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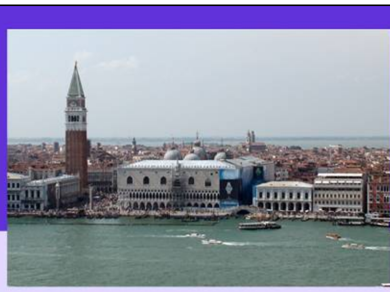

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Spray Pyrolyzed Polycrystalline Tin Oxide Thin Film as Hydrogen Sensor

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Abstract: Polycrystalline tin oxide (SnO_2) thin film was prepared by using simple and inexpensive spray pyrolysis technique (SPT). The film was characterized for their phase and morphology by X-ray diffraction (XRD) and scanning electron microscopy (SEM), respectively. The crystallite size calculated from the XRD pattern is 84 nm. Conductance responses of the polycrystalline SnO_2 were measured towards gases like hydrogen (H_2), liquefied petroleum gas (LPG), ethanol vapors (C_2H_5OH), NH_3, CO, CO_2, Cl_2 and O_2. The gas sensing characteristics were obtained by measuring the sensor response as a function of various controlling factors like operating temperature, operating voltages (1 V, 5 V, 10 V 15 V, 20 V and 25 V) and concentration of gases. The sensor response measurement showed that the SnO_2 has maximum response to hydrogen. Furthermore; the SnO_2 based sensor exhibited fast response and good recovery towards hydrogen at temperature 150 ^{o}C. The result of response towards H_2 reveals that SnO_2 thin film prepared by SPT would be a suitable material for the fabrication of the hydrogen sensor. *Copyright © 2010 IFSA.*

Keywords: SnO_2 thin film**,** Gas sensor, Operating voltages, Selectivity, Response and recovery time.

1. Introduction

In last two decades there exists a worldwide strong interest in realizing inexpensive oxide thin films to be used, for example, in solar cells, as gas sensor devices or as coating to heat glass windows. Gas sensor have a great influence in many areas such as environmental monitoring, domestic safety, public security, automotive applications, space crafts, houses and sensor networks. It has some advantages over other possible materials such as SnO_2, In_2O_3, ZnO or Cd_2SnO_4 due to its unique combination of interesting properties: non-toxicity, good electrical, optical and piezoelectric behaviour and its low price. Tin oxide (SnO_2) is a versatile material having applications in the areas like transparent electrodes in photoelectric conversion devices namely amorphous silicon solar cells, liquid crystal display, gas sensor and many more [1-3], mainly due to their outstanding properties.

Hydrogen is mainly used for energy generations in the near future due to the fact that fuel cell electricity generators are clean, quiet and more efficient than any other known technology. It is therefore straightforward that in all these applications safety measures should be of highest concern due to the explosive properties of hydrogen. To this end, there is an emerging scientific interest in developing solid state hydrogen sensors based on different operation principles [4], as catalytic-type chemiresistive [5], thermoelectric [6] and micromechanical gas sensors [7]. Recent works have shown that SnO_2 is a promising material for applications as sensors towards various gases. Moon et al [8] reported SnO_2 exhibiting good response towards CO. Patil and co-workers [9] synthesized SnO_2 which gives sensitivity towards H_2 gas. While Jain [10] reported Ni doped to be sensitive towards LPG. Most of the researchers have focused on detection of LPG, H_2S, H_2 and NH_3 because of their toxicity, their relation with atmospheric composition or to their high levels in some environments.

Spray pyrolysis technique (SPT) is useful alternative to the traditional methods for obtaining thin films of pure SnO_2. It is of particular interest because of its simplicity, low cost and minimal waste production. In this technique, a solution consists of the ions of required compound is sprayed on a heated substrate and their pyrolysis results in deposition of the compound in thin film form. The chemical reactants are selected such that the products other than the desired compound are volatile at the temperature of deposition [11].

In the present work, we report polycrystalline thin SnO_2 thin film prepared by spray pyrolysis technique. The prepared film has been studied for their gas sensing properties under different conditions.

2. Experimental

SnO_2 thin film was deposited by spray pyrolysis technique (SPT) onto ultrasonically cleaned glass substrate using tin dichloride pentahydrate as precursor solution. The solution of $SnCl_4$.5H2O (0.075 M) was prepared in de-ionized water and methanol in a volume ratio of 1:1. The solution was sprayed continuously through a glass nozzle of 0.1 mm inner diameter onto substrate kept at 350 ^oC. The deposition parameters like spray rate 5 mL/min. was adjusted using air as a carrier gas, nozzle to substrate distance (25 cm) were kept constant, and to and fro frequency of the nozzle (18 cycles min^{-1}) were kept constant at the optimized values indicated in brackets. Due to the air pressure of the carrier gas, a vacuum is created at the tip of the nozzle to suck the solution from the tube after which the spray starts. The precursor solution was sprayed on to the substrate in the air as small drops and around a high temperature zone where thermal decomposition and possible reaction between solutions occur, through compressed air, which is used as carrier gas with a flow rate controlled through air compressor regulator. To achieve uniform deposition the moving arrangement has been used. For this substrate is kept stationary, while the nozzle is free for to and fro motion with microcontroller based moving arrangement as stepper motor has been advantageous, so we don't have to spend energy moving the table with the hot plate and all electrical connections. As prepared SnO_2 thin film was annealed at 550 ^oC for 30 min.

3. Characterization

3.1. Phase Identification

X-ray diffraction pattern was recorded on diffractometer (Miniflex Model, Rigaku, Japan) using CuK_{α} radiation with a wavelength λ =1.5418 A^o at 2θ values between 20^o and 80^o. The average crystallite size (D) was estimated using the Scherrer equation [12] as follows:

$$D = \frac{0.9\lambda}{\beta \cos\theta} \quad (1)$$

where λ, β and θ are the X-ray wavelength, the full width at half maximum (FWHM) of the diffraction peak, and Braggs diffraction angle, respectively.

3.2. Surface Morphology

A JEOL 2300 model (Japan) was used to examine the surface morphology of the sample by scanning electron microscopy (SEM) and the percentage of constituent elements was evaluated by the energy dispersive X-rays analysis (EDAX) technique.

3.2. Gas Sensing Characterization

Sensor response (S) is defined as the ratio of change in conductance of the sample on exposure to a test gas to the conductance in air [13, 14].

$$S = \frac{(Ra - Rg)}{Ra} = \frac{\Delta R}{Ra} \quad (2)$$

where *Ra* and *Rg* are the resistance of a sensor in air and the test gas, respectively. The sensor was examined under different gases such as LPG, H_2, CO, CO_2, NH_3, O_2, Cl_2 and ethanol.

4. Result and Discussion

4.1. Structural Analysis

The X-ray diffraction pattern of the SnO_2 thin film is shown in Fig. 1. It shows well defined diffraction peaks, indicating formation of polycrystalline phases. The diffraction peak indexing, done by matching with the Joint Committee on Powder Diffraction Standard (JCPDS) (no. 72-1147), clearly revealed formation of the SnO_2 phases with tetragonal structure. The average crystallite size was determined using Scherrer equation which was observed to be 84 nm.

4.2. Scanning Electron Microscopy (SEM) and Energy Dispersive X-rays Analysis (EDAX)

Fig. 2 shows SEM images of the SnO_2 thin film on glass substrates at three different magnifications. It exhibit uniform and granular morphology, covering the entire substrate area. The average particle size is found to be ~115 nm.

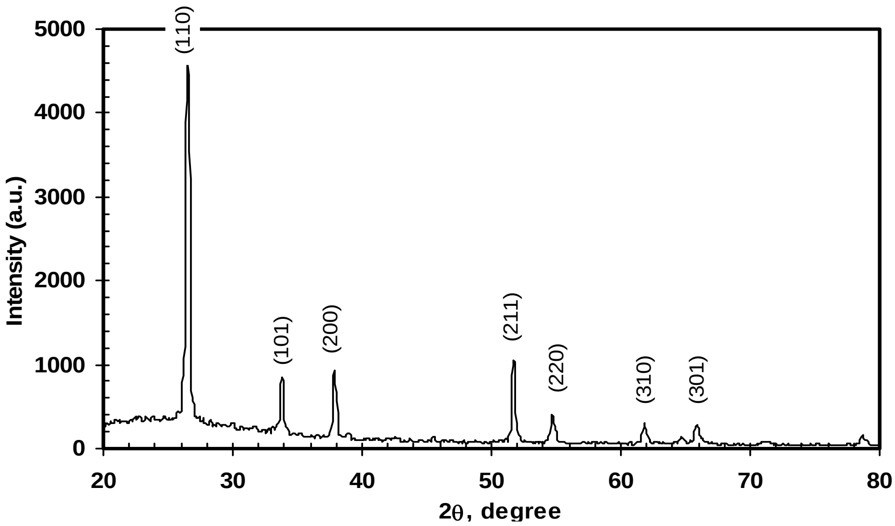
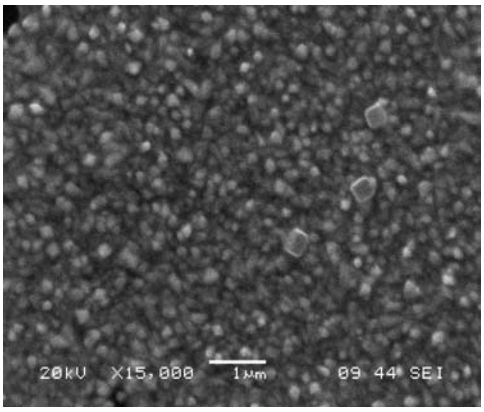
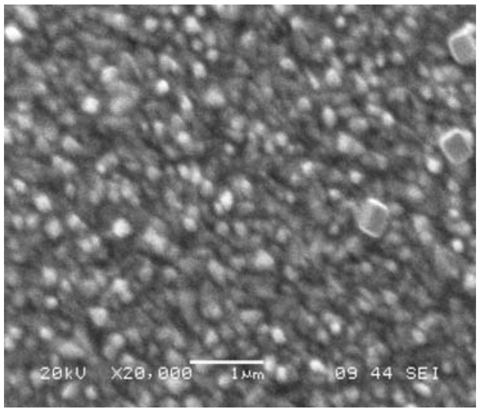


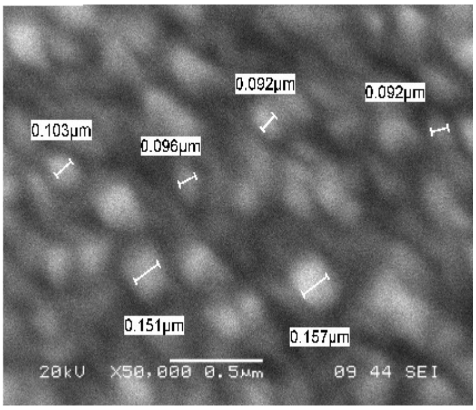
Fig. 1. X-ray diffraction pattern of SnO_2 thin film.



(a)



(b)



(c)

Fig. 2. SEM images of SnO_2 thin film at three different magnifications of (a) 15,000×; (b) 20,000×; and (c) 50,000×.

The EDAX analysis was used to examine the composition of the deposited materials. Fig. 3 shows the EDAX spectra for SnO_2 thin film composition. It is seen that the major peaks are of tin and oxygen and no other impurity. Stoichiometric mass % of Sn and O in SnO_2 are 78.77 and 21.23, respectively. The mass % of Sn and O in our sample are 83.64 and 16.36 which is not as per the stoichiometric proportion and observed to be the oxygen deficient, leading to the semiconducting nature of SnO_2.

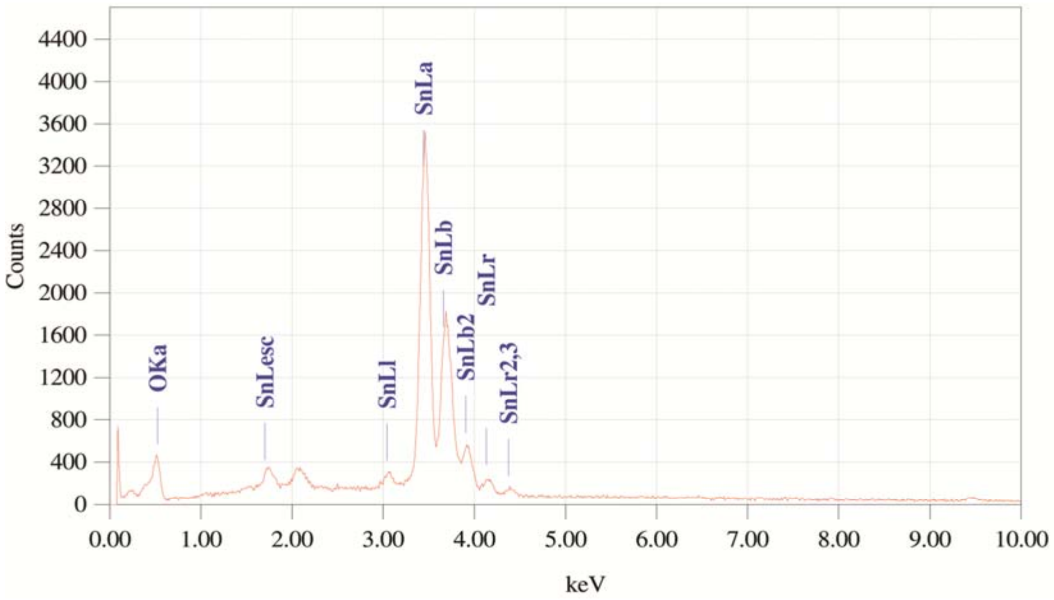


Fig. 3. EDAX spectra of SnO_2 thin film.

4.3. I-V Characteristics

Fig. 4 represents I-V characteristics of the SnO_2 thin film at room temperature. It was clear from the I-V characteristics that the contacts fabricated on the film were ohmic in nature [15]. The non-linear I-V characteristics may be due to semiconducting nature of the films. The voltage was applied in the range 1 V-25 V.

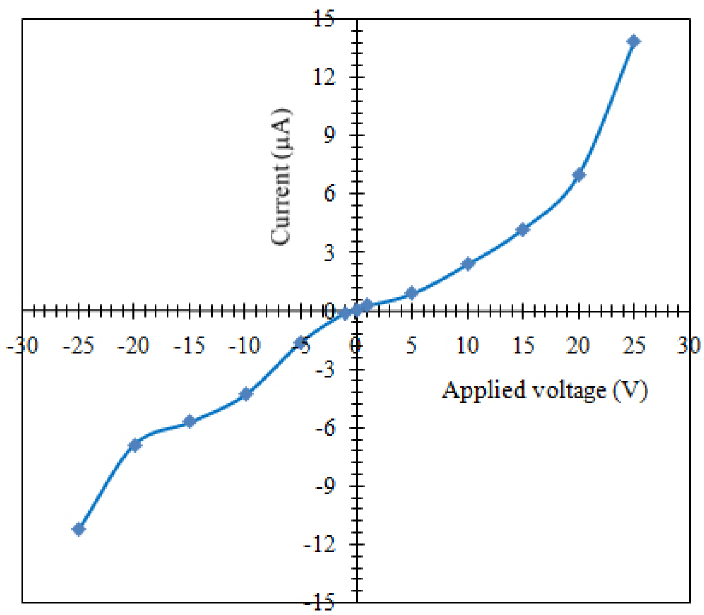


Fig. 4. I-V characteristics of the SnO_2 thin film.

4.4. Gas Sensing Properties

4.4.1. Variation of Sensor Response with Operating Temperature

Fig. 5 shows the variation of response of SnO_2 thin film sensor to 300 ppm H_2 gas with operating temperature. It is seen that for temperatures lower and higher than150 ^oC, the sensor response is less indicating 150 ^oC to be an optimum temperature to have high sensing response at different operating voltages (1 V, 5 V, 10 V, 15 V, 20 V, 25 V). The response for our sample is found to be better than that reported in literature [16, 17]. Also the operating voltage is one of the main factors that influence the sensor response to H_2 gas. The operating voltage is necessary for driving electrons in gas sensing film. However, when the operating voltage is too high, the response to H_2 gas will be decreased. The decrease of the sensor response can be explained as follows: An increase in operating voltage results in increase in electron concentration in the film and at grain boundaries. I-V characteristic (Fig. 4) shows the current in the sensor is a non-linear increase with an increasing operating voltage. The increase of current indicates increasing electric charge in the film. Since there is electrostatic repulsion between electronic excess negative charges in the film and gaseous H_2 molecules, the H_2 adsorption quantities amount in the film decreases. As a consequence, a decrease in response to H_2 of the gas sensing film is observed and shows higher response at 150 ^oC for all operating voltages.

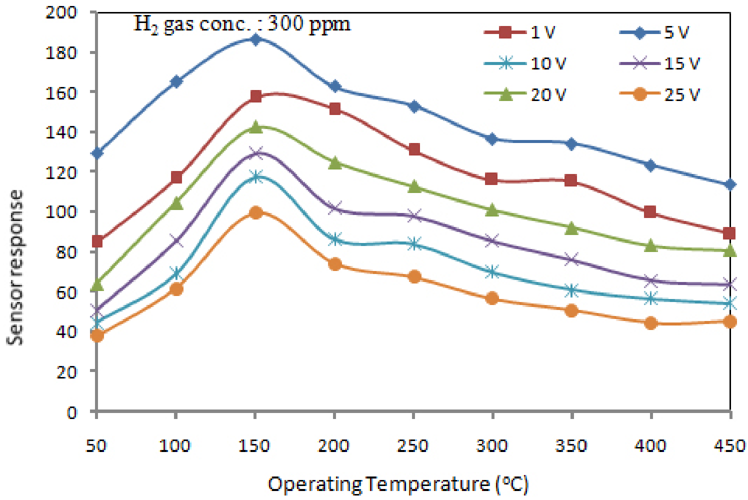


Fig. 5. Variation of sensor response to H_2 with operating temperature.

4.4.2. Sensor Response with H_2 Gas Concentration

To test the H_2 gas concentration characteristics, the sensor was exposed to H_2 gas of ten different concentrations at a constant operating temperature. The sensor responses to H_2 in concentration range (100-1000 ppm) are shown in Fig. 6 at 150 ^oC operating temperature and six operating voltages (1 V, 5 V, 10 V, 1 5V, 2 0V, 25 V). It indicates that the sensor has good response to H_2 at the operating voltage 5V. For all the operating voltages, the response values were observed to increase continuously with increasing the gas concentration up to 1000 ppm at 150 ^oC. The rate increase of response was relatively larger up to 300 ppm, smaller during 300-1000 ppm and then saturates after 300 ppm. Thus the active region of the sensor would be between 100-300 ppm. At lower gas concentration, the monolayer of gas molecules would be expected to be formed on the surface which would interact with the surface more actively giving larger responses [18]. There would be multilayer of gas molecules on the sensor surface at the higher gas concentration resulting in saturation in response.

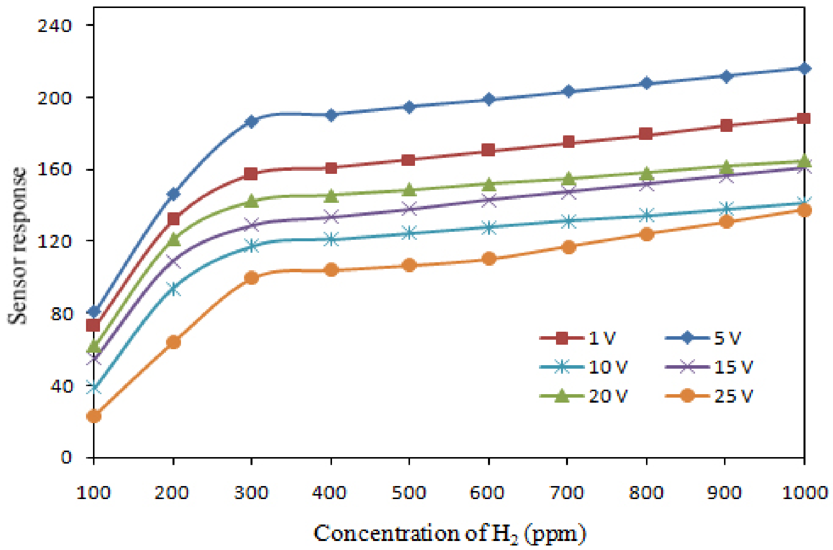


Fig. 6. Sensor response with H_2 gas concentration at 150 °C.

4.4.3. Selectivity of Sensor against Different Gases

Selectivity can be defined as the ability of a sensor to respond to a certain gas in the presence of different gases [19]. Fig. 7 depicts the selectivity of SnO_2 thin film sensor for H_2 (300 ppm) at 150 °C and at the operating voltage 5 V. The sensor showed the high selectivity for H_2 among the following gases: LPG, NH_3, CO, CO_2, C_2H_5OH, H_2S, O_2 and Cl_2.

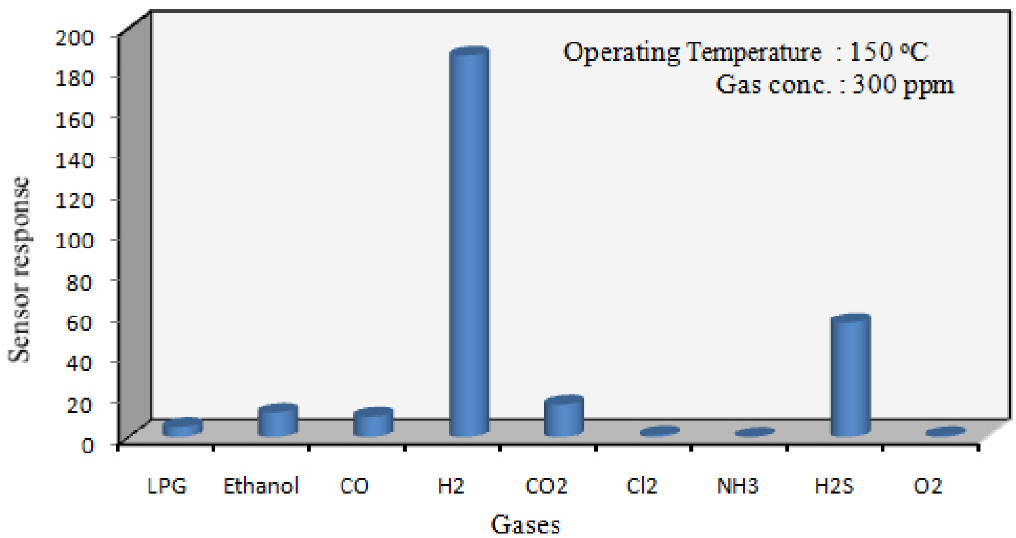


Fig. 7. Selectivity of sensor for various gases.

4.4.4. Response Time and Recovery Time

The response/ recovery time is an important parameter used for characterizing a sensor. It is defined as the time required to reach 90 % of the final change in current, when the gas is turned on and off respectively. The sensor response vs. time is shown in Fig. 8 for 300 ppm of H_2. From the plot, it is seen that the response time is 90 sec and the recovery time is 130 sec.

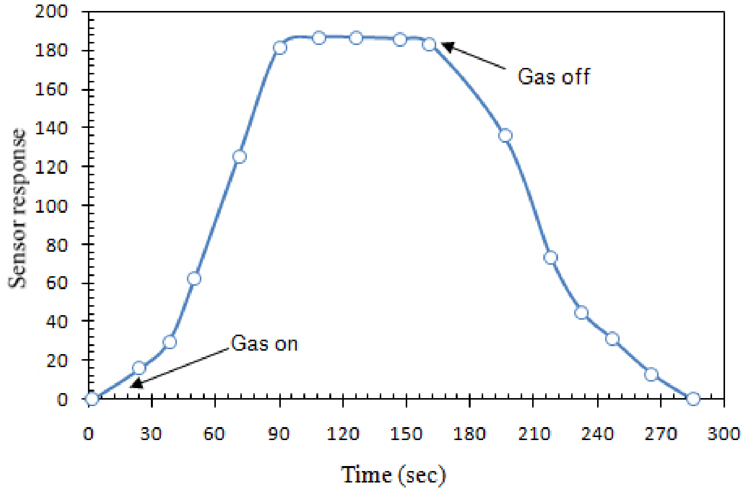


Fig. 8. A dynamic response of SnO_2 thin film sensor.

Fig. 9 shows the response and recovery time vs. concentration of H_2. It is revealed that response time decreased from 160 sec to 56 sec when H_2 concentration is increased from 100 ppm to 1000 ppm. This may be due to the presence of sufficient gas molecules at the interface for reaction to occur. From the same graph, it is found that for higher concentrations of H_2, the recovery time was long. This may be due to the reaction products are not leaving from the interface immediately after the reaction.

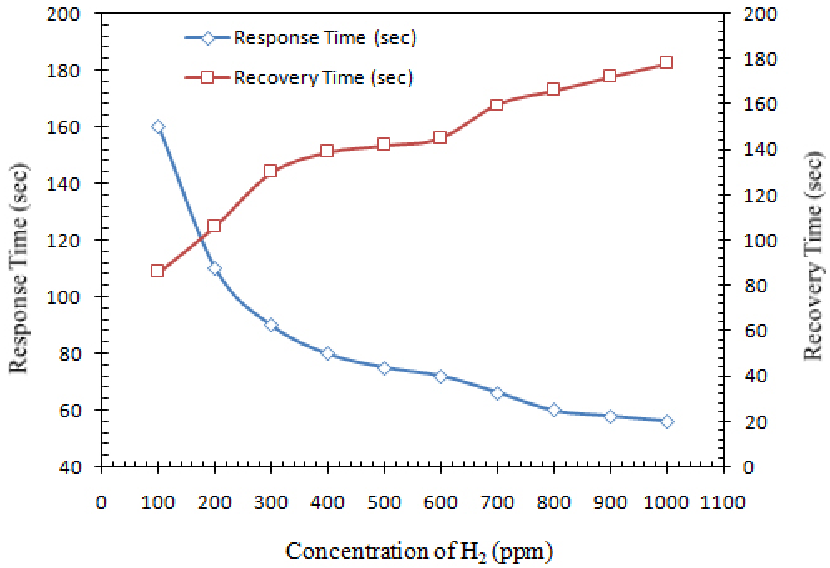


Fig. 9. Variation of response and recovery time with concentration of H_2.

4.4.5. Gas Sensing Mechanism

The gas sensing mechanism of an n-type tin oxide is based on electrical conduction results from point defects, such as oxygen vacancies and interstitial tin atoms (i.e. non-stoichiometric composition), that acts as donor [20-22]. When exposed to air, freshly prepared tin oxide particles adsorb oxygen atoms on the surface [23]:

$$O_{2(gas)} \leftrightarrow O_{2(ads)} \tag{3}$$

$$O_{2(ads)} + e^- \leftrightarrow O^-_{2(ads)} \quad (4)$$

$$O_{2(ads)} + e^- \leftrightarrow 2O^-_{(ads)} \quad (5)$$

Thus the oxygen atoms picks up the electrons from the conduction band of the tin oxide and are adsorbed on the particle surface as O^- and also other oxygen adsorbates such as O^{2-} and O_2^- are also to cover the surface.

Under H_2 exposure, a surface reaction reduces the coverage of oxygen, causing returning electrons to SnO_2 [9] by the following reactions:

$$2H_{2(gas)} + O^-_{2(ads)} \leftrightarrow 2H_2O_{(vap)} + e^- \quad (6)$$

$$H_{2(gas)} + O^-_{(ads)} \leftrightarrow H_2O_{(vap)} + e^- \quad (7)$$

The extra electrons released in this process enhance the surface conductance of the thin film. The model proposed above, explains the gas sensing mechanism of the SnO_2 thin film sensor qualitatively. But it does not explain the increase in gas sensitivity with rise in operating temperature (Fig. 5). 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 SnO_2 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. The minimum operating temperature found in the present work is only 150 ^oC. At such a low temperature, the reaction region is mostly limited to the sensor surface (Fig. 5) or at the most up to the gas-solid interface. Therefore, it is suggested that uniform granular distribution would cause high response at such a low temperature as has been observed in the present work.

5. Conclusions

From the results, following statements can be made for the sensing performance of the SnO_2 thin film sensor.

1. Polycrystalline SnO_2 thin film could be prepared by simple and inexpensive spray pyrolysis technique (SPT).
2. SnO_2 thin film sensor shows a high response to H_2 gas in a wide concentration range at operating temperature 150 ^oC and at an operating voltage 5V.
3. The sensor showed good selectivity to H_2 gas against LPG, NH_3, C_2H_5OH, CO, CO_2 and Cl_2 gases.
4. The polycrystalline SnO_2 thin film exhibits rapid response–recovery which is one of the main features of this sensor.
5. The results obtained by SPT are promising for the preparation of sensitive and low cost hydrogen sensor operating at low temperatures.

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- Sensor-based localization and tracking technologies

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The Fifth International Conference on Sensor
Technologies and Applications

SENSORCOMM 2011

August 21-27, 2011 - French Riviera, France

Important deadlines:

Submission deadline	March 23, 2011
Notification	April 30, 2011
Registration	May 15, 2011
Camera ready	May 22, 2011

Tracks:
- APASN: Architectures, protocols and algorithms of sensor networks
- MECSN: Energy, management and control of sensor networks
- RASQOFT: Resource allocation, services, QoS and fault tolerance in sensor networks
- PESMOSN: Performance, simulation and modelling of sensor networks
- SEMOSN: Security and monitoring of sensor networks
- SECSED: Sensor circuits and sensor devices
- RIWISN: Radio issues in wireless sensor networks
- SAPSN: Software, applications and programming of sensor networks
- DAIPSN: Data allocation and information in sensor networks
- DISN: Deployments and implementations of sensor networks
- UNWAT: Under water sensors and systems
- ENOPT: Energy optimization in wireless sensor networks

http://www.iaria.org/conferences2011/SENSORCOMM11.html

The Fourth International Conference on Advances
in Circuits, Electronics and Micro-electronics

CENICS 2011

August 21-27, 2011 - French Riviera, France

Important deadlines:

Submission deadline	March 23, 2011
Notification	April 30, 2011
Registration	May 15, 2011
Camera ready	May 22, 2011

Tracks:
- Semiconductors and applications
- Design, models and languages
- Signal processing circuits
- Arithmetic computational circuits
- Microelectronics
- Electronics technologies
- Special circuits
- Consumer electronics
- Application-oriented electronics

http://www.iaria.org/conferences2011/CENICS11.html

Guide for Contributors

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 additional, 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

Articles should be written in English. Authors are invited to submit by e-mail editor@sensorsportal.com 8-14 pages article (including abstract, illustrations (color or grayscale), photos and references) in both: MS Word (doc) and Acrobat (pdf) formats. Detailed preparation instructions, paper example and template of manuscript are available from the journal's webpage: http://www.sensorsportal.com/HTML/DIGEST/Submition.htm Authors must follow the instructions strictly when submitting their manuscripts.

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