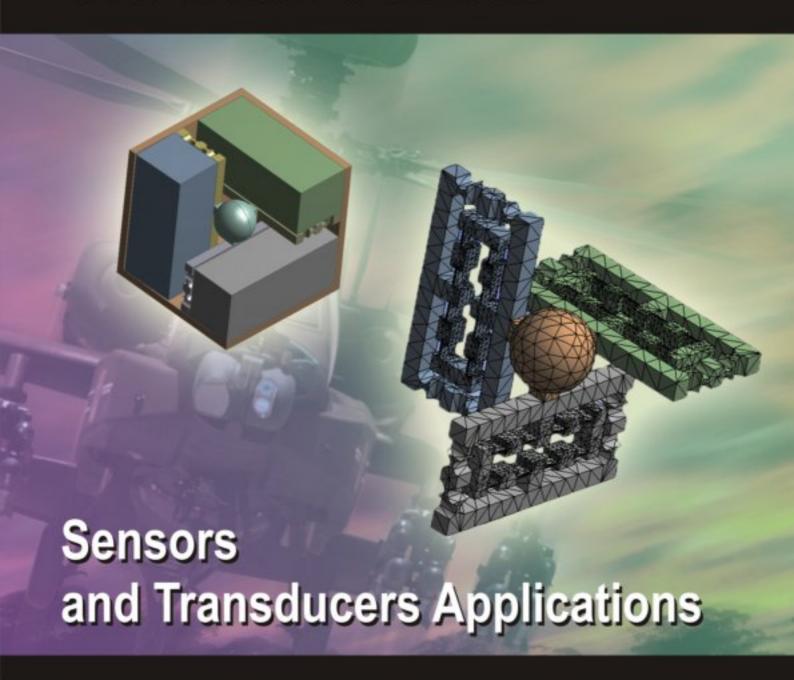
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Contents

Volume 119 Issue 8 August 2010

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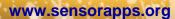
Research Articles

Fully Decoupled Compliant Parallel Mechanism: a New Solution for the Design of Multidimensional Accelerometer Zhen Gao and Dan Zhang	1
Optical Temperature Probe Based on the Fluorescence Decay Time of Tris- (dibenzoylmethane) mono (5-amino-1,10-phenanthroline)-europium(III) Hung T. Lam, Leah Tolosa, Govind Rao	14
Applications of InAs Hall Effect Sensors H. Bourbaba, C. Benachaiba, S. Kadri, A. Saidane	22
A Simple Model for Complex Fabrication of MEMS based Pressure Sensor: A Challenging Approach Himani Sharma, P. A. Alvi and J. Akhtar	30
Acoustic Temperature Transducer Tariq M. Younes, Mohammad A. K. Alia, Shebel Al-Sabbah.	46
Theoretical Performance of GaAs Solar Cell, with Band Gap Gradient Layer on the Back Region Hassane Benslimane, Hemmani Abderahman, and Helmaoui Abderrachid	58
Design of Simple Instrumentation System for the Quality Analysis of Milk (Casein Analysis) V. G. Sangam, M. Sandesh., S. Krishna., S. Mahadevanna	65
Development of a Portable Water Quality Analyzer Germán Comina, Martin Nissfolk, José Luís Solís	72
A Modified Technique of Active Power Measurement for Industrial Frequency Applications Satish Chandra Bera and Dhritinandan Kole	82
New Organic Solvent Free Three-Component Waterproof Epoxy-Polyamine Systems C. M. Lacnjevac, S. Zlatković, S. Cakić, J. Stamenković, M. B. Rajkovic, G. Nikolić and S. Jelic	91
Measurement Equation is to be Extensively Used: What One May Expect from Dynamic Measurements Kristina Abramchuk, George Abramchuk	104
Preparation and study the Electrical, Structural and Gas Sensing Properties of ZnO Thick Film Resistor M. K. Deore, V. B. Gaikwad, N. K. Pawar, S. D. Shinde, D. D. Kajale, G. H. Jain	117
Amperometric Glucose Biosensor based on Immobilization of Glucose Oxidase in Polyethylenemine and Poly (carbamolylsuphonate) Polymer Matrix U. B. Trivedi, D. Lakshminarayana, I. L. Kothari, N. G. Patel, H. N. Kapse, P. B. Patel, C. J. Panchal	129
Development of Bio-analyzer for the Determination of Urinary Chloride R. Vasumathi , P. Neelamegam	142

Prasanta Sarkar, Sagarika Pal, Swadhin Sambit Das	151
Parameter Estimation and Speed Control of PMDC Servo Motor Using Method of Time Moments Prasanta Sarkar, Sagarika Pal, Swadhin Sambit Das	162
Determination of the Region of Stabilizing Controller Parameters of Polytopic Polynomials I. Thirunavukkarasu, V. I. George, Mukund Kumar Menon, S. Shanmuga Priya	174
Identification of Natural Ventilation Parameters in a Greenhouse with Continuous Roof Vents, using a PSO and GAs Abdelhafid Hasni, Belkacem Draoui, Mahieddine Latfaoui and Thierry Boulard	182
Dynamic Modeling of Step Climbing Wheeled Robot Srijan Bhattacharya, Neeta Sahay, Sagarika Pal, Subrata Chattopadhyay	193
ECG Signal Denoising and QRS Complex Detection by Wavelet Transform Based Thresholding Swati Banerjee, Dr. Madhuchhanda Mitra	207
Admittance, Conductance, Reactance and Susceptance of New Natural Fabric Grewia Tilifolia V. V. Ramana C. H., Jayaramudu J., Jeevan Prasad Reddy D., Madhusudhana Rao K. and Varadarajulu A.	215
Role of Catecholamine in Tumor Angiogenesis Linked to Capacitance Relaxation Phenomenon Guangyue Shi, Guanjie Sui, Jianxia Jiang, T. K. Basak	223

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- 15 October 2010: Abstract submission deadline
- 15 November 2010: Notification of acceptance

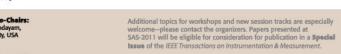
• 10 January 2011: Final manuscript submission deadline



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Preparation and Study the Electrical, Structural and Gas Sensing Properties of ZnO Thick Film Resistor

^{1*}M. K. DEORE, ²V. B. GAIKWAD, ³N. K. PAWAR, ²S. D. SHINDE, ⁵D. D. KAJALE, ⁵G. H. JAIN

^{1*}Dept. of Physics, Arts, Commerce and Science College, Ozar (Mig) – 422 206, India
 ²Dept. of Chemistry, K.T.H.M. College, Nashik -422 005, India
 ³Dept. of Physics, K.A.A.N.M.Sonawane College Satana, India
 ⁵Dept. of Physics, Arts, Commerce and Science College, Nandgaon- 423 106, India, E-mail: ^{1*}deoremadhav@rediffmail.com, ⁵gotanjain@rediffmail.com

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Abstract: Thick films of AR grade ZnO were prepared on glass substrate by screen-printing technique. These films were dried and fired at different temperatures between 550 °C, 600 °C and 650 °C for one hour in air atmosphere. The gas sensing performance of thick films was tested for various gases. ZnO films showed larger response (sensitivity) to H₂S gas (100 ppm) at 250 °C for firing temperature 650 °C. The Morphological, Compositional and Structural properties of the ZnO thick films were performed by Scanning electron microscopy (SEM), Energy dispersive spectroscopy (EDX) and XRD technique respectively. Chemical composition of ZnO film samples changes with firing temperature showing non-stoichiometric behaviours. XRD study indicated the formation of polycrystalline ZnO films with hexagonal wurtzite structure. The gas response (sensitivity), selectivity, response and recovery time of the sensor were measured and presented. *Copyright* © 2010 IFSA.

Keywords: ZnO gas sensor, Thick films, Sensitivity, Selectivity, Response and recovery time.

1. Introduction

Growing industrialization and ever increasing pollutants from vehicular exhaust have resulted into increased air pollution. The problems related to air quality monitoring are important issues of the current research activity. In fact, a key component in many process controls, product development, environmental monitoring etc. is the measurement of concentration of one or the other gaseous component of the ambient. In such situations suitable sensors can provide the necessary interface

between the ambient and the back up electronic instrumentation to detect the target gas. Solid-state sensors have dominated this field for over the past three decades [1, 2].

A wide range of gas sensors are developed using metal oxide semiconductors (MOS) because of its advantageous features like sensitivity to the ambient conditions, low material cost, easy processing and simplicity in fabrication.[3, 4]. The most commonly used n-type MOS in gas sensors are based on ZnO [5–8], SnO₂ [9], TiO2 [10], In2O3 [11], known for their changes in electrical conductivity when exposed to test gases. At elevated temperatures (100 °C –600 °C), The choice of n-type semiconductors is mainly related to their increased resistance during first oxygen adsorption and the subsequent decrease of resistance after oxygen removal by ambient reducing agents; this is usually the preferred direction for sensing reducing agents, since it requires very simple circuitry.

Among the n-type semi conducting oxides, ZnO is one of the metal oxide semiconducting material of wurtzite structure, with a direct large-band gap of about 3.37 eV at low temperature and 3.30 eV at room temperature [12-16]. It is sensitive to many sorts of gases at moderate temperature, ZnO is one of the most widely applied oxide-gas sensor. ZnO gas sensing materials owe to their high chemical stability, low cost, and good flexibility in fabrication. ZnO sensor elements have been fabricated in various forms including single crystal [17, 20, 21, 34], sinter pellet [22, 23, 24], thin film [19, 25, 26-30, 32], and thick film [18, 31, 33, 35].

The aim of this work is to describe the behavior of thick-film gas sensors obtained by screen-printing a ZnO-based paste on glass substrate the thick film semiconductor sensors typically porous and sintered metal oxides. The advantages are well known: versatility in the conception, miniaturization, mass production at low cost, etc.

In this work the ZnO film show the good response to the Hydrogen sulphide (H_2S) gas. H_2S is a toxic gas with a peculiar foul smell. It is corrosive and is naturally occurring due to decomposition of some organic matter in wastewater. It is also used in large quantities to extract heavy water. Monitoring and control of H_2S in ambient is therefore important in laboratories and industrial areas where it is used as a process gas, generated as a byproduct or produced naturally in wastewater swamps.

2. Operating Principle of Gas Sensor

The most general working principle of the semiconductor gas sensors proposed is a simple receptivity change due to adsorption – desorption of gases. The electrical conductance of a highly porous semi conducting thick-film sensor changes in the presence of toxic gases due to two main reactions occurring on the surface [36]. In the first reaction atmospheric oxygen molecules are physisorbed on the surface sites, which while moving from site to site, get ionized by taking an electron from the conduction band and is thus ionosorbed on the surface as O ads [37] (see Fig. 1). This leads to a decrease in conductance of the sensor material.

The resulting equation is

$$O_2 + 2e^- \rightarrow 2O^- ads$$
 (1)

In the case of ZnO films, electrons are also extracted from the interstitial zinc atoms Zn_i^{2+} (which act as an electron donor) [38]. The interstitial zinc atoms in such cases are ionized via the following reversible reaction

$$\operatorname{Zn}_{i}^{2+} \longleftrightarrow \operatorname{Zn}_{i}^{+} + e^{-}$$
 (2)

In the second step the reducing gas (*R*) like H₂S present in the air ambient, reacts with the chemisorbed oxygen, thereby releasing an electron back to the conduction band and increasing the conductance of the semiconductor.

$$R + O^{-} ads \rightarrow RO + e^{-}$$
 (3)

At higher temperature *RO* desorbs.

If the operating temperature is low, the product *RO* will not desorb, thus passivating the surface for further adsorption of oxygen. On the other hand if the operating temperature is too high, oxygen will not physically adsorb [39]. Therefore, the sensor performs only within a specific temperature window.

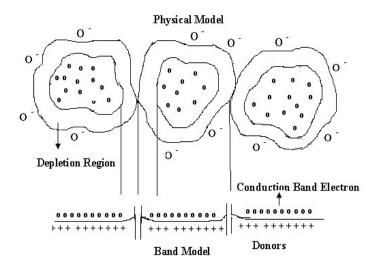


Fig. 1. Physical and band model of semiconductors.

3. Experimental

3.1. Preparation of ZnO Films

The AR grade ZnO powder (99.9 % pure) was milled for 2 hr to obtain fine-grained powder. The thixotropic paste was formulated by mixing the fine powder of ZnO with a solution of ethyl cellulose (a temporary binder) in a mixture of organic solvents such as butyl cellulose, butyl carbitol acetate and terpineol, etc. The ratio of the inorganic to organic part was kept at 75:25 in formulating the paste. This paste was screen printed [40, 41] on a glass substrate in a desired pattern. The films were fired at 550 °C for 30 min. Silver contacts are made for electrical measurements.

3.2. Details of Gas Sensing Unit

The sensing performance of the sensors was examined using a 'static gas sensing system' [42], shown in Fig. 2. 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. The required gas concentration inside the static system was achieved by injecting a known volume of a test gas using a gas-injecting syringe. A constant voltage

was applied to the sensor, and the current was measured by a digital Pico ammeter. The air was allowed to pass into the glass chamber after every gas exposure cycle.

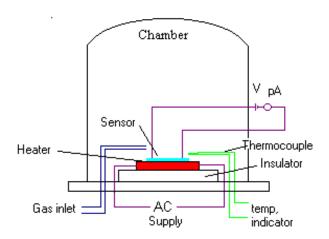


Fig. 2. Block diagram of gas sensing unit.

4. Material Characterization

4.1. X-ray Diffraction

In Order to understand the structural properties of ZnO film samples fired at different temperatures in air atmosphere, the X-ray diffraction study was undertaken. X-ray diffraction analysis of ZnO film samples were carried out in the $20-80^{-0}$ range using Cuk α radiation. Fig. 3 shows an XRD pattern of ZnO film samples plotted in the range $20-80^{-0}(2\theta)$ verses intensity having several peaks of zinc oxide indicating random orientation for the polycrystalline nature and measured interplaner distances agreed with the values reported for ZnO in the literature. The observed peaks match well with the reported ASTM data of Zinc 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 [43].

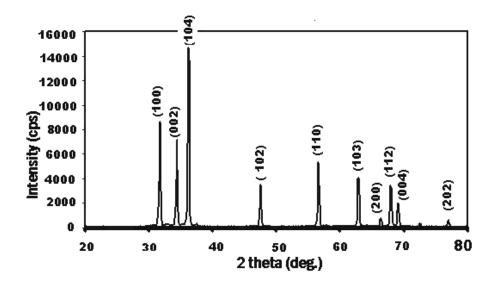


Fig. 3. XRD Pattern of ZnO.

4.2. Micro Structural Analysis

Micro structural chacterization was carried out by using scanning electron microscopy. Fig. 4 (a, b, c) shows SEM images of ZnO thick film fired at 550 $^{\circ}$ C, 600 $^{\circ}$ C and 650 $^{\circ}$ C respectively. The micrographs consists of large number of grain sizes ranging from 0.1 μ m to 1 μ m, leading to high porosity and large effective surface area available for the adsorption of oxygen species.

The comparison of these micrographs shows the interesting changes in morphology. It is found that the grain size and the crystalline quality increased with increase in firing temperature. The firing temperature increases the atomic mobility; the atoms can be moved to more energetically favoured sites such as voids, grain boundaries and interstitial positions. An increase in temperature improves the crystallinity and thus increases the mobility of atoms at the surface of films.

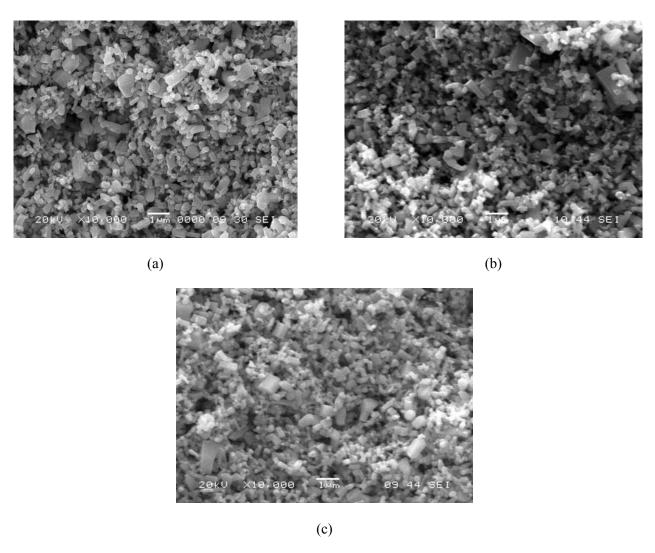


Fig. 4. SEM image of ZnO film fired at (a) 550 °C; (b) 600 °C; and (c) 650 °C.

4.3. Elemental Analysis

The Composition of ZnO thick films fired at different temperatures was analyzed by energy dispersive spectrometer (6360LA) (EDX). Table 1 shows the composition of the films fired at different temperatures. The EDAX spectrum showed the presence of only Zn and oxygen. The mass percentage

of oxygen was found to increases with increasing firing temperature and it shows the adsorption of oxygen increases with temperature on the surface of film. From the analysis it was found that ZnO films are non-stoichiometric.

Firing Temperatures	Element	At.Wt. %	Mass %
550 °C	0	46.90	17.77
	Zn	53.10	82.33
600 °C	0	47.87	18.35
	Zn	52.13	81.65
650 °C	0	48.52	18.74
	7n	51.48	81.26

Table 1. Composition of the films at different firing temperature.

5. Electrical Properties

5.1. I-V Characteristics

Fig. 5 shows the I-V characteristics of pure ZnO film at room temprature in the air atmospere. The linearity in the graph indicate the ohmic nature of contact.

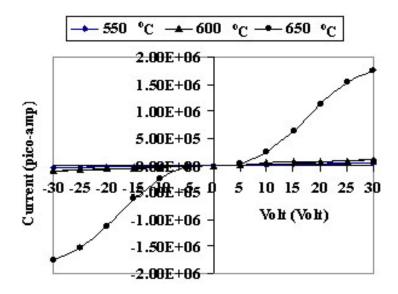


Fig. 5. I-V Characteristics of pure ZnO.

5.2. Electrical Conductivity

Fig. 6 represents the variation of conductivity with temperature for ZnO films in air ambience for different temperatures. The conductivity of these films goes on increasing with increase in temperature, indicating negative temperature coefficient (NTC) of resistance. This shows the semi conducting nature of the films. The film fired at 550 °C shows the large conductivity than other firing temperatures because as the temperature increase the adsorption of oxygen species on surface of film are more and hence the conductivity decrease.

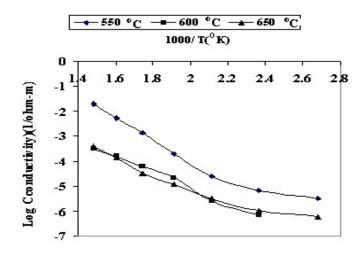


Fig. 6. Variation of conductivity with temperature.

6. Gas Sensing Performance of ZnO Film

The conductance of the sensor in dry air was measured by means of conventional circuitry by applying constant voltage and measuring the current by Pico ammeter. The conductance was measured both in the presence and absence of test gas. The gas response(s) (sensitivity with temperature) is defined as the ratio of change in conductance in gas to air to the original conductance in air

$$S = Gg - Ga / Ga, \tag{4}$$

where Ga= conductance of sensor in air;

Gg = Conductance of sensor in gas.

6.1. Gas Response with Temperature

The gas sensing performances of thick films were tested for various gases. Fig. 7 represents the variation in the gas response at different firing temperature for various gases at 100 ppm with operating temperatures ranging from 50 °C to 450 °C. It is noted from the graph that response increases with further increase in temperature from 150 °C to 300 °C and then decreases with further increase in operating temperature.

The film fired at 650 °C show the maximum response H₂S gas at 250 °C as compared to the other firing temperatures.

6.2 Selectivity of Pure ZnO Film

The selectivity or specipity is defined as the ability of a sensor to respond to certain gas in the presence of other gases.

Fig. 8 represents the bar diagram indicating selectivity of pure ZnO film fired at 650 °C and operated at different temperatures for different gases. It is clear from the bar diagram that the ZnO film is the more selective to H₂S gas against the other gases.

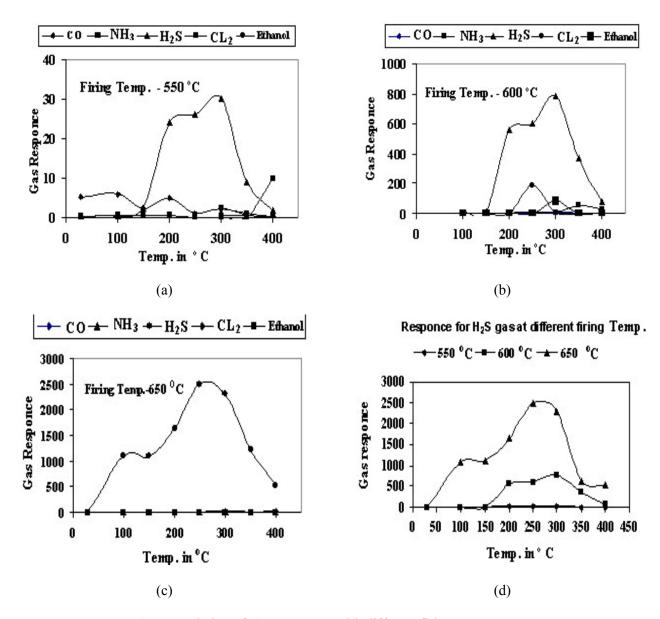


Fig. 7. Variation of Gas Response with different firing temperatures.

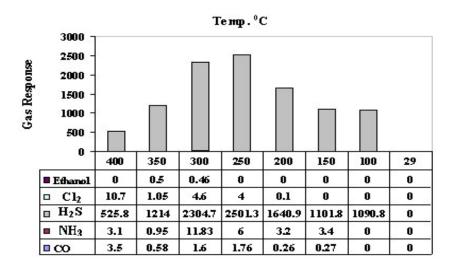


Fig. 8. Selectivity of ZnO film.

7. Response and Recovery Time of Pure Film

The time taken for the sensor to attain 90 % of maximum change in resistance on exposure to gas is the response time. The time taken by the sensor to get back 90 % of the original resistance is the recovery time [44]. The response and recovery time of pure ZnO film was 4 s and 60 s respectively. The large recovery time would be due to lower operating temperature. At lower temperature O_2^- species is more prominent adsorbed on the surface, it is less reactive compared to other species of oxygen, O_2^- and O_2^- .

8. Result and Discussion

The gas sensing mechanism of the semiconductor based sensors belongs to the surface controlled type, which is based on the change in conductance of the semiconductor. The oxygen adsorbed on the surface directly influences the conductance of semiconductor based sensors. The amount of oxygen adsorbed on the sensor surface depends on operating temperature, partial size and specific surface area of the sensor [45]. The atmospheric oxygen molecules adsorbed on the surface of n-type ZnO in the form of O,O₂ and O², there by decreasing the conductance. The state of oxygen on the surface of ZnO sensor undergoing the following reactions [46].

$$O_2 (gas) \rightarrow O_2 (ads)$$
 (5)

$$O_2 (ads) + e \rightarrow O_2^- (ads)$$
 (6)

$$O_2^-(ads) + e^- \to 2O^-(ads)$$
 (7)

$$O^{-}(ads) + e \rightarrow O^{2} - (ads)$$
 (8)

The oxygen species captures electron from the material, which results in the concentration changes of holes or electrons in ZnO semiconductor.

Fig. 9 (a) shows the adsorption of oxygen species on the surface of ZnO, abstracting electrons and thus causing an increase in potential barrier of the grain boundaries.

When ZnO films are exposed to the reducing gases, the reductive gas reacts with the oxygen adsorbed on the surface. Then the electrons are released back to the semiconductor, resulting in the change tin the electrical conductance of ZnO sensor. On the surface of the ZnO film there are two kinds of oxygen, one is the adsorption oxygen, and the other is the lattice oxygen.

Fig. 9 (b) shows when ZnO comes in contact with H₂S gas the potential barrier would decrease as result oxidative conversion of H₂S gas and desorption of oxygen. Then the electrons are released back in to the semiconductor, resulting in the change in the electrical conductance of ZnO film. It can be expressed in the following reaction [47, 48]

$$H_2S + 3O^2 \rightarrow H_2O + SO_2 + 6e^-$$
 (9)

The oxidation of H_2S is a process of thermal activation, but the desorption of oxygen is enhanced with the increase of temperature. So a comprised reaction rate of H_2S oxidation must take place at a proper temperature. In the present study, the optimized temperature was found to be 250 °C.

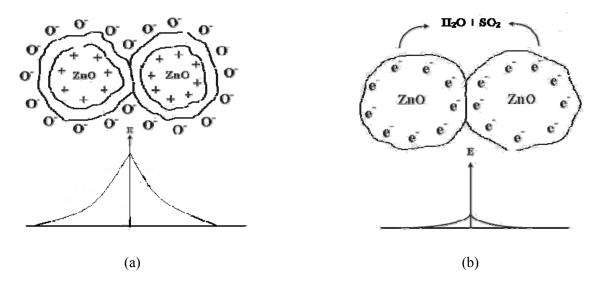


Fig. 9. Oxygen (a) adsorption, (b) desorption on surface of pure ZnO when react with H₂S gas.

The Films are fired at different temperatures. Then it shows that the mass percent of oxygen increase on the surface of the film with increased firing temperature. Hence the film surface becomes more reactive to the reductive gas and there is desorption of oxygen. Then the electrons are released back in to the semiconductor, resulting in the change in the electrical conductance of ZnO film. For low operating temperature the product RO(R-Reducing Gas) will not desorbs , thus passivating the surface for further adsorption of oxygen ,on the other hand for high operating temperature oxygen will not physically adsorbed . Hence the ZnO film shows the performance only within a specific temperature window. Here the ZnO film showed highest response to H_2S gas (100ppm) at 250 °C. It can be completely recovered by heating to a temperature of over 450 °C.

9. Summary and Occlusions

- a) The ZnO material is oxygen deficient, the increase in firing temperature increases the oxygen. Species and this temperature tend to become a material stoichiometric.
- b) The larger oxygen deficiency would enable ZnO to adsorb more oxygen ions helping the sensor to recover fastly.
- c) The pure ZnO film shows the highest response to the H₂S gas over the operating temperature 250 °C at 100 ppm level for firing temperature 650 °C.
- d) The film shows the very rapid response (4 sec.) and recovery (60 sec.) time to H₂S gas.
- e) The ZnO thick film was observed to be highly selective to H₂S gas.
- f) The quick response of the sensor could be attributed to larger oxygen deficiency in ZnO material.
- g) A quick response and fast recovery were the special characteristics of the ZnO material.

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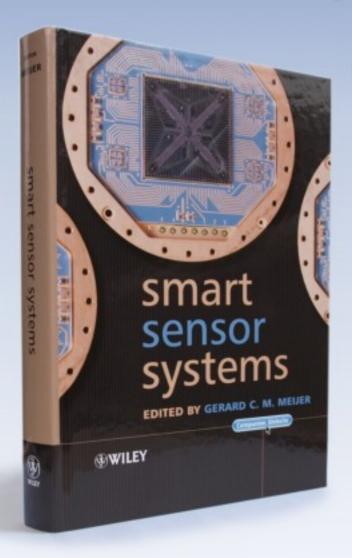
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