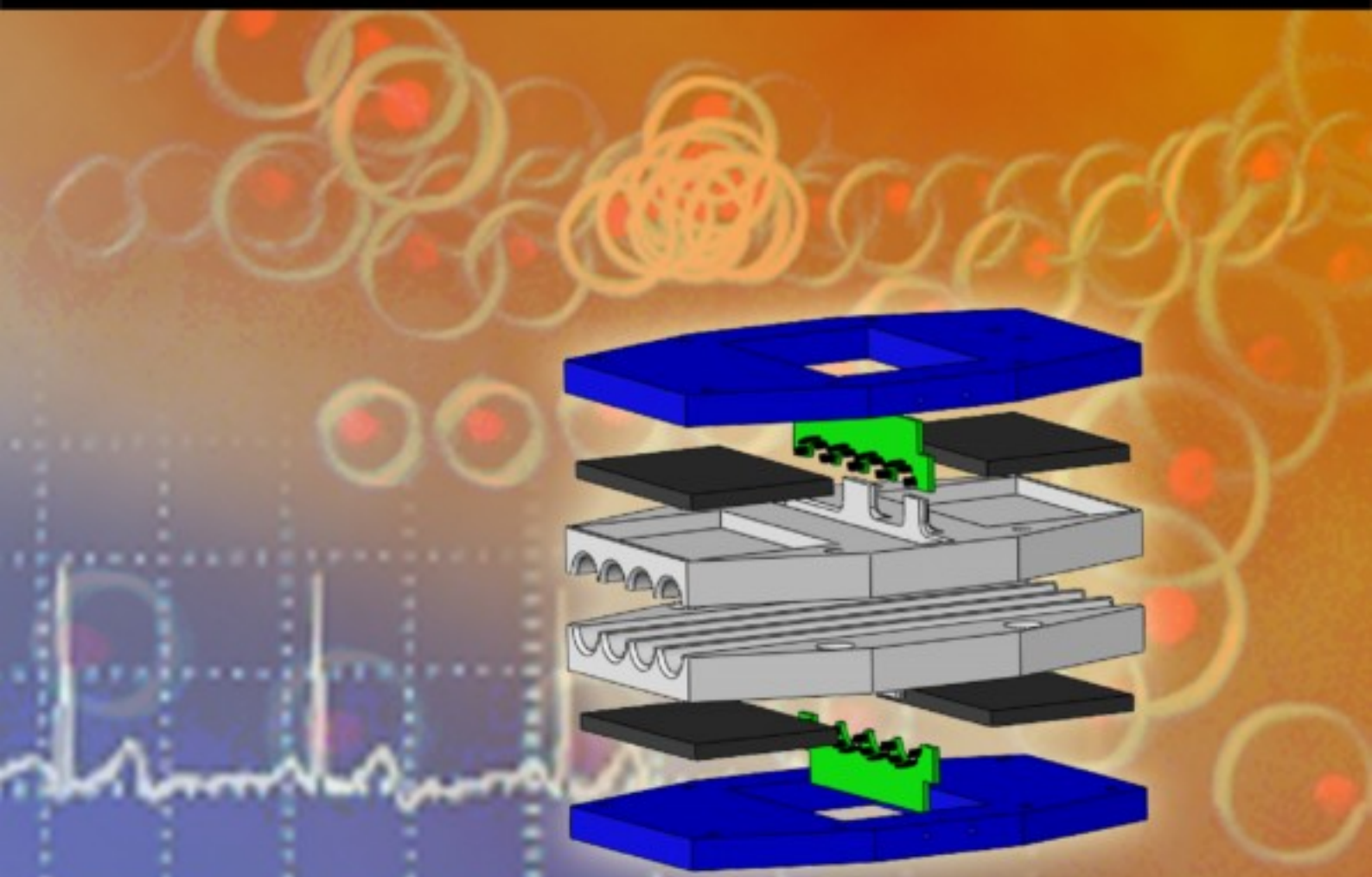


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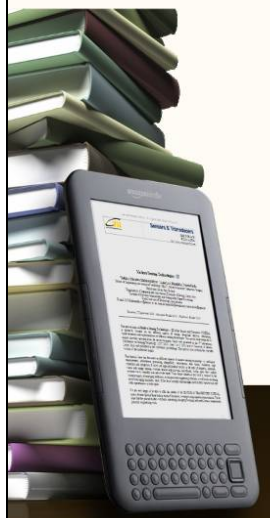
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- Chemoinformatics
- Bioimaging
- Neuroinformatics

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- Biocomputing
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- Molecular and Cellular Biology
- Microbiology

**C. Biotechnologies and biomanufacturing**

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- Biodevices
- Biomedical technologies
- Biological technologies
- Biomanufacturing

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Submission (full paper)	January 10, 2011
Notification	February 20, 2011
Registration	March 5, 2011
Camera ready	March 20, 2011

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- COMAN: Network Control and Management
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- GRIDNS: Grid Networks and Services
- EDNA: Emergency Services and Disaster Recovery of Networks and Applications
- IPv6DFI: Deploying the Future Infrastructure
- IPDy: Internet Packet Dynamics
- GOBS: GRID over Optical Burst Switching Networks



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- Safety in industrial systems
- Complex Systems

## Effect of Annealing Temperature on Gas Sensing Performance of SnO<sub>2</sub> Thin Films Prepared by Spray Pyrolysis

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**Abstract:** The effect of variation of annealing temperature on the gas sensing characteristics of SnO<sub>2</sub> thin films, which have been prepared by spray pyrolysis on alumina substrate at 350 °C, is investigated systematically for various gases at different operating temperature. The XRD, UV-visible spectroscopy and SEM techniques were employed to establish the structural, optical and morphological characteristics of the materials, resp. The X-ray diffraction results showed an increase in the crystallinity at higher annealing temperature. A high value of sensitivity is obtained for H<sub>2</sub>S gas at an optimum temperature of 100 °C is improved considerably. A SnO<sub>2</sub> gas sensor annealed at 950 °C with sensitivity as high as 24 %, 4 times higher than that of sensor annealed at 550°C, are obtained for 80 ppm of H<sub>2</sub>S. The degree of crystallinity and grain size calculated from the XRD patterns has been found increasing with annealing temp. *Copyright © 2010 IFSA.*

**Keywords:** Spray pyrolysis, SnO<sub>2</sub> thin films, Degree of crystallinity, H<sub>2</sub>S gas sensor

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### 1. Introduction

Due to the development of industry activity, pollution of environment has become one of the most important problems all over the world and thus leads to the requirement in the toxic gas detection and air pollution control. One of the most simple and reliable methods to detect gases is by measuring the change in electrical conductivity induced by the adsorption of gas molecules on the surface of a

semiconductor. Gas sensors have found such type of wide applications [1–5]. Among the various types of gas sensors, semiconducting gas sensors are the promising candidates for sensor development giving their sensitivity to many gases and ability to fabricate them readily in many configurations, e.g. single crystals, thick and thin films [6]. Thin film technology, in particular, is being actively applied in the development of semiconducting gas sensor devices given such that sensors depend largely on gas-surface interaction. Thin film gas sensors have potential advantages of fast response times, and importantly, the potential for miniaturization via integration with IC-based technology leading to low power consumption, high reliability, improved selectivity and reduced cost.

In 1962, semiconducting oxides, SnO<sub>2</sub> and ZnO were first demonstrated gas sensing devices [7]. Among the sensors investigated and developed, SnO<sub>2</sub> based sensors received much attention since they can detect a wide variety of gases with high sensitivity, good stability and also low production cost [8–14].

Different deposition technology like PVD, CVD, sol-gel, spray pyrolysis and other can be used for the fabrication of thin film based gas sensors [15, 16]. Among these spray pyrolysis is a versatile technique for obtaining thin films of pure SnO<sub>2</sub> [17]. It is of particular interest because of its simplicity, low cost and minimal waste production. The spray pyrolysis process allows thin film formation by spraying, drying and pyrolytically decomposing onto a heated substrate a solution of precursor salts of the desired constituent ions [18]

The present study demonstrates first, to prepare SnO<sub>2</sub> thin films by the spray pyrolysis technique and to investigate the influence of annealing temperature on the structural, microstructural, optical properties and then, annealing effects on the sensing properties of SnO<sub>2</sub> thin films.

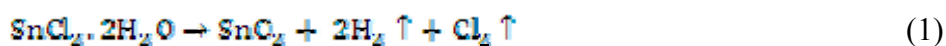
## **2. Experimental**

### **2.1. Substrate Cleaning**

The substrate cleaning is very important in the deposition of thin films. Commercially available alumina slides with a size of 25 mm×25 mm×1 mm were washed using soap solution and subsequently kept in hot chromic acid and then cleaned with deionized water followed by rinsing in acetone. Finally, the substrates were ultrasonically cleaned with deionized water for 20 min and wiped with acetone and stored in a hot oven.

### **2.2. Preparation of SnO<sub>2</sub> Thin Films**

The tin oxide thin films were prepared by using tin (II) dichloride dihydrate (SnCl<sub>2</sub>·2H<sub>2</sub>O) in de-ionized water as a precursor. A precursor solution of 0.075M concentration was sprayed through a specially designed glass nozzle of 0.5 mm inner diameter onto the ultrasonically cleaned alumina substrates. The deposition parameters like substrate temperature (350 °C), rate of spraying solution (5 mL/min.), nozzle to substrate distance (25 cm), quantity of the solution sprayed (50 ml), pressure of carrier gas, and to and fro movement of the nozzle were kept constant at the optimized values. The pyrolytic reaction takes place on a heated substrate, leading to a poly-crystalline metal oxide. The usual expression for this reaction is:



### 2.3. Annealing of SnO<sub>2</sub> Thin Films

As-prepared SnO<sub>2</sub> thin films were subjected to annealing in the air atmosphere for further characterization at different annealing temperature for 30 min with the help of tubular furnace. Three samples were annealed at 550 °C, 750 °C and 950 °C respectively and the furnace was cooled to room temperature.

### 2.4. Characterization

The structural, microstructural and optical properties have been studied. The crystalline structure of the thin films, obtained at different annealing temperature were examined by X-ray diffractometer (Miniflex Model, Rigaku, Japan) using CuK<sub>α</sub> radiation with a wavelength  $\lambda = 1.5418 \text{ \AA}$ . The microstructures of the films were analyzed using a scanning electron microscope (SEM model JEOL 2300-LA, Japan). The optical absorbances of the films were measured using UV-visible-2450 spectrophotometer (Shimadzu) in the wavelength range 200-700 nm at room temperature. The static gas sensing system had been employed for testing of the films to gases, which is explained elsewhere [19-20]. The sensitivity (*S*) is defined as,

$$S = \frac{(R_a - R_g)}{R_a} = \frac{\Delta R}{R_a} \quad (1)$$

where *R<sub>g</sub>* is the resistance in presence of test gas and *R<sub>a</sub>* is the film resistance in dry air, measured at respective temperatures.

## 3. Results and Discussion

### 3.1. Structural Properties of the Films

The XRD patterns of films deposited on alumina substrate annealed with different temperature shown in Fig. 1. Films annealed at 550 °C shows very small peaks indicating their predominantly amorphous nature. As the annealing temperature is increased, structural evolution is found to occur yielding polycrystalline films, characterized by an increase in intensity of peaks. The intensity of peaks is seen to increase indicating an improvement in crystallinity with annealing temperature. The (110), (101), (200) and (002) peaks match well with the standard data [21]. All the peaks correspond to the tetragonal phase. Films on alumina substrate are seen to have preferred orientation along (022) and (002) planes. The peaks (\*) marked are attributed to alumina. The area under the crystalline and amorphous portions was determined in arbitrary units and the degree of crystallinity (*D<sub>c</sub>*) was calculated using the relation [22].

$$D_c = \frac{I_c}{I_c + I_a} \quad (2)$$

where *I<sub>a</sub>* and *I<sub>c</sub>* are the integrated intensity corresponding to amorphous and crystalline phases, respectively. Also, the grain size (*t*), interchain distance (*r*), interplanar distance (*d*) and distortion parameter (lattice strain) (*g*) were calculated as follows [22-24]:

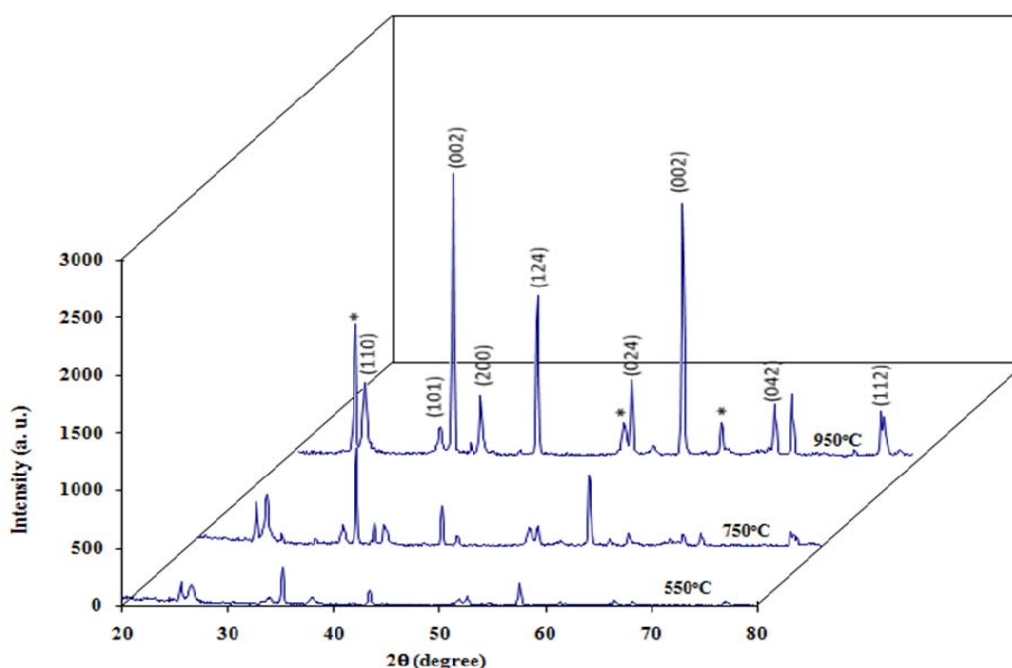


Fig. 1. XRD patterns of SnO<sub>2</sub> thin films.

$$t = \frac{0.9\lambda}{\beta \cos \theta} \quad (3)$$

$$r = \frac{3\lambda}{8\sin \theta} \quad (4)$$

$$d = \frac{\lambda}{2\sin \theta} \quad (5)$$

$$g = \frac{\beta}{\tan \theta}, \quad (6)$$

where  $t$  is the crystallite size,  $\lambda = 1.542 \text{ \AA}$  (X-ray wavelength), and  $\beta$  is the peak FWHM in radian and  $\theta$  is diffraction peak position.  $t$ ,  $r$  and  $d$  are calculated with respect to the most intense crystalline peak at the angular range of 35.2- 35.4°.

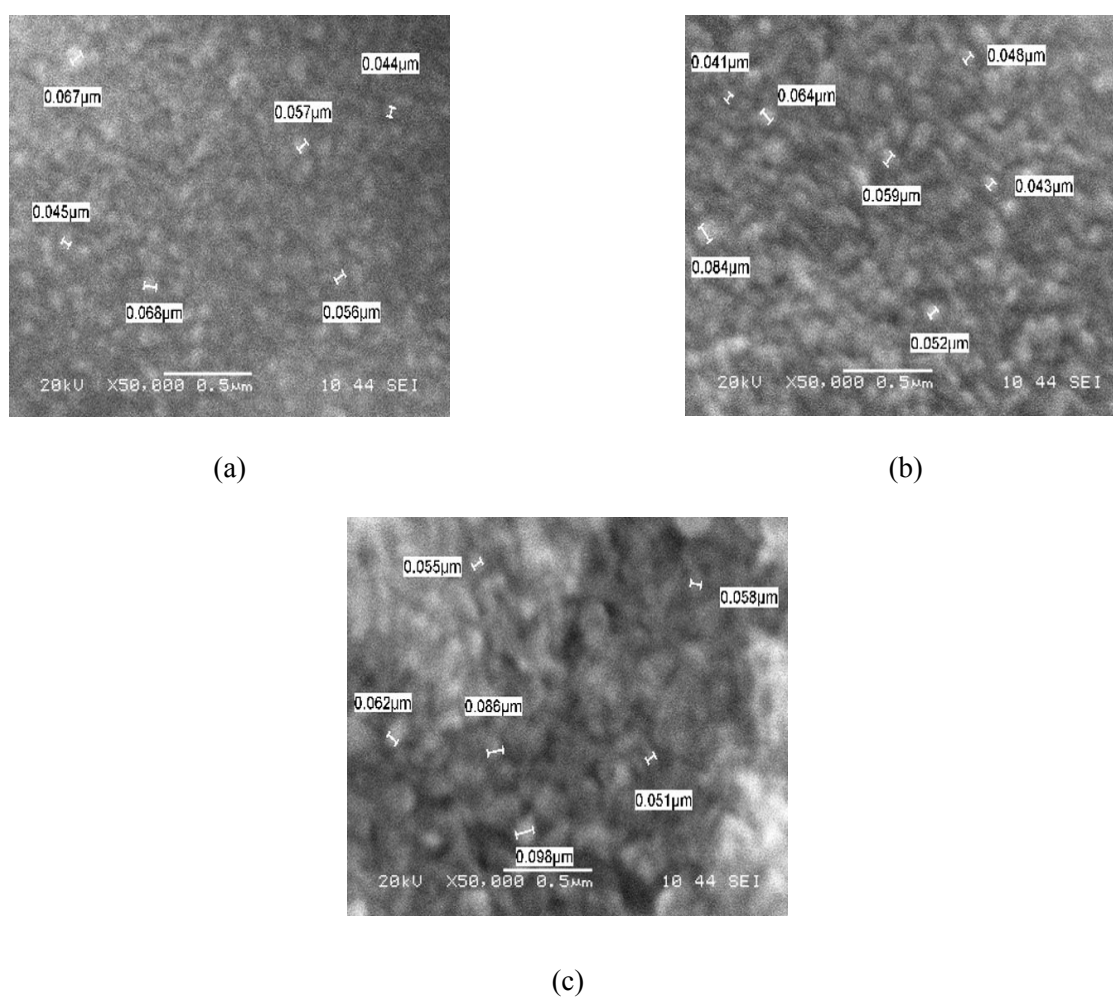
Table 1 shows the different XRD parameters for all the films. It can be seen that the percentage of crystallinity and grain size systematically increased with increase in annealing temperature. Similar reports of increase in crystallinity with different annealing temperature can be seen in literature [25]. Interplanar and interchain distances were marginally changed because the angle of the peak ( $\theta$ ) did not vary significantly. Also the lattice strain decreases with increase in annealing temperature.

Table 1. XRD parameters for SnO<sub>2</sub> thin films.

Annealing temp. (°C)	Peak angle $\theta$ (degree)	Degree of crystallinity $D_c$ (%)	Grain size $t$ (nm)	Interplanar distance $d$ (Å)	Interchain distance $r$ (Å)	Lattice strain $g$ (%)
550	17.6	67.32	40.0	1.015	0.8126	0.22
750	17.6	93.44	43.2	1.015	0.8126	0.19
950	17.7	96.38	56.1	1.056	0.8449	0.11

### 3.2. Surface Morphology of the Films

Fig. 2 consists of SEM images representing surface morphology of the SnO<sub>2</sub> thin films with different annealing temperature. It is seen that the microstructure of these films quite similar except for a small increases in particle size. The average particle sizes obtained from the SEM images are 56.1-68.3 nm. It is found that the SnO<sub>2</sub> films have relatively smooth morphology. The SEM images reveal the increase of particle size with increasing annealing temperature, up to an approximate average particle size of 68.3 nm at 950 °C. However, as determined from XRD data, the average grain size ranged from 40 to 56.1 nm, which was substantially smaller than the 56.1-68.3 nm dimensions of grains observed in SEM.

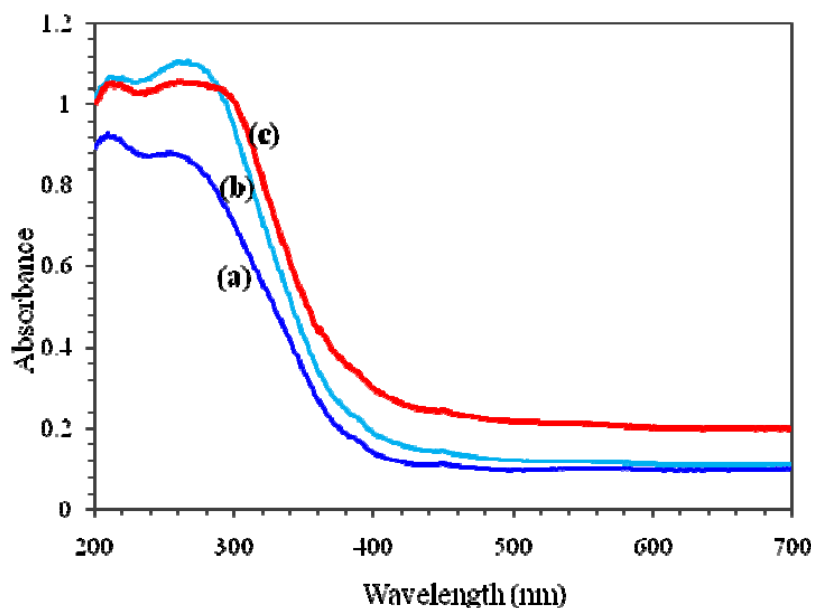


**Fig. 2.** SEM photographs at annealing temperature (a) 550°C, (b) 750 °C and (c) 950 °C.

### 3.3. Optical Properties

Optical characterization of SnO<sub>2</sub> thin films gives information about other physical properties, e.g. band gap energy and band structure and optically active defects etc. The effects of annealing temperature on the optical absorbance and the band gap ( $E_g$ ) values of the SnO<sub>2</sub> films have been studied. The optical absorbance of the SnO<sub>2</sub> films formed with different annealing temperature is shown in Fig. 3. The absorption at higher wavelengths in the visible region is low and at wavelength 350-380 nm an intense absorption can be seen. A shift of the absorption edge proportional to the annealing temperature

values, towards higher energies is evident from the spectra. In order to obtain the band gap, the absorption coefficient ( $\alpha$ ) was calculated from the absorption data.



**Fig. 3.** Variation of absorbance with the wavelength ( $\lambda$ ) nm for annealing temperature (a) 550 °C, (b) 750 °C and (c) 950 °C.

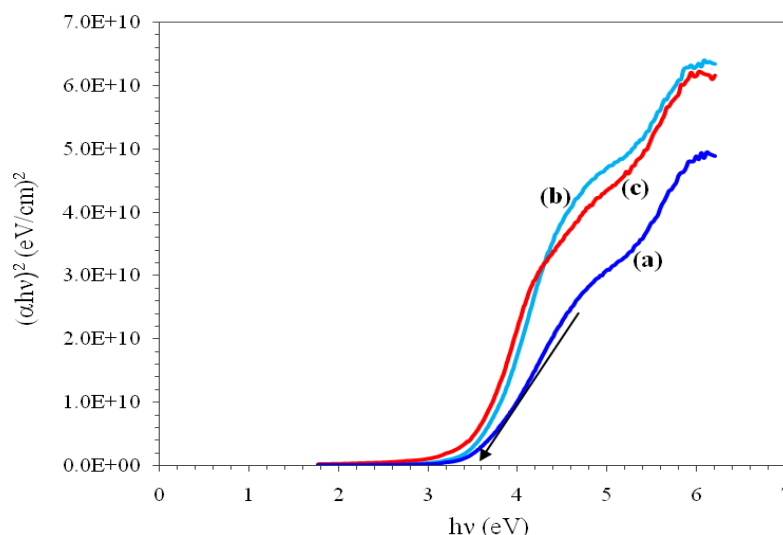
The value of absorption coefficient ( $\alpha$ ) is of the order of  $10^4 \text{ cm}^{-1}$ . Absorption coefficient decreases with increase in annealing temperature. The band gap of the films corresponding to annealing temperature 550°C to 950°C was calculated by plotting  $(\alpha h\nu)^2$  vs.  $h\nu$  using the relation [26],

$$\alpha h\nu = A(h\nu - E_g)^n, \quad (7)$$

where  $\alpha$  is absorption coefficient,  $A$  is constant,  $E_g$  is the optical band gap energy,  $h\nu$  is the photon energy and  $n$  is constant. The value of  $n$  is 1/2 or 2 depending on presence of the allowed direct and indirect transitions. Fig. 4 shows the plots of  $(\alpha h\nu)^2$  versus  $h\nu$  for films at different annealing temperature. The nature of the plots suggests direct interband transition. The band gap is determined by extrapolating the straight line portion of the plot to the energy axis. The intercept on energy axis gives the value of band gap energy for all the samples and decreased from 3.62 to 3.5 eV by increasing the annealing temperature from 550 °C to 950 °C.

**Table 2.** Variation of grain size and band gap energy with annealing temperature.

Annealing temperature (°C)	Average grain size from XRD (nm)	Average particle size from SEM (nm)	Band gap energy (eV)
550	40.0	56.1	3.62
750	43.2	58.0	3.53
950	56.1	68.3	3.50

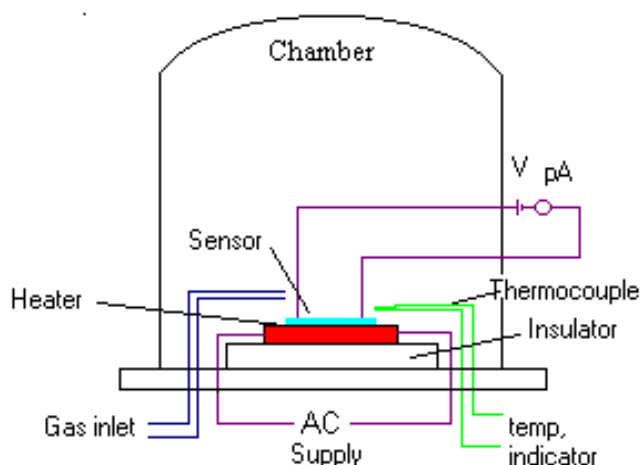


**Fig. 4.** Plot of the  $(\alpha hv)^2$  vs. photon energy ( $hv$ ) for the annealing temperature (a) 550 °C, (b) 750 °C and (c) 950 °C.

### 3.4. Gas Sensing Properties of SnO<sub>2</sub> Thin Films

#### 3.4.1. Details of Static Gas Sensing System

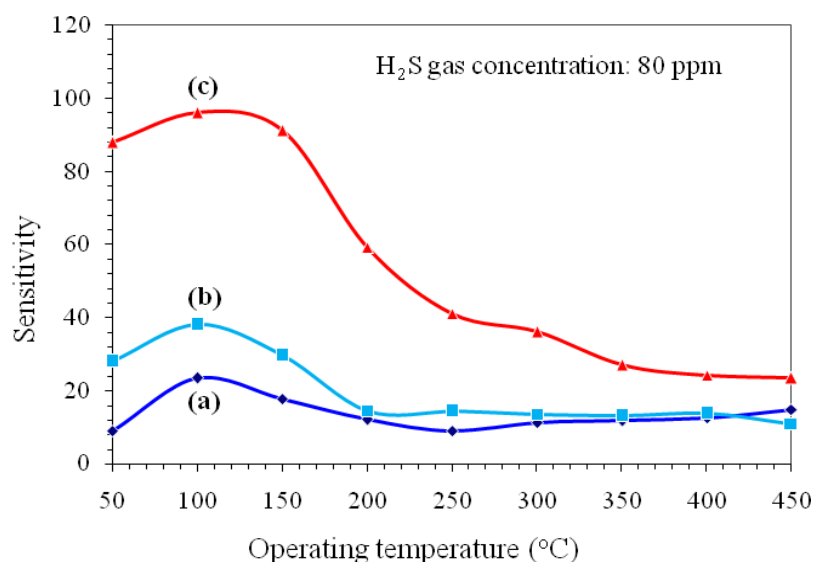
The sensing performance of the films was examined using a 'static gas sensing system', shown in Fig. 5. In this system, the sensor element is mounted in an enclosed test chamber of a known volume. In order to measure the sensor resistance in a desired concentration of the analyte gas, a known amount of gas is injected into the housing using a micro-syringe. There were electrical feeds through the base plate. The heater was fixed on the base plate to heat the sample under test up to required operating temperatures. The current passing through the heating element was monitored using a relay operated with an electronic circuit with adjustable ON-OFF time intervals. A Cr-Al thermocouple was used to sense the operating temperature of the sensor. The output of the thermocouple was connected to a digital temperature indicator. A gas inlet valve was fitted at one of the ports of the base plate. A constant voltage was applied to the thin film sensor, and the current was measured by a digital picoammeter. The air was allowed to pass into the glass chamber after every gas exposure cycle [27, 28].



**Fig. 5.** Block diagram of static gas sensing system.

### 3.4.2. Sensitivity of SnO<sub>2</sub> Films to H<sub>2</sub>S with Operating Temperature

Experiments were performed against operating temperature in order to investigate the influence of the annealing temperature on the sensing properties of the SnO<sub>2</sub> thin films. For comparison, the H<sub>2</sub>S sensing properties of the SnO<sub>2</sub> films at different annealing temperature were also studied under identical experimental conditions. The annealing temperature is an important parameter for gas sensing materials and in designing of sensors [29-30]. The sensing materials have to be annealed at various temperatures to achieve crystallization and structural evolution. A sufficient degree of crystallinity is required to attain the desired electronic properties necessary for gas sensor application. The dependence of the sensitivity of the prepared SnO<sub>2</sub> to 80 ppm of H<sub>2</sub>S at annealing temperature 550 °C, 750 °C and 950 °C on the operating temperature is shown in Fig. 6. The sensitivity is found to be maximum when the annealing temperature was 950 °C. The annealing in air renders more oxygen vacancy generation, which enhances the gas sensitivity. Also the high percentage of crystallinity of SnO<sub>2</sub> thin films caused by the influence of the annealing temperature which is probably responsible for the improvement of the sensing properties of these films in comparison with the properties of the films annealed at 550 °C and 750 °C. It is observed that the sensitivity increases 50 °C to 100 °C and then decreases with the further increase in the operating temperature. It showed the maximum sensitivity of 23.4, 39 and 96 to 80 ppm of H<sub>2</sub>S at annealing temperatures 550 °C, 750 °C and 950°C respectively.



**Fig. 6.** Effect of annealing temperature on the sensitivity of SnO<sub>2</sub> thin films at (a) 550 °C, (b) 750 °C and (c) 950 °C to 80 ppm of H<sub>2</sub>S gas.

### 3.4.3. Variation in Sensitivity with H<sub>2</sub>S Gas Concentration

The dependence of the sensitivity of the SnO<sub>2</sub> on the H<sub>2</sub>S concentration at an operating temperature 100 °C is shown in Fig. 7. It is observed that the sensitivity increases linearly as the H<sub>2</sub>S concentration increases from 10 to 80 ppm and then decreases with further increase in the H<sub>2</sub>S concentration. The linear relationship between the sensitivity and the H<sub>2</sub>S concentration at low concentrations may be attributed to the availability of sufficient number of sensing sites on the film to act upon the H<sub>2</sub>S. The low gas concentration implies a lower surface coverage of gas molecules, resulting into lower surface reaction between the surface adsorbed oxygen species and the gas molecules. The increase in the gas concentration increases the surface reaction due to a large surface coverage. Further increase in the surface reaction will be gradual when saturation of the surface coverage of gas molecules is reached. Thus, the maximum sensitivity was obtained at an operating temperature of 100 °C for the exposure of

80 ppm of H<sub>2</sub>S. The SnO<sub>2</sub> is able to detect up to 10 ppm for H<sub>2</sub>S with reasonable sensitivity at an operating temperature 100 °C. The linearity of the sensitivity in the low H<sub>2</sub>S concentration range (10-80 ppm) suggests that the SnO<sub>2</sub> can be reliably used to monitor the concentration of H<sub>2</sub>S over this range.

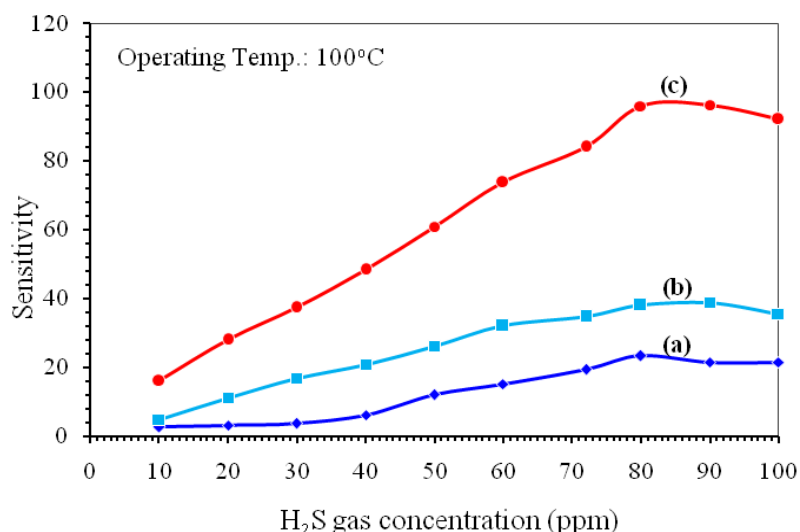


Fig. 7. Dependence of the sensitivity of the SnO<sub>2</sub> on the H<sub>2</sub>S concentration at 100°C.

### 3.4.4. Selectivity of SnO<sub>2</sub> Thin Films for Various Gases

Besides H<sub>2</sub>S, the sensing performances of the sensor to some other gases were also examined to depict its selectivity. Selectivity can be defined as the ability of a sensor to respond to a certain gas in the presence of different gases [19]. Fig. 8 shows the histogram of the selectivity of SnO<sub>2</sub> thin films to various gases. The table attached to histogram shows the sensitivity values to various gases at different annealing temperature. The films showed highest selectivity for H<sub>2</sub>S (80 ppm at 100 °C) against all other tested gases: NH<sub>3</sub>, LPG, Cl<sub>2</sub>, CO, CO<sub>2</sub>, O<sub>2</sub>, H<sub>2</sub> and ethanol. The selectivity also increases with annealing temperature.

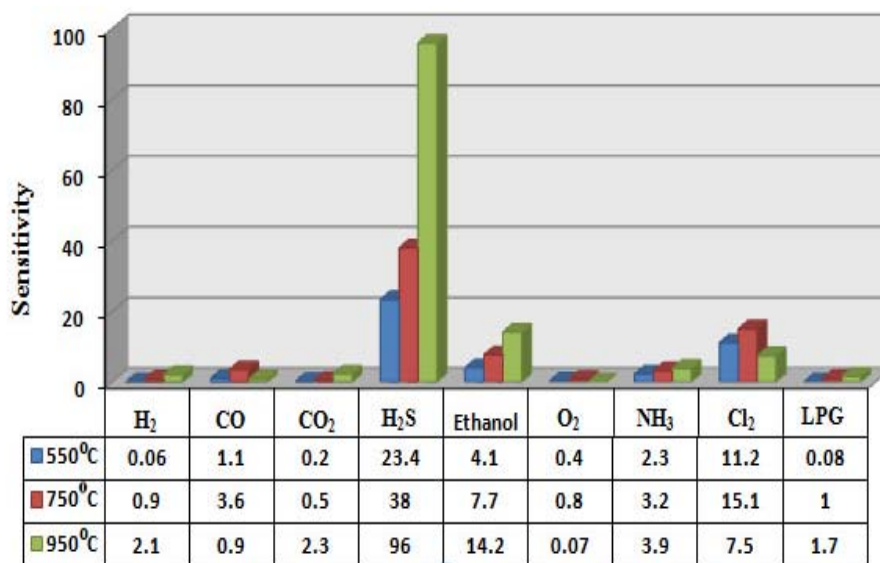


Fig. 8. Selectivity of SnO<sub>2</sub> thin films for various gases.

### 3.4.5. Response Time and Recovery Time

The response time and recovery time (defined as the time required to reach 90 % of the final equilibrium value) with sensitivity values at different annealing temperature are represented in Table 3 on exposure to 80 ppm of H<sub>2</sub>S. The response was quick (32 s) even to a trace amount (80 ppm) of H<sub>2</sub>S, while the recovery was fast (< 88 s). The quick response may be due to faster oxidation of gas. The sensitivity is found to be maximum at around 350 °C. The maximum in sensitivity is also observed from Fig. 6.

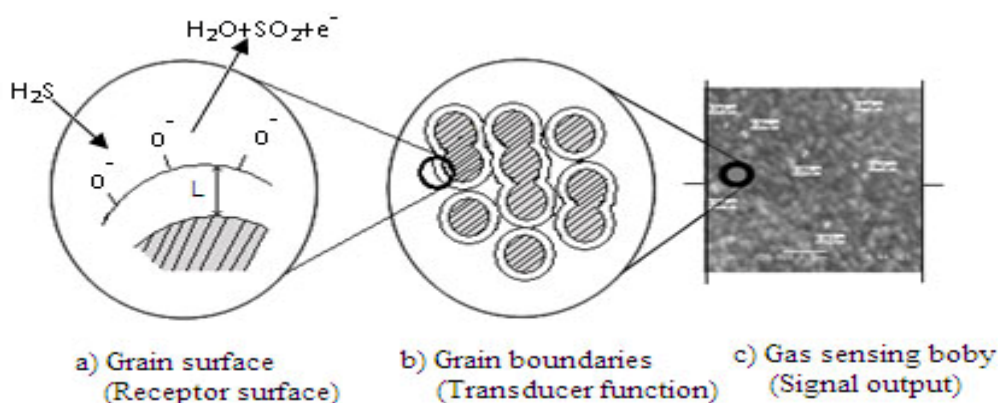
**Table 3.** Response and recovery characteristics for 80 ppm of H<sub>2</sub>S.

Annealing temp. (°C)	Sensitivity	Response time (s)	Time for complete recovery (s)
550	23.4	98	Incomplete
750	38	60	100
950	96	32	< 88

### 3.4.6. Gas Sensing Mechanism

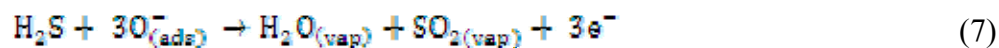
The exact fundamental gas sensing mechanisms of SnO<sub>2</sub> is still not fully understood. However, the principle of operation of SnO<sub>2</sub>-based sensor lies on detecting the conductivity changes experienced by an n-type material when surface chemisorbed oxygen reacts with reducing gases, such as H<sub>2</sub>S, CO, etc. In the detection mechanism, in clean air the conductivity of SnO<sub>2</sub> is low because the conduction electrons are bound to surface oxygen, whereas in the presence of a reducing gas, electrons are no longer bound to surface states and the conductivity increases. Therefore, the adsorption of gaseous species controls the resistance of the SnO<sub>2</sub> as well as that of the grain boundaries.

The three basic factors of SnO<sub>2</sub> gas sensors are schematically shown in Fig. 9.



**Fig. 9.** Model for gas sensing mechanism of the SnO<sub>2</sub> thin film.

Receptor function concerns the ability of the oxide surface to interact with the target gas. The surface oxygen, especially adsorbed oxygen of the oxide acts as receptor. In air, oxygen is adsorbed on the oxide grains as negatively charged ion, inducing a surface space charge layer deplete of electrons or increasing the work function of the grains. Upon exposure to the H<sub>2</sub>S gas, the adsorbed oxygen is consumed and decreased down to a steady state level, resulting in a corresponding decrease in space charge layer or work function causing returning electrons to SnO<sub>2</sub> [31] by the following reactions:



The model proposed above, explains the gas sensing mechanism of the SnO<sub>2</sub> thin film sensor qualitatively. But it does not explain the increase in gas sensitivity with rise in operating temperature (Fig. 6). In this connection, the microstructure, particularly the size and distribution of surface porosity may also have a significant role. This is so because the surface of SnO<sub>2</sub> thin film may provide suitable chemisorption sites, and hence can influence the extent as well as the kinetics of the oxidation reaction (Eq. 7) between the sensor surface and the surrounding gas ambient. Transducer function concerns the ability to convert the change in the work function of grains into a change in electrical resistance. The grain sizes of the SnO<sub>2</sub> increased with annealing temperature. When the grain size of the material is small enough, the material resistivity of the device is determined by the grain control and the material conduction type is surface conduction dominant [32-33]. The grain sizes of SnO<sub>2</sub> with annealing 950 °C is around 68.3 nm; this dimension is smaller compared to the reported range of L for the semiconducting oxide materials chemisorbed oxygen [34], hence it is surface conduction dominant and the surface-to-bulk ratio for the material is much greater than that for coarse material [35]. The grain size of the material is therefore one of the key factors to control the gas sensing properties of the material [35]

#### 4. Conclusions

SnO<sub>2</sub> thin films were prepared by spray pyrolysis technique using SnCl<sub>2</sub>.2H<sub>2</sub>O as a precursor on alumina substrates. The influence of annealing temperature on the films surface morphology, crystalline status, optical and gas sensing properties has been investigated. The percentage of crystallinity and grain size were increased with the increase in annealing temperature. The band gap values obtained from the absorption spectra was found decreases from 3.62 eV to 3.5 eV. The gas sensing characteristics of these films are strongly influenced by surface morphology. The maximum sensitivity was obtained at an operating temperature of 100 °C for the exposure of 80 ppm of H<sub>2</sub>S. The results of the H<sub>2</sub>S sensing studies reveal that the SnO<sub>2</sub> films prepared by spray pyrolysis method are a suitable material for the fabrication of the H<sub>2</sub>S sensor.

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## Guide for Contributors

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### Aims and Scope

*Sensors & Transducers Journal* (ISSN 1726-5479) provides an advanced forum for the science and technology of physical, chemical sensors and biosensors. It publishes state-of-the-art reviews, regular research and application specific papers, short notes, letters to Editor and sensors related books reviews as well as academic, practical and commercial information of interest to its readership. Because it is an open access, peer review international journal, papers rapidly published in *Sensors & Transducers Journal* will receive a very high publicity. The journal is published monthly as twelve issues per annual by International Frequency Association (IFSA). In addition, some special sponsored and conference issues published annually. *Sensors & Transducers Journal* is indexed and abstracted very quickly by Chemical Abstracts, IndexCopernicus Journals Master List, Open J-Gate, Google Scholar, etc.

### Topics Covered

Contributions are invited on all aspects of research, development and application of the science and technology of sensors, transducers and sensor instrumentations. Topics include, but are not restricted to:

- Physical, chemical and biosensors;
- Digital, frequency, period, duty-cycle, time interval, PWM, pulse number output sensors and transducers;
- Theory, principles, effects, design, standardization and modeling;
- Smart sensors and systems;
- Sensor instrumentation;
- Virtual instruments;
- Sensors interfaces, buses and networks;
- Signal processing;
- Frequency (period, duty-cycle)-to-digital converters, ADC;
- Technologies and materials;
- Nanosensors;
- Microsystems;
- Applications.

### Submission of papers

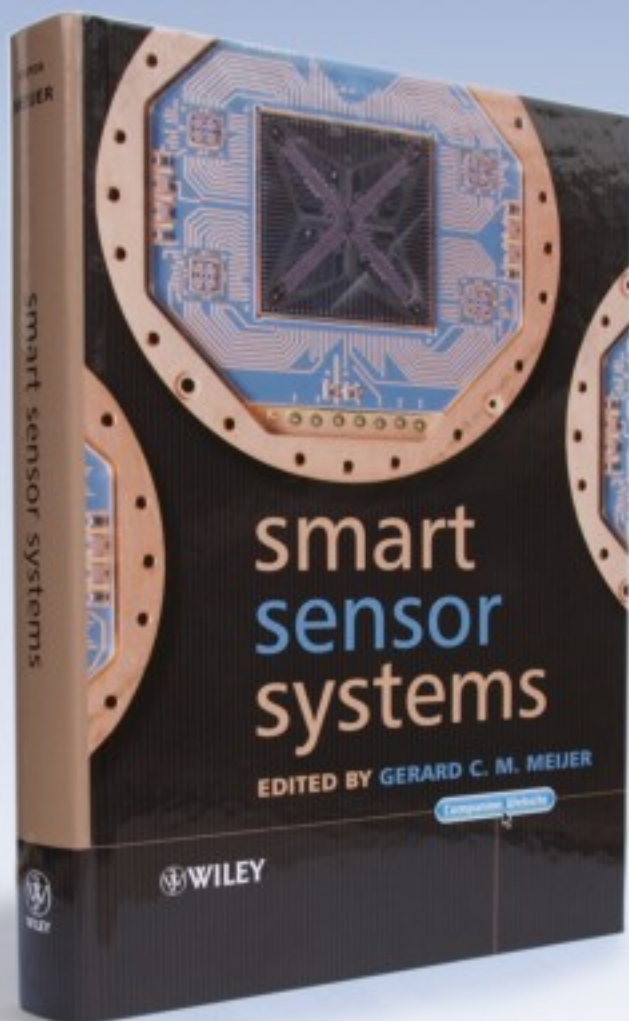
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