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SYNTHESIS AND PROPERTIES OF SNO_2 THIN FILMS

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Abstract: Multilayered SnO_2 thin films were prepared by sol-gel spin coating method. The deposited films were characterized with X-ray diffraction and UV-visible spectroscopy. The synthesized films showed crystalline nature. The band gap of the films agreed to their theoretical value.

Keywords: Thin films; sol-gel processes; optical properties; X-ray diffraction

1. Introduction

Among various semiconducting metal oxides used for sensing methane gas, Tin oxide (SnO_2) is one such material. SnO_2 is n-type, wide band gap (3.6 eV) semiconductor with high thermal and chemical stability in air. Most of the SnO_2 based methane sensors operate at temperature higher than room temperature [1-6]. In order to reduce the operating temperature nanostructures of SnO_2 such as nanorods [7] and nanofibers [8] have been used.

Therefore, the development of a CH_4 gas sensor operated at a relatively low temperature is important. Our study deals with the deposition of doped multilayered SnO_2 films on glass substrates followed by their rapid thermal annealing.

2. Experimental details:

Reagent grade(Merck) $SnCl_2.2H_2O$ was used as precursor material. 8.374 gm of $SnCl_2.2H_2O$ was dissolved in 100 ml of absolute ethanol. The mixture was refluxed and stirred at 353 K for 3 hours and then it was allowed to cool to the room temperature for 1½ hours with continuous stirring. A commercial spin coater (Apex SCU 2005) was used for coating the sol on the glass substrates. The speed of the spin coater was fixed at 2100 rpm. Six drops (0.8 ml = 6 drops) of sol were dropped on approximately 1 inch x 1 inch sized clean glass slide. The coated glass slide was air annealed at 673 K for 10 minutes. The slide was then allowed to cool to the room temperature to produce a transparent SnO_2 film. Using the above procedure multilayered

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films (with increased film thickness upto twelve-layers) were prepared. All layers were deposited repeating the same procedure as before and were repetitively annealed with the above mentioned condition after every single layer deposition.

These multilayered films were characterized with XRD, optical studies and SEM. Optical studies were done using UV-Vis-NIR Spectroscope and their results were used to calculate the band gap of the films.

3. Results and Discussion

3.1 Sample characterization- X-ray diffraction patterns of some representative undoped SnO_2 films (one-layered, three-layered, six-layered, ten-layered and twelve-layered films) deposited for the initial phase of the experiment. The undoped films showed amorphous nature up to three-layered deposition (figure 2b) whereas with increased film layers, crystalline nature sets in. Reflections from the tetragonal crystallographic phase (cassiterite) of SnO_2 became more defined and progressively more intense and sharp for films with more than five-layered depositions. The shows increase in mean grain size and improved crystallinity. Debye-Scherrer formula was employed to calculate the particle size of the prepared undoped SnO_2 films from their XRD spectras. The average particle thickness 'd' calculated for ten-layered and twelve-layered undoped SnO_2 films is 48 nm.

The spectral dependence of the transmittance (T) for one, three, six, eight, ten and twelve-layered films. The average transmission of the undoped SnO_2 films deposited on glass substrates is more than 80% over the range 450 to 800 nm. A sharp fall in transmission at about 310 nm is due to the absorption of the glass substrate. The transparency of the films decreases in major portions of the visible range with the addition of layers. This may be due to the built-up thickness which is also clear from the fringes in the transmission spectra. The absorption edge also shifted slightly to higher wavelengths with the increase in film layers. The transmittance falls rapidly in the low wavelength region. With increase in number of layers, the onset of absorption edge becomes less sharp, this may be due to the presence of bigger crystalline sizes and increased scattering due to the surface roughness (Mahmoud et al 2002; Jiménez-González et al 2000**)**.

The deposited SnO_2 films' thicknesses were calculated from their optical data using the equation (Sarkar et al 1991):

$$t = (N_o \lambda_1 \lambda_2) / \{2(n_1 \lambda_2 - n_2 \lambda_1)\}$$

Where, N_o= Number of oscillations between two extrema and n_1 and n_2 represent the refractive indices of the film at wavelengths λ_1 and λ_2 respectively. The refractive index was obtained from the relation

$$n^2 = [(n_a^2+n_g^2)/2 + 2n_a n_g T_o] + [\{(n_a^2+n_g^2)/2 + 2n_a n_g T_o\}^2 - n_a^2 n_g^2]^{1/2}$$

where,

$$T_o = (T_{max} - T_{min})/ T_{max}.T_{min}$$

The films' thicknesses were calculated from the above expressions and were found to vary from 0.18 to 1.75 μm for 1-layered and 12-layered undoped SnO_2 films respectively. After palladium doping the calculated films' thicknesses ranged between 0.24 to 1.8 μm for 1-layered and 12-

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layered SnO_2 films respectively. Film thickness depends upon the preparation techniques (ie. numbers of drops, drop size, rotation speed of the spin coater, etc.) and hence the thicknesses of the films are not always exactly proportional to the number of layers.

Surface morphologies as obtained through Scanning Electron Microscope (SEM) of undoped SnO_2 films are shows comparatively smoother film surface for three-layered undoped SnO_2 which is amorphous in nature (also observed earlier from the XRD spectra). The nanostructures displayed granules with particle sizes in the range of (~) 30-40 nm and this non-uniform particle size is caused due to the uneven distribution of temperature and mass flow during the synthesis. It is also reflected in the XRD studies that the average particle thickness of the prepared ten-layered and twelve-layered undoped films are about 30 nm. The difference between the values of the particle size obtained by the XRD and SEM are due to the fact that the former is the measure of the diffraction from the crystals, whereas the SEM results correspond to the microscopic view of the deposited sample surface.

Conclusions

SnO_2 thin films were successfully deposited over glass substrates. The X-ray diffraction of films showed crystalline nature. The prepared films of SnO_2 showed band edge absorption.

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