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Synthesis And Characterization Of SnO_2 Thin Film By Spray Pyrolysis Technique

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Abstract
In this paper I reported tin dioxide (SnO_2) thin film, deposited on to glass substrate by spray pyrolysis technique. Deposition parameters were optimized. The film was characterized by X-ray diffraction (XRD), atomic force microscopy (AFM), Scanning Electron Microscope (SEM) and UV-VIS spectrophotometer. The XRD analysis shows SnO_2 thin film has tetragonal structure; SEM micrograph shows uniform deposition on to glass substrate and optical band gap was found to be 3.18 eV. The results of structural, surface morphological and optical investigation show that, the film is suitable for gas sensing application.
Keyword: *spray pyrolysis technique; gas sensor; thin film*

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I. Introduction

The fast-growing industrialization and technological advancements have led to increased emissions of toxic gases including volatile organic compounds (VOCs) into the atmosphere. This causes serious health issues. Highly sensitive gas sensor devices are required to detect harmful gasses in the atmosphere. The feasible electrical and morphological properties of metal oxides make them more suitable for gas sensor applications because of their tunable electrical properties [1]. A sensor that uses the change in resistance as the means to detect the presence and concentration of target gas is the chemi-resistive gas sensor. SnO_2 is widely used as a gas sensor due to the significant changes in conductivity associated with gas molecules chemisorbed on its surface. Tin dioxide (SnO_2) is an n-type semiconductor material which has wide large band gap energy of 3.1 –3.8 eV and gas sensing property [2]. It also exhibits very interesting physical properties like high optical transparency, electrical conductivity, and chemical stability. Tin oxide is of great importance in many technological applications such as transparent conducting electrodes [3] solar cell fabrication [4, 5], energy storage devices [6], optoelectronic application [7], and gas sensor [8, 9]. For gas sensor and other applications, it is necessary to grow SnO_2 thin films that possess good thermal, chemical, and mechanical stability.

SnO_2 thin films have been synthesized many researchers with various methods such as Sol gel [10], hydrothermal methods [11], chemical bath deposition [12] and spray pyrolysis [13, 14]. However, for industrial applications, thin films are of low cost coating is generally required. The spray pyrolysis technique is a very simple and cost-effective coating technique will grow thin films at very higher temperatures [14]. The spray pyrolysis technique is a low-cost technique for creating thin films, ceramic coatings, and powders by spraying a solution of chemical precursors onto a heated substrate, where the heat causes the solvent and volatile byproducts to evaporate and the remaining components to react and form a solid coating. This versatile and scalable process is used to deposit a variety of materials, including metal oxides, semiconductors, and luminous compounds, with deposition parameters like substrate temperature and solution concentration being key to controlling the quality and morphology of the final product.

II. Experimental

SnO_2 thin films were deposited by the spray pyrolysis technique from aqueous solutions containing 2 M Stannic Chloride ($SnCl_4.5H_2O$) as a precursor, using compressed air as a carrier gas. Stannic Chloride ($SnCl_4.5H_2O$), supplied by S. D. Fine Limited (India), dissolved in deionized water and methanol (aqueous and non-aqueous medium). 0.01 M oxalic acid was added to solution to make solution clear and increase transmittance of the film. A homemade spray pyrolysis assembly has been used to prepare thin film. Ultrasonically cleaned glass slide substrate was placed on a solid uniform thermal conductor surface to provide proper heating with uniformity to film. A heater is used as heat source to provide temperature of around 400 $^{\circ}$C. After spraying, films on glass slides were deposited at 400 $^{\circ}$C for 5min inside the furnace. Total volume of the solution sprayed was 20mL.

Optimized preparative parameters for SnO_2 thin film deposited by spray pyrolysis are given in table 1

Table 1: Optimized parameters

Initial Ingredients	Stannic Chloride ($SnCl_4.5H_2O$), Deionized Water (H_2O), Methanol (CH_3OH)
Substrate temperature	500 ^{0}C
Concentration of solution	2M
Composition (Volume Ratio)	1:1
Spray Rate	4 mL/min
Nozzle to substrate distance	25 cm
Carrier Gas	Air
Gas flow rate	10 lit/min
Gas Pressure	1.35 Kg/ m^2

The deposited thin film was characterized by X ray diffraction (XRD), scanning electron microscopy (SEM), and optical absorption spectra. X-ray diffraction pattern was recorded on Bruker AXS D8 advanced X-Ray diffractometer using Cu-Kα radiation wavelength (**λ** = 1.5405Å) within range from 20^o to 100^o. The SEM micrograph was recorded on JOEL-JSM-5600 SEM instrument model. The optical absorption spectra of the film was measured in the wavelength range of 350–850nm on a Shimadzu UV-2450 spectrophotometer. Film thickness was measured by weight difference method.

III. Results And Discussions

XRD Analysis

The structural determination and crystallite size SnO_2 thin film was carried out by X-ray diffraction (XRD) pattern. Fig. 1 shows X-ray diffraction pattern of as-deposited SnO_2 thin film. One small and two large peaks are observed. More than one XRD peaks indicate that the as deposited film is polycrystalline in nature. The 2θ peaks at 51.35°, 57.90° and 80.80° corresponding to (211), (002) and (400) planes. These peaks are compared with the JCPDS file no. 41-1445, confirms the formation of SnO_2 thin film with tetragonal structure. The crystallite size (D) was calculated by Scherrer's formula [15] from the full width at half maxima (β) of the peaks expressed in radians and found to 70.65 nm.

$$D = \frac{K\lambda}{\beta Cos\theta} \qquad (1)$$

where 'K' is constant dependent on crystallite shape (0.89), 'λ' is wavelength of CuKα1 radiation, and 'θ' is angle between the incident and scattered X-rays. The average crystallite size (derived from Fig. 1) is found 70.65 nm.

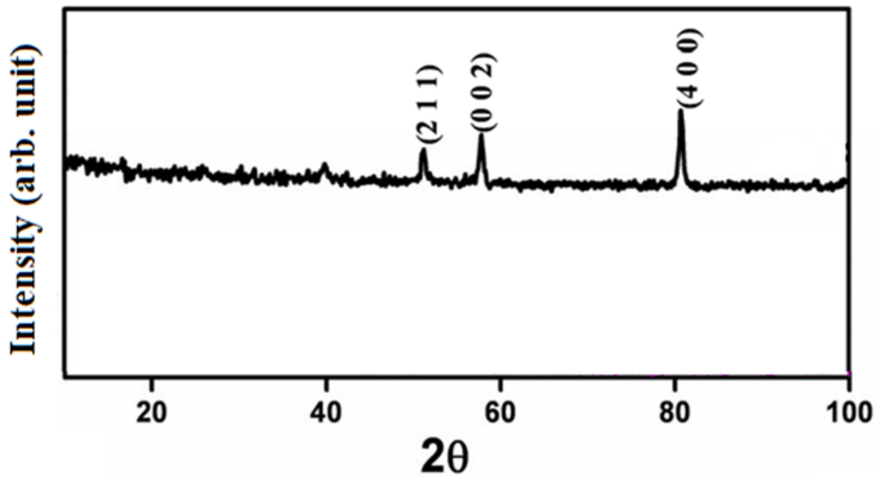
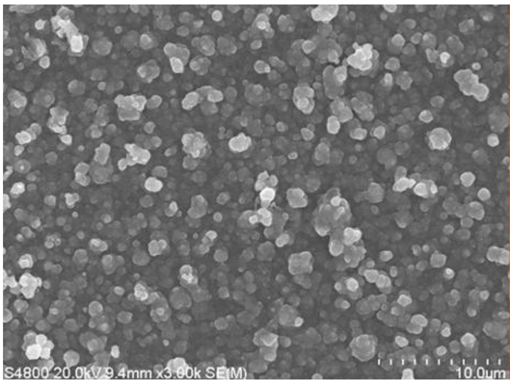


Fig. 1 XRD pattern of as deposited SnO_2 thin film

Surface Morphology Analysis

Surface morphology of the films was studied by scanning electron microscope (SEM) and atomic force microscopy (AFM) images. Fig. 2 (a) shows SEM image of as-deposited ternary SnO_2 thin films. It is observed that the particles are not perfect spherical and were covered on the entire substrate surface with some gaps.



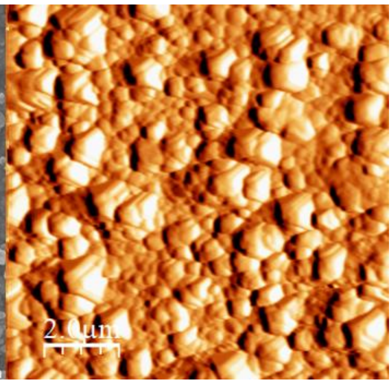


Fig. 2(a) SEM image of as deposited SnO_2 thin film

Fig. 2(b) AFM image of as deposited SnO_2 thin film

Fig. 2 (b) shows AFM image of as-deposited SnO_2 thin film. From the image it is clear that the film is uniform and substrate surface is well covered by fine spherical or elliptical grains. The average cluster size was determined to be 137 nm and average surface roughness was 0.0003 nm.

The cluster size and root mean square (rms) surface roughness were determined by using the software which was provided with the microscope. The surface roughness of the film is very small in our case due to particles are non-spherical in shape.

Optical Analysis

Energy band gap is calculated by using UV-VIS absorbance spectra of as-deposited SnO_2 thin film. The absorbance spectra were used to study the optical transition in the films, which were studied at room temperature in the wavelength range of 300–1100 nm. The optical absorption studies revealed that the film is highly absorptive and have direct type of transitions, which allowed the determination of optical band gap by the following Urbach relationship

$$\alpha h\nu = A\left(h\nu - E_g\right)^n \quad (2)$$

where 'A' is the constant; depending upon the transition probability for direct transition, n = 1/2 for direct allowed transition and 'E_g' is the optical band gap of the material.

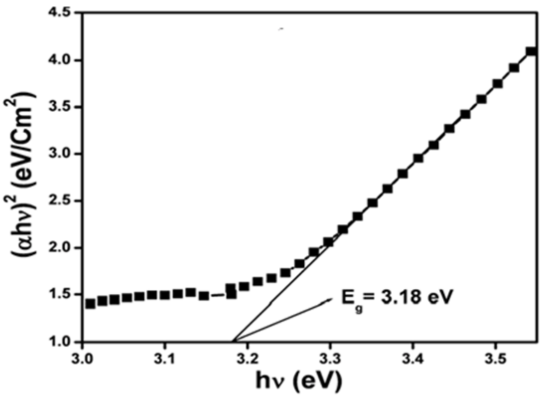


Fig. 3 plot of $(\alpha h\nu)^2$ vs hν of as deposited SnO_2 thin film

Fig. 3 shows the variation of $(\alpha h\nu)^2$ versus hν. Extrapolating the straight-line portion of the plot of $(\alpha h\nu)^2$ versus hν for zero absorption coefficient value gives the band gap energy, which is found to 3.18 eV.

IV. Conclusion

The thin films of SnO_2 are deposited on to glass substrates by using spray pyrolysis technique. The XRD studies showed that the as-deposited film has polycrystalline tetragonal structure. SEM and AFM images showed uniform deposition of film on to the glass substrate. Optical study showed band gap energy equal to 3.18 eV. Optical band gap energy is the more important physical property of semiconductor material that is useful for gas sensing applications

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