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Indium tin oxide thin films prepared by dc magnetron sputtering for transparent heating

Jianhua Li[1, 2], Xueshen Wang[2], Pingwei Lin[2], Xinhua Chen[3], Longfa Zhang[3], and Shuo Liu[3]

[1]*Yanshan Branch of Beijing Research Institute of Chemical Industry, Sinopec, Beijing, China*
[2]*National Institute of Metrology, Beijing, China, wangxs@nim.ac.cn*
[3]*Beijing Key Laboratory of Performance Guarantee on Urban Rail Transit Vehicles, University of Civil Engineering and Architecture, Beijing, China*

***Abstract* – This paper reported a research on conducting tin-doped indium oxide (ITO) films fabricated by dc magnetron sputtering for transparent heating. ITO films with a thickness of 130 nm were deposited on BF33 glass substrates, followed by an annealing process in N_2 atmosphere for 2 h at 450 ℃. The crystal structure, surface morphology, optical and electrical properties of ITO films were characterized. The predominant crystal face orientation was (222). The transmittance from 400 nm to 800 nm was 92.5%. The sheet resistance and resistivity increased, and the temperature coefficient of resistance (TCR) decreased with the larger oxygen flow rate. A low TCR and proper resistivity were the key factors for the application of conducting transparent heating.**

Keywords – ITO, Dc sputter, Transparent, Conducting

I. INTRODUCTION

Due to the low resistivity and high transmittance within the visible light range, ITO films as conducting transparent heating materials have been widely used in the optoelectronic fields [1-4]. As the heating unit trends to millimetres even micrometres scale (e.g., chip scale atomic clocks, chip scale atomic magnetometers and MEMS device fields) [5-8], ITO films with designed resistance and structures are becoming more and more necessary.

A variety of techniques have been used to fabricate ITO films such as dc [9-11] and rf [12] magnetron sputtering, electron beam evaporation [3], sol-gel [13], and so on. Magnetron sputtering was most widely used due to the good film adhesion, uniformity, reproducibility, and the compatibility for the micro-fabrication. ITO films were formed by Ar/O_2 reactive magnetron sputtering of an In-Sn alloy target followed by an annealing process to improve the crystallization [10, 13-14]. X-ray diffraction (XRD), surface morphology, optical transmission, resistivity and TCR were the mainly characterization methods.

In this work, ITO films were fabricated on BF33 glass substrates by dc magnetron sputtering with different O_2 flow rates, and annealed in N_2 at 450 °C for 2 h. Then the films were characterized in detail.

II. EXPERIMENTS

The BF33 glass substrates which provided convenience for the optical transmission characterization were cleaned ultrasonically in acetone, isopropyl alcohol (IPA) for 5 mins and then dried by N_2. An alloy target (In_2O_3-SnO_2, 90-10 wt.%) was used as the sputtering source. The base pressure of the sputter chamber was lower than 1.0×10^{-5} Pa. High purity Ar (30 sccm) and O_2 (0.2 sccm-1.8 sccm) were then introduced and the pressure was kept to be 0.3 Pa. The dc sputter power was 100 W, and the thickness of the ITO films was controlled to be 130 nm by adjusting the sputtering time. The annealing process was performed in a home-made thermal system with N_2 flow for 2 h at 450 ℃.

The crystallographic structure of the films was measured with Cu $K\alpha$ radiation (λ = 1.5418 Å) and the 2θ range from 10° to 70°. The morphology and surface roughness were characterized by atomic force microscope (AFM). Optical transmittance of the films was achieved by a spectrophotometer within the visible light range from 400 nm to 900 nm. The sheet resistance ($R_{\square}$), the resistivity (ρ) and TCR were measured using a four-point probe method.

III. RESULTS AND DISCUSSION

A. XRD pattern

The XRD patterns of the ITO thin films for different flows of O_2 are shown in Fig .1 from which we can see that all the samples present (222) and (400) crystal orientations and (222) is the preferential one which will benefit the optical transmittance. The shift of the (222) peak positions maybe due to the stress of the ITO films [15].

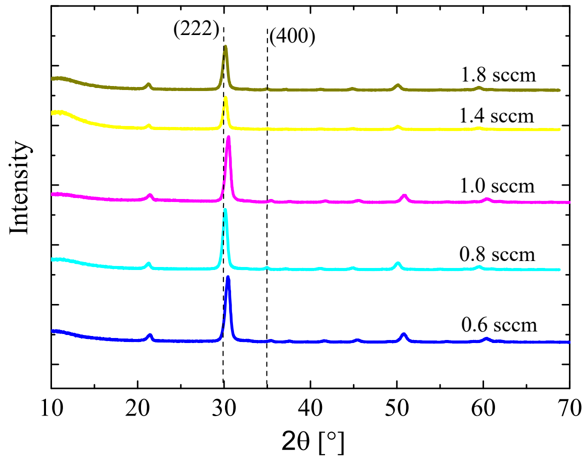

Fig. 1. XRD patterns of ITO films for different flow rates of O_2.

The crystal size of the (222) crystal orientation D_{222} and intensity ratio $I_{(222)}/I_{(400)}$ are shown in Fig. 2. The crystal size changes a little as the flow of O_2 below 1 sccm and the intensity ratio becomes smaller. According to the crystal size and intensity ratio, 1.4 sccm is the proper O_2 flow rate with a consideration of the precise controlling.

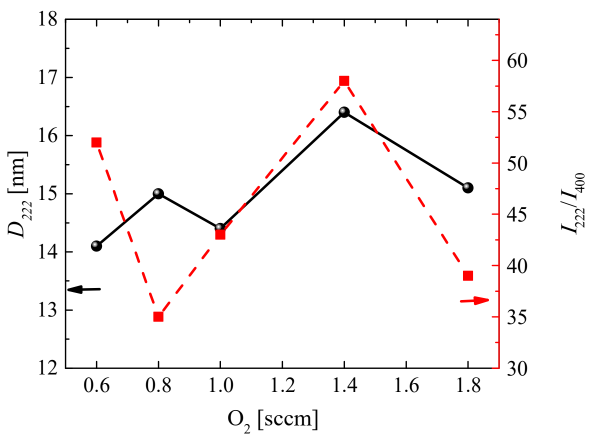

Fig. 2. The Crystal size D_{222} and intensity ratio $I_{(222)}/I_{(400)}$ of ITO films for different flow of O_2.

B. Surface morphology characterization

The surface morphologies characterized by AFM in a 2 μm×2 μm scale are shown in Fig. 3. The surface roughness R_q becomes larger as the flow rate of O_2 increases to 1.4 sccm and then decreases. The particle size shows the same trends as the R_q and is obviously large at 1.4 sccm. The particles aggregates and make the R_q larger. This also will raise the resistance.

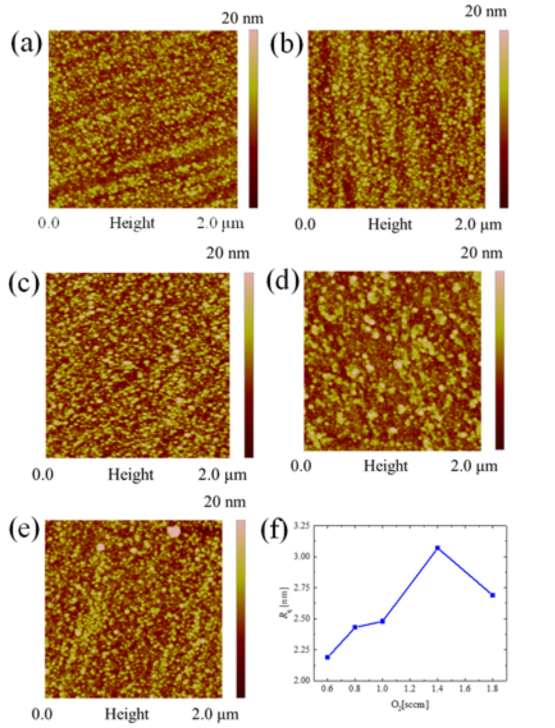

Fig. 3. The AFM images of ITO thin films. (a) 0.6 sccm; (b) 0.8 sccm; (c)1.0 sccm; (d)1.4 sccm; (e) 1.8 sccm; (f) the surface roughness.

C. Optical properties

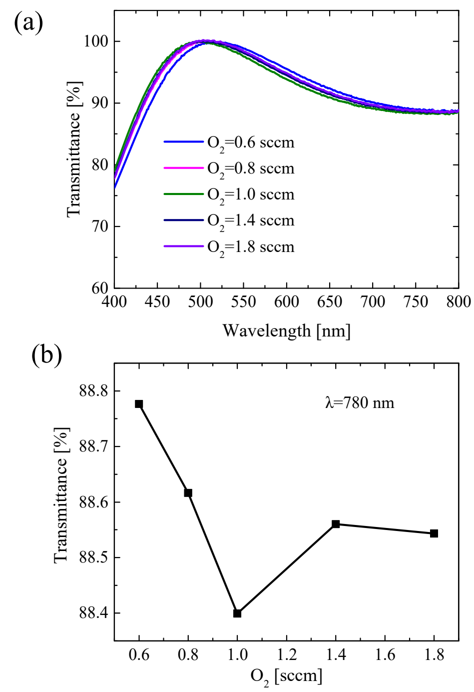

Fig. 4. (a) Transmittance spectra of ITO thin films. (b) Transmittance at 780 nm.

Optical properties are the most important factors for the application of ITO films. The transmittance spectra from 400 nm to 800 nm are shown in Fig. 4(a). The wavelength with the highest transmittance is about 500 nm. The average value from 400 nm to 800 nm is above 92.5% for all the flow rates of O_2.

Specifically, the application of ITO films on MEMS chip scale atomic clock (CSAC) attracted great attentions due to the conducting transparent property. For the CSAC using Rb^{87}, the characteristic wavelength is 780 nm. Results from the corresponding transmittance is demonstrated in Fig. 4(b). The transmittance, above 88.4%, is favourable in the transparent heating application of the Rb^{87} vapor cell [6].

D. Electrical conductivity

Fig. 5 shows the sheet resistance ($R_□$) and resistivity (ρ) of the ITO films. The $R_□$ and ρ do not change remarkably and show the same trends because there is a little difference in the thicknesses for different flow rates of O_2. The largest $R_□$ and ρ are 38.9 Ω/□ and 5.4 × 10^{-4} Ω·cm for 1.4 sccm O_2, respectively, which is consistent with the surface roughness. Both the $R_□$ and ρ are comparable with the values in [16] and are in the suitable scale for electro-optic applications.

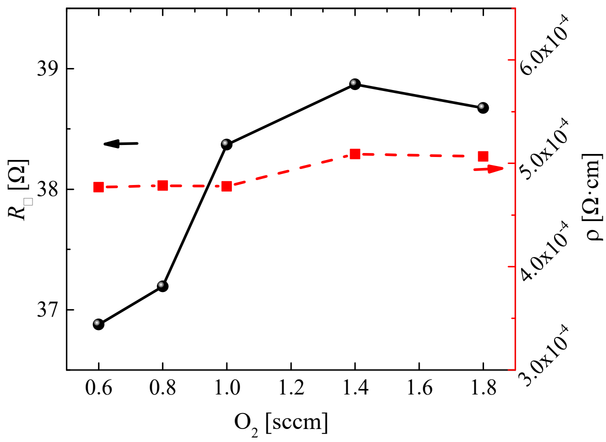

Fig. 5. The sheet resistance and resistivity of ITO thin films.

TCR was an important factor for the application of heating. A small TCR meant that the resistance changed small as the temperature increased, and could ensure the stability of the heating power. The TCR of the deposited ITO films was only from 340 ppm/K to 290 ppm/K as shown in Fig. 5.

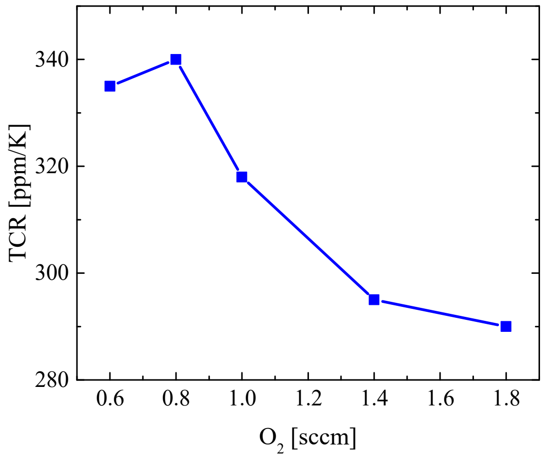

Fig. 5. The TCR of ITO thin films.

IV. CONCLUSIONS

In this paper, ITO thin films were deposited on BF33 glass substrates by dc magnetron sputtering with different flow rates of O_2 and annealed at 450 °C. The ITO films showed a polycrystalline structure and the predominant crystal face orientation was (222). The crystal sizes of ITO thin films were between 14.1 nm and 16.4 nm. The surface roughness (R_q) was from 2.43 nm to 3.07 nm. The average transmittance was above 92.5% for the visible light and was above 88.4% for the characteristic wavelength 780 nm of Rb^{87} vapor cell CSAC application of the Alkali vapor cell heating. The sheet resistance and resistivity were in suitable scale for the electro-optic application. The TCR was also small enough for the heating application. In conclusion, the deposited ITO films were applicable for conducting transparent heating.

V. ACKNOWLEDGEMENT

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