

Room temperature preparation of high performance AZO films by MF sputtering

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Abstract

Aluminum-doped zinc oxide (AZO) thin films have been deposited by MF magnetron sputtering from a ceramic oxide target without heating the substrates. This study has investigated effects of sputtering power on the structural, electrical and optical properties of the AZO films. The films delivered a hexagonal wurtzite structure with (002) preferential orientation and uniform surface morphology with 27–33 nm grain size. The results indicate that residual stress and grain size of the AZO films are dependent on sputtering power. The minimum resistivity of $7.56 \times 10^{-4} \Omega \text{ cm}$ combined with high transmittance of 83% were obtained at deposited power of 1600 W. The films delivered the advantages of a high deposition rate at low substrate temperature and should be suitable for the fabrication of low-cost transparent conductive oxide layer.

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1. Introduction

As an important transparent conductive oxide (TCO) material, ZnO based film has been widely applied in solar cells due to its excellent optoelectronic properties with the advantages of low cost, good stability and environmental friendly [1]. Generally, Al, F and Ga doped ZnO are considered as alternatives for thin-film transparent electrode applications, in which Al doped ZnO (AZO) films are regarded as more promising materials with low resistivity in order of $10^{-4} \Omega \text{ cm}$ [2,3]. Numerous studies have proved that the performances of AZO films are dependent on their preparation technology.

Park et al. investigated the influences of growth condition (oxygen pressure and substrate temperature) on

structure and properties of AZO films which prepared by pulse laser deposition [4]. The results showed that the resistivity decreased with the increasing of oxygen pressure and deposition temperature, respectively. Musat et al. discussed effects of Al dopant concentration, heating and annealing treatment on microstructure as well as the optoelectronic properties of ZnO thin films by the sol–gel method [5]. The film exhibited the excellent transmittance of 95% and the minimum resistivity of $1.3 \times 10^{-3} \Omega \text{ cm}$. In addition, AZO films can also be prepared by magnetron sputtering technology including direct current (DC), mid-frequency (MF) and radio frequency (RF) sputtering. Ting and Tsai [6] studied the optoelectronic properties of AZO films prepared by DC reactive sputtering using metallic targets, indicating that the resistivity values are dependent on the oxygen partial pressure and Al doping levels.

Among these fabrication techniques, magnetron sputtering has an attractive attention because of its high deposition rate, no toxic gas emission and easy to expand to large scale glass substrate. However, the utilization of metallic or alloy targets in reactive atmosphere hinders the control

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of structure and composition of AZO films by DC and MF reactive sputtering, therefore limits its optoelectronic properties. In the case of RF sputtering using a ceramic target, the films can yield a good quality and accurate composition whereas delivering a low deposition rate. Moreover, substrate heating or post annealing treatment above 200–500 °C are necessarily employed to achieve high transparent and conductive ZnO based films [7,8]. However, further development of the optoelectronic devices such as light emitting diodes (LEDs) and flexible displays requires large area deposition with high efficiency as well as simplified strategy [9,10].

As a result, the preparation of AZO films with high deposition rate and low deposition temperature is now considered as an important issue. In this study, we prepared the AZO films at room temperature by MF magnetron sputtering from a ceramic oxide target and investigated effects of sputtering power on its optoelectronic performance.

2. Experimental

Transparent conductive Al doped ZnO thin films were deposited on glass substrate by mid-frequency (MF) magnetron sputtering (SHENGPU sputtering source consisting of two cathodes operating at 40 kHz). Glass substrates were ultrasonically cleaned with distilled water, acetone and ethanol. ZnO containing 2 wt.% Al_2O_3 in dimension of 300×75 mm was used as a sputtering target. Chamber vacuum was evacuated to a base pressure of 4×10^{-3} Pa. The films grew at the pressure of 0.1 Pa in ambient of Ar and the sputtering power was varied from 120 to 1800 W. In present experiment, substrates were not heated and hence the temperature was considered as 30 °C. In order to uniform the film thickness, deposition duration was changed from 5 to 15 min based on sputtering power. Before deposition, the substrates were plasma etched for 5 min by ion source at power of 300 W and bias voltage of 800 V.

The microstructure of the as-deposited films was characterized by X-ray diffraction (XRD) using a Philips X'pert MPD diffractometer. Thickness measurement was carried by a 6JA photic interferometer. To facilitate the thickness measurement, a film sidestep on the substrate was necessary. We firstly marked the substrate with white-out before film growth and then eliminated it by acetone after the deposition process. Cross-sectional and surface morphology were assessed by field-emission scanning electron microscopy (SEM) using Nova NanoSEM 430. The film composition was performed by an energy dispersive spectrometer (EDS) using Bruker XFlash 5010 operating at EHT of 20 kV. Optical measurements were determined by a SP-752PC spectrophotometer and photon wavelength ranged from 300 to 800 nm. The electrical resistivity was measured by a four-point probe instrument at room temperature.

3. Results and discussion

XRD patterns of the as-deposited AZO thin films at various sputtering power was given in Fig. 1, from which only diffraction peaks of (002) and (004) located at $2\theta = 34^\circ$ and 72° were detected. XRD results are in good agreement with the standard wurtzite ZnO (JCPDS 79-0208). All films are highly oriented with their crystallographic c -axis perpendicular to the substrate. The (002) preferred growth is caused by the lowest surface free energy. Single phase ZnO with the absence of Al peaks in XRD patterns indicates that Al is well dissolved in ZnO lattice. Furthermore, (002) and (004) peaks become sharper and more intense with the increasing of sputtering power, attributing to the enhancement of the crystallization of the films. However, further increasing deposition power to 1800 W lead to a reduction of the (002) peak intensity. It is known that high power can degrade the preferred orientation, resulting in the weak diffraction intensity and poor optoelectronic properties [11]. Table 1 gives crystal parameters of the as-deposited AZO films at various sputtering power. The full width at half maximum (FWHM) in the radian of (002) peak firstly decreases and then increases. The average crystallite dimension of the AZO films is about 27–33 nm calculated from the FWHM according to Scherrer's formula [12].

It is worth to notice that 2θ values of (002) peak for all AZO films are smaller than that of standard ZnO. Generally, Al doped ZnO would lead to larger 2θ values according to solid solution principle [13]. It is attributed to smaller radius of Al^{3+} comparison with Zn^{2+} , resulting in the shrink of the ZnO crystal lattice and high angle shift of (002) peak position. We consider that the low angle shift of the (002) peak is mainly caused by the existence of residual stress in the films. For hexagonal crystals with a highly c -axis preferred orientation, the calculation of film stress is based on biaxial strain model [14]

$$\sigma_{\text{film}} = \frac{2c_{13}^2 - c_{33}(c_{11} + c_{12})}{2c_{13}} \frac{c_{\text{film}} - c_{\text{bulk}}}{c_{\text{bulk}}} \quad (1)$$

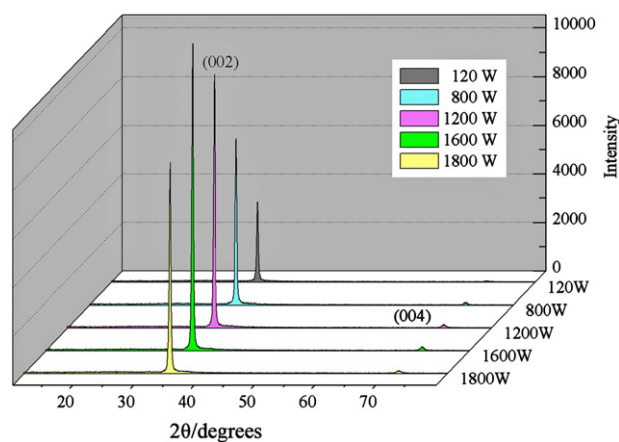


Fig. 1. XRD patterns of the AZO films deposited at various sputtering power.

Table 1
Crystal structure parameters of AZO film deposited at various sputtering power.

Power/W	(hkl)	$2\theta/^\circ$	d/nm	FWHM/ $^\circ$	Grain size/nm	Stress/GPa
120	002	34.128	0.2624	0.314	27.1	−1.339
800	002	34.212	0.2617	0.303	30.4	−0.714
1200	002	34.225	0.2616	0.290	31.6	−0.625
1600	002	34.241	0.2615	0.283	32.9	−0.536
1800	002	34.149	0.2622	0.313	29.3	−1.161

where C_{ij} is the elastic stiffness constant, $C_{11}=208.8$ GPa, $C_{33}=213.8$ GPa, $C_{12}=119.7$ GPa, $C_{13}=104.2$ GPa which obtained by single crystalline ZnO, C_{film} represents the lattice constant. Owing to the c -axis direction of the film strain $\varepsilon=(c_{\text{film}}-c_{\text{bulk}}/c_{\text{bulk}})$ measured by XRD, the inter-layer spacing d of the strained film is therefore substituted to c_{film} [15,16]. d_{bulk} is that in bulk ZnO (0.2609 nm) and the values of d_{film} were calculated using Bragg formula, $2d\sin\theta=n\lambda$.

According to Eq. (1), the stress values have been calculated and shown in Table 1. Negative sign for calculated stress means that the films are under compressive stress. With the increase of sputtering power, the values of compressive stress decrease from 1.339 GPa to 1.161 GPa with a minimum of 0.536 GPa at 1600 W. There are several reasons for the existence of stress: (1) physical characteristics such as lattice mismatch and the difference in thermal expansion coefficient between amorphous substrates and the film, (2) deposition under non-equilibrium conditions and (3) the dopant and composition gradient across film surface. Furthermore, Vinodkumar et al. believe that lost of the lattice periodicity at the interfaces or film surface can result in surface stress during the deposition process [17].

From XRD results, it can be concluded that film structure is mainly dependent on sputtering power. Lower lattice stress and good crystallinity were obtained when the film deposited at power of 1600 W. In our experiment, the stress variation in the films can be explained by atomic peening effect, which plays an important role in compressive stress for films prepared by magnetron sputtering [18]. High discharge power provides more sputtered particles and collides the substrate with high energy, indicating compressive stress is mainly caused by the growth process itself. However, the reduction of compressive stress was observed at high sputtering power of 1600 W. This is attributed to the combined effect of grain size and film thickness [19]. Larger grain size is helpful for the release of compressive stress, in which atoms diffuse rapidly from the compressively strained areas to tensile grain boundary regions [20]. Thus, the direction of stress could be changed when sputtered at high discharge power. Furthermore, increasing thickness can also cause the decrease of compressive stress [21]. As a result, the film exhibits the lowest compressive stress at 1600 W. After further increasing sputtering power to 1800 W, the high kinetic energy of atoms would induce severe structural defects and lattice distortion in the film.

Fig. 2 shows the influence of sputtering power on morphology of the AZO films. The as-deposited films are composed by close-packed nano-particles without regular geometric form, exhibiting uniform and dense surface morphology. The particle size grew with the increase of sputtering power and the results matched well with XRD analysis. However, some small and disordered particles were observed in the interface of large grains for the film deposited at 1800 W as shown in Fig. 2e. It indicates the film was partly damaged and the grains agglomerated or shrunk into small clusters. It is believed that enough sputtering power and material supply are essential to the large grains. When film deposited at power of 120 W, low kinetic energy of the atoms would obstruct grain growth of textured film. Larger gains can be obtained after increasing sputtering power due to the improvement of kinetic energy of atoms. However, the overspeed atoms probably disorder the interface layer at AZO/substrate interface and destroy the preformed particles.

Thickness of the as-deposited films is in the range 243–378 nm as a function of deposition power as given in Table 2. The cross-sectional morphology of the AZO film deposited at 1600 W is shown in the inset of Fig. 2d. The film is very dense with a columnar grained structure perpendicular to substrate. The thickness was measured as 390 nm, very closing to 378 nm obtained by a photic interferometer. The result confirms the reliability of thickness value measured by a photic interferometer. Al content in the film was detected as 1.2 wt% and the ratio of Al/Zn was 3.6 at.% which is slightly higher than that of the target. The similar result was also observed by Cai et al. [22]. The increase of Al content could be attributed to the fact that the sputtering threshold energy of Zn atoms is lower than that of Al atoms, leading to Zn atoms deposited on substrate can be easily resputtered by the negative ions [22,23].

Fig. 3a illustrates the transmission spectra of the AZO thin films deposited at different sputtering power, from which the evident variation was not observed. The average transmittances of the films at optical wavelength region of 400–800 nm are given in Table 2. The transmittances retain about 83% when the films deposited lower than 1600 W. Fig. 3b shows plot of α^2 as a function of photon energy of the AZO films. As a direct band gap n-type semiconductor, the optical energy gap (E_g) of the AZO films can be determined by the following equation which extrapolated

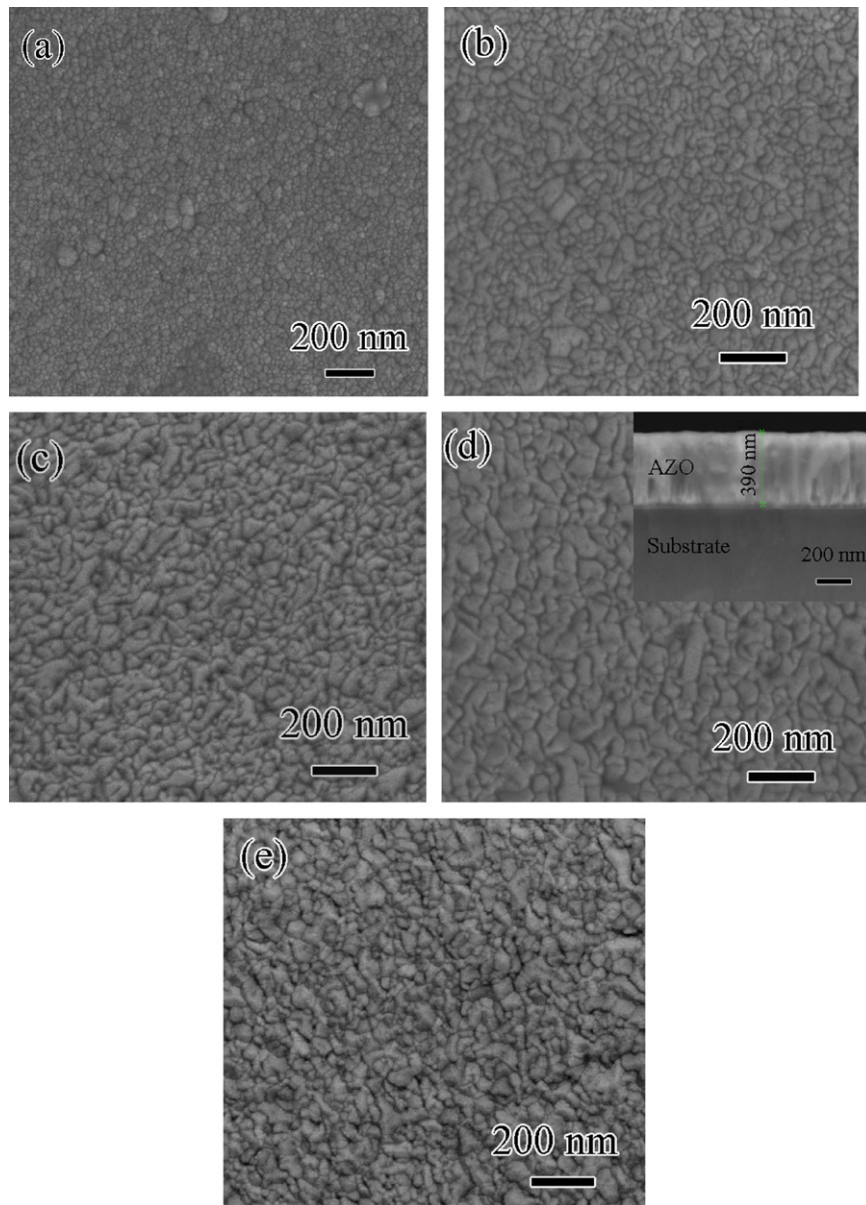


Fig. 2. SEM images of the AZO films deposited at different sputtering power (a) 120 W, (b) 800 W, (c) 1200 W, (d) 1600 W and (e) 1800 W.

Table 2
Performances of AZO film deposited at various sputtering power.

Power/W	Al concentration/wt%	Al/Zn/at%	Thickness/nm	Resistivity/ Ω cm	Average transmittance/%	Band gap/eV
120	1.6	4.9	243	1.15×10^{-3}	86	3.70
800	1.6	4.9	270	8.02×10^{-4}	84	3.85
1200	1.1	3.3	351	7.72×10^{-4}	83	3.88
1600	1.2	3.6	378	7.56×10^{-4}	83	3.91
1800	1.2	3.4	324	8.16×10^{-4}	84	3.79

from the absorption edge [24]:

$$\alpha = A(h\nu - E_g)^{1/2} \quad (2)$$

where α is the absorption coefficient, A is α constant dependent on the electron–hole mobility, $h\nu$ is the photon

energy which represents the Planck's constant and radiation frequency, respectively. To obtain E_g , absorption coefficient α should be firstly calculated by the formula [25]

$$\alpha = -\frac{\ln(T/100)}{d} \quad (3)$$

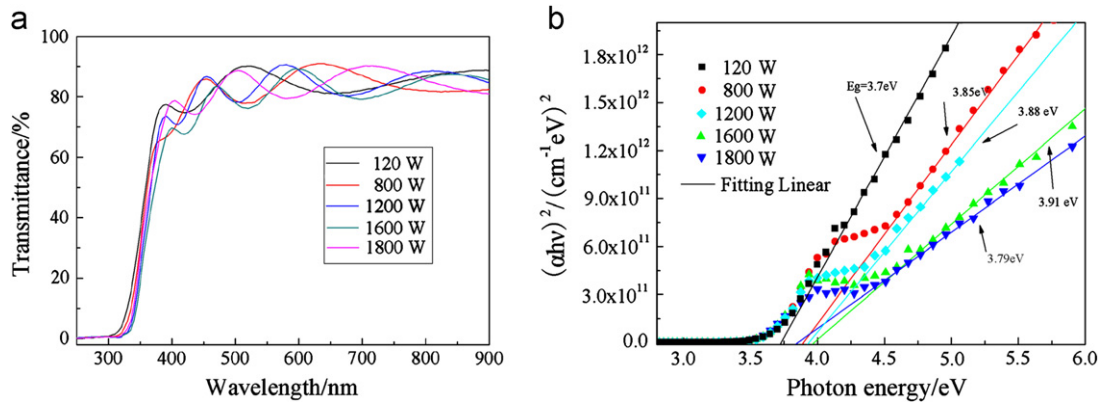


Fig. 3. (a) Plots of optical transmittance for the AZO films prepared at different sputtering power and (b) α^2 vs. photon energy plots of the AZO films.

where T is the optical transmittance and d is thickness of the film. According to eq. (2), the photon energy is equal to E_g when α is zero. The energy gap therefore can be obtained from the intercept of the linear absorption edge portion to the energy axis. The optical band gaps are 3.7, 3.85, 3.88, 3.91 and 3.79 eV which deposited at 120, 800, 1200, 1600 and 1800 W, respectively. Higher band gap of the present AZO film comparison with pure ZnO film (3.37 eV) is attributed to the Burstein–Moss effect caused by the increased free electron concentration after Al³⁺ doping.

Fig. 4 presents the sputtering power dependence of the resistivity for the AZO films. The resistivity varies from 1.15×10^{-3} to $8.16 \times 10^{-4} \Omega \text{ cm}$ and the minimum of $7.56 \times 10^{-4} \Omega \text{ cm}$ is appeared at 1600 W. As up-mentioned, deposition power plays an important role in film growth and affects its properties. It is quite clear that the film sputtered at 1600 W delivers the best crystal growth with low residual stress and large grain size. Generally, the resistivity of doped ZnO films is decided by the doping concentration, O vacancies, grain boundaries, residual stress and ionized impurity scattering [26]. In our case, the improvement of the resistivity as a function of sputtering power is impossible caused by Al content according to EDS analysis in Table 2. Thus, morphology and residual compressive stress seem to be the key factors for AZO film electrical performances. As shown in Table 1, residual compressive stress decreases with the increase of sputtering power. Li et al. suggest that internal stress suppresses the electronic conduction and its release can enhance the electron mobility [27]. Stress will distort the unit cell and introduce crystal defects, resulting in the decrease of carrier concentration [28,29]. According to optoelectronic analysis, it would conclude that high compressive stress leads to a low optical band gap and a high resistivity. Furthermore, the good preferential c -axis orientation and larger grain size produce less grain boundary, reducing the carriers scattering at the grain boundaries and crystal defects. As a result, the electrical property of the AZO film is improved at 1600 W.

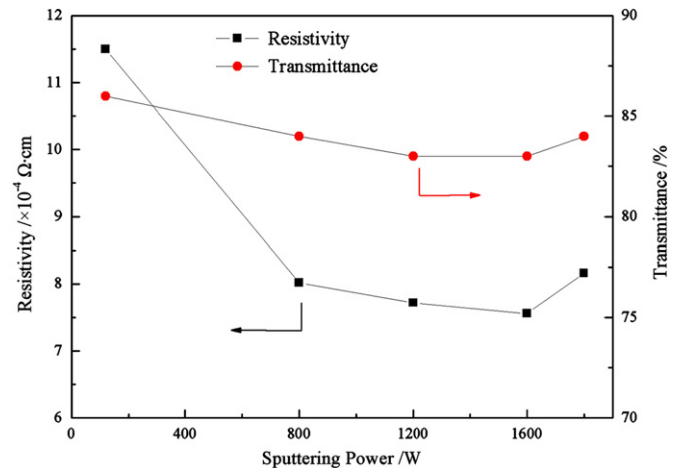


Fig. 4. Resistivity and transmittance of the AZO films as a function of sputtering power.

In this study, the AZO films were deposited by MF sputtering at room temperature using a ceramic target. The characteristics of the AZO films prepared by different deposition technology are so different and their comparisons are given in Table 3. Studies show that AZO films can be prepared at room temperature, however delivered the poor electrical performance [30]. MF sputtering technology has been employed to prepare AZO films, reaching a low resistivity of $3.5 \times 10^{-4} \Omega \text{ cm}$. Nevertheless, the film was growth at high temperature of 310 °C, limiting the wide application of TCOs [31]. In our study, high deposition rate of 54 nm/min and a low resistivity of $7.56 \times 10^{-4} \Omega \text{ cm}$ at room temperature were obtained. The present study can refrain from low deposition rate when using a ceramic target and guarantee the superior electrical performance even prepared at room temperature. As the appeal of wild applications for TCO films, low temperature and high rate deposition technology come to be an urgent requirement. Table 3 proved that the present deposition technique is superior to others [31–33]. Considering the suitable preparation procedure and good optoelectronic properties in this study, we believe that the MF sputtering

Table 3

Comparison of electrical performance for AZO films obtained at various deposition conditions.

Sample	Method	Target	Deposition rate/nm min ⁻¹	Temperature/°C	Resistivity/Ω cm
[30]	RF	AZO ceramic	–	Room temperature	10 ⁻²
[31]	MF	Zn/Al	40	310	3.5 × 10 ⁻⁴
[32]	RF	AZO ceramic	27.5	250	5.63 × 10 ⁻⁴
[33]	RF	AZO ceramic	18	Room temperature	20 × 10 ⁻⁴
Our	MF	AZO ceramic	54	Room temperature	7.56 × 10 ⁻⁴

technology can be used to prepare high performance AZO films on flexible substrate. Further research is still under discussion and will be a valuable area for TCO films.

4. Conclusions

The structural, optical and electrical properties of the AZO films were studied using MF magnetron sputtering method at room temperature. Sputtering power was optimized with the purpose of achieving highly transparent and conductive films from a ceramic target. All of the deposited films delivered a *c*-axis preferred orientation with grain size ranging from 27 to 33 nm. Results showed that compressive stress and grain size were evidently dependent on sputtering power. As a result, an improvement of the structural, optical and electrical properties was achieved at deposition power of 1600 W. The deposition technique with good optoelectronic performance, high deposition rate and low substrate temperature have demonstrated that MF sputtered AZO films might be suitable for application in low cost and flexible optoelectronic devices.

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