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Tunable physical properties of Al-doped ZnO thin films by O₂ and Ar plasma treatments

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Abstract

Al-doped ZnO (AZO) is a promising transparent conducting oxide that can replace indium tin oxide (ITO) owing to its excellent flexibility and eco-friendly characteristics. However, it is difficult to immediately replace ITO with AZO because of the difference in their physical properties. Here, we study the changes in the physical properties of AZO thin films using Ar and O₂ plasma treatments. Ar plasma treatment causes the changes in the surface and physical properties of the AZO thin film. The surface roughness of the AZO thin film decreases, the work function and bandgap slightly increase, and the sheet resistance significantly decreases. In contrast, a large work function change is observed in the AZO thin film treated with O₂ plasma; however, the change in other characteristics is not significant. Therefore, the results indicate that post-treatment using plasma can accelerate the development of high-performance transparent devices.

1. Introduction

Transparent conducting oxides (TCOs) are used as electrodes in touch screens, transparent displays, and organic solar cells [1–5]. Recently, owing to the advent of new technologies, such as flexible and stretchable electronics, augmented reality, and virtual reality, extensive research on functional transparent electrodes is ongoing [6–9]. Currently, indium tin oxide (ITO) is the most widely used material in the transparent electrode industry. However, its high price and brittle characteristics limit its application in transparent electrodes [9–11]. Al-doped ZnO (AZO) can be used as a flexible transparent electrode owing to its high conductivity, good flexibility, low cost, and eco-friendly characteristics [12–15].

Since AZO differs from ITO in terms of several physical properties, such as work function and bandgap, it is difficult to directly replace ITO with AZO. For example, the work function of ITO is approximately 3.6–5.3 eV; whereas, the work function of AZO is approximately 3.1–4.5 eV, which is lower than that of ITO [16]. A large work function mismatch between the transparent electrode and the active layer of the device can cause difficulty in injecting charges from the electrode to the device, which leads to the deterioration of the device characteristics [17, 18].

The physical properties of the oxide thin film can be slightly adjusted using post-treatment processes, such as doping and defect control [19–21]. Helander et al increased the work function by covering the ITO surface with single-layer Cl, which significantly increased the efficiency of organic light-emitting diodes (OLEDs) [22]. This change in work function is due to the large difference in the polarity of the surface of ITO thin film. In addition, Kuo et al reported that Ar sputtering, and oxygen plasma treatment increase the work function of ZnO thin films. They hypothesize that the work function of the ZnO film increased with the removal of carbon contamination by the Ar sputtering, and with the surface oxygenation by oxygen plasma treatment [23]. According to previous studies, the high electrical conductivity of the oxide transparent electrode is due to oxygen vacancies and/or metal interstitials [24, 25]. Hwang et al increased the oxygen vacancy of the ITO thin film using...
Ar plasma treatment, which resulted in a threefold decrease in sheet resistance without any significant change in the bandgap [26]. These results suggest that the physical properties of the oxide thin film can be tuned by post-treatment.

In this study, we investigated various changes in the physical properties of AZO thin films using Ar and O₂ plasma treatments. The physical properties of the AZO thin film prepared by Ar and O₂ plasma treatment exhibited the opposite properties. The AZO thin film was etched by the Ar plasma treatment, resulting in a smooth surface and change in the surface composition. In addition, the work function and bandgap slightly increase, and the sheet resistance decreased considerably. In contrast, the O₂ plasma treatment did not significantly change the surface characteristics, bandgap, and sheet resistance of the AZO thin film, but it significantly increased the work function. This is because Ar and O₂ plasma treatment have contrasting effects on the AZO thin film. This study provides useful information for the design of devices using transparent electrodes.

2. Experiment

Prior to the deposition of the AZO films, Si and glass substrates were sequentially sonicated in isopropyl alcohol (IPA) and DI water for 10 min to obtain a clean surface. The AZO thin films were deposited on p-type Si wafer (100) and soda lime glass substrates using magnetron RF sputtering. The distance between the sputtering target and the substrate was 8 cm. A target of ZnO:Al₂O₃ (98:2 wt%) was used for the deposition of the AZO thin film, and its size and thickness were 2 and 1/8 inches, respectively. The chamber was pumped out to a base pressure of 5.5 × 10⁻⁵ Torr, followed by sputtering. The AZO thin films were deposited under pure Ar flow of 30 sccm, RF power of 60 W, operating pressure of 3 mTorr, and substrate temperature of 300 °C for 1 h. The thickness of the sputtered AZO thin film was approximately 300 nm. According to XRD pattern of the deposited AZO thin film, the sample contains wurtzite ZnO structure as the main phase, as shown in figure S1 (available online at stacks.iop.org/MRX/5/126402/mmedia).

The adaptively coupled plasma (ACP) system for plasma treatment comprised the main process chamber, a load lock chamber, two RF generators for high-density plasma and ion as well as radical flux, a pumping system for high vacuum, and a cooling system to maintain the substrate temperature during the process. The antenna of the upper RF generator for plasma generation was prepared by a mixed-type capacitively coupled plasma method and an inductively coupled plasma method. The frequencies of the upper and lower RF generators were 13.56 and 2 MHz, respectively. Before proceeding with the plasma treatment, the chamber was pumped out to a base pressure of 5 × 10⁻⁶ Torr for 30 min to proceed with the process under pure gas conditions. The AZO thin films deposited by sputtering were treated with Ar or O₂ plasma in an ACP system under the following conditions: total gas flow of 100 sccm, RF power of 500 W, bias power of 150 W, process pressure of 15 mTorr, and substrate temperature equal to room temperature.

The surface chemical compositions of the AZO thin films before and after plasma treatment were investigated using x-ray photoelectron spectroscopy (XPS, NEXSA, Thermo Fisher Scientific). The base pressure of the analysis chamber was initially reached below 10⁻⁶ mbar using two turbomolecular pumps with an automated titanium sublimation pump and backing pump. Prior to XPS analysis, the sample surface was treated by Ar⁺ ion gun for 10 s to reduce the contaminants. The XPS spectra were obtained in fixed retarding ratio mode using monochromated low-power Al Kα x-ray source (1486.6 eV) with a spot size of 400 μm diameter, and all results were determined with reference to C 1s (284.8 eV) component. The curve fitting of the spectra was performed using a Gaussian–Lorentzian peak after Shirley background correction. The work function of the AZO thin films was measured by ultraviolet photoelectron spectroscopy (UPS, NEXSA, Thermo Fisher Scientific) using He-Iα (21.22 eV) UV light. For UPS analysis, the samples were biased at −15 Vdc. The work function was corrected using a pure gold specimen to measure the Fermi edge of the oxide-based thin films. The optical transmittance and bandgap energy of the AZO thin films were investigated and determined using a UV–vis spectrophotometer (Lambda 35, Perkin Elmer) in the wavelength range of 300–1100 nm. The surface morphology and roughness were analyzed using atomic force microscopy (AFM, NX-10, Park system) and field-emission scanning electron microscopy (FE-SEM, Sigma 300, Carl Zeiss). The sheet resistance was measured using a four-point probe (802M, Guardian Scientific). The AZO thin films on Si substrates were used for etching rate, XPS, UPS, and AFM analysis, and the AZO thin films on glass substrates were used to determine the transmittance and bandgap using a UV–vis spectrophotometer. All samples for analysis were immediately vacuum-packed for transfer to the measuring equipment.
3. Results and discussion

Ions and radicals formed by the plasma are accelerated to the surface of the thin film by bias power, which etches away atoms or molecules on the surface of the thin film. This causes a change in the surface composition of the thin film, leading to changes in its overall properties. Therefore, it is necessary to study the composition change of the thin-film surface according to the plasma treatment.

Figure 1 shows the composition change of the AZO thin film according to the Ar plasma treatment using XPS analysis. The bar chart in figure 1(a) shows the change in the atomic concentration of the AZO thin film surface according to the Ar plasma treatment. Before the Ar plasma treatment, the atomic concentrations of the AZO thin films were 4.09% for Al, 48.7% for O, and 47.21% for Zn. As the Ar plasma treatment time was increased to 60 s, the Al atomic concentration increased from 4.09 to 9.73%, and the O atomic concentration increased from 48.7 to 57.44%. In contrast, the Zn atomic concentration decreased from 47.21 to 32.83%.

Figure 1(b) shows the XPS of O 1s narrow-scan spectra depending on the plasma treatment time. Deconvolution was performed to study the change in the oxygen binding by the plasma treatment. Three peaks were observed at binding energies of 530.1, 531, and 532.1 eV and were named as OI, OII, and OIII, respectively. OI, OII, and OIII are attributed to metal-oxide bonds, oxygen vacancies, and chemisorbed/dissociated/hydroxyl (OH) bonds, respectively [27]. The intensity of the OI peak gradually decreased while that of the OII peak slightly increased as the Ar plasma treatment time increased to 60 s (figure S2(a)). This result indicates that the metal-oxide bond (mainly ZnO) was broken and that structural oxygen vacancies were formed by the ion bombardment of the Ar plasma. Based on the results shown in figures 1(a) and (b), it can be concluded that the Ar ion bombardment in the Ar plasma can easily break the Zn–O bond, which has a weak bonding strength and it mainly removes Zn atoms from the surface of the AZO thin film. The change in the Zn spectra can be seen in figure S3. Figure 1(c) shows the XPS narrow-scan spectra of the Al 2p peak. After the Ar plasma treatment, the peak shifted from 73.65 to 74.5 eV, and the peak indicating that metallic Al, binding energy of 72.8 eV, was not observed. [27]. Here, the binding energy of 73.65 eV corresponds to AlOx bonding, and the binding energy of 74.5 eV corresponds to the Al2O3 structure. In other words, the AlOx bond, which is unstable on the surface after sputtering deposition, is etched away by the bombardment of Ar ions, and the close-packed Al2O3 structure remains. Thus, the Ar plasma treatment removes metal–oxide bonds with unstable structures, such as Al–Ox and Znx–Oy. Furthermore, it removes metal atoms (mainly Zn) after breaking the metal-oxide bonds.

The O2 plasma treatment is mainly used to remove organic residues from the thin-film surface or to improve the adhesion with metal electrodes [28–30]. It is also used to oxidize the surface of the thin film or to remove defects, such as oxygen vacancies in the oxide thin film [31–33]. XPS analysis was performed to determine the surface changes of the AZO thin film under O2 plasma. Figure 2 shows the composition change of the AZO thin
film according to the O$_2$ plasma treatment. As shown in figure 2(a), the O atomic percentage slightly increased from 48.7 to 49.24% as the O$_2$ plasma treatment time increased to 60 s. Accordingly, the atomic percentages of Al and Zn slightly decreased from 4.09 and 47.21% to 3.81 and 46.95%, respectively. This is because the oxygen ions by the O$_2$ plasma were adsorbed on the surface of the AZO thin film or filled the oxygen vacancies of the AZO thin film, which was confirmed by the deconvolution of the XPS narrow-scan spectra of the O 1s peak in figure 2(b). Although no significant change was observed compared to the Ar plasma treatment, the intensity of the O$_{II}$ peak, which indicates an oxygen vacancy, decreased by approximately 2%, and the intensity of the O$_{I}$ and O$_{III}$ peaks slightly increased. This is because the oxygen radicals in the O$_2$ plasma accelerated to the surface of the AZO thin film by bias power, and oxygen was filled in the oxygen vacancies on the surface of the AZO thin film. Figure 2(c) shows the XPS narrow-scan spectra of the Al 2p peak. After the O$_2$ plasma treatment, only a slight peak shift was observed from 73.65 to 73.7 eV. In other words, because the energy of the oxygen ion bombardment to the AZO surface is lower than the Ar ion energy, it is considered that oxygen fills the oxygen vacancies on the surface of the AZO thin film and exhibits a low etch rate. In conclusion, the O$_2$ plasma treatment shows a completely different trend from that of the Ar plasma treatment. The O$_2$ plasma treatment does not cause defects, such as oxygen vacancies in the AZO thin films and does not remove unstable bonds, such as AlO$_x$. However, defects such as oxygen vacancies are removed by the oxygen injection\cite{34}.

Because the surface roughness of the transparent conductive thin film affects the haze factor, it is considered as an important parameter in optical devices, such as OLEDs and solar cells\cite{35, 36}. Figure 3 shows the surface roughness of the AZO thin film according to the plasma treatment. Figures 3(a) and (b) show a bar chart of the surface roughness change according to the Ar and O$_2$ plasma treatment times, respectively, and figures 3(c)–(e) show the surface SEM images of the AZO thin film before and after the plasma treatment, respectively. The AFM image for the bar chart is shown in figure S4. The surface of the as-deposited AZO thin film features pyramid-shaped crystals, as shown in the SEM image in figure 3(c). The roughness of the AZO thin film decreased from 7.63 to 4.21 nm as the Ar plasma treatment time increased from 0 to 60 s, as shown in figure 3(a). Figure 3(d) shows a smooth AZO thin film surface planarized by the Ar plasma treatment. Electrons generated by the Ar plasma accumulate on the surface of the AZO thin film, especially on the top edges of the pyramid crystals. The Ar ions accelerate to the AZO thin film by the bias power, and these accelerated ions are focused on the top edges of the pyramid crystal\cite{37}. Therefore, the etch rate is higher on the top edge of the pyramid crystal than in other regions, resulting in the formation of a smooth surface. Figures S5(a) and (b) show cross-sectional SEM images of the AZO thin films without Ar plasma treatment and with Ar plasma treatment for 60 s. An AZO thin film of approximately 54 nm was etched by the Ar plasma for 60 s. As shown in the bar chart of figure 3(b) and SEM image of figure 3(e), no significant change was observed in the surface roughness of the AZO thin film. The surface roughness is within the range of 7.02–7.63 nm depending on the O$_2$ plasma.
treatment time. The oxygen radicals in the O$_2$ plasma experience a lower acceleration effect compared to the Ar ion owing to the bias power. Consequently, the bombardment energy is low, resulting in less surface roughness changes. Figure S5(c) shows the cross-sectional SEM image of the AZO thin film obtained by the O$_2$ plasma treatment (60 s). Compared to the as-deposited thin film, the thickness of this film is almost unchanged, indicating that etching barely proceeds in the O$_2$ plasma treatment.

We previously observed that the surface composition of the AZO thin film was changed by the Ar and O$_2$ plasma treatments, as shown in figures 1 and 2. The changes in the composition of the thin film surface affect various physical properties of the thin film, such as the work function, electrical conductivity, and bandgap [38]. These physical properties are important for the design of devices and systems. Therefore, we investigated the changes in various physical properties of the AZO thin films due to the plasma treatment. Figure 4 shows the changes in the work function of the AZO thin film due to Ar and O$_2$ plasma treatments as determined by UPS analysis. The work function was evaluated using the following equation.

$$\varphi = h\nu - |E_{cutoff} - E_F|$$

Here, $\varphi$ is the work function and $h\nu$ is the photon energy corresponding to 21.2 eV, which is the wavelength of the UV source. $E_{cutoff}$ and $E_F$ are the cutoff energy and Fermi level energy, respectively.

Figure 4(a) shows the change in the work function of the AZO thin film according to the Ar plasma treatment. The cutoff energies for the as-grown, Ar 30 s, and Ar 60 s films are 17.01, 16.85, and 16.84 eV, respectively. The corresponding work function in the as-grown AZO thin film is 4.19 eV. When Ar plasma treatment is performed for 30 s, the work function increases to 4.35 eV, and when further processed for 60 s, it increases to 4.36 eV, which is not a significant change. We have already observed the surface of the AZO thin film through XPS analysis. In particular, we can confirm that the structure of AlO$_x$ was removed through the change in the Al 2p peak (figure 1(c)). Furthermore, as shown in the SEM images in figure S5, the AZO thin film was etched using the Ar plasma. This suggests that unstable metal-oxide structures remain on the AZO thin film surface during the initial deposition, and the work function changes as the Ar ions accelerated by bias power in the Ar plasma remove unstable AlO$_{x,y}$, ZnO$_y$, and carbon contaminations from the surface of the AZO thin film [23].

The changes in the work function of the AZO thin films with respect to the O$_2$ plasma treatment time are shown in figure 4(b). The cutoff energy for O$_2$ 30 s, and O$_2$ 60 s is 16.99 and 16.67 eV, respectively. The corresponding work function in the O$_2$ 30 s, and O$_2$ 60 s is 4.21 and 4.53 eV, respectively. As the O$_2$ plasma treatment time increases, the work function increases from 4.19 to 4.53 eV. This is because of the reduction of crystal defects, such as oxygen vacancies during the O$_2$ plasma treatment, and the oxygenation on the surface of the AZO thin film. This result causes the thin film surface to become more electronegative [23]. Therefore, the
The work function of the AZO thin film increases to a greater extent under the O₂ plasma than under the Ar plasma [23, 39]. Work function diagram of AZO thin films by Ar and O₂ plasma treatment is summarized in figure S6.

UV–vis spectrophotometer measurements were performed to investigate the changes in optical properties, such as optical transmittance and bandgap, by the Ar and O₂ plasma treatments. Figures 5(a) and (b) show the change in optical transmittance after the Ar and O₂ plasma treatments. All samples exhibited sharp absorption edges between 350 and 400 nm. While most samples with the glass substrate showed similar transmittance characteristics of over 80%, the AZO thin film treated with the O₂ plasma for 60 s showed slightly lower transmittance than the other samples. The optical bandgap was estimated using the Tauc relation based on the transmittance results as follows:

\[(a\nu)^2 = A(\nu - E_g)\]

where \(a\), \(A\), and \(E_g\) are the absorption coefficient, constant, and optical bandgap, respectively. Figures 5(c) and (d) show the change in the optical bandgap of the AZO thin film after the Ar and O₂ plasma treatments. In the as-grown AZO thin film before plasma treatment, the bandgap was estimated as 3.47 eV (figure S7). As the Ar plasma treatment time increased to 60 s, the optical bandgap increased by 0.07 eV from 3.47 to 3.54 eV (figure 5(c)). The increase in defects in the AZO thin film owing to the Ar ion bombardment led to an increase in the charge carrier concentration, thereby increasing the optical bandgap [40]. In contrast, as the treatment time increased in the O₂ plasma, the optical bandgap slightly decreased by 0.03 eV from 3.47 to 3.44 eV. It can be considered that defects such as oxygen vacancies generated during the deposition of the AZO thin film were recovered by the oxygen injection during the O₂ plasma treatment; consequently, the bandgap decreased. Band diagram of AZO thin films by Ar and O₂ plasma treatment is summarized in figure S7.

Figure 6 shows the change in sheet resistance with the Ar and O₂ plasma treatments. The as-grown AZO thin film exhibited a sheet resistance of approximately 102 kΩ sq⁻¹. When the Ar plasma treatment was performed, the sheet resistance of the AZO thin film significantly decreased to approximately 2.45 kΩ sq⁻¹. In general, the generation of defects such as oxygen vacancies (for n-type) or metal vacancies (for p-type) in the oxide thin film generated free electrons or holes, which increased the conductivity of the oxide thin film [41–43]. Here, the decrease in the sheet resistance during the Ar plasma treatment may be due to the increase in simultaneous structural defects of the AZO thin film. In contrast, when the O₂ plasma treatment proceeded for up to 60 s, the sheet resistance increased to approximately 110 kΩ sq⁻¹, which may be due to the reduction of oxygen vacancies during the O₂ plasma treatment.

4. Conclusion

In this study, we investigated the changes in the surface and physical properties of AZO thin films, which are referred to as TCOs, after Ar and O₂ plasma treatment. The AZO thin films treated with Ar and O₂ plasma exhibited contrasting properties. The Ar plasma treatment significantly affected the surface composition of the
Figure 5. Transmittance of AZO thin film according to plasma treatment time: (a) Ar plasma treatment, (b) O₂ plasma treatment. Band gap energy of AZO thin film with (c) Ar plasma treatment and (d) O₂ plasma treatment according to plasma treatment time.

Figure 6. Sheet resistivity of AZO thin film according to plasma treatment time.
AZO thin film by removing the unstable metal oxide and breaking the metal-oxide bond. Accordingly, the surface roughness significantly decreased from 7.63 to 4.21 nm, and the work function slightly increased from 4.19 to 4.36 eV. In addition, the bandgap increased from 3.47 to 3.54 eV, and the sheet resistance significantly decreased from 102.73 to 2.45 kΩsq⁻¹. In contrast, the O₂ plasma treatment did not significantly affect the surface composition of the AZO thin film, but it reduced defects, such as oxygen vacancies. Consequently, the surface roughness did not vary significantly, while the work function increased considerably from 4.19 to 4.53 eV. In addition, the bandgap slightly decreased, and the sheet resistance did not vary significantly. This difference is considered to be due to the different mechanisms of Ar ions and O₂ radicals in the plasma. The property changes of the AZO thin film by Ar and O₂ plasma treatment can significantly improve the properties of various optoelectronic devices.

As a result, the plasma treatment as a post-process can tune the physical properties of the AZO thin film, which will improve the properties of various devices. For example, the bottom electrode of the touch panel is required a transparent electrode with low surface roughness and high conductivity, which can be implemented by Ar plasma treatment. In the case of OLEDs requiring high work function values, O₂ plasma treatment may be applied. Therefore, we expect that the plasma treatment as a post-process can be a robust platform for the development of high-performance optoelectronic devices.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

CRediT authorship contribution statement

Young-Hee Joo: Conceptualisation, Methodology, Investigation, Data Curation, Visualization, Writing-Original draft. Doo-Seung Um: Conceptualisation, Validation, Resources, Visualisation, Writing-Review & Editing, Supervision, Funding acquisition. Chang-Il Kim: Conceptualisation, Writing-Review & Editing, Supervision, Project administration, Funding acquisition.

Conflicts of interest

The authors declare no conflicts of interest.

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