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SYNTHESIS AND CHARACTERIZATION OF NANOCRYSTALLINE TIN OXIDE THIN FILMS

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ABSTRACT
This paper describes the structural, morphological and optical properties of nanocrystalline tin oxide thin films synthesized by sol-gel spin coating method. The as-synthesized samples are thin, uniform and adherent to the substrate support. The XRD studies show synthesis of phase pure tetragonal SnO_2 thin films. The lattice parameters are a=4.7284Å and c=3.2673Å. The average crystallite size is 7 nm. SEM studies show that growth of the film takes place with porous structure embedded with fine nanograunules, increasing the open surface area of the film. Optical study revealed that band gap of SnO_2 is 3.96 eV with direct band to band transitions.

KEY WORDS: SnO_2 thin films, structural characterization, optical properties

INTRODUCTION

Tin oxide with a wide band gap of 3.6 eV is one of the most important and extensively used metal oxide semiconductor materials which are usually used as gas sensors and transparent electrodes in optoelectronic devices (Zhurbina et al 2012). The method and condition of SnO_2 nanoparticles preparation are very important for the control of the microstructure and thus expected to influence the optical and electrical properties. Both chemical and physical methods have been widely investigated, such as sonochemical (Sedghi et al 2010) chemical vapor deposition (Zhao et al 2010), solid state reaction (Firooz et al 2009), sol-gel (Wang et al 2010), spray pyrolysis (Patil et al 2009), etc. In fact, many authors proved that the sintering of nanoparticles under standard conditions led to a dramatic growth of the particles and to a loss of the nanostructure in the sintered sample. The synthesis of SnO_2 nanoparticles with better control of the microstructure using cost-effective techniques still remains a future challenge. Among the various methods, the sol-gel process is a wet-chemical technique widely used in the fields of materials science and ceramic engineering. The sol-gel approach is a cheap and low-temperature technique that allows for the fine control of the product's chemical composition. This paper describes the structural, morphological and optical properties of nanocrystalline tin oxide thin films synthesized by sol-gel spin coating method.

MATERIALS AND METHODS

The thin films of SnO_2 were deposited onto glass substrates by sol-gel method. For synthesis, AR grade $SnCl_4$ $5H_2O$ was dissolved in double distilled water to get 8.9 M solution. The solution was stirred for 20 minutes at 70^{o}C using magnetic stirrer to get clear and homogeneous solution. This was then added drop wise into 40 ml isopropyl alcohol with continuous stirring to form a gel. The films were deposited from this gel using spin coating unit (MILMAN-XT56) and heated in the furnace at 400^{o}C for one hour. The thin film was then characterized through X-ray diffraction technique for its structural analysis. A Philips PW-3710 X-ray diffractometer with CuK_α radiation (λ = 1.54056 Å) was used for this purpose. The morphology of the films was observed on SEM (JEOL JSM 6360) operating at 20 kV. The UV-VIS spectra of these films were recorded using Shimadzu UV-VIS-NIR spectrophotometer (UV-3600)

RESULTS AND DISCUSSION

The X-ray diffractogram of SnO_2 thin film is shown in Fig.1. The pattern was analyzed to get the information about crystal structure, lattice parameters, induced strain and grain size. The d values and intensities of the observed diffraction peaks match with the single crystalline form of the tetragonal SnO_2 (JCPD card no. 41-1445). No diffraction peaks from any other impurities are detected indicating that synthesized samples are pure tin oxide with tetragonal structure. The lattice parameters are a=4.7284Å and c=3.2673Å. The observed lattice parameters slightly

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differ from the standard values. The reason is that the sample is ultrafine in nature and it is possible that lattice may be under strain induced forces. The strain induced in the material and the crystallite size was determined by Williamson-Hall method;

$$\beta \cos\theta = 4\varepsilon \sin\theta + \lambda/D \quad \text{--- (1)}$$

where, D is the crystallite size, λ is the wavelength of the X-ray, β is full width at half maximum (FWHM) measured in radians, ε is the induced strain in system, and θ is the Bragg angle. Fig. 2 shows the variation of β cos θ versus 4 sin θ for as-synthesized sample plotted by linear fit method. The induced strain and crystallite size were calculated from these plots and the values are 7 nm and 0.0008 respectively.

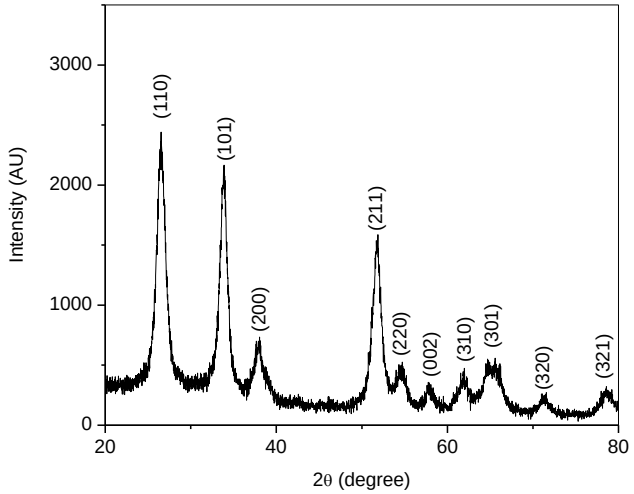

Fig.1: X-ray difffractogram of SnO_2 thin film.

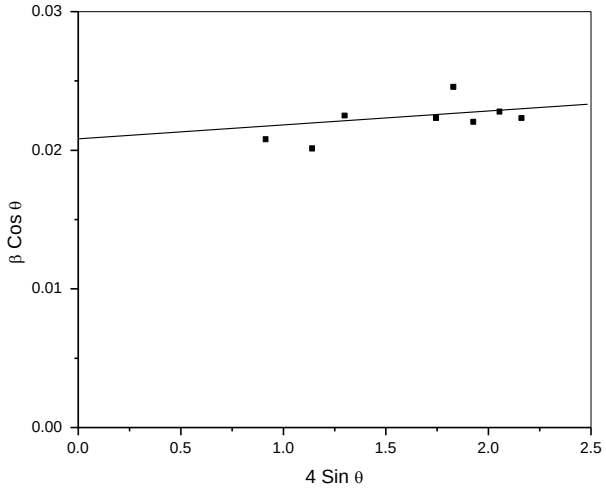

Fig.2: Williamson-Hall plot for SnO_2 thin film.

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The surface morphology of SnO_2 thin film was studied using scanning electron microscope. The SEM images of SnO_2 thin film is shown in Fig. 2. It is seen that the growth of the film takes place with porous structure embedded with fine nanograunules, increasing the open surface area of the film. This type of growth supports the application of these films as gas sensor since the film has large open surface area to be exposed for the gas. The results are in close agreement with XRD studies.

Fig.3: SEM image of SnO_2 thin film

The optical absorption spectra of SnO_2 thin film recorded over the wavelength range 280 -900 nm at room temperature is shown in Fig. 3.

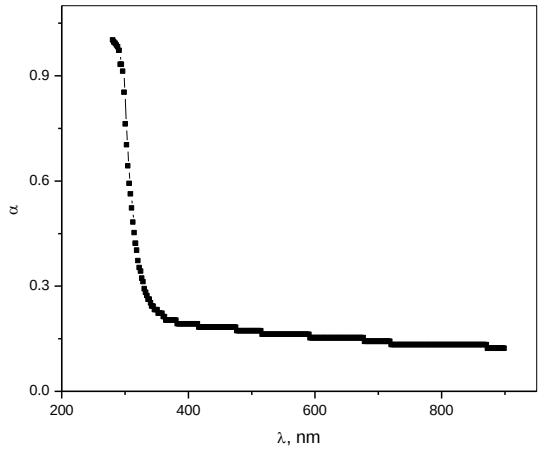

Fig.4: Optical absorption spectrum of SnO2 thin film.

A sharp ultraviolet absorption edge at approximately 290 nm is observed. The spectra was studied to evaluate the absorption coefficient (α) energy gap (E_g) and nature of transition involved. It is found that the optical absorption coefficient is higher for all the composites (10^4 cm^{-1}). The absorption coefficient (α), energy gap (E_g), and photon energy ($h\nu$) are related as (Patil et al 2001):

$$\alpha h\nu = A(h\nu - E_g)^{n/2} \quad \text{--- (2)}$$

Assuming the mode of transition to be of the direct allowed type (n = 1), the band gap energies have been calculated from the variation of $(\alpha h\nu)^2$ versus $h\nu$ (Fig. 4). The $(\alpha h\nu)^2$ versus $h\nu$ plots shows straight line behavior on the higher

energy side that confirm direct type of transitions involved in these films. The band gap (E_g) of the pure SnO_2 thin film is 3.96eV. The observed band gap energy is higher than the bulk value which can be attributed to the size quantization effects due to decreased grain size as revealed from the XRD studies (Raut et al 2012).

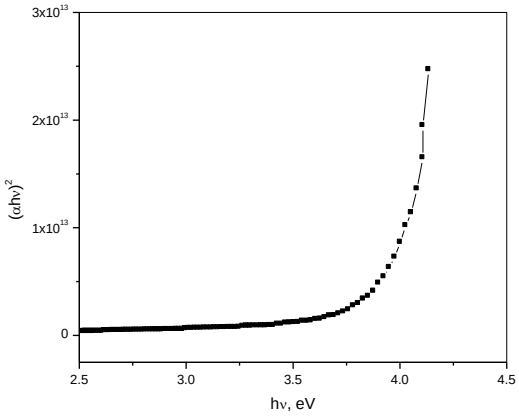

Fig.5: Band gap determination of SnO_2 thin film.

CONCLUSION

A sol gel technique has been used to synthesize nanocrystalline SnO_2 thin films. The synthesized sample is pure SnO_2 pure zinc oxide with tetragonal structure. The lattice parameters are: a=4.7284Å and c=3.2673Å. The average crystallite size is 7 nm. SEM studies show that growth of the film takes place with porous structure embedded with fine nanograunules, increasing the open surface area of the film. Optical study revealed that band gap of SnO_2 is 3.96 eV with direct band to band transitions.

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