

Research Article

Effects of Hydrogen Plasma on the Electrical Properties of F-Doped ZnO Thin Films and p-i-n α -Si:H Thin Film Solar Cells

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1.5 wt% zinc fluoride (ZnF_2) was mixed with zinc oxide powder to form the F-doped ZnO (FZO) composition. At first, the FZO thin films were deposited at room temperature and 5×10^{-3} Torr in pure Ar under different deposition power. Hall measurements of the as-deposited FZO thin films were investigated, and then the electrical properties were used to find the deposition power causing the FZO thin films with minimum resistance. The FZO thin films with minimum resistance were further treated by H_2 plasma and then found their variations in the electrical properties by Hall measurements. Hydrochloric (HCl) acid solutions with different concentrations (0.1%, 0.2%, and 0.5%) were used to etch the surfaces of the FZO thin films. Finally, the as-deposited, HCl-etched as-deposited, and HCl-etched H_2 -plasma-treated FZO thin films were used as transparent electrodes to fabricate the p-i-n α -Si:H thin film solar cells and their characteristics were compared in this study. We would show that using H_2 -plasma-treated and HCl-etched FZO thin films as transparent electrodes would improve the efficiency of the fabricated thin film solar cells.

1. Introduction

ZnO is a nontoxic, abundant, and inexpensive material; in addition, impurity-doped ZnO can have electrical and optical properties comparable to the expensive indium tin oxide (ITO) currently used commercially. For that, impurity-doped ZnO is a possible candidate for transparent conducting oxides (TCOs) for potential applications as a transparent electrode in flat panel displays (FPDs) and photovoltaic solar cells [1]. TCO thin films have been widely used in the applications of solar cells, flat panel displays, and more optoelectronic products. TCO thin films show a good combination of electrical conductivity at ambient temperature and optical transparency in a visible region [2]. Some especially interesting properties of ZnO-based thin films are their low cost, ready availability, and high chemical stability. ZnO is a II-VI n-type semiconductor with a wide band gap of approximately 3.3 eV at room temperature. Group III donor elements, such as B [3], Al [4], and Ga [5], are added to

improve the electrical properties of ZnO thin films. Fluorine, the ionic radius (0.136 nm) of which is similar to that of oxygen (0.132 nm), may be an adequate anion doping candidate due to lower lattice distortion compared with Al, Ga, and In, but comparatively few studies on fluorine-doped ZnO (F-doped ZnO) can be found in the past researches [6, 7].

In this study, 1.5 wt% zinc fluoride (ZnF_2) was mixed with zinc oxide powder to fabricate the F-doped ZnO (FZO) ceramic target for sputtering process [8]. In the past, Al-doped ZnO (AZO) thin films were deposited on glass substrate by RF magnetron sputtering by changing the substrate temperature from room temperature (RT) to 300°C [4]. From this literature, we would know that hydrogen (H_2) has large effect on the ZnO-based TCO thin films. When H_2 was used as deposition atmosphere during the deposition process [4] or used as plasma treatment [9, 10] after the deposition process, the ZnO-based TCO thin films will enhance their carrier concentration and improve their resistivity. In the

past, we also found that the H_2 -plasma treatment on the deposited AZO thin films has large influence on their physical (transparency ratio and grain size) and electrical (energy gap and resistivity) properties [11]. For that, the plasma enhanced chemical vapor deposition (PECVD) H_2 was treated on the as-deposited FZO thin films (abbreviated as plasma-treated FZO thin films). The effects of H_2 plasma on the properties of FZO thin films were compared by observing the carrier concentration, carrier mobility, and resistivity. The FZO thin films under differently treated parameters were also used as the transparent electrodes of the α -Si thin film solar cells. The current-voltage characteristics of the fabricated α -Si thin film solar cells were measured to determine the effects of treated parameters of the FZO thin films on the characteristics of the fabricated α -Si solar cells.

2. Experimental

1.5 wt% zinc fluoride (ZnF_2) (99.995%) was mixed with zinc oxide powder (99.999%) to form the F-doped ZnO (abbreviated as FZO) composition. After being dried and ground, the FZO powder was calcined at $600^\circ C$ for 1 h, then ground again, and mixed with polyvinyl alcohol (PVA) as binder. The mixed powders were uniaxially pressed into pellets of 5 mm thickness and 54 mm diameter using a steel die. After debinding, the FZO pellet was sintered at $1060^\circ C$ for 3 h. Glass substrates (Corning 1737) with an area of $3.3 \times 3.3 \text{ cm}^2$ were cleaned ultrasonically with isopropyl alcohol (IPA) and deionized (DI) water and then dried under a blown nitrogen gas. The base pressure of the sputtering chamber was below 5×10^{-6} Torr and the working pressure was maintained at 5×10^{-3} Torr in pure Ar (99.995%) ambient (abbreviated as-deposited FZO thin films). FZO thin films with a thickness of about $650 \text{ nm} \pm 10\%$ were deposited by RF magnetron sputtering on glass substrates at room temperature (RT) by controlling deposition time to find the deposition parameters at which FZO thin films had optimally electrical characteristics. At the first, the FZO thin films were deposited by changing RF power from 50 W to 150 W at RT. Thicknesses of the FZO thin films were measured using a SEMF-10 ellipsometer and confirmed by field emission scanning electron microscopy (FESEM). The crystalline structure of the FZO thin films was identified by X-ray diffraction (XRD), while the thin films' Hall-effect coefficients were determined by a Bio-Rad Hall set-up. The optical transmission spectrum was recorded using a Hitachi U-3300 UV-Vis spectrophotometer in the 300–800 nm wavelength range. The as-deposited FZO thin films with optimally electrical characteristics were treated by the plasma enhanced chemical vapor deposition (PECVD) hydrogen (H_2) (abbreviated as plasma-treated FZO thin films). The working pressure was maintained at 1 Torr under the 300 sccm H_2 flow rate. The plasma power was changed from 0 W (without H_2 plasma treatment) to 100 W, and the plasma-treated temperature and time were $200^\circ C$ and 60 min.

After that, the thicknesses of the as-deposited and plasma-treated FZO thin films were extended to 1000 nm $\pm 10\%$, and then their surfaces were etched by wet etching performed in diluted HCl solution with concentrations of 0.1%, 0.2%, and 0.5% in H_2O to acquire the textured FZO thin

films. The thickness of the etched FZO thin films was around 650 nm, which was obtained by controlling the etched time. Also, the surface texture of the etched FZO thin films was observed by FESEM. The etched as-deposited and plasma-treated FZO thin films were used as substrates to fabricate the superstrate p-i-n α -Si:H thin film solar cells. Thin film solar cells were fabricated using a single-chamber plasma-enhanced chemical vapor deposition unit at $200^\circ C$, with a working pressure of 700×10^{-3} Torr and a deposition power of 20 W. The p-type α -Si thin films (thickness was about 20 nm) were deposited by controlling the gas flowing rates for $H_2 = 100 \text{ sccm}$, $SiH_4 = 20 \text{ sccm}$, $CH_4 = 10 \text{ sccm}$, and $B_2H_6 = 40 \text{ sccm}$. The i-type α -Si thin films (400 nm) were deposited by using $H_2 = 100 \text{ sccm}$ and $SiH_4 = 10 \text{ sccm}$, and the p-type α -Si (50 nm) thin films were deposited by using $H_2 = 100 \text{ sccm}$, $SiH_4 = 20 \text{ sccm}$, and $PH_3 = 20 \text{ sccm}$, respectively. The current-voltage characteristic of the fabricated solar cells was measured under an illumination intensity of 300 mW/cm^2 and an AM 1.5 G spectrum.

3. Results and Discussion

An increase in orientation during crystalline growth is possible according to the model of Van der Drift, which is often referred to as "survival of the fastest" model. Even the grain sizes are not really formed, this model is based on different growth rates of the crystal planes. At first, the thin films start growing in random crystal orientation at the initial layer, they grow fastest in the plane parallel to the substrate and then grow at the cost of the others [12]. This "survival of the fastest" model implies a related increase in crystal orientation, which is called figure of merit in a growth of thin films, but the model cannot explain an increase in grain sizes with increasing thin films' thickness [13]. Figure 1 shows that even different deposition powers were used, all FZO thin films exhibited a strong (002) peak at $2\theta = 34.24^\circ$ and a weak (004) peak, which indicate that the c-axis is predominantly oriented parallel to the substrate normal. The results in Figure 1 prove that the deposited FZO thin films have the figure of merit in c-axis orientation. The absence of additional peaks in the XRD patterns excludes the possibility of any extra phases and/or large-size precipitates in the FZO thin films. The diffraction intensity of the (002) peak increased as the deposition power increased from 50 W to 150 W.

The results of the carrier mobility, carrier concentration, and resistivity shown Figure 2 indicate that the electrical properties of the FZO thin films were dependent on deposition power. When plasma molecules are deposited on a glass substrate, many defects result and inhibit electron movement. As different deposition powers are used during the deposition process, two factors are believed to cause an increase in the carrier mobility of the FZO thin films. First, higher deposition power provides more energy and thus enhances the motion of plasma molecules, which will improve the crystallization and grain size growth of the FZO thin films, and also the defects in the thin films will be decreased. Second, as deposition power is raised, the defects in the FZO thin films decrease, and that will cause the decrease in the inhibiting of the barriers electron transportation

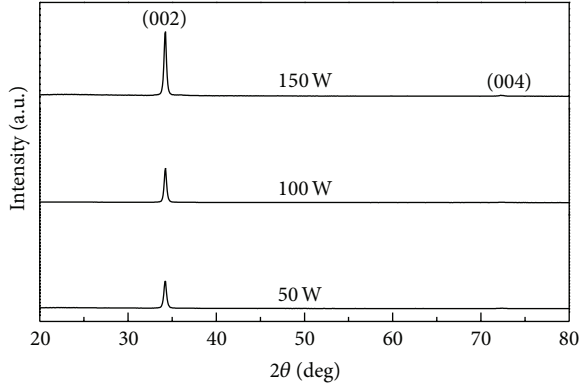


FIGURE 1: X-ray diffraction (XRD) patterns of the FZO thin films as a function of deposition power.

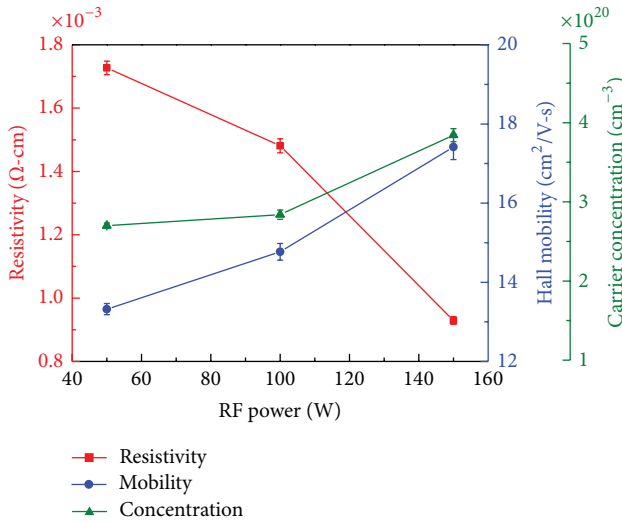


FIGURE 2: Carrier mobility, carrier concentration, and resistivity of the FZO thin films as a function of deposition power.

[14]. The results in Figure 1 suggest that this condition will dominate the characteristics of the deposited FZO thin films. As Figure 2 shows, both the carrier concentration and carrier mobility of the FZO thin films linearly increased with deposition power and reached the maximum concentration and carrier mobility at 150 W. The resistivity of the TCO thin films is proportional to the reciprocal of the product of carrier concentration N and mobility μ :

$$\rho = \frac{1}{Ne\mu}. \quad (1)$$

Both the carrier concentration and the carrier mobility contribute to the conductivity. The resistivity decreased from $1.72 \times 10^{-3} \Omega\text{-cm}$ to $9.29 \times 10^{-4} \Omega\text{-cm}$ as the deposition power increased from 50 W to 150 W. The minimum resistivity of the FZO thin films at deposition power of 150 W is mainly caused by the carrier concentration and mobility being at their maximum.

Because of the maximum mobility and carrier concentration and the minimum resistivity, the 150 W-deposited FZO thin films are used for further treatment by H_2 plasma.

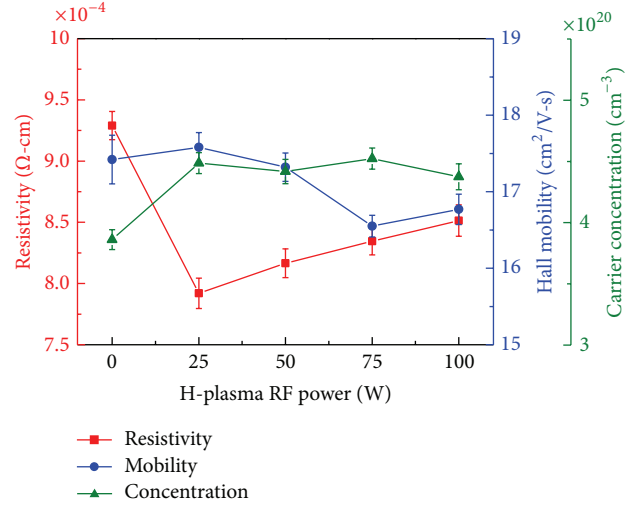


FIGURE 3: Carrier mobility, carrier concentration, and resistivity of the FZO thin films as a function of H_2 -plasma-treated power.

Figure 3 shows the carrier mobility, carrier concentration, and resistivity of the FZO thin films as a function of H_2 plasma power. The mobility slightly increased as plasma power of H_2 increased from 0 W (without H_2 plasma treatment) to 25 W and then it decreased as plasma power was further increased. The carrier concentration first increased as plasma power of H_2 increased from 0 W (without H_2 plasma) to 25 W and then it reached a saturation value as plasma power was further increased. However, resistivity is really improved as plasma process is used because the carrier concentration is increased as H_2 plasma is used. 25 W-plasma-treated FZO thin films have the minimum resistivity of $7.92 \times 10^{-4} \Omega\text{-cm}$ because the mobility and carrier concentration have their maximum. As the plasma power is higher than 25 W, the mobility of the FZO thin films decreases because their surfaces are destroyed by H_2 plasma.

Rough interfaces are usually introduced into solar cells by using substrates with textured surface [15]. It is well known that the front TCO thin films with textured surface can scatter more light at the TCO/ α -Si:H(p) interface and haze ratio is strongly influenced by the surface morphologies of TCO thin films or/and glass substrates. Therefore, the surface structures of glass substrate and TCO thin films play a vital role to improve the performance of thin film solar cells. A suitable textured surface is very important to increase the haze ratio and scatter an incident light, particularly the long wavelength light (red and near-infrared), to extend the effective path length within the active silicon layer and subsequent light trapping inside the absorber material of the solar cell [15]. In this study, diluted HCl etching of the as-deposited and plasma-treated FZO thin films has been carried out at room temperature to develop the textured surfaces for enhancing the efficiency of solar cells. FZO thin films deposited at 150 W, room temperature, and 5×10^{-3} Torr with thickness of 1000 nm were used as the etching samples. Figure 4 shows the etching rate of the FZO thin films as a function of concentration of HCl solution; the etched time was dependent on the concentration of HCl solution. As the

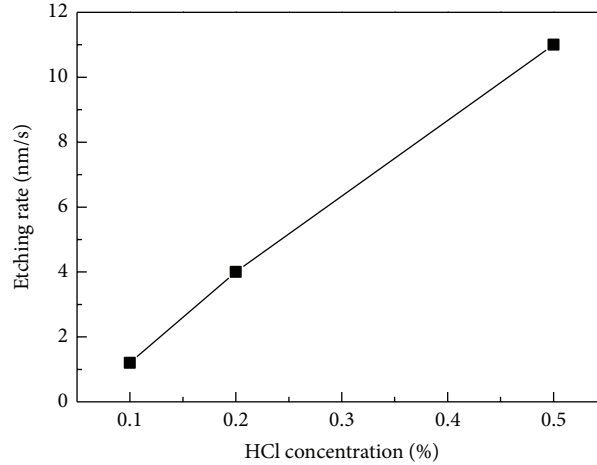


FIGURE 4: Etching rate of the FZO thin films as a function of HCl solution concentration.

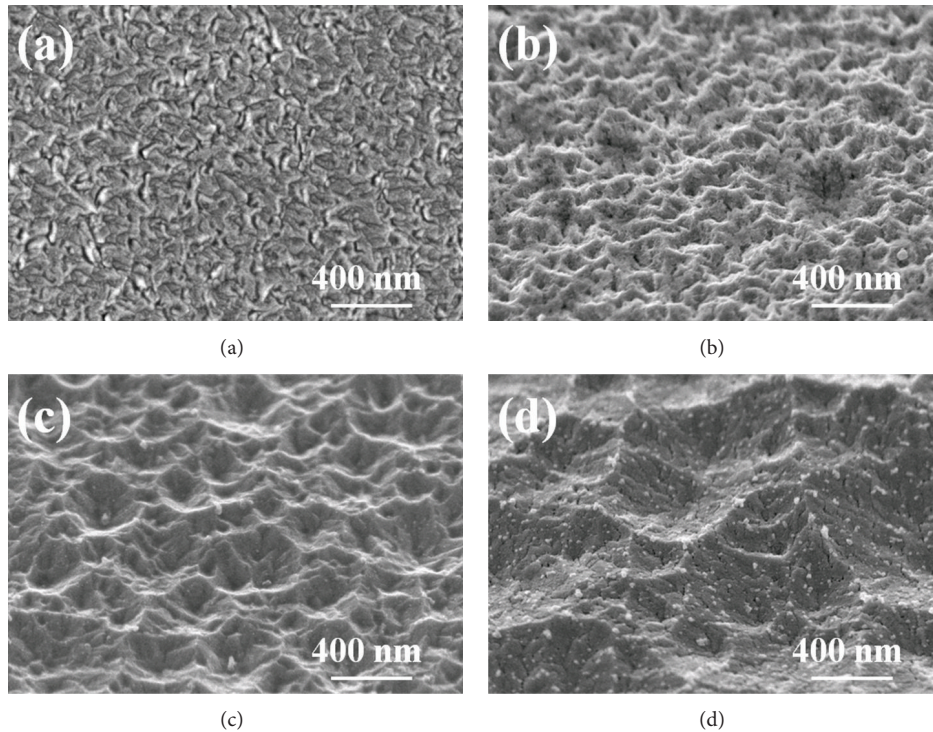


FIGURE 5: Surface morphology of the FZO thin films (a) as-deposited and etched at different HCl concentrations (b) 0.1%, (c) 0.2%, and (d) 0.5%, respectively.

concentration of HCl solution increased from 0.1% to 0.5%, the etched rate linearly increased from 1.2 nm/s to 11 nm/s, and the thickness of the etched FZO thin films was controlled at around 650 nm.

Figure 5 shows the surface morphology of the etched FZO thin films, and also the etched time was dependent on the concentration of HCl solution. Compared with the results shown in Figure 5, the surface roughness increased significantly after HCl etching. The etching process causes the thin films' surfaces to develop a crater-like cave and the size of cave increases with increasing HCl concentration. Figures 6(a) and 6(b) depict the total and diffused transmittance of the textured FZO thin films for various concentrations of

HCl solution. The average total transmittance of as-deposited FZO thin film substrates was 82.8%, whereas the textured FZO thin films showed that the average total transmittance decreased with increasing concentration of HCl solution in the visible (400~800 nm) wavelength region. The average total transmittances of 81.1%, 79.2%, and 78.8% were shown by the textured FZO thin films with various concentrations of HCl solution, including 0.1%, 0.2%, and 0.5%, respectively. In general, the diffused transmittance is related to the surface structure and roughness of the textured FZO thin films. The average diffused transmittance of the as-deposited FZO thin films was around 1.7%, whereas it varied from 0.7% to 4.0% in the visible wavelength region. As the concentrations

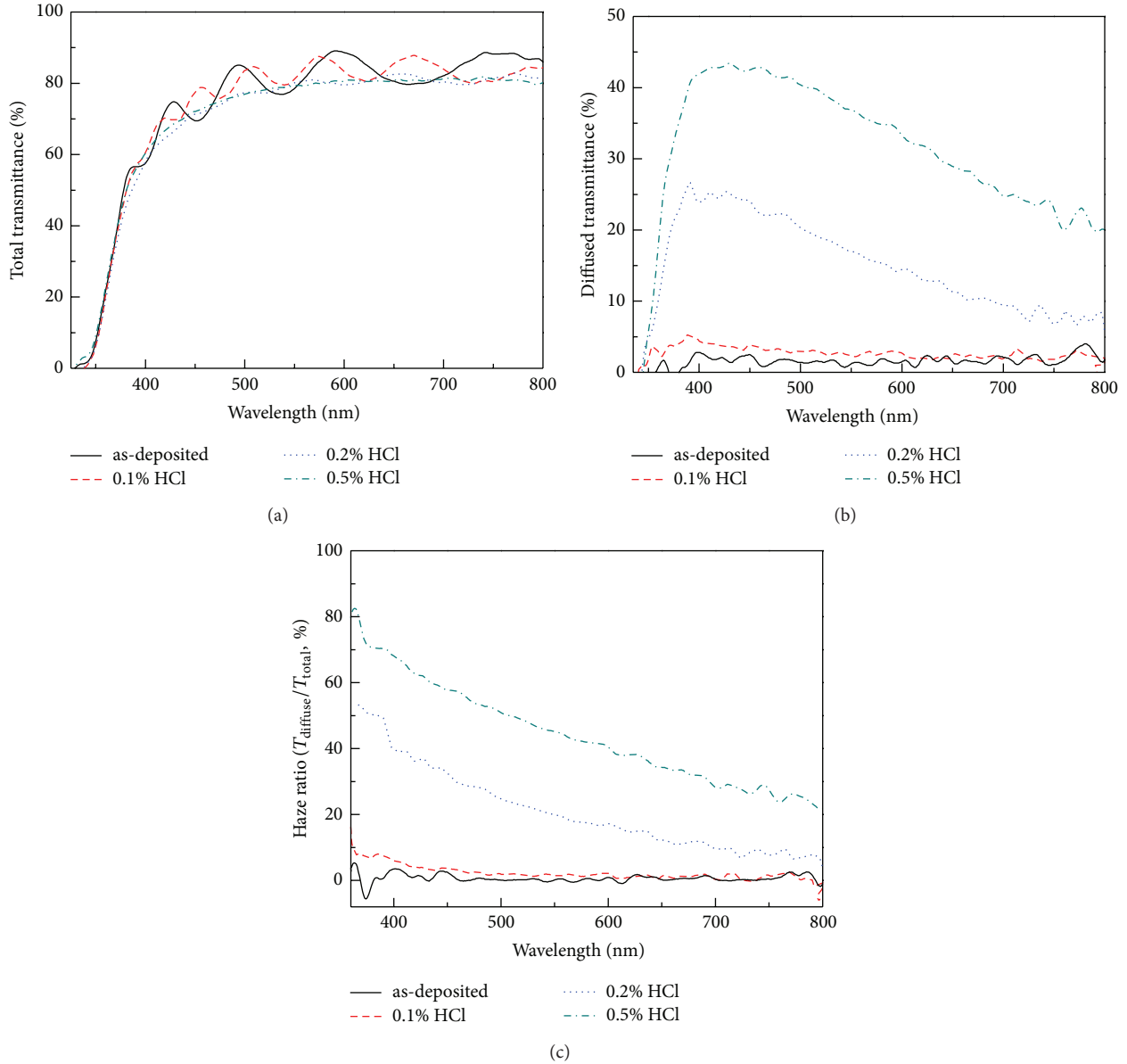


FIGURE 6: The optical characteristics: (a) total transmittance, (b) diffused transmittance, and (c) haze ratio of the HCl etched FZO substrates.

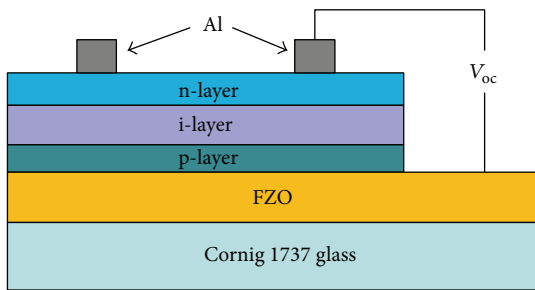


FIGURE 7: Structure configuration of the hydrogenated amorphous silicon thin film solar cells.

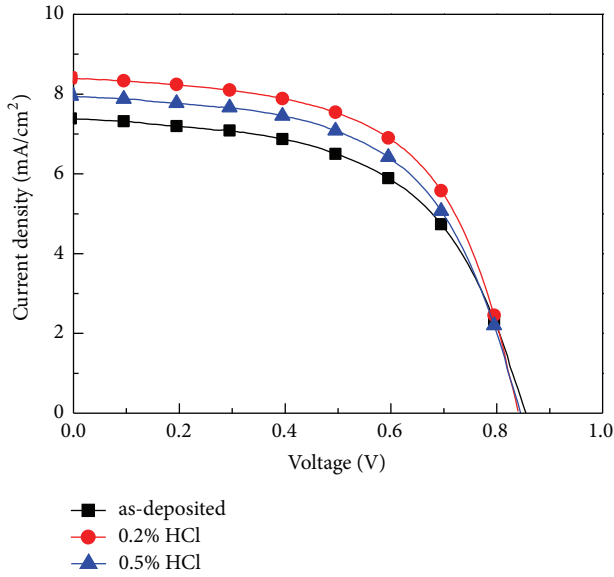
of HCl solution were 0.1 mol %, 0.2 mol %, and 0.5%, the average diffused transmittances were 2.6%, 15.0%, and 32.8%, whereas, in the visible wavelength region, they varied from

1.3% to 4.1%, from 1.3% to 25.3%, and from 20.0% to 43.4%, respectively. As the concentrations of HCl solution were 0.1 mol%, 0.2 mol%, and 0.5%, the maximum diffused transmittances were 5.4% (at 390 nm wavelength), 26.7% (at 390 nm wavelength), and 43.4% (at 430 nm wavelength), respectively. Figure 6(c) shows the haze ratio of the textured FZO thin films with various concentrations of HCl solution. The ratio of diffused to total transmittance is known as haze ratio. The average haze ratio of the textured FZO thin films in visible wavelength region was varied from 0.65%, 1.74%, 18.4%, and 41.2% as the HCl solution concentrations were 0.1 mol%, 0.2 mol%, and 0.5%, respectively.

A p-i-n structure is usually used for α -Si thin film solar cells, as opposed to an n-i-p structure. This is because the mobility of electrons in α -Si:H is roughly 1 or 2 orders of magnitude larger than that of holes, and thus the collection

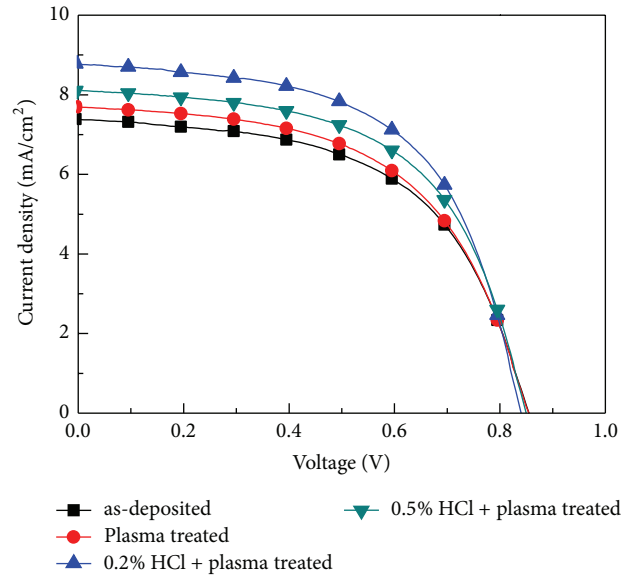
TABLE 1: V_{oc} value, J_{sc} value, and F.F. value of the fabricated amorphous silicon thin film solar cells.

FZO parameters	Abbreviated	V_{oc} (V)	J_{sc} (mA/cm ²)	F.F.	Efficiency (%)
as-deposited	A-FZO	0.855	7.380	0.558	3.52
0.2% HCl	A2-FZO	0.843	8.394	0.585	4.14
0.5% HCl	A5-FZO	0.846	7.942	0.570	3.83
plasma treated	P-A-FZO	0.854	7.692	0.552	3.63
0.2% HCl + plasma treated	P-A2-FZO	0.840	8.764	0.578	4.26
0.5% HCl + plasma treated	P-A5-FZO	0.850	8.109	0.574	3.95

FIGURE 8: Current-voltage characteristics of the p-i-n α -Si:H thin film solar cells under illumination.

rate of electrons moving from the n- to p-type contact is better than holes moving from p- to n-type contact. For that, the superstrate p-i-n hydrogenated α -Si thin film solar cells were fabricated using a single-chamber PECVD unit at 200°C, and the structures of the designed solar cells are shown in Figure 7; no antireflective coatings were deposited on the cells. Although there are several reports related to the etching of glass substrates to improve the efficiency of the α -Si thin film solar cells, texturing of patterned FZO thin films for high haze ratio in thin film solar cells is focused. At the first, as the as-deposited and etched as-deposited FZO thin film substrates are used, the values of open-circuit voltage (V_{oc}), short-circuit current density (J_{sc}), fill factor (F.F.), and efficiency (η) are measured for the devices fabricated on those substrates.

Table 1 shows the measured current-voltage characteristics of the solar cells (substrate size 3.3×3.3 cm²) under illumination as a function of differently treated processes. The abbreviated names of the FZO thin films under different processes are also shown in Table 1. Figure 8 shows that as the A-FZO, A2-FZO, and A5-FZO were used to fabricate the thin film silicon solar cells, the V_{oc} values of the solar cells were 0.855 V, 0.843 V, and 0.846 V; the J_{sc} values were 7.380 mA/cm², 8.394 mA/cm², and 7.942 mA/cm²; the F.F. values were 0.558, 0.585, and 0.570; and the efficiencies were

FIGURE 9: Current-voltage characteristics of the p-i-n α -Si:H thin film solar cells under illumination.

3.52 ± 0.17 , 4.14 ± 0.13 , and 3.83 ± 0.15 , respectively. Those results suggest that the surface texture (or the haze ratio) of the FZO thin films is the key parameter to improve the performance of the α -Si:H thin film solar cells.

Figure 9 shows that as the P-A-FZO, P-A2-FZO, and P-A5-FZO were used to fabricate the thin film silicon solar cells, the V_{oc} values of the solar cells were 0.854 V, 0.840 V, and 0.850 V; the J_{sc} values were 7.692 mA/cm², 8.764 mA/cm², and 8.109 mA/cm²; the F.F. values were 0.552, 0.578, and 0.574; and the efficiencies were 3.63 ± 0.15 , 4.26 ± 0.11 , and 3.95 ± 0.13 , respectively. As Figure 9 shows, the V_{oc} value and F.F. value had no changes, the J_{sc} value increased, and the efficiency increased as the H₂ plasma was used to treat the nonetched and etched FZO thin films. Those measured results of the fabricated α -Si thin film solar cells are also compared in Table 1. The greater efficiencies in etched FZO substrates are mainly ascribable that as the haze ratio increases, the absorption of light increases, and then the short-circuit current density increases.

4. Conclusions

In this study, resistivity is really improved as H₂-plasma process was used because the carrier concentration was increased

and 25 W plasma-treated FZO thin films have the minimum resistivity of $7.92 \times 10^{-4} \Omega\text{-cm}$. The average haze ratio of the etched as-deposited FZO thin films increased from 0.65% to 41.2% as the concentration of HCl solution increased from 0.0% (nonetched) to 0.5%. As the as-deposited FZO thin films in the fabricated $\alpha\text{-Si:H}$ thin film solar cells were changed to the 0.2 HCl-etched FZO ones, the J_{sc} values were improved from 7.380 mA/cm^2 to 8.394 mA/cm^2 , the F.F. values were improved from 0.558 to 0.585, and the efficiencies were improved from 3.52 ± 0.17 to 4.14 ± 0.13 , respectively. When the 0.2 HCl-etched H_2 -plasma-treated FZO thin films were used as the electrodes, the fabricated solar cells had the optimal characteristics of J_{sc} value of 8.764 mA/cm^2 , F.F. value of 0.578, and efficiency of 4.26 ± 0.11 , respectively. For the FZO thin films, the haze ratio increased with increasing concentration of HCl solution and the carrier concentration increased as H_2 plasma was used. That would effectively enhance light trapping and increase the electrons inside the absorber material of solar cells and then improve the efficiency of the fabricated thin film solar cells.

Conflict of Interests

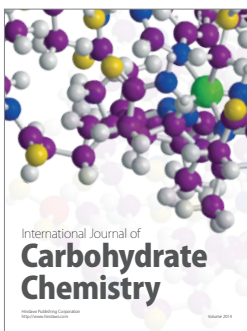
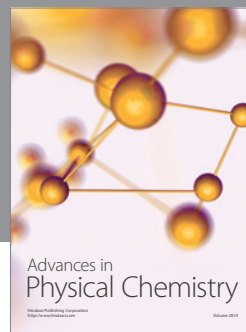
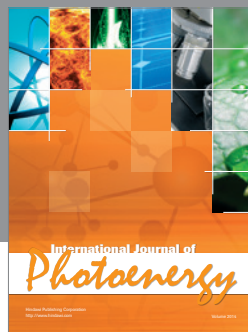
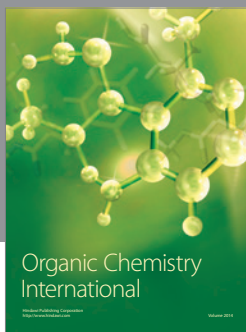
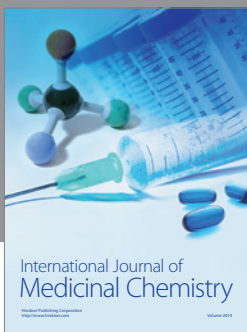
The authors declare that there is no conflict of interests regarding the publication of this paper.

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