Oxygen-Controlled Seed Layer in DC Sputter-Deposited ZnO:Al Substrate for Si Thin-Film Solar Cells

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Abstract—Oxygen-controlled seed layer in Al-doped ZnO (ZnO:Al) thin films deposited by the industrially compatible dynamic dc magnetron sputter results in both enhanced electron mobilities and appropriate etched morphologies for the Si thin-film solar cells. At the relatively low deposition temperature of 300 °C, optimized ZnO:Al film grown on the seed layer has the carrier mobility of 45 cm²/V·s and proper postetching morphology with around 1–2-μm crater size. Reduced angular distribution of the (002) grains analyzed by the diffraction rocking curve is shown as the key structural feature for the improved carrier mobility. Finally, the performance of the microcrystalline Si solar cell on the developed ZnO:Al substrate shows high-efficiency potential of the tandem solar cell adapting this transparent conductive oxide substrate.

Index Terms—a-Si-based thin-film solar cells, Al-doped ZnO, sputtering, transparent conducting oxide.

I. INTRODUCTION

ALUMINUM-DOPED zinc oxide (ZnO:Al) has had significant roles in Si thin-film solar cells [1]–[7]. For the high-efficiency Si thin-film solar cell, ZnO:Al front transparent conductive oxide (TCO) should have high transparency throughout the wide optical spectrum from visible to near-infrared (NIR) region, sheet resistance of $\sim 10 \Omega/\square$ (depending on the module design), and proper postetching surface morphology of uniformly distributed craters with 1–2-μm lateral size to scatter and trap the incident light into the Si absorber layers. Electron mobility is an important characteristic to fulfill the high transparency and low sheet resistance simultaneously [2], [8], [9], and deposition and etching processes should be precisely controlled to obtain the multifunctional materials properly [2], [5], [10]–[14].

One of the most serious issues for the application of the ZnO:Al front TCO into the practical manufacturing process is

its high deposition temperature. Most of the high-performance ZnO:Al films have been obtained in the substrate temperature of 350 °C or higher, which is not easy to implement to industrial in-line sputtering system [15], [16], [24] with the large substrate size over 1 m². Very special and tricky heat management technologies should be adapted to overcome many issues occurred over 350 °C.

One tried method to acquire better ZnO:Al thin film at lower deposition temperature is a seed or buffer-layer scheme to enhance both the crystallinity and the electrical properties of the overgrown ZnO:Al film [17]–[20]. In our previous work at LG Electronics, it has been shown that predeposition of ZnO:Al seed layer with extra oxygen can result in the improved crystallinity of overgrown ZnO:Al bulk layer and thereby reduced resistivity [20]. The oxygen-controlled ZnO:Al seed layer has an advantage compared with other seed layer schemes [17]–[19] in that it does not increase the process costs or manufacturing steps.

In this paper, deposition of ZnO:Al thin film with oxygen-controlled seed layer using the dynamic dc magnetron sputtering system was adjusted to obtain superior carrier mobility of 45 cm²/V·s at relatively low deposition temperature of 300 °C. The reduced angular distribution of the (002) preferred grains is suggested as the main origin of the improved carrier mobility. In addition, microstructural features for the appropriate postetching morphology are also discussed. Finally, the improved current density and higher efficiency of the Si thin-film solar cell were obtained by adopting the optimized ZnO:Al substrate with oxygen-controlled seed layer.

II. EXPERIMENTAL PROCEDURE

The ZnO:Al films were deposited on Corning 1737 glass by a dynamic dc magnetron sputtering system equipped with a moving magnet using ZnO:Al₂O₃ targets having 1.0 wt% Al₂O₃ in the deposition temperature of 300 °C. Deposition was performed in a dynamic deposition mode, where moving magnet bar is located in a local position of the target in a certain moment, and deposition occurs in the position just beneath the magnet. The deposition process mode is similar to that of the industrial in-line sputter system. The films were deposited by a two-step growth method [20] with a seed layer and a bulk layer. With the 9:1 ratio of Ar:O₂ for the seed layer, the optimum thickness of the seed layer was ~ 40 nm, which was previously shown appropriate for the improved electrical conductivity of the overgrown

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bulk layer and superior optical haze after wet etching [20]. In addition, the effect of deposition pressure from 0.5 to 0.2 Pa was also investigated in this experiment.

The deposited thin films were analyzed by X-ray diffraction (XRD: Rigaku D/MAX-IIIC) both in the $\theta - 2\theta$ and rocking-curve modes. The electrical resistivity, mobility, and carrier concentration of the films were measured by a four-point probe and a Hall measurement system (HL5500PC: BIO-RAD). The thickness of the deposited ZnO:Al film was examined by measuring a spectroscopic ellipsometry (M2000-U) with parameters following the Tauc-Lorentz model [21].

To obtain a light-scattering rough surface, wet etching was carried out with diluted hydrochloric-acid-based aqueous solution. Etching time was adjusted to obtain proper sheet resistance of 8–10 $\Omega/\square$ for the application to the *a*-Si:H based thin-film solar cell. The postetching surface morphology was observed by scanning electron microscopy (SEM: JEOL JSM-6700F), and the root-mean-square (RMS) values of surface roughness was deduced by atomic force microscopy (AFM: Nanoscope IIIa, Digital Instrument). Transmittance (*T*), reflectance (*R*), and the absorptance (*A*) calculated by the formula $A = 100\% - T - R$ was obtained in the range from 350 to 1100 nm by spectrophotometer (Cary 5000, Varian), and haze values were evaluated with the ratio of the diffused transmittance over the total integrated transmittance.

The optimized ZnO:Al film was adopted as a front TCO substrate for a single-junction microcrystalline Si ($\mu\text{c-Si:H}$) thin-film p-i-n solar cell prepared by plasma-enhanced chemical vapor deposition. The thickness of the *i*-layer was around 2.5 μm , and sputtered ZnO:Al/Ag/Al stack was used as a back reflecting electrode. As a reference, commercial $\text{SnO}_2\text{:F}$ substrate was also used as a front TCO. Details of silicon deposition and cell preparation are described elsewhere [22]. Light *J-V* characterization of the solar cells was carried out using a solar simulator (Wacom) at standard test conditions (AM 1.5, 100 mW/cm^2 at 25 $^\circ\text{C}$). Finally, the enhanced solar cell efficiency was analyzed by external quantum efficiency (EQE) measurement by spectral response analyzer (Bunko Keiki).

III. RESULTS AND DISCUSSION

A. Improvement of the Carrier Mobility

The mobility of the ZnO:Al thin film is one of the most important properties governing the electrical and optical losses for the solar cell [1], [2], [8]. Fig. 1 shows electrical resistivity, mobility, and carrier concentration of ZnO:Al thin films in terms of deposition pressure and with/without the seed layer. The resistivity of the ZnO:Al thin film decreased with the decreased deposition pressure from 0.5 to 0.2 Pa, due to the increased mobility. In each deposition pressure, the resistivity of the ZnO:Al thin film further decreased when the seed layer was included due to the increased mobility. The carrier concentration of the films showed almost similar values in the range of 4.5 to 5.0 $\times 10^{20} \text{ cm}^{-3}$. Eventually, the highest mobility of 45.0 $\text{cm}^2/\text{V}\cdot\text{s}$ was obtained at the deposition pressure of 0.2 Pa with the seed layer. This magnitude is nearly the upper limit of the mobility in the doped polycrystalline ZnO thin film with that

Fig. 1. Resistivity, mobility, and carrier concentration of ZnO:Al thin films, in terms of deposition pressure, and with/without oxygen controlled seed layer. Samples *a* and *b* are thin films without the seed layer, and *c*–*e* are those with the seed layer.

specific carrier concentration [23]. It is remarkable because such a high mobility of the ZnO:Al film has been reported almost at the deposition temperature of 350 $^\circ\text{C}$ or higher in in-line dc or middle-frequency magnetron sputtering deposition [10].

B. Nanostructural Analysis

In order to systematically investigate the structural change with the deposition condition and the seed-layer adoption, XRD was performed with two different scan methods: the $\theta - 2\theta$ scan and rocking curve by ω scan.

Fig. 2(a) shows the XRD results by the $\theta - 2\theta$ scan. The films with seed layer had nearly perfect (002) preferred orientation, while the films without seed layer had mixed grains of (002) and (101) planes. In general, the ZnO:Al film deposited by static-type RF sputter or dc sputter with enough high temperature had (002) preferred orientation [19], [25], [28]. However, in this study using the dynamic sputtering system, the deposition process is interrupted periodically due to the magnet movement, which possibly inhibit the evolution of (002) preferred grain structures. On the other hand, once the (002) orientation of the initial seed texture is formed, the growth is less affected by the deposition process [19], [25]. Therefore, it can be inferred that the oxygen-controlled seed layer provided the (002) textured platform for the growth of bulk layer.

Diffraction rocking curves by ω scan in Fig. 2(b) show higher peak intensity and narrower full-width at half-maximum (FWHM) as deposition pressure decreased (with the seed layer). The narrower FWHM of the ω peak (FWHM_ω) means that the angular distribution of the (002) grains becomes smaller [8], [19], [25], that is, the film has more vertically aligned (002) grains. As oxygen-controlled seed layer provides the (002) textured platform, it is reasonable that the overgrown film has more

Fig. 2. XRD with (a) θ - 2θ scan and (b) ω scan. (c) Mobility in terms of the FWHM from (a) and (b).

TABLE I
VERTICAL GRAIN SIZE ESTIMATED BY THE θ - 2θ DIFFRACTION

Sample	a	b	c	d	e
Grain Size (nm)	43.3 ± 0.6	43.0 ± 0.7	34.7 ± 0.6	37.8 ± 0.7	38.2 ± 0.8

aligned (002) texture by the epitaxy-like growth. As the energy of the adatoms becomes lower due to the higher deposition pressure and shorter mean free path, the epitaxy-like growth becomes distorted, and the angular distribution among the grown (002) grains increases.

In Fig. 2(c), the carrier mobility values of the films were plotted in terms of $\text{FWHM}_{2\theta}$ and FWHM_{ω} . From the left graph of the mobility in terms of the $\text{FWHM}_{2\theta}$, we cannot find any consistent relationship between the mobility and the grain size estimated by the $\text{FWHM}_{2\theta}$ with the well-known Scherrer's equation [26]. Table I shows the vertical grain size estimated by the θ - 2θ diffraction. On the other hand, the mobility in terms of the FWHM_{ω} shows monotonic increase of the carrier mobility from 22.0 to 45.0 $\text{cm}^2/\text{V}\cdot\text{s}$ as the FWHM_{ω} reduces from 11.9° to 2.7°. The angular distribution of (002) grains estimated by the FWHM_{ω} can be associated with the Seto's carrier-transport model [27] that traps existed in the grain boundaries act as a barrier against the intergrain carrier transport. It is reasonable that the ZnO:Al films with less FWHM_{ω} have lower angle grain boundaries, which gives less defects or traps, and thus lower energy barrier against the carrier transport yielding improved

Fig. 3. SEM images of textured ZnO:Al and SnO₂:F substrates with sheet-resistance values.

carrier mobility. Therefore, the reduced angular distribution of the (002) columnar grains can be the main origin for the improvement of the carrier mobility by the seed layer adoption.

C. Textured Surface of ZnO:Al by Wet Etching

Fig. 3 shows SEM images of the surface morphology of the etched ZnO:Al and a commercial SnO₂:F-coated glass samples. There appeared many craters on the surface of the ZnO:Al films, and the overall crater size increased as the deposition pressure decreased. The commercial SnO₂:F film had no crater but had relatively small randomly distributed pyramids. For the light trapping of NIR wavelength which is very weakly absorbed by $\mu\text{c-Si}$ thin film [1], the rough surface dimension should be comparable with the wavelength, i.e., approximately 1 μm or larger. Therefore, the ZnO:Al films can be more beneficial than the commercial SnO₂:F substrate for the $\mu\text{c-Si}$ thin-film solar cell.

Clear difference of the ZnO:Al films with/without the seed layer is the microroughness of the inner crater surface. The wet-etched ZnO:Al films *a* and *b* (without seed layer) had relatively rough inner surface in the craters, whereas the ZnO:Al films *c-e* (grown on the seed layer) had relatively smooth inner surface in their etched craters. It is very important because the sharp valleys of the TCO substrate cause crack-like defects in the absorbing Si layers, causing decreased V_{oc} and FF [13], [14]. Owen *et al.* [28] showed that the vertically preferred (002) orientation is essential for the formation of the large craters with smooth inner surface in the polycrystalline ZnO films. Therefore, the existence of the vertical (101) grains without the seed layer [as shown in

Fig. 4. Schematic for the different textured surface morphologies between the etched ZnO:Al films, without a seed layer (upper), and those grown on a seed layer (lower). The ZnO:Al film without a seed layer has many irregularly fragmented (002) and (101) grains with roughnesses inside craters. In contrast, the film with a seed layer has well-oriented (002) grains with relatively smooth inner surface in each etched crater.

Fig. 2(a)] can be the origin of the microroughness evolution in the wet-etched craters.

Fig. 4 shows schematic images to explain the grain-structure evolution and its effects on the wet-etched ZnO:Al surface morphology. The upper image shows the ZnO:Al film without seed layer, in which the (002) and (101) grains are coexisted. Because of the repetitive interruption during the film growth in the dynamic sputtering process without the initial (002) texture formation, the transition of the growth mode from the nucleation-merge stage into the epitaxy-like growth stage was delayed, and thus, (101) grains were able to survive during the thick film growth. The survived (101) planes near the surface caused the irregular postetched surface morphology in the craters. On the other hand, the films with (002) seed layer, in which epitaxy-like growth occurred in spite of the periodical growth interruption, (002) preferred orientation near the surface led to the 1–2- μm -sized craters with smooth inner surface.

Optical haze and absorbance spectra of the textured TCO samples were measured as shown in Fig. 5. As expected from the wet-etched morphology from Fig. 3, the overall haze level increased as the deposition pressure decreased, irrespective of whether the seed layer was adopted or not. Remarkably, the haze levels of the samples that were deposited at the same pressure were similar irrespective of the existence of the seed layer. For example, the samples *a* and *c* (deposited at 0.5 Pa) had similar haze levels (*a* with the seed layer, while *c* without seed layer). The samples *b* and *d* were analogous. The absorbance of the ZnO:Al films [see Fig. 4(b)] can be considered as a criterion for the optical absorption loss by the front TCO in the thin-film solar cell. From samples *a*–*e*, the overall absorbance decreased. The improvement of the mobility resulted in the reduced free-carrier

Fig. 5. (a) Optical haze and (b) absorbance spectra of the textured ZnO:Al samples and commercial SnO₂:F substrate. (c) Plots of haze (at 850 nm) in terms of RMS vertical roughness of ZnO:Al and SnO₂:F.

absorption and less film thickness with similar sheet resistance level.

D. Solar Cell Results

The microcrystalline Si ($\mu\text{c-Si:H}$) thin-film solar cell was fabricated on the optimized ZnO:Al substrate. For comparison, the commercial SnO₂:F-coated glass was also used as a front TCO contact. Fig. 6 shows the EQE of the two solar cells, with the open-circuit voltage (V_{oc}), short-circuit current density (J_{sc}), fill factor (FF), and the conversion efficiency (η), extracted from the photocurrent–voltage (J – V) curve of the solar cell. As expected from superior optical transparency and haze ratio of the in-house ZnO:Al substrate to those of commercial SnO₂:F substrate (as shown in Fig. 5), the EQE of the solar cell grown on ZnO:Al was larger than that of the SnO₂:F substrate, especially in the wavelength range longer than 600 nm. Additionally, the solar cell on the ZnO:Al substrate had larger V_{oc} and FF values than that on the SnO₂:F substrate, which resulted from the less shunt paths by the crack-like defects. From high J_{sc} of 26 mA/cm², and superior V_{oc} and FF compared with the cell grown on the SnO₂:F substrate, we can predict that the a -Si:H/ $\mu\text{c-Si:H}$ double-junction tandem cell using the developed ZnO:Al substrate by this research can potentially reach up to $\sim 13\%$ conversion efficiency [5].

Fig. 6. Comparison of the EQE of the μc -Si:H thin-film solar cells made of commercial SnO₂:F and in-house ZnO:Al substrates with optimized process conditions. Light J - V parameters of the solar cells are also compared.

IV. CONCLUSION

The oxygen-controlled seed layer in the dynamic dc magnetron sputtered ZnO:Al thin film, which is compatible with practical industrial processes, has resulted in the enhanced electrical mobility and appropriate etched morphology for Si thin-film solar cells. Although it has been generally reported that deposition temperature higher than 350 °C is necessary for superior properties, ZnO:Al film grown on the seed layer showed the carrier mobility of 45 cm²/V·s, which approached the upper limit of the ZnO-based material, even at the relatively low deposition temperature of 300 °C. The main origin of the improved carrier mobility was found to be the reduced angular distribution of the vertically elongated (002) grains from 11.9° to 2.7°, as confirmed by the diffraction rocking curve. It was further discussed that obtaining perfect (002) preferred orientation was important for the smooth crater surface of the wet-etched ZnO:Al, which leads to a better performance of the Si thin-film solar cells. The μc -Si:H thin-film solar cell with the optimized ZnO:Al substrate showed high J_{sc} of 26.3 mA/cm², which means that a -Si:H/ μc -Si:H thin-film solar cells adopting this superior ZnO:Al substrate can potentially reach ~13% conversion efficiency.

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