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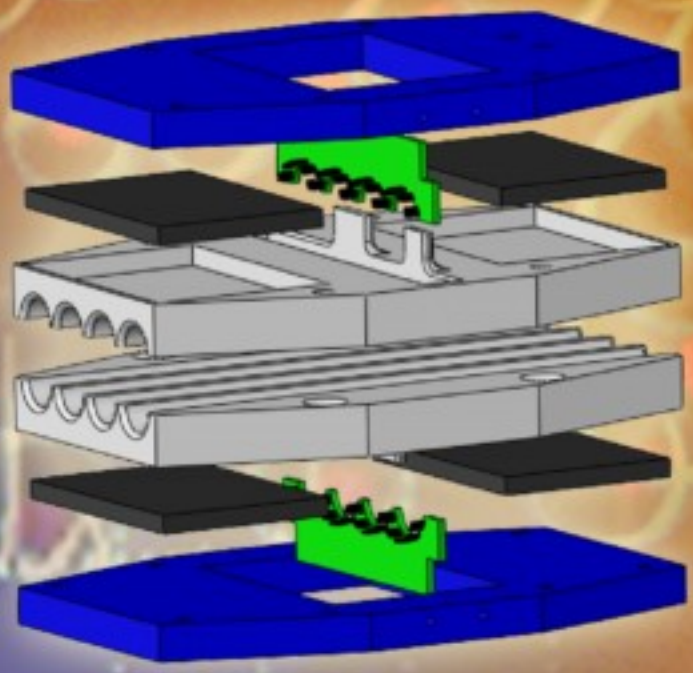

Modern Sensing Technologies III

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- Chemoinformatics
- Bioimaging
- Neuroinformatics

B. Computational systems
- Bio-ontologies and semantics
- Biocomputing
- Genetics
- Molecular and Cellular Biology
- Microbiology

C. Biotechnologies and biomanufacturing
- Fundamentals in biotechnologies
- 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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ICNS 2011

May 22-27, 2011 - Venice, Italy

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Submission (full paper)	January 10, 2011
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Tracks:
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- COMAN: Network Control and Management
- SERVI: Multi-technology service deployment and assurance
- NGNUS: Next Generation Networks and Ubiquitous Services
- MPQSI: Multi Provider QoS/SLA Internetworking
- 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

The Sixth International Conference on Systems

ICONS 2011

January 23-28, 2011 - St. Maarten,
The Netherlands Antilles

Important deadlines:

Submission (full paper)	September 25, 2010
Notification	October 20, 2010
Registration	November 5, 2010
Camera ready	November 5, 2010

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- System instrumentation
- Embedded systems and systems-on-the-chip
- Target-oriented systems [emulation, simulation, prediction, etc.]
- Specialized systems [sensor-based, mobile, multimedia, biometrics, etc.]
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- Security and protection systems
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- Application-oriented systems [content, eHealth, radar, financial, vehicular, etc.]
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- Complex Systems

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Synthesis and Characterization of Nanostructured ZnO Thick Film Gas Sensors Prepared by Screen Printing Method

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Abstract: Nanosized ZnO was prepared by self propagating solution combustion synthesis method. The synthesized ZnO thick films were deposited on alumina substrate by using standard screen printing technique and fired at 700 ^{0}C. The films were characterized by X-ray diffractometer (XRD), Scanning Electron Microscopy (SEM) and energy dispersive analysis of X-ray (EDAX). The electrical behaviors of ZnO thick films were investigated. From XRD spectra it is revealed that ZnO films are polycrystalline in nature. The average grain size of 87.44 nm has been estimated for the film fired at 700 ^{0}C using Scherrer's formula. EDAX clearly shows the peaks corresponding to Zn and O element which confirms the successful growth of ZnO films. Gas sensing study for these samples shows high sensitivity and selectivity towards NO_2 at all operating temperatures. The resistivity, TCR and activation energy of the ZnO films have been evaluated and discussed.

Keywords: Nanostructures, Dextrose, ZnO, LPG sensor, Sensitivity.

1. Introduction

Zinc Oxide is a wide-band gap semiconductor metal oxide with wide range of optical and electronic applications. ZnO is an n-type semiconductor of wurtzite structure with direct band gap of about 3.37eV at room temperature. Polycrystalline ZnO has found numerous applications such as related to surface acoustic wave devices, piezoelectric devices, varistors, planar optical waveguides, transparent electrodes, UV photo detectors, facial powders, gas sensors, etc. Out of these applications of ZnO, gas sensor devices have the sensitivity to various gases, high chemical stability, and suitability for doping, non-toxicity and low cost [1, 2].

Zinc oxide (ZnO) is a multi functional material with a wide range of applications. ZnO films have attracted considerable attention because they can be made to have high electrical conductivity, high infrared reflectance and high visible transmittance. Low resistive zinc oxide films have been achieved by doping with different group III elements like aluminium, boron, indium, gallium or with group VII elements like fluorine [1]. Many techniques including evaporation, chemical vapour deposition, spray pyrolysis, sputtering, etc can be employed to deposit these films [2-5]. Due to the transparency in the visible range, high electrical stability, direct band gap (3.37 eV), absence of toxicity, abundance in nature, etc., ZnO is one of the versatile and technologically importance materials [6]. Controlled synthesis of semiconductor nanostructures in terms of size and shape has been strongly motivated and novel applications can be investigated dependent on their structural properties [7–10]. Among various semiconductor nanostructures, variety of nanostructures of ZnO has been investigated presenting it as richest family of nanostructures. It crystallizes in a wurtzite structure and exhibits n-type electrical conductivity [11]. ZnO nanomaterials with one-dimensional structure, such as nanowires or nanorods, are especially attractive due to their tunable electronic and opto-electronic properties, and the potential applications in the nanoscale electronic and opto-electronic devices [12].

Window layer [13], varistor [14], gas sensor [15-17], etc., are the reported applications. Researchers are now probing on this material as one of the alternative photoanode for dye-sensitized solar cells [18-20]. Zinc oxide has proven itself as one of the competitive and promising candidates to replace expensive materials like CdS, TiO_2, GaN, SnO_2, and In_2O_3 for applications such as solar cells [21], photocatalysis [22], ultraviolet laser [23, 24], transparent conductive oxides [25], spintronics [26], and gas sensors [27]. For gas sensor application, SnO_2 has been the most investigated material. However, ZnO is particularly applicable to gas sensors because of its typical properties such as resistivity control over the range 10^{-3} to 10^{-5} cm, high electrochemical stability, absence of toxicity, and abundance in nature [28].

Zinc Oxide nanostructures could be synthesized by several techniques such as vapor deposition, oxidation, sputtering, and pulse laser deposition. We prepared nano-size ZnO powder by self propagating solution combustion synthesis method. The powder extracted is characterized and a thick film paste was prepared by adding suitable binder and solvent. Screen printing is a viable and economical method to produce thick films of various materials. The ZnO thick films are screen printed onto alumina substrate [29, 30].

Herein we report tailoring of various structural and morphological changes of ZnO using dextrose as fuel combustion. Their electrical and gas sensing study has also been carried out at various operating temperatures and is found to be good reducing gas sensor.

2. Experimental

2.1. Preparation of ZnO Nano Powder

Zinc oxide nano structured powder was prepared by self propagating solution combustion technique. The starting materials are Zinc nitrate and Dextrose. Proper amount of zinc nitrate and dextrose are dissolved in water contained beaker and placed on a hot plate for 15 minutes as the solution dehydrates to form a deposition like a gel. Then the beaker was placed in a preheated muffle furnace at 400 ^{0}C. The solution boils, ignites with a flame and the entire reaction was completed within 5 minutes. The powder is amorphous in nature. Then the powder was calcinated at 650 ^{0}C to get nanocrystalline ZnO powder. The XRD pattern of this confirms the formation of ZnO [31].

2.2. ZnO Thick Film Preparation

ZnO thick films were prepared on alumina substrate by using standard screen-printing technique. The calcinated nanosized ZnO powder was crushed and mixed with glass frit and ethyl cellulose. The mixture was then mixed with butyl carbitol acetate to make the thixotropic paste. The paste was then screen printed on the alumina substrate. The films were dried under IR-lamp for 30 min and then fired at 700 ^{0}C for 30 min.

2.3. Structural and Morphological Studies

2.3.1. X-Ray Diffraction Method

The SnO_2 thick films were characterized by X-ray diffraction technique from 20-80^o [diffractometer (Miniflex Model, Rigaku, Japan) with CuKα, λ = 0.1542 nm radiation] with a 0.1o/step (2θ) at the rate of 2 s/step. The average crystallite size was determined using Scherrer formula [31],

$$D = \frac{0.94\lambda}{\beta \cos\theta}, \tag{1}$$

where D is the average crystalline grain size; β is the full angular width of diffraction peak at the half maximum peak intensity (FWHM); λ is the wavelength of X-ray diffraction (1.542 Å); θ is the angle of diffraction.

The surface morphology 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).

2.3.2. Electrical Behaviours and Gas Response

The D.C. Resistance of the films was measured by using half bridge method in atmosphere at different temperatures [32, 33]. The gas sensing studies were carried out on a static gas sensing system [34, 35] under normal laboratory conditions. The NO_2 gas response of ZnO thick films were studied in test assembly. The electrical resistances of a ZnO film in air (R_a) and in the presence of NO_2 gas (R_g) were measured to evaluate the gas response (S) given by the relation:

$$S = \frac{R_a - R_g}{R_a}, \tag{2}$$

where R_a is the resistances of the ZnO thick film sample in air and Rg is the resistances of the ZnO thick film sample in NO_2 gas atmosphere.

3. Results and Discussion

3.1 Calcination, Drying and Firing of the films

The calcination of the powder before the paste preparation and the firing process of the printed film can determine the sensitivity of the active layer of the film if it is used as a gas sensor. With calcination, grain boundaries are developed and the powder sinters to bigger agglomerates. This causes

a higher surface area after firing and therefore a higher sensitivity of a layer. This powder was milled after calcination. The calcination took from 1 h to 10 h [36]. A drying stage is required to remove the organic solvents, make the printed film adhere to the substrate and be relatively immune to smudging. After printing, the film was allowed to settle in air for a few minutes so that some of the volatile solvents were evaporated slowly at room temperature. The organic agent was still present in the paste at this stage. Drying took place at temperatures between 70-100 ^{o}C by placing films under infrared radiation [36].

The high temperature firing cycle is designed to remove the remaining organic binders, to develop the structural and electrical properties of the film and bond the film to the substrate. During this firing process the glass frit melts and grains of the functional materials are held together and also the film becomes bonded firmly to the substrate. There are three distinct regions in this firing cycle. Firstly the temperature slowly was increased towards the peak firing temperature. During this time the remaining organics were removed. This occurred at 350-400 ^{o}C. As the temperature reached 580-780 ^{o}C, the glass frit softens. Secondly the temperature remained constant for about 30 minutes. During this time the active material sintered and various reactions took place. The electrical properties of the film began to develop. Finally there was a cooling stage to room temperature that allows the glass frit to solidify [36].

3.2. Structural and Morphological Studies

3.2.1. Composition of ZnO Thick Film

Table 1 shows the composition of the films fired at 700 ^{o}C. The EDX spectrum showed the presence of only Zn and Oxygen along with Al and Si. From the analysis it was found that the ZnO films are nonstoichiometric. The deficiency or excess of any type of atom in the crystal results in a distorted band structure, with a corresponding increase in conductivity. Tin oxide looses oxygen on heating so that tin is then in excess. The oxygen, of course, evolves as an electrically neutral substance so that it is associated with each excess tin ion in the crystal; there will be two electrons that remain trapped in the solid material, thus leading to nonstoichiometry in the solid. This leads to the formation of the n-type semiconductor [37].

Table 1. Composition of the ZnO films at 700 ^{0}C firing temperature.

Element	Mass %	At.%
O	14.85	40.89
Zn	83.23	56.08
Al	0.30	0.48
Si	1.62	2.54
Total	100	100

3.2.2. X-ray Diffraction Analysis

Fig. 1 shows X-ray diffraction patterns of ZnO thick film deposited on alumina substrates and fired at 700 ^{o}C. XRD pattern show the different peaks of ZnO phases. It has been observed that [101] reflections corresponding to $2\theta = 36.17^{o}$ is of maximum intensity for all film samples thereby a strong orientation with stacking of the plane along the c-axis, which indicates ZnO film had preferred orientation in the direction of [101] plane. The ZnO diffraction peaks for (100), (002), (101), (102), (110), (103), (200), (112), (201) crystal orientation are identified for film at angles 31.69, 34.20, 36.17,

47.41, 56.67, 62.84, 66.50, 68.17, 69.58 respectively. The XRD was carried out by PHILIPS PW1729 X-ray generator Recorder PW 1840 diffractometer control**,** PM 8203A one line recorder. The standard interplaner spacing JCPDS data card Number 21-1486 matches with calculated values [38]. This clearly indicates that the structure of ZnO film is polycrystalline in nature.

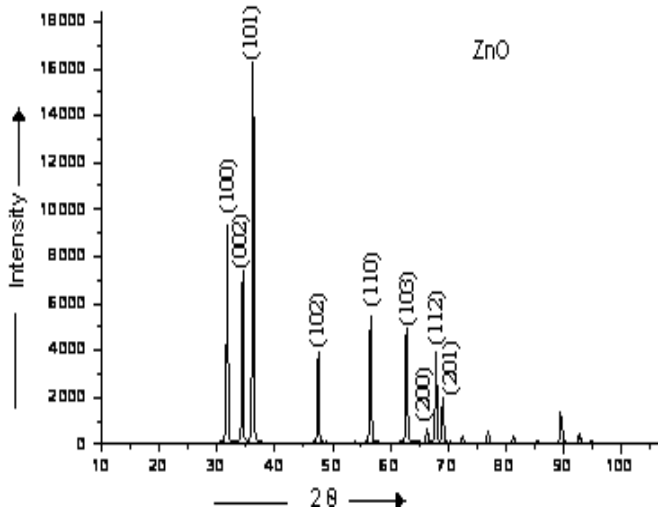

Fig. 1. X-ray Diffraction Pattern of ZnO Thick Film.

The XRD pattern was used to calculate the crystallite size of ZnO by using Debye Scherrer's formula [31]. The average crystallite sizes of ZnO thick film is 86 nm (±2 nm) at 700 ^{o}C (±2 ^{o}C).

3.2.3. Scanning Electron Microscopy

Fig. 2 shows SEM images of ZnO thick film fired at 700 ^{o}C. Microstructural chacterization was carried out by using scanning electron microscopy. SEM indicated rod type microstructure with negligible porosity. However some residual, intragranular porosity was seen. The film fired at 700 ^{o}C has good adhesion. Therefore it is used for gas sensing.

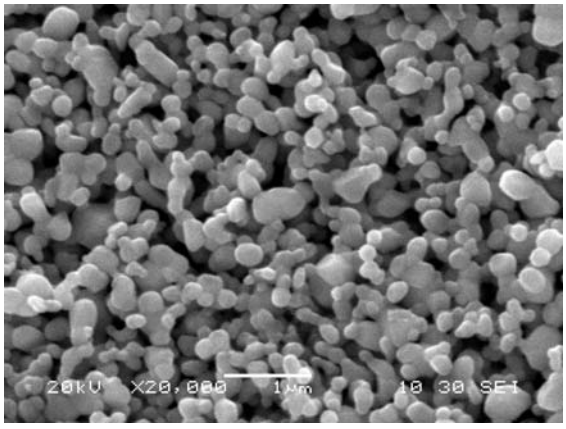

Fig. 2. SEM Micrograph of ZnO Thick Film.

3.2.4. Thickness Measurement

The thickness of thick film samples measured by Tally-step method ranging from 8.14 μm, 21.86 μm and 35.64 μm for single layer, double layer and three layer sample respectively. The thickness, resistivity and resistance of the single layer, double layer and three layer samples were given below in Table 2.

Table 2. The thickness, resistivity and resistance of the single layer, double layer and three layer samples.

Thick film layer	Film Thickness μm	Resistivity 10^4 (Ωm)	Resistance 10^4 (Ω)
Single layer	8.14	16.58	32.59
Double layer	21.86	5.275	3.861
Three layer	35.64	2.65	1.19

3.3. Electrical Characterization

3.3.1. Electrical Resistivity

Variation of resistance of ZnO film with temperature is shown in Fig. 3. The resistance of the film decreases as temperature increases. The TCR was calculated for temperature range 90 – 400 ^{0}C.

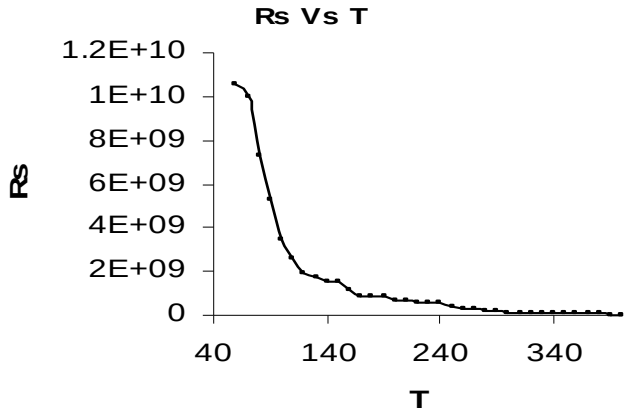

Fig. 3. Resistance vs. Temperature Variation of ZnO Thick Film.

The Fig. 4 shows the graph of TCR versus temperature of the film.

Fig. 5 shows graph of logR versus 1/T. The graph indicates two regions, low temperature region and high temperature region. The activation energy was calculated by this plot.

TCR, resistivity and activation energy of ZnO thick film fired at 700 ^{0}C having thickness 35.64 μm for three layer sample is given in Table 3.

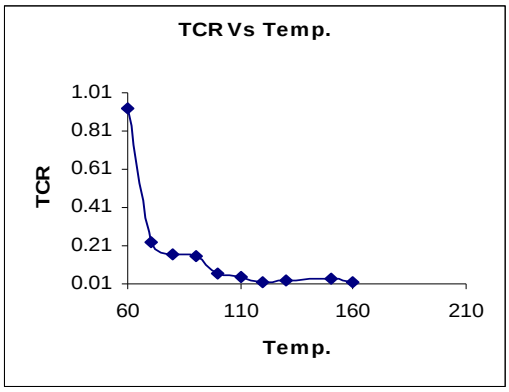

Fig. 4. TCR vs. Temperature of ZnO Film.

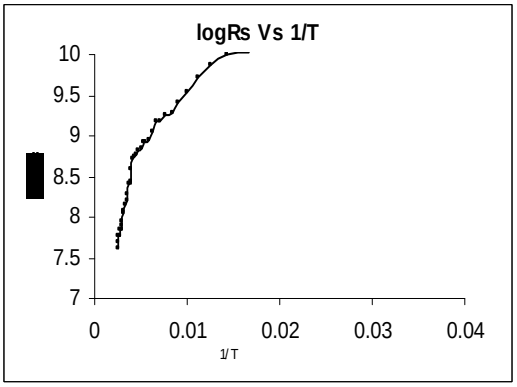

Fig. 5. LogR vs. 1/T of ZnO Film.

Table 3. Electrical Parameters.

TCR (/°c)	Resistivity ρ (Ωm)	Activation Energy ΔE(ev)	
		LT region	HT region
0.22	2.65 x 10^4	0.02161	0.11839

3.4. Gas Sensing Response

3.4.1. NO_2 Gas Sensing

Fig. 6 shows the variation of response of pure ZnO fired at 700 °C to 1000 ppm NO_2 gas with operating temperature. The gas response increases with temperature from 150 to 250 °C and then decreases with a further increase in temperature. The response of pure ZnO to NO_2 gas is 20.29 at 250 °C. In present work, every time prior to exposing the ZnO film to NO2, it was allowed to stabilize at an operating temperature for 15 min and the stabilized resistance was taken as Ra. After exposing the film to the NO_2 gas, the changed resistance was taken as Rg. NO_2 is oxidizing gas. It reacts with surface oxygen ions of the film. Oxidation of film decreases the number of free carriers. Therefore resistance of the film increases with oxidizing gas [39-41].The result of reaction of NO_2 with polycrystalline ZnO is adsorbed NO_3 with little NO_2 or NO present on the surface of the oxide. The Zn↔NO_2 interactions on ZnO are strong and Zn sites probably get oxidized and nitrated [42].

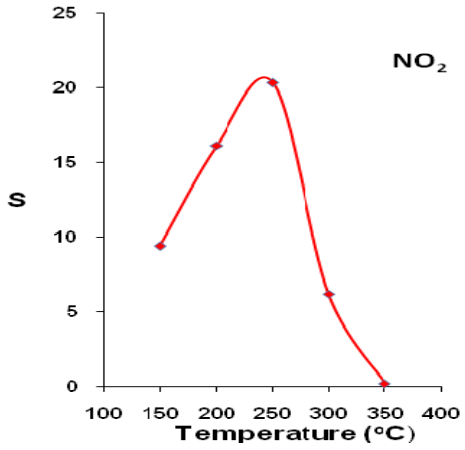

Fig. 6. Variation of Response with Operating Temperature for NO_2 Gas at 1000 ppm.

3.4.2. Gas Response and NO_2 Concentration

The variation of gas response of the ZnO film sample with NO_2 gas concentration at 250 ^oC temperature is represented in Fig. 7. This film was exposed to different gas concentrations of NO_2. The sensitivity values were observed to increase continuously with increasing the gas concentration up to 1000 ppm.

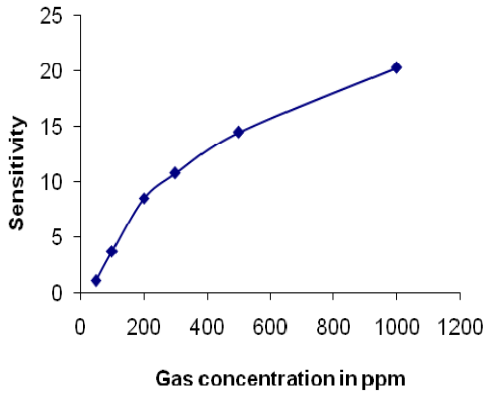

Fig. 7. Variation of Gas Response of ZnO Thick Film with NO_2 Gas Concentration.

3.4.3. Selectivity for NO_2 against Other Gases

It is observed from Fig. 8 that the ZnO sample shows maximum response to NO_2 (1000 ppm) at 250 ^oC. Sample showed highest selectivity for NO_2 against all other tested gases viz: NH_3, LPG. Ethanol vapours, CO_2, H_2S.

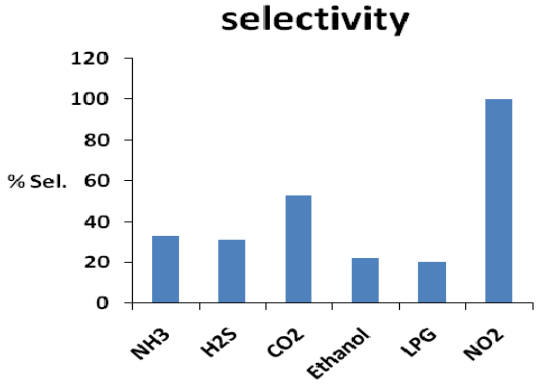

Fig. 8. Selectivity of ZnO Thick film sample for various gases.

3.4.4. Response and Recovery Time

The response and recovery times of ZnO film sample are represented in Fig. 9. The response was quick (~ 28 s) to 1000 ppm of NO_2 while the recovery was fast (~ 35 s). The quick response may be due to faster oxidation of gas. Its high volatility explains its quick response and fast recovery to its initial chemical status.

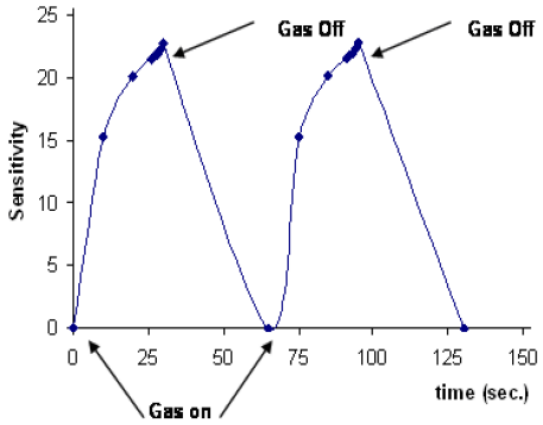

Fig. 9. Response and Recovery of ZnO Thick Film Sample.

4. Conclusions

This work demonstrated the successful preparation of ZnO screen printed thick film shows good adhesive to alumina substrate employing a simple, inexpensive method and capability of the ZnO films for NO_2 sensing. The film fired at 700 ºC exhibited good sensing performance to NO_2.

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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.

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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:

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