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Study on physical properties of indium-doped zinc oxide deposited by spray pyrolysis technique

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ABSTRACT

Transparent conducting indium doped zinc oxide (IZO) thin films have been deposited on soda-lime glass substrates by the spray pyrolysis technique. The structural, electrical, and optical properties of these films were investigated as a function of substrate temperature. In this work the substrate temperature was varied between 350 °C and 500 °C. X-ray diffraction pattern reveals that at 350 °C dominant peak is (100) orientation. By increasing substrate temperature from 350 °C to 450 °C, sheet resistance decreases, from 302 Ω/□ to 26 Ω/□, then at 500 °C increases to 34 Ω/□. In the useful range for deposition (i.e. 450 °C to 500 °C), the orientation of the films was predominantly (002). The lowest sheet resistance (26Ω/□) is obtained at substrate temperature of about 450 °C with the transmittance of about 75%. Study of scanning electron microscopy images shows that films deposited at 400 °C, have grain size as large as 574 nm, while with increasing substrate temperature to 450 °C, grain size becomes smaller and reaches to a value of about 100 nm with spherical shape. At 500 °C grain size value would be around 70 nm with the same spherical shape.

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1. Introduction

A fundamental role is played by highly transparent conducting oxide (TCO) thin films in photovoltaic technology. Materials such as tin oxide, indium tin oxide, and zinc oxide, have been extensively studied and applied as electrodes in thin film solar cells. These materials possess a wide energy band gap (E_g>3.4 eV) and are therefore transparent in the visible region, where the long wavelength cutoff depends on the charge carrier concentration [1]. TCO films can be prepared with resistivities in the region of 1 to 5×10^{-4} Ω cm [2].This makes these materials very well studied for transparent electrodes in flat panel displays(e.g. liquid crystal, electroluminescence and plasma displays), thin film solar cells, microwave oven windows, low emissivity glass thermal solar collectors, etc. ZnO films are particularly interesting because of their advantageous properties, such as low cost, non-toxicity and high stability after heat cycle during the fabrication process of amorphous silicon solar cells [3].

Zinc oxide (ZnO) thin films have emerged as an attractive option in the design of transparent electrodes in thin films solar cells due to the simultaneous occurrence of high transmittance in the visible region and a low resistivity [4].

Some other singular characteristics, such as the stability under strongly reducing environments [5], the n-type character and its piezoelectric effect [6], make ZnO thin films very suitable as gas sensors [7] and surface acoustic devices [8]. For many of these applications it is very important to control the ZnO physical properties by doping. Usually, n-type doping is obtained by Al, Ga or In. On the other hand, p-type doping is not easily obtained. A wide-variety of physical and chemical methods such as reactive evaporation [9], sputtering [10], laser ablation [11], sol–gel [12] and spray pyrolysis [13], have been used in the deposition of highly conductive and transparent ZnO thin films on several substrates. Considering the manufacture of transparent electrodes, spray pyrolysis is not only adequate but easy, safe and cheap as compared with other sophisticated techniques [14–16].

In this work, we report on the effect of substrate temperature on the electrical, optical and structural properties of In-doped ZnO thin films. The (100) orientation obtained in this work is one the rare case to be reported. Most of the workers exhibited (002) and (101) to be the predominate orientation [17–20]. The aim of the work is to show the effect of substrate temperature using a simple way of optimization of the samples with the help of two probes and XRD technique.

2. Experimental details

The spray pyrolysis apparatus used in this work consists of a home made spraying unit, substrate holder with heater, and enclosure. The custom glass spray gun having a nozzle diameter of 0.2 mm was positioned at a distance of 25 cm above the substrate. The whole assembly was kept in an enclosure connected to an exhaust. The soda-lime glass substrate is kept on a stainless steel plate. The heater is

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Table 1
Variation of physical properties for ZnO:In thin films at different substrate temperature.

Substrate temperature (°C)	Sheet resistance (Ω/□)	Percent transmittance (λ = 550 nm)
350	302	52
400	72	63
450	26	75
500	34	74

capable of heating the substrate up to a temperature of 700 °C [21]. In order to prepare indium doped zinc oxide (IZO) thin films, the following procedure was adapted. The films were prepared from a 0.4 M solution of zinc acetate dissolved in a mixture of deionized water, acetic acid and methanol in a volume proportion of 3:1:5 respectively. Indium chloride was added to the starting solution as a dopant. The In concentration in the starting solution was selected after a previous experimental work made in our laboratory, which showed that the lowest sheet resistance for ZnO:In films were obtained at [In]/[Zn] = 1% [22].The solution was sprayed at a flow rate of 18 L min^{-1} using purified compressed air.

The crystalline structure was obtained by means of an X-ray diffractometer with Cu-Kα radiation (Philips-pw-1830). In addition, to evaluate the electrical and optical properties of films, a two-probe measurements and a UV–VIS spectroscopy (Cary 100 Scan Varian) were carried out respectively. Surface morphology was investigated using scanning electron microscopy (SEM) (Stereo Scan S360-Cambridge).

3. Result

3.1. Electrical characteristic

The variation of sheet resistance and room temperature transmission in the visible region (at λ = 550 nm) of In-doped ZnO films as a function of substrate temperature are shown in Table 1. As shown in the table by increasing the substrate temperature from 350 °C to 450 °C, sheet resistance decreases, from 302 Ω/□ to 26 Ω/□, then at 500 °C increases to a value of 34 Ω/□.

The minimum sheet resistance is obtained at 450 °C; out of this range higher sheet resistance values are observed. This behavior is explained on the basis of stoichiometric variation of ZnO with the temperature. In the low-temperature regime, the chemical reaction of ZnO synthesis takes place in an incomplete manner, yielding high resistivity films, but as the substrate temperature is increased, an optimum stoichiometric deviation is reached, namely, an oxygen deficiency along with an excess in zinc, partly substituted by In ions, yielding ZnO films with sheet resistance values as low as 26 Ω/□ [18]. As the substrate temperature increased, the activation energy of the reaction is fully reached, and not only a high-resistance ZnO stoichiometric phase is obtained, but an increase in the diffusion of alkaline type ions coming from the substrate occurs to an increase in the sheet resistance.

3.2. Structural properties

We employed the X-ray diffraction technique to get a first impression of the main crystalline phases and the possible orientation of crystalline in the films prepared at optimum conditions. Fig. 1 shows the XRD patterns of IZO films prepared at different substrate temperature with a fixed gas flow rate 18 L min^{-1} on soda-lime glass. When the substrate temperature during deposition is low (less than 250 °C), the resultant films exhibit a single broad unstructured XRD line. Lower substrate temperature leads to lower deposition rate, less stoichiometry of the film, more impurity incorporation, poor crystallinity and small grain size [23].These properties can be improved by increasing substrate temperature.

At higher substrate temperature (Fig. 1a, Ts = 350 °C) peaks related to ZnO is observed. Secondary peaks presented for film deposited at 350° are (002), (101), (102) and (110). By further increase in substrate temperature (T_s = 400 °C, Fig. 1b) the intensity of (002) diffraction peak increased in the XRD pattern. For samples deposited at 350 °C and 400 °C, (100) and (110) peaks are dominant orientations respectively. Increasing substrate temperature to 450 °C and 500 °C (Fig. 1c and d) causes the IZO thin films exhibit a strong orientation along (002). Other peaks, (100), (102) and (110) are vanished.

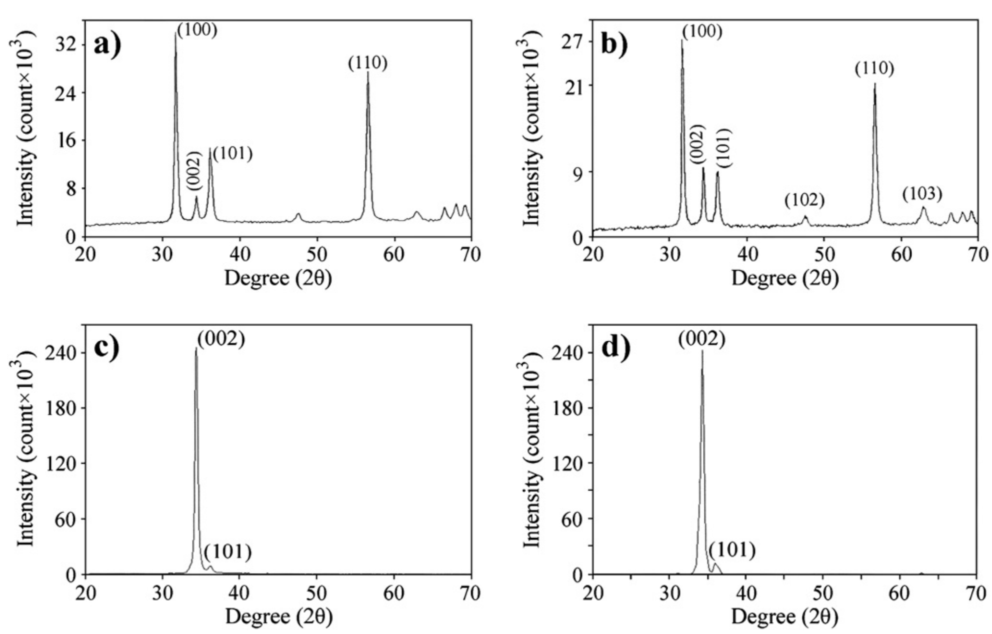

Fig. 1. X-ray diffraction patterns for ZnO:In nanostructure thin films at substrate temperature: a) 350 °C, b) 400 °C, c) 450 °C and d) 500 °C.

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