_{1-x}$Cu/CdTe/Cu-Au. The current–voltage J–V curves measured under the standard AM 1.5 illumination are shown in Fig. 8(a). All the solar cells were fabricated with the same processing procedures except that the CdS layers were doped with different Cu concentrations. It can be seen that with increased Cu doping, the cell performance deteriorated dramatically. The values of the open-circuit voltage ($V_{oc}$) and the fill factor (FF) were decreased rather quickly with increased Cu doping concentrations, shown in Fig. 8(b) and 8(c). This was induced by the increased space-charge recombination near the p-n junction and the worsened junction crystalline quality, where the impurity Cu acted as an efficient recombination center. The variation of the short-circuit currents was not as sensitive to the Cu doping as that of the $V_{oc}$ and the FF. With increased Cu doping concentrations the series and shunt resistances increased and decreased, respectively, leading to much decreased fill factor. The shape of the J–V curves developed roll-over phenomenon at high voltage near the open-circuit voltage with increased Cu doping. This is attributed to an additional front energy barrier generated by the presence of Cu in CdS.$^{33}$ The deteriorated cell performance was induced by both the impurity Cu in CdS and the relatively bad CdS/CdTe junction quality, as shown in Fig. 9. It was reported that the presence of Cu in CdS:Cu created trapping levels and decreased the lifetime of the photocarriers.$^{34}$

![FIG. 7. Film absorption measurements, $(\alpha h\nu)^{1/2}$ versus photon energy $h\nu$, of CdS films with different Cu-doping concentrations.](image)
FIG. 8. (a) Current-voltage curves of CdTe solar cells prepared with CdS window layers with different Cu doping concentrations; (b) the $V_{oc}$ and the fill factor versus the Cu concentrations in CdS; (c) the efficiency versus the Cu concentrations in CdS; (d) series and shunt resistance versus the Cu concentrations in CdS.

FIG. 9. SEM cross-sectional microstructure of the CdS/CdTe interfaces of two solar cells (a) without and (b) with Cu doping.
FIG. 10. Current-voltage curves before and after the J–V–T measurements for the CdTe solar cell fabricated with a Cu-doped CdS window layer.

As shown in Fig. 3, with increased Cu doping in CdS, the crystalline quality of CdS after the CdCl$_2$ annealing became worse compared to the CdS film without Cu doping. This led to the formation of much low-quality p-n junctions, as shown in Fig. 9(b). The CdS/CdTe junction with a CdS window layer without Cu doping showed a densely packed uniform interface, which had almost no pinholes at the interface, see Fig. 9(a), while for that fabricated with a CdS:Cu layer, pinholes with a high density were observed. The presence of pinholes hindered the transport of photo-generated carriers and also decreased the intensity of the electric field at the interface, which was disadvantageous for the carrier separation and transportation. As discussed above, the doped Cu mainly formed an acceptor Cd$_{Cu}$, which trapped electrons and thus hindered the electron transport in CdS and near the CdS/CdTe interface. The formation of Cd$_{Cu}$ and pinholes at the p-n junction effectively presented an energy barrier for the carrier transport leading to the roll-over phenomenon at high voltage near the open-circuit voltage.

We found that one of the most important facts for Cu impurity in CdS was that Cu atom/ion was not stable in CdS, and it became mobile at high temperature. We did temperature dependent current–voltage curve measurements (J–V–T) from room temperature to 200 °C, shown in Fig. 10. It can be seen that when the device was heated to 200 °C, both the short-circuit current and the fill factor were improved. This was induced by the Cu diffusion from CdS into the p-type CdTe absorber to form acceptor Cd$_{Cu}$, which decreased the electric resistance of the CdTe, thus increased the short-circuit current and the fill factor. It was interesting to find that after the J–V–T measurement and after the cell had been stored in air at room temperature for 40 days, the device J–V characteristics was almost fully recovered, indicating that the Cu, which was diffused into the CdTe, diffused back into the CdS window layer and the CdS/CdTe interface. We suggested that the electric field presented at the CdS/CdTe p-n junction enhanced the Cu back diffusion.

CONCLUSIONS

In this study Cu was intentionally doped into CdS to study its effect on the microstructural, the optical and the electrical properties of the CdS films. The photoluminescence intensity of the Cu-doped CdS films was dramatically decreased or quenched, both for the as-deposited, the air- and the CdCl$_2$-annealed films. The intensities of the V$_{Cd}$ and the surface-state-related PL emissions were decreased, this was ascribed to Cu occupancy at the Cd lattice vacancy as V$_{Cd}$. The Cu doping hindered the recrystallization/coalescence of the nano-sized grains in the as-deposited CdS films during the air/CdCl$_2$ annealing. CdTe thin film solar cell fabricated with Cu-doped CdS window layers showed much decreased fill factor and open-circuit voltage, which were induced by the increased space-charge recombination near the p-n junction and the worsened junction crystalline quality. Temperature dependent current–voltage curve measurement indicated that the doped Cu in
the CdS window layer was not stable, and it became mobile at high temperature. This confirms that the presence of Cu is indeed a vital factor inducing CdTe solar cell device instability.

ACKNOWLEDGMENTS

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