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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 $7.56 \times 10^{-4}\,\Omega$ 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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Keywords: Electrical properties; Al-doped zinc oxide; MF Sputtering; Transmittance

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 $10^{-4}\,\Omega$ 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 $1.3 \times 10^{-3}\,\Omega$ 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]

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

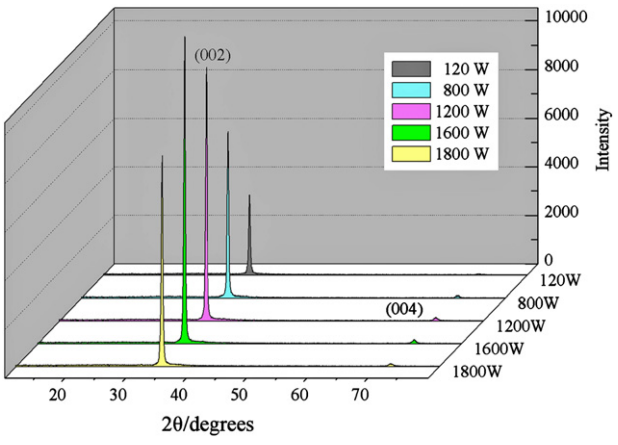

Fig. 1. XRD patterns of the AZO films deposited at various sputtering power.

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