



Fiber-like stripe ATO ($\text{SnO}_2\text{:Sb}$) nanostructured thin films grown by sol–gel method: Optical, topographical and electrical properties



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ABSTRACT

Sb-doped SnO_2 thin films were deposited on glass substrates by sol–gel dip coating technique. The effects of dopant concentration on structural, morphological, electrical, and optical properties of ATO thin films were investigated. The compositional analysis of the films carried out by using EDX and XPS. The analyses confirmed the presence of Sb in SnO_2 thin films. The XRD results showed a tetragonal rutile structure of SnO_2 and the crystallite size of the films decreased with increasing dopant concentration. The surface morphology of the films is found to depend on the doping concentration. The wrinkle network with uniformly protruding fiber like stripes arranged randomly on the film surface can be observed at a higher Sb content (above 7 mol%). The surface topographical image exhibited that the surface roughness of films increased by Sb doping. A minimum resistivity of $1.72 \times 10^{-2} \Omega \text{cm}$ was obtained for the ATO (10 mol% Sb) film. The Hall mobility of the ATO films decreased with increasing the Sb concentration. The optical study revealed that transmittance of the films was diminished by doping and also a gradual increase in the optical band gap was observed by enhancement of dopant concentration. The compositional dependence of optical parameters such as refractive index, extinction coefficient, dispersion energy, real and imaginary parts of the dielectric constant and optical conductivity were also investigated. The PL measurements illustrated the photoluminescence quenching with increasing Sb content up to 10 mol%.

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1. Introduction

Tin oxide (SnO_2) nanostructures as transparent conductive oxide (TCO) with interesting combination of multifunctional properties including low resistivity, high transmittance in visible region and chemical stability have acquired considerable attention among researchers [1–4]. The SnO_2 thin films can be doped with a wide variety of ions to meet the demands of several practical applications such as gas sensors [5], spintronics [6] and solar cell windows [7]. Among the various dopants, Sb^{5+} and F [8] were found to be more effective to improve the n-type conductivity of SnO_2 thin films. The Sb doped SnO_2 thin films exhibit high conductivity while maintaining its transparency in the visible optical range which are significant for the fabrication and operation of solar cells. A number of methods such as magnetron sputtering [9], chemical vapor deposition [10], pulsed laser deposition [11], electron beam evaporation [12], spray pyrolysis [13] and sol–gel methods [14–21] have been reported for the deposition of ATO thin films. In recent studies on Sb doped SnO_2 films, Liu et al. [19] reported that Sb-doped SnO_2 thin films with different Sb concentration were pre-

pared by spin coating technique and characterized by XRD, SEM and UV–vis spectroscopy. They observed a significant decrease in resistivity of thin films. The optical band gap reduced quickly first and increased slowly afterwards with Sb-doping concentration with a minimum at 15 at.% Sb content. Benrabah et al. [20] reported that undoped and Sb doped SnO_2 thin films were prepared by sol–gel dip coating technique. They investigated the effects of Sb incorporation on the structural, optical and electrical properties. Complex impedance spectroscopy revealed that effect of grain boundaries was dominant in the conduction mechanism. The activation energy for conduction (E_a) of the material was found to be 0.87 eV. Floriano et al. [21] deposited Sb doped SnO_2 thin films by using sol–gel method and reported a decay of photo-induced conductivity in thin films after irradiation with monochromatic light of about band gap. The results showed that the physical properties of prepared films were strongly related to the preparation conditions and the resulted surface morphology of the films. Recently, some researches indicated that unique textures of thin films such as nanofiber [22–25] and nanorod [26,27] can be synthesized by sol–gel method. This special shape of nanostructures, providing a higher surface area, might exhibit some interesting physical and chemical properties. Although, there have been a lot of reports on synthesis and characterization of Sb-doped SnO_2 thin films, there is

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no regular and detailed study on the preparation and characterization of fiber-like stripes ATO thin films by sol-gel route. In this paper, for the first time, fiber like stripes ATO thin films with wrinkle network have been deposited on glass substrates by sol-gel dip coating technique and the influence of Sb incorporation on the structural, compositional, morphological, optical, electrical and photoluminescence properties of films were investigated. Moreover, the optical parameters such as refractive index (n), extinction coefficient (k), packing density, dispersion oscillator energy (E_o , E_d), optical band gap (E_g) and electrical parameters were also calculated and comprehensively discussed in order to show the composition dependence of Sb-doped tin oxide films.

2. Experimental details

2.1. Sample preparation

SnO₂:Sb thin films were deposited by sol-gel method using dip coating technique on Corning 2947 glass substrates. The following procedure was adopted for the preparation of the films. The sol was prepared by dissolving 0.7 mol of SnCl₂·2H₂O in 50 ml absolute ethanol as a solvent. The mixture was well stirred and refluxed at 80 °C for 2 h. Antimony chloride (SbCl₃) was then added into the solution as a dopant source and nominal concentration of dopant ($\frac{Sb}{Sn+Sn}$) was varied as 2, 5, 7 and 10 mol%. The homogenous mixing of the solution was refluxed at 80 °C for 4 h and then aged in air for 48 h, i.e. until the formation of a clear and homogenous sol with a stabilized viscosity and pH. The Sn_{1-x}Sb_xO₂ thin films were deposited on ultrasonically cleaned glass substrates using the dip coating technique. The withdrawal speed of the substrate from the sol was 90 mm/min. Then, the films were dried at 150 °C for 20 min. to evaporate the solvent and remove organic residuals. The coating and drying procedure was repeated several times to obtain suitable thickness. Finally, the samples were annealed at 500 °C for 1 h in air for crystallization.

2.2. Sample characterization

Thermogravimetric-derivative thermogravimetry (TG-DTG) of concentrated gel was performed using Perkin Elmer (Pyris Diamond) thermal analyzer. 1.66 mg of the sample was analyzed in N₂ with increasing the temperature at a speed of 20 °C/min. up to a maximum of 700 °C. The chemical structure information of the samples was collected by using Fourier transform infrared (FTIR) spectrometer (Nicolet Magna-IR560). The prepared films composition (Sn, O, Sb) was determined by quantitative energy dispersive X-ray analysis (EDX, VEGAII TESCAN instrument). The chemical state of each constituent element in the ATO thin film was investigated by X-ray photoelectron spectroscopy (XPS, VG Microtech, Twin Anode XR3E2, X-ray Source Systems using Al K α = 1486.6 eV). The structural properties were determined with Philips PW-1840 diffractometer by using Cu K α radiation ($\lambda_{Cu\ K\alpha}$ = 0.15406 nm). The surface morphology and roughness parameter of the films was characterized using SEM, LEO1430VP. AFM analysis of the films was performed in noncontact (NC) operational mode (Veeco CP Research instrument). The topographical inhomogeneity of the surfaces can be explained from the AFM topography images using surface roughness parameters, while the compositional inhomogeneity was elucidated from AFM phase images.

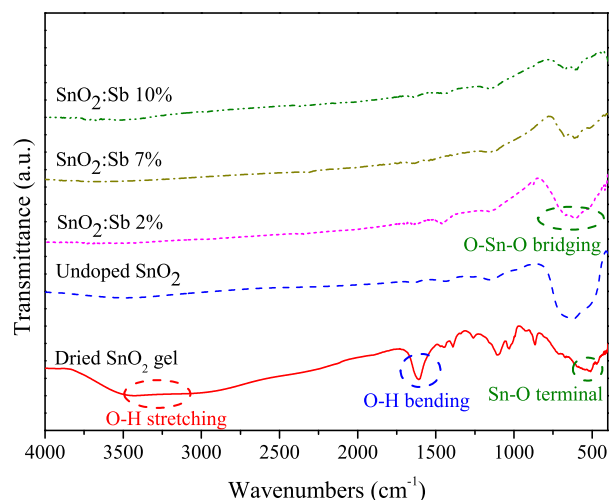


Fig. 2. FTIR spectra of ATO samples.

The electrical parameters such as resistivity, carrier concentration, Hall mobility of the films were determined by Hall effect measurements system (RH2010) using the Vander-Pauw technique at RT. The transmission spectra of the films were measured with Varian Cary100 UV/Visible spectrophotometer. The optical constants (refractive index and extinction coefficient) and thickness of the films were calculated from the experimental spectral transmittance by using pointwise unconstrained minimization approach [28]. The single oscillator model was used in analyzing the obtained spectral refractive index dispersion. The photoluminescence (PL) spectra of the prepared films were recorded by PerkinElmer (LS 55) Fluorescence spectrometer with an excitation wavelength of 270 nm.

3. Results and discussion

3.1. Thermogravimetric and FTIR (chemical) studies

Fig. 1 shows the TG/DTG curves of the Sb doped SnO₂ xerogel. Two weight losses were observed at 70–100 °C and 110–190 °C in TG curve which may be caused from the evaporation of water, alcohol and combustion of resultant organics produced by alcoholysis reactions (sharp endothermic peaks in DTA curve), respectively. The combustion and decomposition of organic components accompanied by the reaction between gel and O₂ and crystallization process of ATO powder occurred at the temperature range of 350–500 °C corresponding to broad exothermic peak in DTA curve [29]. No weight loss in TG curve was observed for the

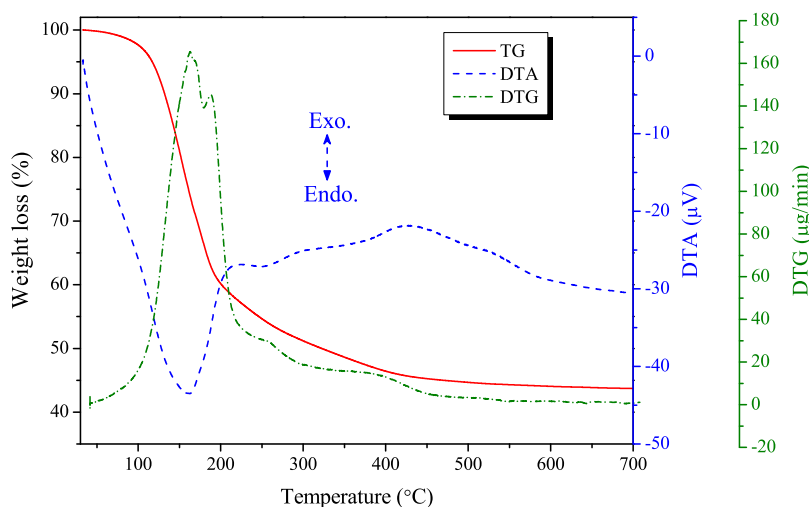


Fig. 1. TGA/DTG curves of the 5 mol% Sb-doped SnO₂ precursor xerogel.

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