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Atomic oxygen irradiation resistance of transparent conductive oxide thin films

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ABSTRACT

One set of tungsten-doped indium oxide thin films (In_2O_3:W, IWO) and another set of tin-doped indium oxide thin films (In_2O_3:Sn, ITO) were prepared on glass substrates by radio frequency (RF) reactive magnetron sputtering method. The as-deposited IWO and ITO films have resistivity values at a level of 10^{-4} Ω•cm. Average transmittance values of these films are above 85% in visible-light region as well as in near-infrared region. All these films were irradiated by atomic oxygen (AO) with different amount of flux in a ground-based simulation system close to the environment of low Earth orbit. Changes in characteristics including surface morphology, mechanical properties, optical and electrical properties were compared between IWO and ITO films after AO irradiation. The effects of AO irradiation on transparent conductive oxide thin films were analyzed. As a result, AO has influences on ITO and IWO thin films through the way of oxidation and erosion. Both ITO and IWO films possess suitable anti-AO properties. IWO films are more appropriate as AO protective coatings due to their compact microstructure, the coexistence of W^{4+} and W^{6+} ions in their chemical systems, and incremental WO_3 protective layer under AO oxidation.

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1. Introduction

Transparent conductive oxide (TCO) thin films are widely used in our daily life and industry. Typical applications include solar cells, solar collectors, liquid crystal displays, and electrochromic glasses [1–5]. In previous reports, efforts were focused on optimizing production parameters [6–8], finding new methods of fabrication or modification [9–11] and investigating novel materials [12–14], in order to achieve best optical and electrical properties and extend applications to near-infrared region (NIR) as well as deep-ultraviolet (UV) region.

As the most prevalent kind among TCO films, tin-doped indium oxide (In_2O_3:Sn, ITO) thin films are widely employed in industrial production for their superiority in optical and electrical properties. However, ITO films are susceptible in hydrogen plasma [15–16], sensitive to high temperature and humidity [17–18]. Moreover, transmittance curve of ITO film declines evidently in infrared range longer than 1200 nm, which means a large part of solar radiation energy cannot be used efficiently. Compared with ITO films, tungsten-doped indium oxide (In_2O_3:W, IWO) thin films possess better optical properties in NIR region [14]. Due to a high valence gap between W^{6+} ions and In^{3+} ions, larger than that of ITO films, IWO films possess higher carrier mobility than ITO films with the same carrier concentration. Consequently, IWO films have potential capabilities to substitute ITO films in practical applications.

With rapid development of technology, more scientific and technical activities are carried out in space. Low Earth orbit (LEO, 200– 700 km) is the most important area for space exploration, including the operation of space station, spacecraft and satellite. In LEO, atomic oxygen (AO) is the decisive role derived from the dissociation of molecular oxygen under the function of solar radiation. The recombination rate of AO is extremely low due to the high vacuum environment. It is investigated from the kinetics of AO reactions that under the same pressure, AO possesses 10 times of oxidation rates than diatomic oxygen (O_2), and AO is easier to have chemical adsorption, which also increases the rate of chemical reactions [19]. AO has erosion and pollution effects on space shuttles due to its strong oxidability and a relative speed of 7– 8 km/s to the forward facing surfaces of space shuttle with energy of nearly 5 eV [20]. For longer period of reaction time, AO can have destructive damage for lifetime degradation. The vulnerable parts are conductive or thermal control coatings. Researches on this filed involve some kinds of materials, mainly related to composite material polymers, inorganic substances, metals, and organic-inorganic hybrid materials, respectively. Oxidation and erosion effects of AO irradiation on these materials were already reported [21–26]. However, more materials employed in this field and various methods to increase the working stability under AO environment are to be explored.

In spacecrafts, ITO films can be used as anti-static discharge protective coatings, thermal control coatings and anti-AO coatings. It was indicated

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that ITO films own a certain degree of anti-irradiation properties [27–29], while they tend to be crisp when on service [30]. In our research, IWO films are speculated to be applied in this field for their similar optical and electrical properties to ITO films. Moreover, tungsten compounds were reported to be resistant to severe oxidation for the formation of compact WO_3 protective layer [31]. As far as can be seen, no relevant researches report the anti-AO irradiation properties of IWO films. Consequently, whether IWO films can substitute ITO films as AO protective coatings is still unknown. In this paper, anti-AO irradiation properties of ITO and IWO films are compared and analyzed, and some referential guides for material selection in spacecrafts are pursued.

2. Experimental details

2.1. Preparation of IWO and ITO films

IWO films and ITO films were prepared by radio frequency magnetron sputtering method. To obtain IWO films, a ceramic target (60 mm in diameter and 3 mm in thickness) made of indium oxide (purity: 99.99%) doped with 8 wt.% metal tungsten (purity: 99.99%) was used. Preparation of ITO films employed a ceramic target made of indium tin oxide (purity: 99.99%, tin doped amount: 6 wt.%) with the same size as IWO target. Soda-lime glasses were boiled in concentrated sulfuric and hydrogen peroxide with the volume ratio being 3:2 for 45 min, then cleaned with acetone, ethanol and deionized water successively in an ultrasonic device each for 15 min before used as substrates for ITO and IWO films. The substrate temperature was 25 °C, and distance between substrate and target was 85 mm. Vacuum chamber was evacuated to lower than $1*10^{-3}$ Pa with a mechanical pump and then a molecular pump before deposition. Sputtering gas was a mixture of Ar and O_2 during deposition for IWO films, and flow rates of argon and oxygen were 100 sccm and 0.6 sccm, respectively. Sputtering gas for ITO film was pure argon with a flow rate of 100 sccm. Working pressure was 1 Pa. Sputtering time and sputtering power was 25 min and 90 W, respectively for both ITO films and IWO films.

2.2. AO irradiation experiments

AO irradiation experiments were conducted with a filament discharging plasma-type atomic oxygen simulation facility built by Beihang University. AO flux was measured by Kapton-loss method according to the formula $f = M/\rho At\delta$, where the f, M, ρ, A and t are the AO flux, the mass loss of Kapton films, the density of Kapton films, the surface area receiving AO flux, the time for irradiation, respectively. δ represents the erosion rate of Kapton which is a constant of $3.0*10^{-24}$ cm^3/atom [20]. AO flux rate was determined to $3.3*10^{15}$ atoms/(cm^2 • s). The as deposited ITO and IWO films were both divided into five groups. One group as deposited without irradiation was used as a comparison, and the other four groups were irradiated with different accumulated flux. The flux values for each irradiated group were 1.2, 1.8, 2.4 and $3.0*10^{20}$ atoms/cm^2, respectively.

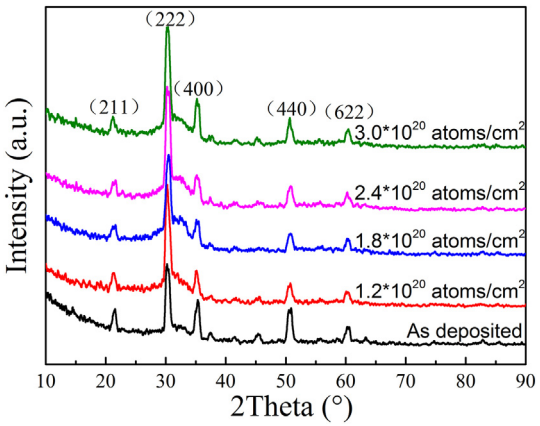

Fig. 2. XRD patterns of ITO films under different AO flux.

2.3. Characterization methods

The crystal structure was characterized by X-ray diffraction (XRD, Rigaku D/max 2500 pc, Japan) with Cu Kα radiation sources (k = 1.5405 Å), and the scanning angle ranged from 10° to 90°. Surface morphology was acquired by scanning electron microscopy (SEM, HITACHI S-4800). Thickness of the films was obtained by three-dimensional white light interferometer (MicroXAM, ADE phase-shift, US). Chemical state analysis of different elements in the films was carried out using an X-ray photoelectron spectroscope (XPS, ESCALAB 250Xi, Thermofisher, UK). Hall-effect measurement (ET-9000, East Changing Technologies Inc., China) was used to detect the electronic properties. Carrier concentration and carrier mobility were measured by ven der Pauw method, while the resistivity was tested by four-point probe method. Spectrophotometer (Jasco V-570) was employed to show the optical properties of the films in the wavelength range of 300– 1700 nm. Critical load of the films was achieved by nano scratch tester (NHT2, CSEM, Switzerland).

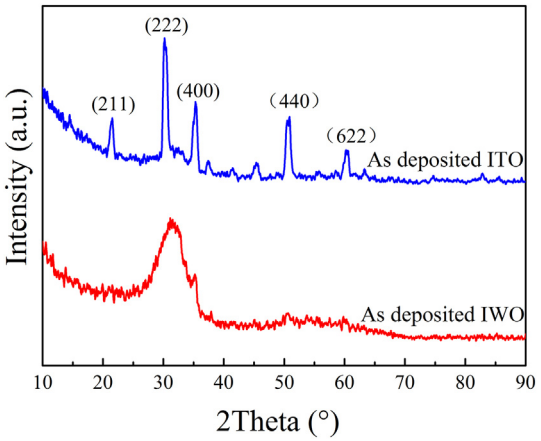

Fig. 1. XRD patterns of ITO and IWO films before irradiation.

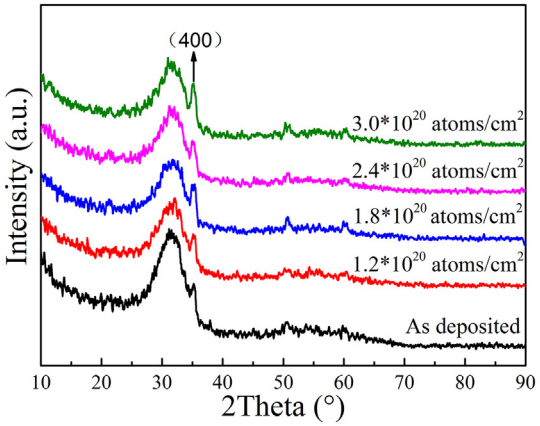

Fig. 3. XRD patterns of IWO films under different AO flux.

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