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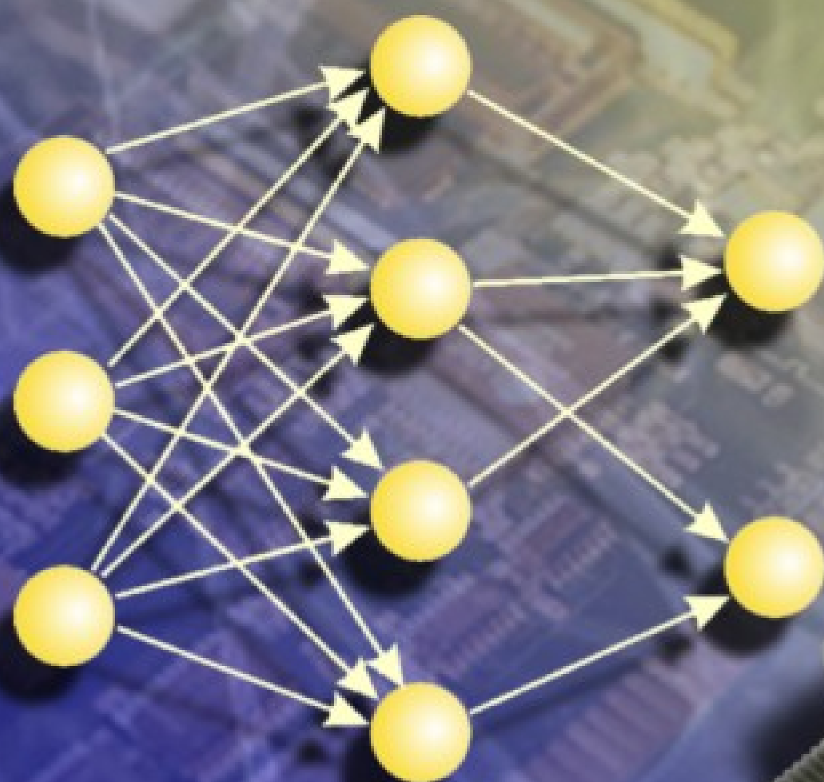

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Electrical and Gas Sensing Properties of SnO_2 Thick Film Resistors Prepared by Screen-printing Method

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Abstract: Thick films of tin-oxide (SnO_2) were deposited on alumina substrates employing screen-printing technique. The films were dried and fired at 680^0C for 30 minutes. The variation of D.C. resistance of thick films was measured in air as well as in H_2S gas atmosphere as a function of temperature. The SnO_2 films exhibit semiconducting behaviour. The SnO_2 thick films studied were also showing decrease in resistance with increase of concentration of H_2S gas. The film resistors showed the highest sensitivity to H_2S gas at 350^0C. The XRD studies of the thick film indicate the presence of different phases of SnO_2. The elemental analysis was confirmed by EDX spectra. The surface morphological study of the films was analyzed by SEM. The microstructure of the films was porous resulting from loosely interconnected small crystallites. The parameters such as grain size, activation energy, sensitivity and response time were described. *Copyright © 2008 IFSA.*

Keywords: Thick film, SnO_2, H_2S gas, Screen-printing technique, Resistivity, EDX.

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1. Introduction

It is well known that the electrical properties of semiconductor metal oxides are sensitive to the gaseous ambient (H_2S, CH_4, CO, CO_2, O_2 etc.).Semiconductor sensors are based on a reaction between the semiconductor surface and the gases in atmosphere. At certain temperature and conditions the atmospheric oxygen chemisorbed on metal oxide causes the space charge layer at the surface and produces a change in surface conductivity [1]. The electrical properties of thick film resistor are functions of several factors [2], such as ingredients, manufacturing technique and sintering history. The

main ingredients of thick film include a conducting paste, such as an oxide powder; a dielectric phase, such as glass frit; and an insulating substrate. The oxides used in thick films can be broadly classified into two groups: metallic oxides where the resistivity usually obeys a power law dependence on temperature, $\rho \propto T^n$; where n > 0; and semiconducting oxides, where the resistivity usually follows an exponential law, $\rho \propto \exp(E/KT)$.

The use of resistive, adsorption-based sensors has been increasing over the past few years for purposes such as detection of smokes, oxidizing or reducing gases and humidity [3-6]. Several materials have been used for gas sensing, including ceramics that consists of combination of metal oxides. The semiconducting oxides of tin and zinc have been widely investigated for sensing reducing gases [7, 8]. SnO_2 is one of the semiconducting materials very widely used for sensing oxidizing/reducing gases due to its high sensitivity to small concentration of gas at ppm level [1, 9-15]. These sensors are not able to differentiate the gases in a mixture, so selectivity is a serious problem. The selectivity can be improved by temperature control, using catalysts on the surface of sensing material and changing thickness of the film of sensing material.

The present work deals with preparation of thick film resistors of pure SnO_2 by the screen-printing technique and studies their electrical, gas sensing and structural properties. Studies were carried out and results are presented on the variation of electrical resistivity, gas sensitivity with different operating conditions. The results are interpreted and summarized in terms of conclusions.

2. Experimental

2.1. Preparation of $Sn0_2$ Thick Films

Tin-oxide thick films were prepared on alumina substrate by using standard screen-printing technique [1, 16-21]. The SnO_2 powder (99.99 %) was weighed and calcined in air at 400 ^{0}C for 1 hr. The calcined SnO_2 powder was crushed and mixed thoroughly with glass frit as permanent binder and ethyl cellulose as a temporary binder. The mixture was then mixed with butyl carbitol acetate as a vehicle to make the paste. The paste was then screen printed onto the surface of alumina substrate. The details of the technique are described elsewhere [18]. After screen printing the films were dried under IR- lamp for 1 hr and then fired at 680 ^{0}C for 30 minutes.

2.2. Thickness Measurements

The thickness of the SnO_2 thick films was measured by using Taylor-Hobson (Taly-step UK) system. The thickness of the films was observed in the range of 10 µm–13 µm.

2.3. Structural and Morphological Studies

Using X-ray diffraction (Miniflex Model, Rigaku, Japan) analysis from 20-80^0, 2θ was carried out to examine the final compositions of the SnO_2 films samples. The average grain sizes of tin oxide thick film samples were calculated by using the Seherrer formula [22]:

$$D = \frac{0.9\lambda}{\beta \cos\theta},$$

where D is the average grain size, λ =1.542 AU (X-ray wavelength), and β is the peak FWHM in radiation and θ is diffraction peak position. The microstructure and chemical composition of the films were analyzed using a scanning electron microscope [SEM model JEOL 6300 (LA) Germany] coupled with an energy dispersive spectrometer (EDS JEOL, JED-2300, Germany).

2.4. Electrical Behaviour and Gas Response

The D.C. Resistance of the films was measured by using half bridge method as a function of temperature.[23, 24] The gas sensing studies were carried out on a static gas sensing system [25, 26] under normal laboratory conditions. The gas sensitivity, S_{ra} of the SnO_2 thick film sensor is calculated by the relation

$$S_{ra} = \frac{R_a}{R_g},$$

where R_a is the resistances of the SnO_2 thick film resistors in air and R_g is the resistances of the SnO_2 thick film resistors in H_2S gas atmosphere gas.

3. Results and Discussions

3.1. X-ray Diffraction Analysis

In Order to understand the phases in SnO_2 film samples, the X-ray diffraction study was undertaken. X-ray diffraction analysis of SnO_2 film samples were carried out in the 20-80^0 range using Cuk$_\alpha$ radiation. Fig. 1 shows an XRD pattern of SnO_2 film samples plotted in the range 20-80^0 (2θ) verses intensity having several peaks of tin oxide phases indicating polycrystalline nature. The observed peaks match well with the reported ASTM data of Tin-oxide, confirming the polycrystalline nature. The higher peak intensities of an XRD pattern is due to the better crystallinity and bigger grain size can be attributed to the agglomeration of particles. The average crystallite size was calculated Using Scherrer equation and was estimated to be about 42.38 *nm*. Table 1 illustrates the percentage of different tin oxide phases in thick film resistor.

Table 1. Presence of different relative phases of the SnO_2 [27].

Firing temperature(^{o}C)	% relative presence of phases				
	T	O	Sn_3O_4	Al_2O_3	Average grain size (±2 nm)
680	29.54	25.58	15.74	29.14	42.38

3.2. Scanning Electron Microscopy

Scanning electron microscopy is convenient technique to study the microstructure of SnO_2 thick film samples. Fig. 2 shows the surface morphology of SnO_2 films observed by SEM. Both the images are recorded at 20000x magnification for the comparison. The SEM in Fig. 2 (a), shows the surface morphology of the unfired film sample which is not uniform throughout the region but it is without any void, pinhole and it covers the substrate well. Fig. 2 (b) shows the SEM for SnO_2 film sample fired at 680 ^{0}C. The micrograph of this sample shows voids between the particles are basically due to

evaporation of the organic solvent during the firing of the film. The micrograph also shows the presence of more agglomeration in the film samples.

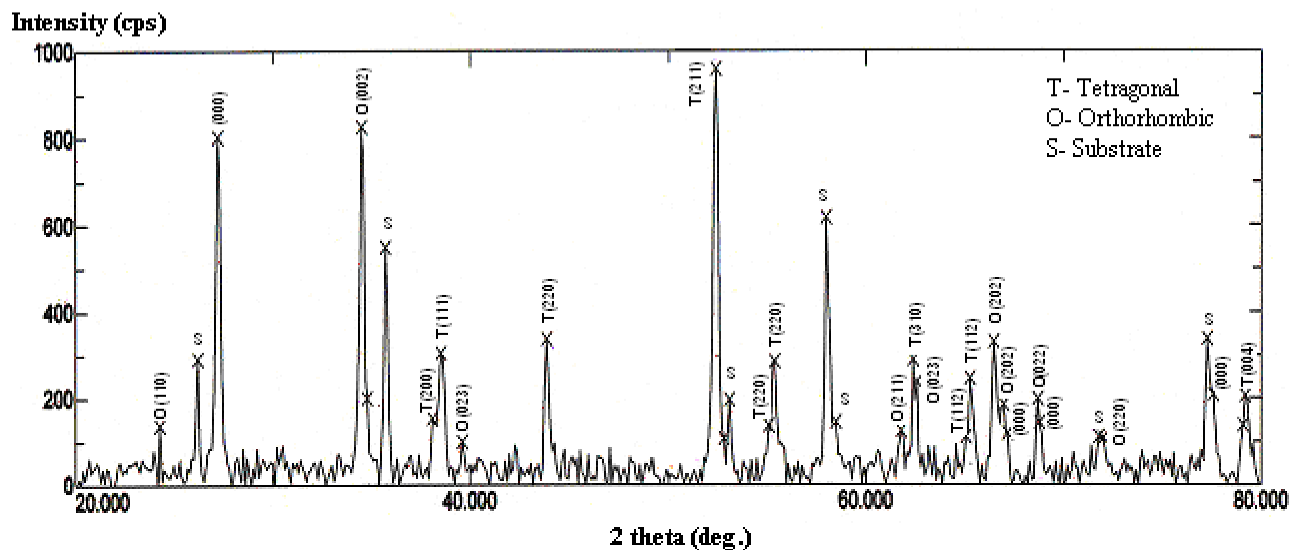
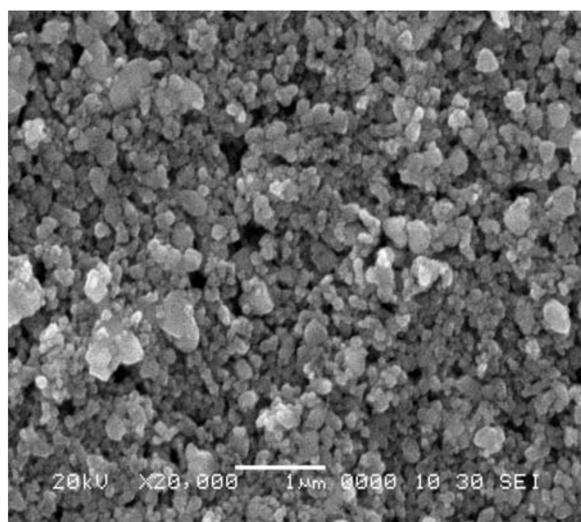
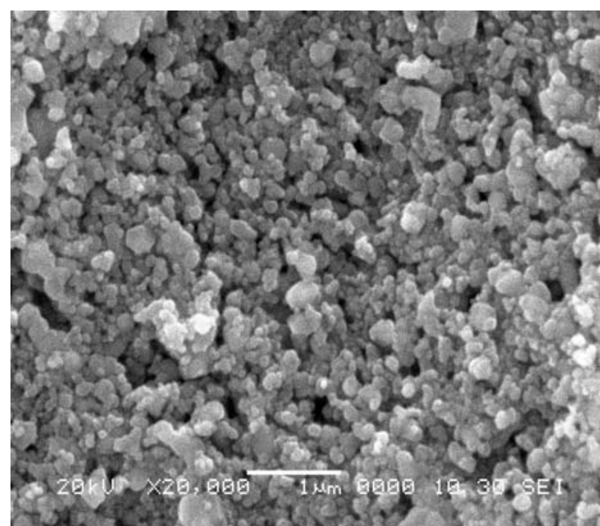


Fig. 1. X-ray diffraction pattern of a SnO_2 Thick Film fired at 680 ^oC.



(a) SEM image of unfired SnO_2 Thick film



(b) SEM image of fired SnO_2 Thick film.

Fig. 2. SEM images of SnO_2 Thick Films.

3.3. Energy Dispersive X-Ray Analysis

The EDX analysis was used to examine the composition of the film materials. Fig. 3 shows the EDX spectra for SnO_2 thick film composition .It is seen that the major peaks are of tin and oxygen and no other impurity elements are presents in the composition. Also from the spectra it is seen that weight % and atomic % are nearly matched illustrated in Table 2.

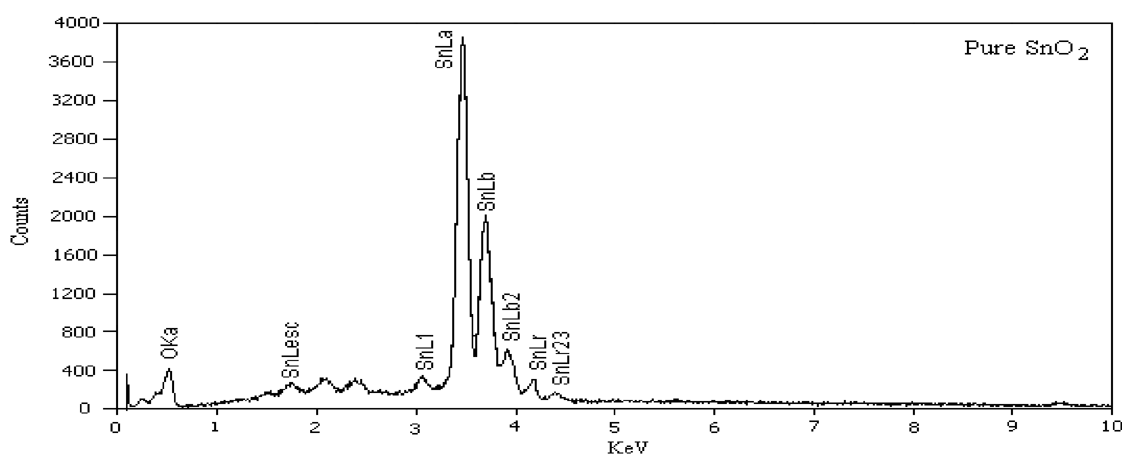


Fig. 3. EDX Analysis spectra of SnO_2 thick films fired at 680^0C.

Table 2. Quantitative elemental analysis.

Sample- SnO_2	Mass%	At. %	Error %
Sn	79.55	34.50	1.11
O	20.45	65.50	2.07
Total	100.00	100.00	---

3.4. Electrical Characteristics

Fig. 4 shows the resistance variation of SnO_2 thick film with temperature in air and H_2S gas (ppm) atmosphere. There is an exponential decrease in resistance with increase of temperature indicating semiconducting behaviour in the temperature range of 100 to 400 ^{0}C. The resistance of $Sn0_2$ film samples upon exposer to H_2S gas was lower than that in air. The initial value of SnO_2 thick film resistance in air atmosphere (at 100^oC) was 499990 M-ohm. The systematic decrease in resistance of film up to 428561 M-ohm upon exposer to H_2S gas is attributed to the decrease in potential barrier at grain boundaries, when H_2S gas comes in contact with grain boundaries. Normally the resistance of SnO_2 thick film samples can be decreased due to the following two reasons [28]: (i) any increase in temperature of thick film resistor causes the electrons to acquire enough energy and cross the barrier. Due to this the effective resistance of thick film resistor drops down to steady levels at high enough temperature. (ii) Any reducing gas at ppm level reacts with the adsorbed oxygen at the grain boundaries of the thick film sample and the potential barrier reduces and free electrons cross the boundary easily. This reduces the effective resistance of thick film resistor. As the gas concentration increases, the resistor resistance decreases. This continues until the concentration of reducing gases is high enough to react with the adsorbed oxygen radical fully. Beyond that, the effective resistance of the resistor remains unchanged.

The relationship between sensor resistance and the concentration of reducing gas can be expressed by the following equation over a certain range of gas concentration: [29].

$$R_s = A[C]^{-\alpha},$$

where R_s is the electrical resistance of the sensor material; A is the constant; [C] is the gas concentration; α is the slope of R_s curve.

Fig. 5 shows the adsorption of oxygen species on the surface of SnO_2, abstracting electrons and thus, causing an increase in potential barrier at the grain boundaries. When reducing gas such as H_2S, comes into contact with the grain boundaries of SnO_2, potential barrier will decrease as a result of oxidation of H_2S and desorption of oxygen. In the present case the decrease in resistivity of SnO_2 thick film sensor in the presence of H_2S gas may be explained by the reaction as [30-31],

$$2H_2S(g) + 3O_2(S) \rightarrow 2SO_2(g) + 2H_2O + 6e^-$$

According to this reaction, the interaction of H_2S with previously adsorbed O^{2-} ions leads to the injection of electrons into the depletion layer of SnO_2 grains.

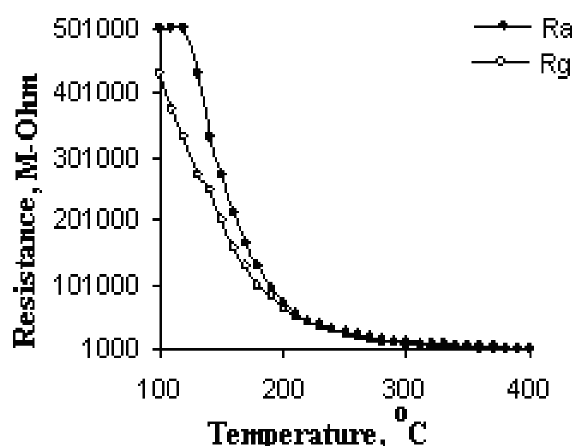


Fig. 4. Variation of resistance with temperature of SnO_2 thick film.

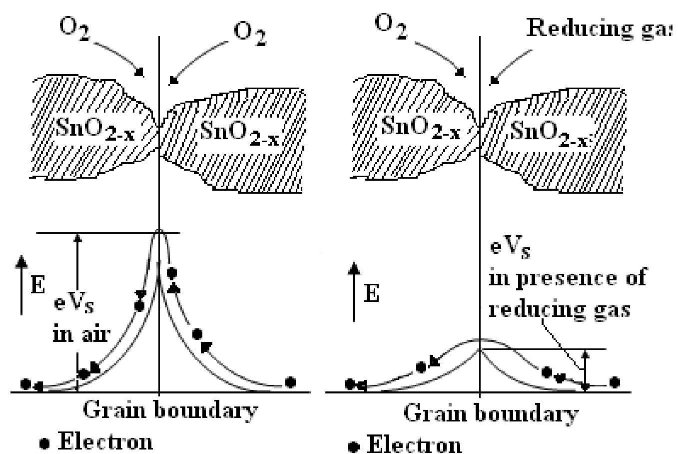


Fig. 5. Gas sensing mechanism of SnO_2 thick resistors to H_2S gas.

To study the conduction mechanism in SnO_2 thick films, resistivity measurements in air as well as in H_2S gas atmosphere were performed at different temperatures. In Fig. 6 the plot of log R versus reciprocal of temperature for SnO_2 thick films are shown. The calculated resistivity was found to follow the Arrhenius equation:

$$R=R_o\, e^{-E/kT},$$

where R_o is the constant, E is the activation energy of the electron transport in the conduction band, K is the Boltzman constant and T is the absolute temperature. Fig. 6 shows two linear regions for SnO_2 films, revealing two activation energies, one for higher temperature (413 to 663 ^{0}K) and other for lower temperature region (363 to 413 ^{0}K). The activation energy in the low temperature region is always less than the energy in the high temperature region because material passes from one conduction mechanism to another [32]. In this region activation energy decreases, because a small thermal energy is quite sufficient for the activation of charge carriers to take part in the conduction process. In other words the vacancies/defects weakly attached in lattice can easily migrate. Hence increase in conductivity in the lower temperature region can be attributed to the increase of charge mobility.

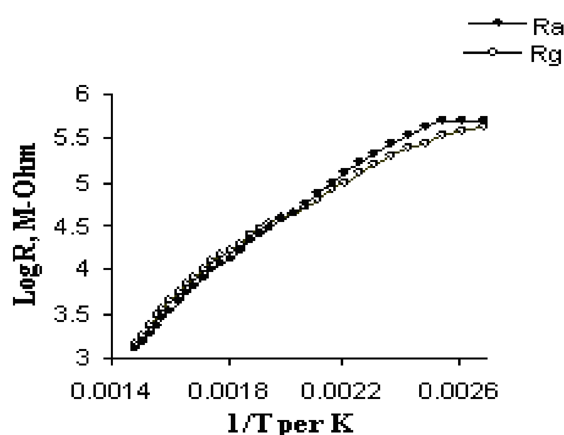


Fig. 6. Plot of Log R versus 1/T for SnO_2 thick film.

In high temperature region, the activation energy is higher than that of low temperature region. In this region the electrical conductivity is mainly determined by the intrinsic defects and hence is called high temperature or intrinsic conduction. The high values of activation energy obtained for this region may be attributed to the fact that the energy needed to form defects much larger than the energy required for its drift. For this reason, the intrinsic defects caused by the thermal fluctuations determine the electrical conductance of the samples only at elevated temperature. Table 2 illustrates the activation energies of SnO_2 film in different temperature regions.

Table2. Activation energy of SnO_2 thick film resistor

	Activation Energy, eV	
Temperature Region → / Medium ↓	Low temperature region	High temperature region
Air	0.4718	0.5289
H_2S gas	0.2695	0.6209

3.5. Measurement of Gas Response

3.5.1. Gas Response (Sensitivity)

The steady-state gas sensing properties of SnO_2 thick films are presented. Initially, the variation of the sensitivity with the temperature was scanned from 100 to 400 ^{0}C. Fig. 7 shows the typical variation of the sensitivity as a function of the operating temperature for SnO_2 thick film exposed to 600 ppm of H_2S in air. The sensitivity raises up to 350 ^{0}C and then falls at higher temperatures [33]. It is generally accepted [34-36] that the surface conductance of the sensor increases with the partial pressure P_g of the test gas in ambient air according the relation

$$G_g = G_a + \gamma (P_g)^{1/2},$$

where G_a is the conductance of the sensor in absence of the test gas in ambient air and γ is the constant proportionality governed by the adsorption process.

Also above 350 ^{0}C temperature, the surface of SnO_2 film sample would be unable to oxidize the H_2S gas so intensively. Therefore, the sensitivity decreases further with increasing temperature [37].

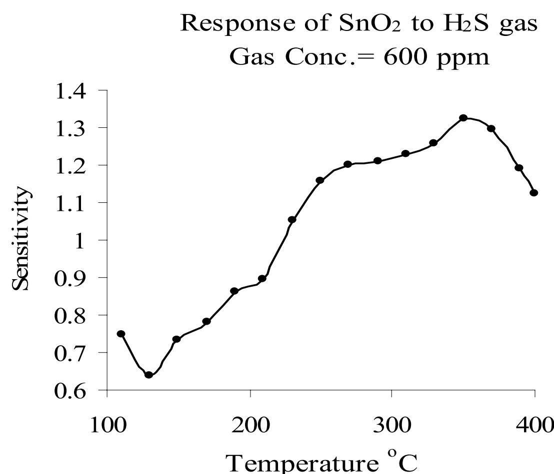


Fig. 7. Variation in sensitivity with operating temperature of SnO_2 thick film resistor.

3.5.2. Response Time and Recovery Time

The time taken for sensor to attain 90 % of maximum change in conductance upon exposure to H_2S gas is the response time. The time taken by the sensors to get back 90 % of original conductance is the recovery time [26]. Response and recovery time of the sensor were measured. Fig. 8 shows the typical change in the response with time for SnO_2 thick film resistor maintained at 350 ^{0}C after 600 ppm of H_2S has been injected into the chamber. The 90 % response and recovery levels were attained within 15 and 40 sec respectively.

3.5.3. Effect of Gas Concentration (Active Region)

The variation of gas response of SnO_2 thick film samples with H_2S gas concentration is represented in Fig. 9.It is clear from the figure that the gas response goes on increasing with gas concentration up to 600 ppm. The rate of increase in response was relatively larger up to 600 ppm and after that reaches to saturation value, so we can infer that the active region of the SnO_2 film resistor would be up to 600 ppm.

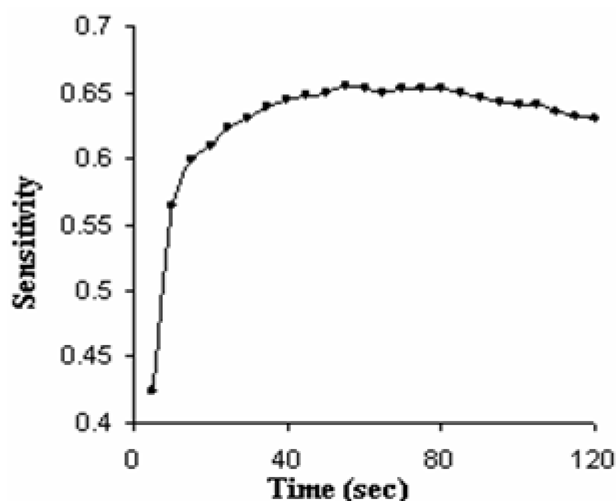


Fig. 8. Transient response of SnO_2 film resistor to H_2S gas (600 ppm) at 350 ^{0}C.

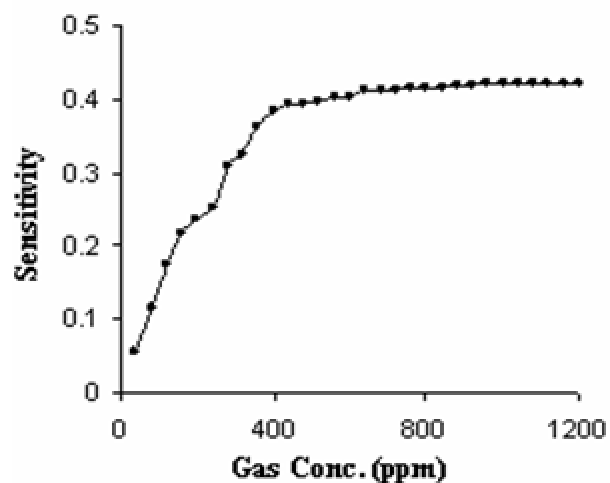


Fig. 9. The sensitivity of SnO_2 film resistor as a function of H_2S gas concentration at 350 ^{0}C.

4. Conclusions

Following conclusions can be drawn from the experimental results.

1. The SnO_2 thick film resistors was observed to be semiconducting in nature and showed a negative temperature coefficient of resistance.
2. The resistance of the SnO_2 thick film resistors upon exposer to H_2S gas was lower than in air due to disorption of oxygen species by reaction with the H_2S gas.
3. The SnO_2 films showed extremely high response to H_2S gas at 350 ^{0}C.
4. The sensing mechanism of the SnO_2 thick film resistors was the surface controlled mechanism (adsorption/desorption).

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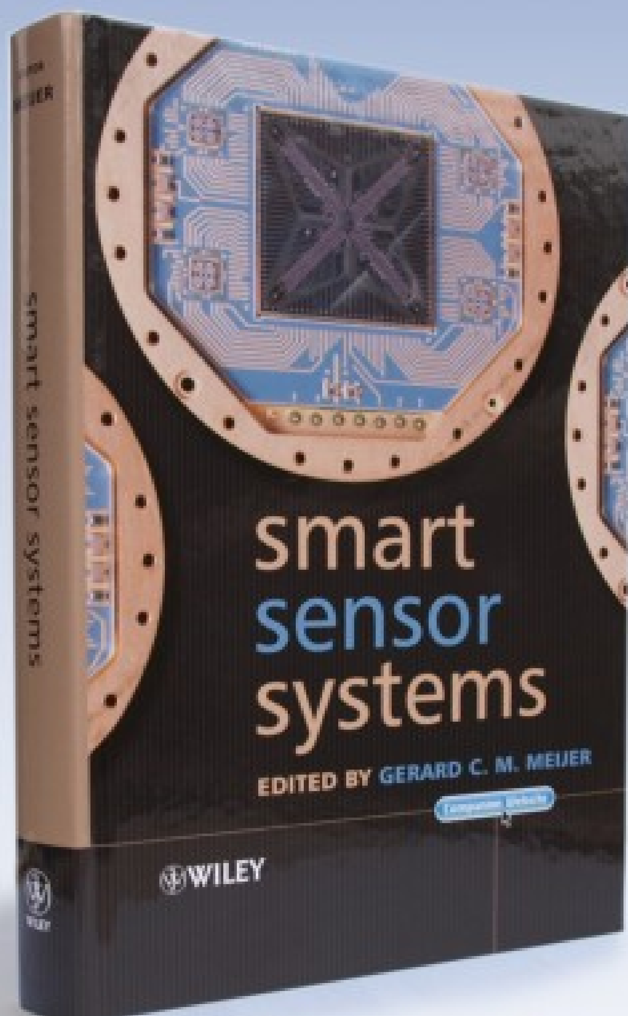

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