

Optimization of Oxygen Flow for Tailoring the Properties of AZO Thin Films Deposited by DC Magnetron Sputtering

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Abstract

This study reports on the preparation of aluminum-doped zinc oxide (AZO) thin films by DC magnetron sputtering using a Zn/Al target. The influence of reactive oxygen gas flow rate (10 – 100 sccm) was investigated while maintaining a constant Ar flow rate of 40 sccm. Films were deposited on both silicon and glass substrates, and further examined in their as-deposited state as well as after annealing at 400 °C in vacuum for 2 h. Morphological, structural, optical, and electrical properties were systematically characterized by FE-SEM, GI-XRD, UV–Vis–NIR spectroscopy, and Hall effect measurements, respectively. The results reveal that increasing O₂ flow rate affects the deposition rate while the films consistently exhibit a hexagonal wurtzite structure with preferred orientations along (002) and (103). The degree of crystallinity strongly depends on the O₂ concentration. The optical transmittance was found to be in the range of 76.10 – 84.79%, with estimated band gap energies between 3.32 and 3.52 eV. Notably, AZO thin films deposited at an O₂ flow rate of 20 sccm exhibited optimum electrical properties, including a resistivity of $4.0 \times 10^{-3} \Omega \cdot \text{cm}$, conductivity of 246 S cm^{-1} , carrier mobility of $5.83 \text{ cm}^2 \cdot \text{V}^{-1} \cdot \text{s}^{-1}$, and carrier concentration of $2.66 \times 10^{20} \text{ cm}^{-3}$. These findings highlight the critical role of O₂ flow control in tailoring the physical properties of AZO thin films, underscoring their potential for various optoelectronic applications.

Keywords: AZO thin films; DC magnetron sputtering; Oxygen control

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1. Introduction

Transparent conductive oxides (TCOs) are essential materials for the development of modern thin-film optoelectronic technologies owing to their unique combination of high electrical conductivity and excellent optical transparency in the visible wavelength region. Generally, TCO materials exhibit low electrical resistivity in the range of 10^{-3} – $10^{-4} \Omega \cdot \text{cm}$, together with optical transmittance exceeding 80% [1]. These advantageous properties have enabled the widespread application of TCOs as transparent electrodes in flat-panel displays and photovoltaic devices, as well as in various thin-film optoelectronic applications, including low-emissivity windows, thin-film transistors, and light-emitting devices [2 – 6]. Commonly used TCO materials include indium tin oxide (ITO), fluorine-doped tin oxide (FTO), and aluminum-doped zinc oxide (AZO) [7,8]. Among these materials, ITO has been the most extensively employed owing to its excellent electrical conductivity and high optical transparency in the visible region. However, its large-scale application is constrained by the high cost and limited availability of indium. In contrast, the fabrication of FTO involves challenges associated with fluorine incorporation during the deposition process. Due to these limitations, considerable research efforts have been devoted to the development of alternative TCO materials that are cost-effective and environmentally benign [9 – 12].

In this context, aluminum-doped zinc oxide (AZO) thin films have emerged as promising candidates to replace ITO because of their high electrical conductivity, good optical transparency, low material cost, and non-toxic nature. AZO thin films can be fabricated using a variety of deposition techniques, including chemical vapor deposition (CVD) [13], sol–gel processing [14], chemical spray deposition [15], pulsed laser deposition (PLD) [16], and DC magnetron sputtering (DCMS). Among these techniques, DC magnetron sputtering is particularly attractive due to its scalability for large-area deposition, cost-effectiveness, and precise control over deposition parameters. This method enables the fabrication of AZO thin films with uniform thickness, strong adhesion, and high atomic density, which are critical requirements for optoelectronic and photovoltaic applications.

Previous studies on AZO thin films prepared by DCMS have primarily focused on the influence of deposition parameters such as sputtering power and working pressure [4],[9],[10]. In addition to conventional DCMS, some studies have employed high-power impulse magnetron sputtering (HiPIMS), demonstrating that variations in pulse frequency can enhance ion energy and film density, thereby improving the crystallinity, optical transparency, and electrical performance of AZO thin films [10]. Other deposition parameters, including target power, target–substrate distance, and substrate temperature, have also been extensively investigated [19]. In most reported works, AZO thin films deposited by DCMS were prepared using ceramic ZnO:Al targets with a fixed aluminum concentration of approximately 2 wt% [17 – 20]. In contrast, studies employing metallic Zn/Al targets have revealed that variations in the oxygen gas flow rate can induce a distinct transition in AZO thin films from a conductive state to an insulating state, highlighting the critical role of oxygen incorporation in determining the electrical properties of the films [21].

In this study, the influence of oxygen flow rate on the surface morphology, crystalline structure, optical properties, and electrical characteristics of AZO thin films deposited by DC magnetron sputtering is systematically investigated. Metallic Zn/Al targets were employed with controlled oxygen gas flow, which directly governs the transition from metallic films to metal oxide films. The primary objective of this work is to identify the optimal deposition conditions for the development of high-performance transparent conducting oxide (TCO) thin films suitable for optoelectronic applications.

2. Materials and Methods

AZO thin films were deposited on n-type Si (100) wafers and glass slide substrates using a DC pulsed magnetron sputtering system. The overall experimental workflow and the schematic of the sputtering system used in this study are illustrated in Figure 1. Prior to deposition, the glass substrates were sequentially cleaned in acetone, 2-propanol, and deionized water for 15 min each using an ultrasonic bath. After cleaning, the substrates were dried under a nitrogen gas flow and subsequently heated on a hot plate at 90 °C for 5 min to remove residual moisture. A 3-inch Al-doped Zn (Zn/Al) sputtering target with a composition of 98:2 wt% and a purity of 99.99% was used for film deposition. The sputtering chamber was evacuated to a base pressure of 6×10^{-6} mbar prior to deposition. AZO thin films were deposited in an argon atmosphere with a purity of 99.99%, while the oxygen flow rate was varied from 10 to 100 sccm at a constant sputtering power of 150 W. Before deposition, the target was pre-sputtered for 3 min to remove surface contaminants. During the initial stage, the films were conditioned using argon gas at a flow rate of 40 sccm. In the first step, AZO thin films were deposited for a fixed duration of 30 min under different oxygen flow rates (10–100 sccm) to determine the deposition rate. In the second step, AZO thin films with a fixed thickness of approximately 400 nm were prepared by adjusting the deposition time, while all other deposition parameters were kept constant. The as-deposited films were subsequently annealed at 400 °C under vacuum for 2 h.

All samples deposited on silicon substrates were used for crystalline structure analysis. The crystal structure of the AZO thin films was characterized by grazing-incidence X-ray diffraction (GIXRD) using a Rigaku TTRAX III diffractometer. Film thickness and surface morphology were examined by field-emission scanning electron microscopy (FE-SEM, Hitachi S-8030). Optical transmittance spectra of AZO thin films deposited on glass substrates were recorded using a UV–Vis–NIR spectrophotometer (Cary 7000, Agilent) over the wavelength range of 200 – 2000 nm. In addition, the electrical properties of the films were characterized at room temperature by Hall-effect measurements using an Ecopia HMS-3000 system.

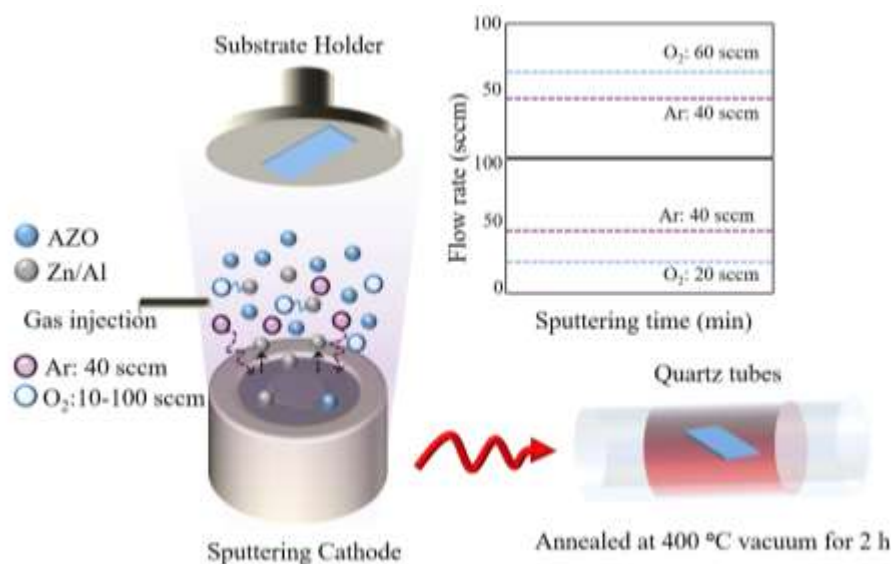


Fig. 1 Schematic diagram of the experimental process for AZO thin film preparation, including the DC magnetron sputtering system with Ar and O₂ flow control, and the post-deposition vacuum annealing setup.

3. Results and Discussions

AZO thin films were deposited using the DC magnetron sputtering (DCMS) technique, with the oxygen flow rate varied from 10 – 100 sccm while maintaining a constant argon flow rate. It is well known that the oxygen flow rate significantly influences the deposition rate of oxide thin films. Therefore, the deposition rate of AZO thin films was first evaluated by fixing the deposition time at 30 min under each oxygen flow condition. The obtained deposition rates were subsequently used to adjust the deposition time in order to fabricate AZO thin films with a uniform thickness of approximately 400 nm. The measured film thicknesses were 602.38, 712.85, 400.99, and 358.41 nm at oxygen flow rates of 10, 20, 60, and 100 sccm, respectively, corresponding to deposition rates in the range of approximately 11.94 – 23.76 nm/min. These results clearly indicate that the deposition rate of AZO thin films is strongly dependent on the oxygen flow rate. The reduction in deposition rate with increasing oxygen flow can be attributed to enhanced scattering of sputtered species. In addition, the introduction of oxygen dilutes the argon plasma and intensifies collisions between sputtered target atoms and oxygen species during transport toward the substrate, leading to a decreased arrival flux at the substrate surface [22]. The optimum electrical performance observed at an O₂ flow rate of 20 sccm is attributed to the ideal balance between forming a stable ZnO lattice and maintaining a sufficient concentration of native donor defects, such as oxygen vacancies (V_O) and zinc interstitials (Zn_i). When the O₂ flow rate increases beyond this point (60 – 100 sccm), the films become more stoichiometric, and excess oxygen fills these V_O sites, leading to a reduction in free carrier concentration [23].

Fig. 2(a) shows the cross-sectional FE-SEM images of AZO thin films. The films prepared after deposition-rate calibration exhibit comparable thicknesses in the range of 386 – 401 nm, confirming that the deposition parameters were successfully adjusted to achieve nearly identical film thicknesses. After annealing at 400 °C under vacuum for 2 h, the films become more densely packed, accompanied by a slight thickness reduction to 370 – 388 nm. This behavior indicates post-annealing-induced atomic diffusion, grain coalescence, and film densification. The surface FE-SEM images in Fig. 2(b) reveal a clear dependence of grain diameter on oxygen flow rate. For the as-deposited films, the largest average grain diameter (85.42 ± 21.8 nm) is observed at an oxygen flow rate of 10 sccm. Increasing the oxygen flow rate to 20 sccm results in a significant reduction in grain size to 53.09 ± 15.6 nm, followed by a gradual increase to 73.27 ± 24.7 nm at higher oxygen flow rates. This trend suggests that at low oxygen flow (10 sccm), incomplete oxidation of Zn/Al species promotes a metallic growth mode, leading to larger grains. With increasing oxygen flow, the growth regime transitions toward a metal–oxide mode, increasing nucleation density and reducing grain size. After annealing, the grain diameters under all oxygen flow conditions decrease slightly, as shown in Fig. 2(d). This reduction is attributed to thermally induced densification and structural relaxation, which compact grain boundaries and reduce voids, resulting in a more uniform and densely packed microstructure. [24]

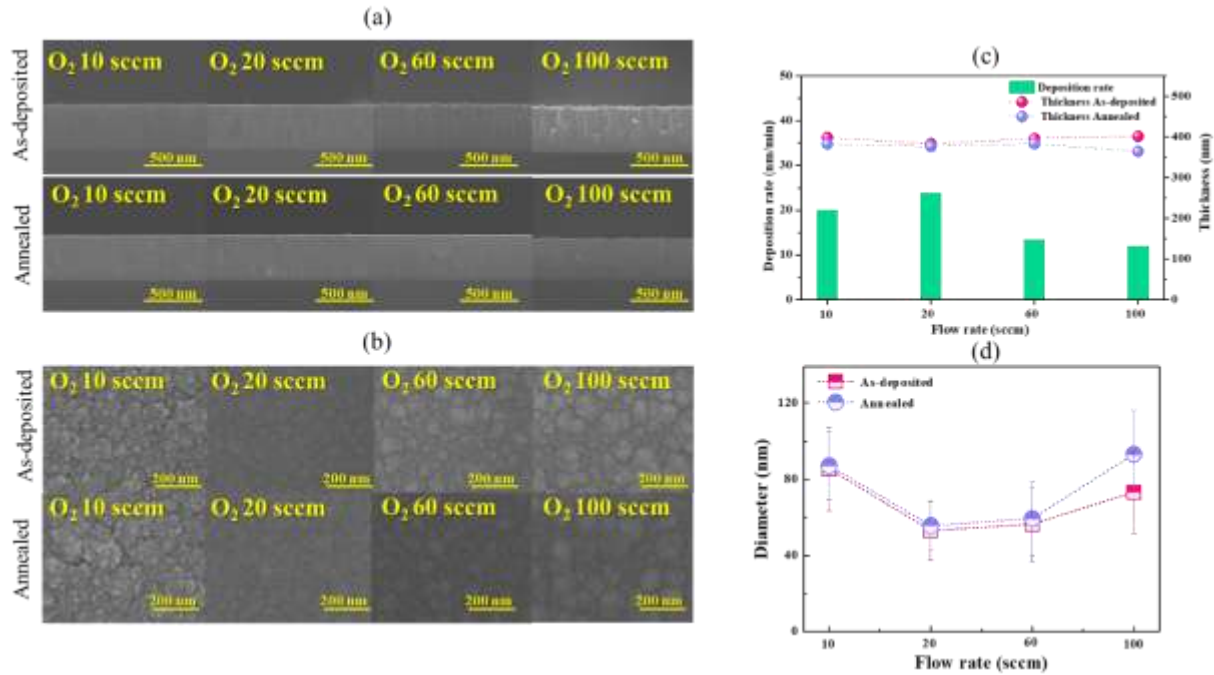


Fig. 2 (a) cross-sectional and (b) Surface FE-SEM images of AZO thin films deposited by DC magnetron sputtering in the as-deposited state and after annealing at 400 °C for 2 h. (c) Film thickness and deposition rate as a function of O₂ flow rate (d) Average grain diameter of AZO thin films as a function of O₂ flow rate.

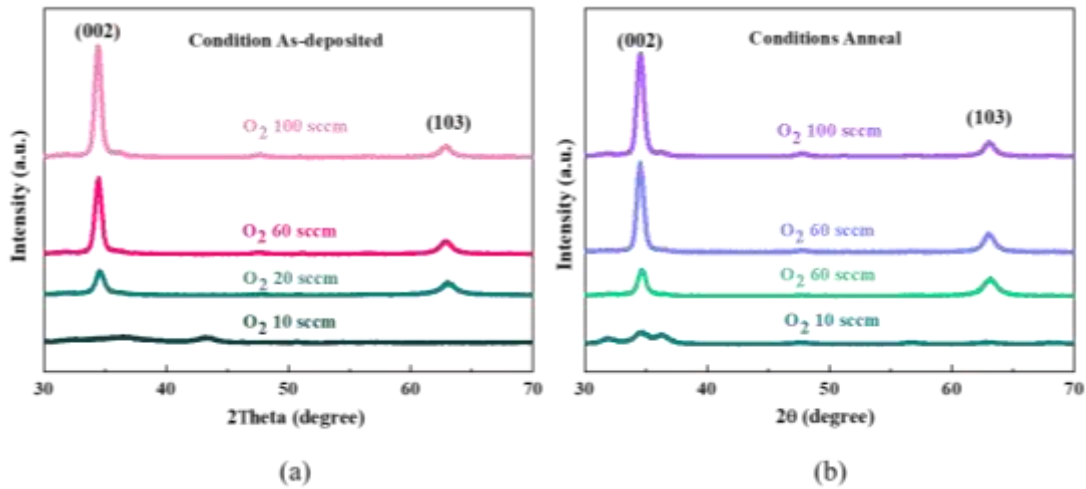


Fig. 3 Grazing-incidence X-ray diffraction (GI-XRD) patterns of AZO thin films deposited on Si substrates under different processing conditions: as-deposited and after annealing at 400 °C for 2 h.

The crystalline structures of AZO thin films are shown in Fig. 3. The results clearly demonstrate that the oxygen flow rate has a significant influence on the crystallinity of the films. At a low oxygen flow rate of 10 sccm, no distinct polycrystalline diffraction peaks corresponding to the wurtzite ZnO structure are observed, indicating that the film is either amorphous or predominantly metallic in nature. In contrast, as the oxygen flow rate increases, diffraction peaks associated with the hexagonal wurtzite structure of ZnO gradually emerge, particularly those corresponding to the (002) and (103) planes. The intensities of these peaks continuously increase with increasing oxygen flow rate, reaching the highest crystallinity at an oxygen flow rate of 100 sccm. This enhancement in crystallinity at higher oxygen flow rates can be attributed to more effective oxidation reactions between oxygen and Zn/Al species, promoting the formation of a well-defined ZnO:Al lattice. In addition, the reduced

deposition rate at higher oxygen flow rates may contribute to a self-annealing effect, allowing sputtered adatoms more time to migrate and occupy energetically favorable lattice sites, thereby improving crystalline ordering. After post-annealing at 400 °C under vacuum for 2 h, all AZO thin films exhibit a slight improvement in crystallinity, as evidenced by the increased intensities of the (002) and (103) diffraction peaks. Notably, for the film deposited at an oxygen flow rate of 10 sccm, a weak (002) peak becomes observable after annealing. This behavior indicates that thermal treatment facilitates the oxidation process and structural rearrangement, leading to a transition from a predominantly metallic film toward a metallic oxide phase.

The optical properties of AZO thin films deposited on glass substrates were investigated for both the as-deposited and post-annealed states. The optical transmittance spectra were measured at normal incidence in the wavelength range of 300–1800 nm using a UV–Vis–NIR spectrophotometer, as shown in Fig. 4(a) and (b). For AZO thin films deposited with oxygen flow rates of 20 – 100 sccm, clear interference fringes are observed in the transmittance spectra. These fringes arise from multiple partial reflections at the air/film and film/substrate interfaces, indicating good optical uniformity and smooth film surfaces [25]. In contrast, the film deposited at an oxygen flow rate of 10 sccm exhibits very low transmittance in the UV–visible region in the as-deposited state, confirming the predominantly metallic nature of the Zn/Al film due to insufficient oxidation. After annealing at 400 °C, this film becomes optically transparent, indicating that thermal treatment promotes oxidation and structural transformation from a metallic phase to a transparent conductive oxide phase [26]. The average optical transmittance in the visible region (380 – 780 nm), denoted as T_{avg} , was calculated from the transmittance spectra, as summarized in Fig. 3(c). After annealing, the T_{avg} values reach 79.83%, 84.79%, 83.10%, and 83.94% for oxygen flow rates of 10, 20, 60, and 100 sccm, respectively, confirming that all films exhibit good optical transparency suitable for TCO applications.

To further evaluate the optical band gap energy (E_g), the transmittance data were converted into the optical absorption coefficient (α) using the relation described in [27]. The optical band gap was determined using the Tauc relation (1):

$$(\alpha h\nu)^n = A (h\nu - E_g) \quad (1)$$

where A is a constant, $h\nu$ is the incident photon energy, E_g is the optical band gap energy, and α is the absorption coefficient. The exponent n represents the nature of the electronic transition; for AZO thin films, a direct allowed transition ($n = 2$) was assumed

Fig. 4(d) – (f) present the optical band gap energies of AZO thin films in both the as-deposited and annealed states. The estimated E_g values for the as-deposited films are approximately 1.40, 3.35, 3.32, and 3.31 eV at oxygen flow rates of 10, 20, 60, and 100 sccm, respectively. After annealing, the optical band gap increases to about 3.23, 3.47, 3.45, and 3.33 eV for the corresponding oxygen flow rates. The significant increase in E_g after annealing, particularly for the film deposited at 10 sccm, is attributed to improved crystallinity, enhanced oxidation, and a reduction in structural defects and localized states within the band gap [28, 29].

Fig. 5 presents the electrical properties of AZO thin films deposited using a Zn/Al metallic target after annealing at 400 °C for 2 h. The electrical properties of the as-deposited films are not shown because only the film deposited at an oxygen flow rate of 10 sccm exhibited electrical conduction due to its metallic nature, whereas the films deposited at oxygen flow rates of 20–100 sccm were electrically insulating owing to insufficient carrier activation and poor oxide crystallization. After annealing, the electrical properties show a strong dependence on the oxygen flow rate. The film deposited at 10 sccm exhibits relatively low carrier concentration and Hall mobility, resulting in high resistivity, which is attributed to incomplete oxidation and a high density of defects. When the oxygen flow rate is increased to 20 sccm, both carrier concentration and mobility reach their maximum values, leading to the lowest resistivity. This indicates that an optimal oxygen supply, combined with thermal annealing, promotes effective oxidation and substitutional incorporation of Al into the ZnO lattice, thereby enhancing free carrier generation. At higher oxygen flow rates of 60 and 100 sccm, excessive oxygen incorporation suppresses donor defects and introduces compensating defects, resulting in reduced carrier concentration and mobility and, consequently, increased resistivity.

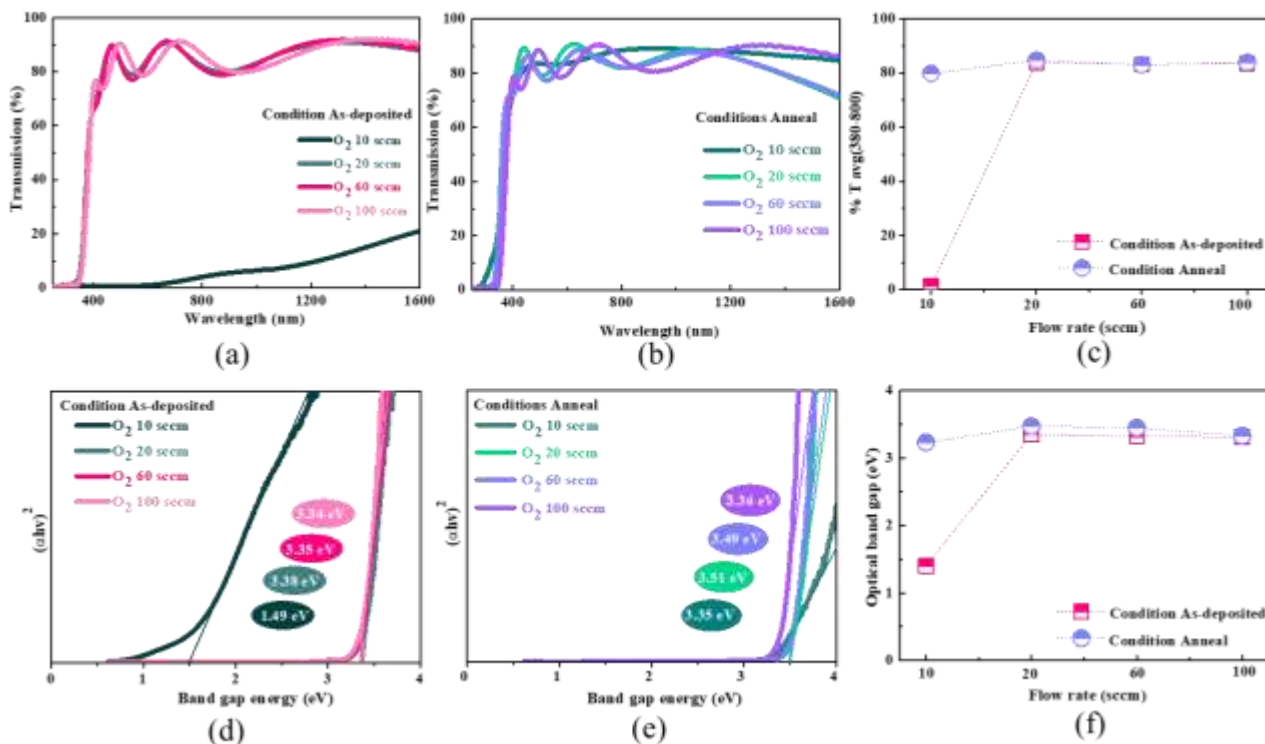


Fig. 4 (a, b) Optical transmittance spectra, (c) average transmittance in the visible region (380 – 780 nm), (d, e) Tauc plots for optical band gap determination, and (f) optical band gap energies of AZO thin films deposited on glass substrates under different processing conditions: as-deposited and after annealing at 400 °C for 2 h.

The transition from an insulating as-deposited state to a highly conductive state after vacuum annealing is driven by three primary mechanisms. First, thermal energy at 400 °C facilitates the substitutional incorporation of Al^{3+} ions into Zn^{2+} lattice sites, thereby activating them as effective donors [30]. Second, annealing in a vacuum environment promotes the desorption of chemisorbed oxygen species from the grain boundaries. This process reduces the potential barrier height for charge carriers, leading to the observed increase in Hall mobility [31]. Finally, the improved crystallinity and reduced structural defects, as confirmed by GIXRD, further minimize carrier scattering. Although the electrical resistivity obtained in this study ($4.00 \times 10^{-3} \Omega \text{ cm}$) is higher than some values reported in previous researches that utilized expensive ceramic targets, the use of a metallic Zn/Al target in this work offers significant advantages in terms of material cost-effectiveness and higher target durability, which are critical for industrial-scale applications.

Overall, the experimental results consistently show that the oxygen flow rate is the key parameter controlling the structural, optical, and electrical properties of AZO thin films deposited using a Zn/Al metallic target. FE-SEM and GI-XRD results confirm a transition from metallic or poorly crystallized films at low oxygen flow to well-crystallized wurtzite AZO films at optimized oxygen conditions, which is further enhanced by post-annealing. This structural improvement leads to high optical transparency and stable electrical conduction, with the lowest resistivity achieved at an oxygen flow rate of 20 sccm. The electrical performance obtained in this work is comparable to previously reported AZO films prepared using ceramic targets, demonstrating the effectiveness of oxygen flow control and post-annealing in producing high-performance AZO transparent conducting films.

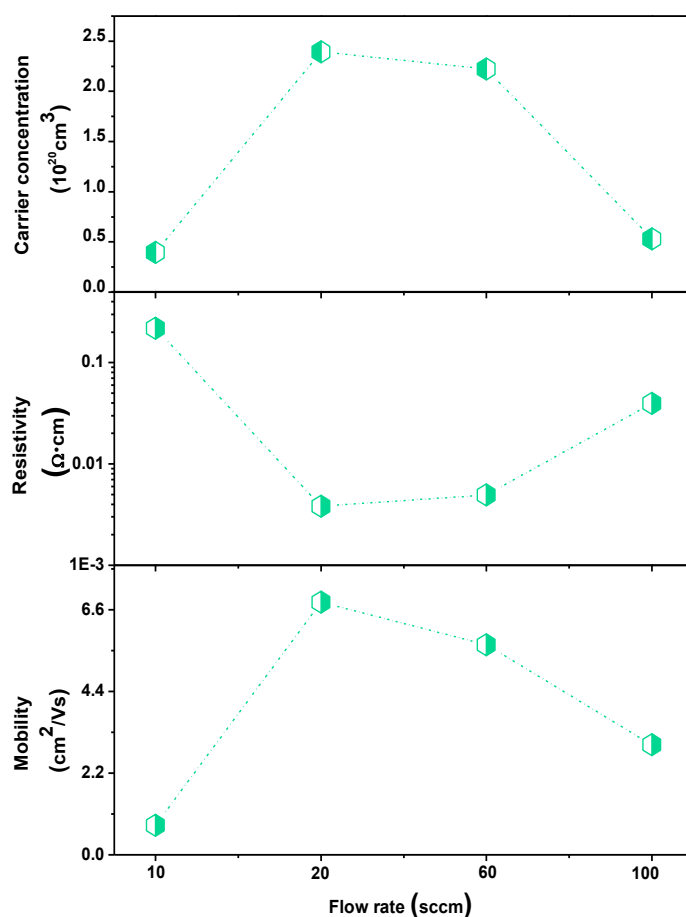


Fig. 5 Hall-effect parameters of AZO thin films, including carrier concentration, electrical resistivity, and Hall mobility, as a function of oxygen flow rate.

Table 1 Comparison between Previous AZO Studies and This Work.

No.	Process	Target	Thickness (nm)	Resistivity ($\Omega \cdot \text{cm}$)	Ref.
1	RF-Sputtered (Annealing)	ZnO-Al (2 wt%)	~ 300	1.05×10^{-3}	[1]
2	DC-Sputtered (GLAD)	ZnO-Al (2 wt%)	~ 200	5.80×10^{-3}	[8]
3	HiPIMS- Sputtered (Duty cycle)	ZnO-Al (2 wt%)	~ 400	3.00×10^{-3}	[10]
4	DC-Sputtered (Substrate Temp.)	ZnO-Al (2 wt%)	~ 200	1.78×10^{-3}	[17]
5	DC-Sputtered (Oxygen flow rate)	Zn/Al	~ 400	3.83×10^{-3}	This work

4. Conclusion

AZO thin films were successfully deposited using DC magnetron sputtering with a Zn/Al metallic target by systematically controlling the oxygen flow rate and post-annealing conditions. The results demonstrate that the oxygen flow rate plays a critical role in governing the structural, optical, and electrical properties of the films. At low oxygen flow rates, the films exhibit metallic or poorly crystallized characteristics, while increasing the oxygen flow promotes the formation of well-

crystallized wurtzite AZO structures, which are further enhanced by thermal annealing. Among the investigated conditions, an oxygen flow rate of 20 sccm combined with annealing at 400 °C yields the optimal performance, providing high optical transparency, enhanced carrier concentration and mobility, and the lowest electrical resistivity. The electrical properties obtained in this work are comparable to those of AZO thin films prepared using conventional ceramic targets and advanced sputtering techniques. These findings confirm that precise oxygen flow control, together with post-annealing, is an effective strategy for fabricating high-performance AZO transparent conducting oxide thin films suitable for optoelectronic applications. Furthermore, this research provides a cost-effective pathway for TCO production by successfully employing metallic Zn/Al targets instead of conventional ceramic ones, offering a practical guideline for large-area industrial coatings where cost-efficiency is a primary concern.

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