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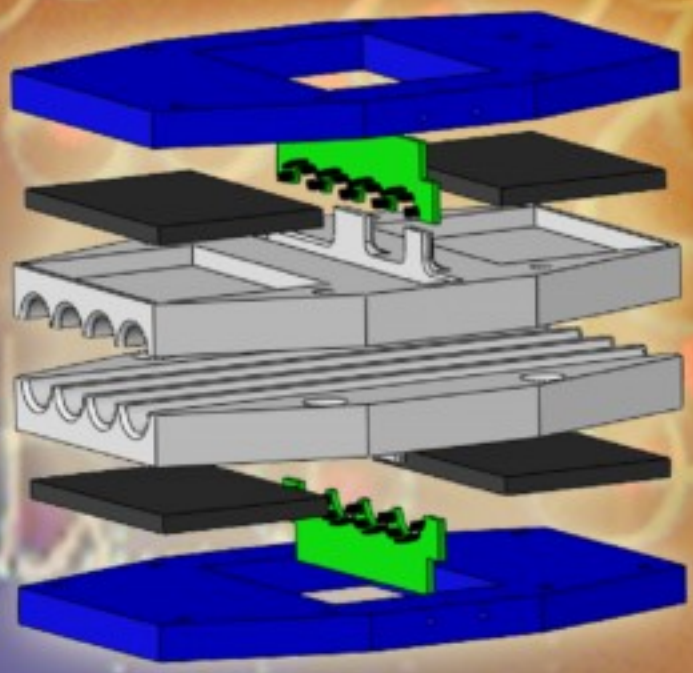

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Notification	February 20, 2011
Registration	March 5, 2011
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- IPDy: Internet Packet Dynamics
- GOBS: GRID over Optical Burst Switching Networks

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Camera ready	November 5, 2010

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- Embedded systems and systems-on-the-chip
- Target-oriented systems [emulation, simulation, prediction, etc.]
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- Security and protection systems
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Formulation and Characterization of Cu Doped ZnO Thick Films as LPG Gas Sensor

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Abstract: Thick films of pure and various concentrations (1 wt. %, 3 wt. %, 5 wt. %, 7 wt. % and 10 wt. %) of Cu-doped ZnO were prepared on alumina substrates using a screen printing technique. These films were fired at a temperature of 700ºC for two hours in an air atmosphere. Morphological, compositional and structural properties of the samples were obtained using the scanning electron microscopy (SEM), Energy dispersive spectroscopy (EDAX) and X-ray diffraction techniques respectively. The LPG gas sensing properties of these thick films were investigated at different operating temperatures and LPG gas concentrations. The surface resistance of thick films decreases when exposed to LPG gas. The Cu doped films show significant sensitivity to LPG gas than pure ZnO film. 5 wt. % Cu-doped ZnO film was found to be more sensitive (87.3 %) to LPG gas exposed at 300 ^{o}C than other doping concentrations with fast response and recovery time.

Keywords: ZnO, Cu, Thick films, Screen printing, LPG.

1. Introduction

Of all the gas sensing solid state materials, metal oxides were one of the first considered [1, 2] and are still the most widely used gas sensing materials. Gas sensors based on metal-oxides are commonly used in the monitoring of toxic pollutants, highly inflammable gas and can provide the necessary

sensitivity, selectivity required by such systems [3]. Commonly used oxides include, zinc oxide, titanium dioxide, iron oxide, tungsten oxide and tin oxide. These materials have successfully been employed to detect a range of gas particularly ethanol, NO_2, H_2S, NH_3, LPG, CO_2 [4–12]. Thick film technology is often used to fabricate such sensors and possesses many advantages, for example, low cost, simple construction, small size and good sensing properties [13]. In addition, this approach provides reproducible films consisting of a well-defined microstructure with grains and grain boundaries that can be studied easily [14].

Zinc oxide (ZnO) is a multifunctional material. Because of its high chemical stability, low dielectric constant, large electrochemical coupling coefficient and high luminous transmittance, ZnO based materials have been widely used as dielectric ceramic, pigment, catalyst and sensing material [6, 7, 8]. As a gas sensing material, it is one of the earliest discovered and most widely used gases sensing material for the detection of hazardous gases [15-19]. It is sensitive to many sorts of gases and has satisfactory stability. The gas sensing performance of the material can be improved by incorporating dopants and additives [20-24] that can modulate the gas sensing characteristics to some extent.

Liquefied Petroleum Gas (LPG) is highly inflammable gas. It is explosively utilized in industrial and domestic fields as fuel. It is referred as town or cooking gas. Cooking gas consists chiefly of butane (55-vol %) [25], a colorless and odorless gas. It is usually mixed with compounds of sulfur (methyl mercaptan and ethyl mercaptan) having foul smell, so that its leakage can be noticed easily. This gas is potentially hazardous because explosion accidents might be caused when it leak out by mistake. It has been reported that, at the concentration up to noticeable leakage, it is very much more than the lower explosive limit (LEL) of the gas in air. So there is a great demand and emerged challenges [26] for monitoring it for the purpose of control and safety applications in domestic and industrial fields. ZnO crystallizes in a wurtzite structure showing n-type semiconductivity. ZnO utilized in wide range of applications .The Cu/ZnO system is of great importance because of its use as the methanol synthesis catalyst in low-temperature water–gas shift reactions and for hydrogen production from methanol in the reverse water–gas shift reaction. No matter what the original motivation, the outcome allows us to demonstrate the usefulness of these kinds of model system studies not only for applications in heterogeneous catalysis but also for gas sensing applications [27].

The aim of the present work is to study the sensor by doping ZnO thick films, which could be able to detect the LPG gas. Among the various additives tested, Cu in ZnO is outstanding in promoting the sensing properties to LPG in air.

2. Experimental

The ZnO: Cu pastes used in screen printing were prepared by maintaining the inorganic to organic materials ratio of 70:30. Inorganic part consists of a functional material (ZnO), dopant (Cu) and glass frit (70 wt. % PbO, 18 wt. % Al_2O_3, 9wt. % SiO_2 and 3wt. % B_2O_3). Organic parts consist of 8 % ethyl cellulose (EC) and 92 % butyl carbitol acetate (BCA). The Analar (AR) grade ZnO with x wt. % Cu (x = 1, 3, 5, 7 and 10%) and 5 wt. % of glass frit were mixed thoroughly in an acetone medium with mortar and pestle. A solution of EC and BCA in the ratio 8:92 was made, which was added drop by drop until proper thixotropic properties of the paste were achieved. ZnO thick films were prepared on alumina substrates using a standard screen-printing technique. A nylon screen (40s, mesh no.355) was used for screen-printing. The required mask (2 x 1.25 cm) was developed on the screen using a standard photolithography process. The paste was printed on clean alumina substrates (5 x 2 cm) with the help of a mask. The pattern was allowed to settle for 15 to 20 minutes in air. The films were dried under infrared radiation for 45 minutes to remove the organic vehicle and then fired at a temperature of 700 ^{o}C for 2 h (which includes the time required to achieve the peak firing temperature , constant firing for 30 minutes at the peak temperature and then to attain the room temperature) in a

muffle furnace. During the firing process glass frit melted and the functional material and dopant were sintered. The function of glass frit is to bind the grains of functional and dopant materials together and also to adhere the film firmly to the substrate surface.

The structural properties of ZnO: Cu films were investigated using X-ray diffraction analysis from 20-80^0 [Rigaku diffractometer (Miniflex Model, Rigaku, Japan) with CuKα, λ=0.1542 *nm* radiation] with a 0.1^o/step (2θ) at the rate of 2 s/step. A scanning electron microscopy (SEM- JOEL JED-2300) was employed to characterize the surface morphology. The composition of ZnO: Cu thick film samples were analyzed by an energy dispersive X- ray spectrometer (JOEL-JED 6360 LA). The thickness of the films was measured using a Taylor-Hobson (Taly-step UK) system and was observed to be uniform in the range of 20 μm to 25 μm. The specific surface area was calculated for spherical particles using the following equation [28],

$$S_w = \frac{6}{\rho d}, \tag{1}$$

where d is the diameter of the particles, ρ is the density of the particles. The D.C. resistance of the films was measured by using half bridge method in an air atmosphere at different temperatures.

The gas sensing studies were carried out on a static gas sensing system under normal laboratory conditions. The electrical resistance of thick films in air (R_a) and in the presence of LPG (R_g) was measured to evaluate the Sensitivity (S) and is given by the relation,

$$Sensitivity(S) = \left|\frac{R_a - R_g}{R_a}\right| = \frac{\Delta R}{R_a}, \tag{2}$$

where R_a is the resistance of the ZnO: Cu thick films in air and R_g is the resistance of the ZnO: Cu thick films in LPG atmosphere. The response of the sensor for LPG was tested in the presence of other gases so that the selectivity can be determined. The response and recovery time of the sensor was measured.

3. Results and Discussion

3.1. Elemental Analysis

The elemental compositions of the pure and doped ZnO films were analyzed using an energy dispersive spectrometer and is shown in Table 1. The mass% of Zn and O in each sample was observed to be non-stoichiometric. The ZnO film doped with 5 wt. % Cu was observed to be most oxygen difficient. The deficiency of the constituent material particles or an excess of it leads to the semiconducting behaviour of the material [29].

Table 1. Composition of Cu doped ZnO films.

Element (mass %)	**Cu**				
	1wt. %	**3wt. %**	**5wt. %**	**7wt. %**	**10wt. %**
O	21.12	21.25	20.95	21.64	22.52
Zn	78.80	78.56	78.53	77.45	75.50
Cu	0.08	0.19	0.52	0.91	1.98

3.2. Structural Analysis

The crystalline structure of the films was analyzed with X-ray diffractogram in the 20-80^0 (2θ) range using Cuk$_\alpha$ radiation. Fig. 1 shows the XRD pattern of undoped and Cu doped ZnO thick films of different concentrations. The observed phases were for ZnO (ASTM card No. 36-1451) and CuO (ASTM Card No. 5-0661). For 1 % and 3 % Cu doping, no peak corresponds to Cu phase has observed due to low Cu content. It means Cu may form solid solution with ZnO. For 5 %, 7 %, and 10 % Cu doping, XRD pattern presents traces of CuO as a secondary phase (insoluble ZnO).These peaks are related to complex reactions involving the production of Cu → Cu2O → CuO during firing in air.

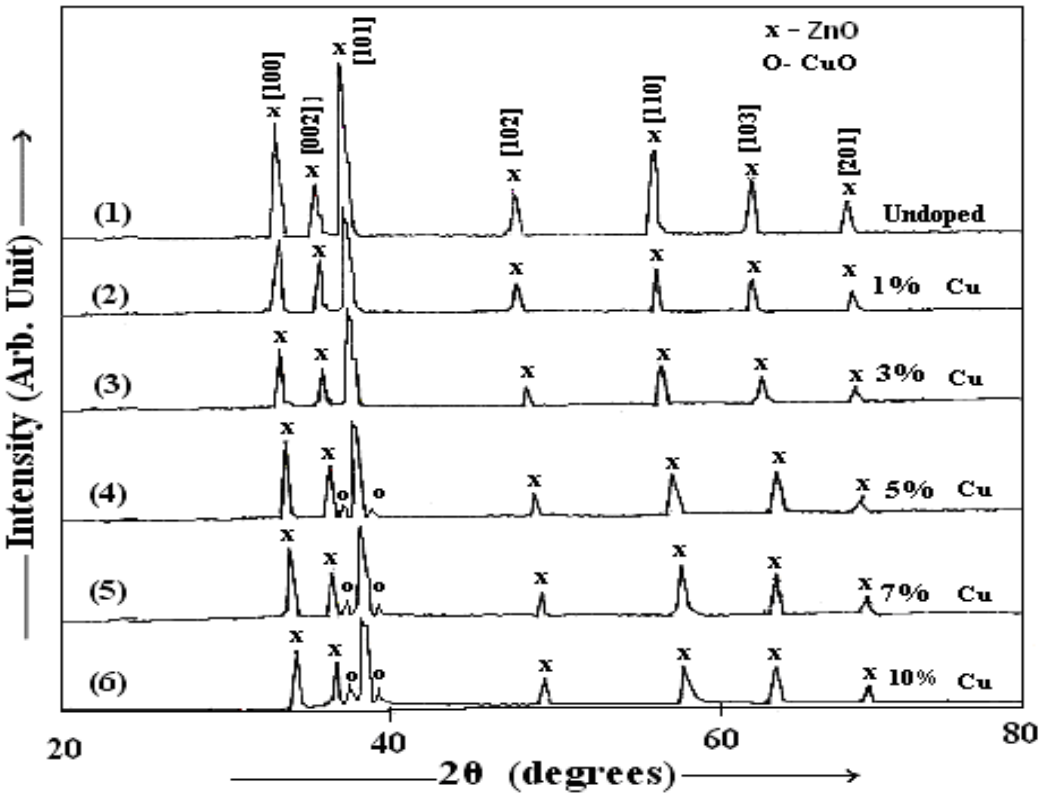

Fig. 1. XRD pattern of pure ZnO and ZnO: Cu films fired at 700 ^{o}C.

3.3. Surface Morphology Analysis

The gas sensing properties of a metal oxide thick film strongly depends on its morphological features. A high surface area facilitates the chemisorptions process by increasing the adsorption and desorption rates [30]. Fig. 2 indicates the SEM images of pure and x wt. % of Cu (x = 1, 3, 5, 7 and 10 wt. %) doped thick films. Fig. 2(a) shows the microstructure of pure ZnO thick film. It showed that the microstructure is nearly uniform with negligible open porosity. Fig. 2(d) shows the microstructure of 5 % Cu doped ZnO thick film which was most sensitive. It showed that the grain size decreases giving large effective surface area. The larger surface area gives more response to react with the target gas. The film seems to be highly porous for oxygen adsorption. The specific surface area was calculated by using equation 1.The specific surface area increases as the size of the grains decreases [28]. Table 2 presents the particle size and surface area of ZnO thick films doped with different wt. % of Cu.

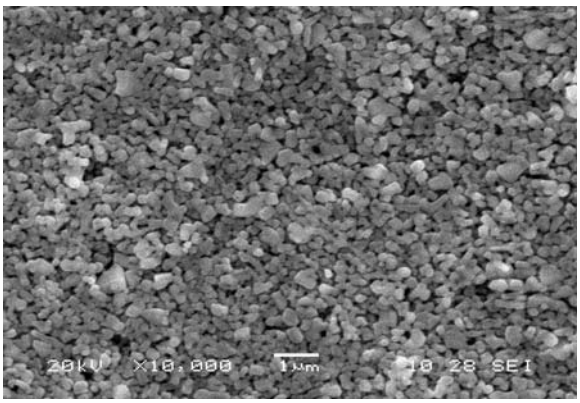

Fig. 2a. SEM of Pure ZnO film.

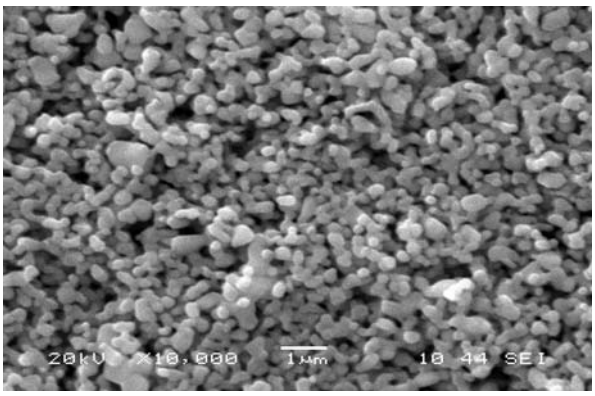

Fig. 2b. SEM of 1 wt. % Cu: ZnO film.

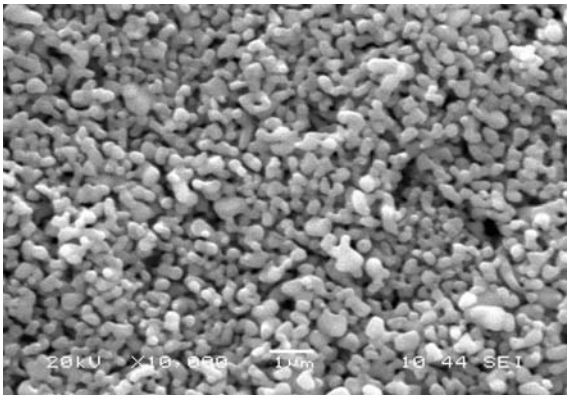

Fig. 2c. SEM of 3 wt. % Cu: ZnO film.

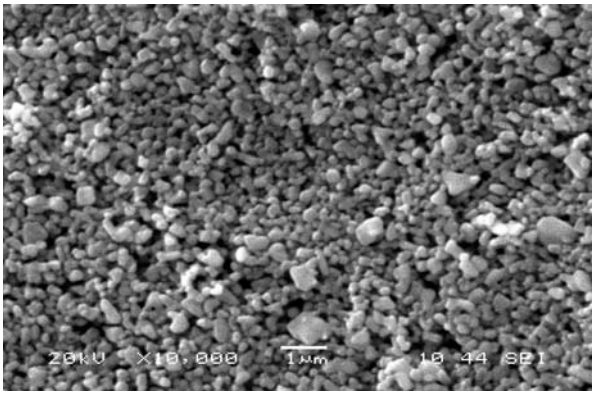

Fig. 2d. SEM of 5 wt. % Cu: ZnO film.

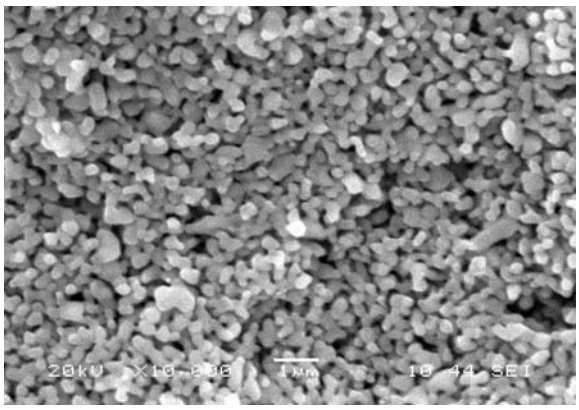

Fig. 2e. SEM of 7 wt.% Cu: ZnO film.

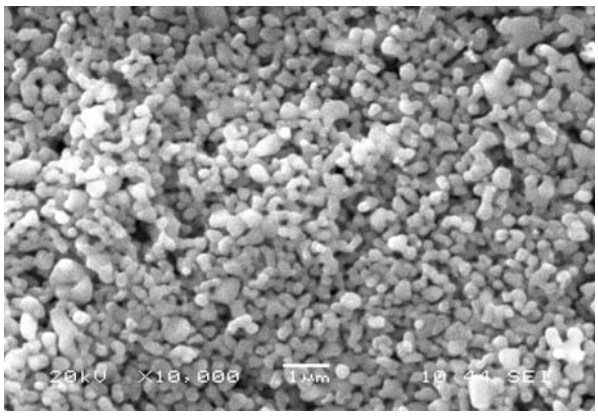

Fig. 2f. SEM of 10 wt.% Cu: ZnO film.

Table 2. Particle size and surface area of ZnO- Cu thick films.

wt. % of Cu	Particle size in nm	Specific Surface Area in m^2/g
Pure ZnO	381	2.752
1	500	2.104
3	433	2.402
5	333	3.088
7	400	2.543
10	433	2.311

3.4. Gas Sensing Characteristics

Fig. 3 shows the gas sensitivity of pure ZnO and different wt. % Cu doped ZnO thick films fired at 700 ^{o}C exposed to 1000 ppm of LPG with operating temperatures. Sensitivity is the device characteristic of perceiving a variation in physical and/or chemical properties of the sensing material under gas exposure.

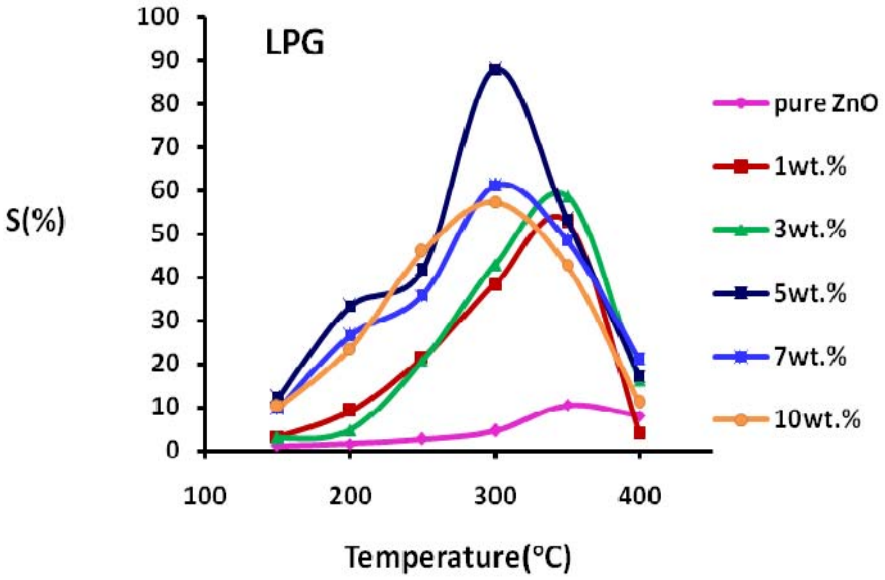

Fig. 3. Gas sensitivity of pure and Cu doped films for 1000 ppm LPG.

The sensitivity of pure ZnO thick film to LPG was found to be 10.5 % at 300 ^{0}C. Pure ZnO is notably less sensitive than doped ZnO. The sensitivity of 5 wt. % Cu doped ZnO film was observed to be 87.3 % at 300 ^{0}C which is higher than other dopant concentrations. Fig. 4 shows histograms indicating the selectivity of 5wt. % of Cu doped ZnO thick film for different gases against LPG. Selectivity or specificity is defined as the ability of a sensor respond to a certain (target) gas in the presence of other gases.

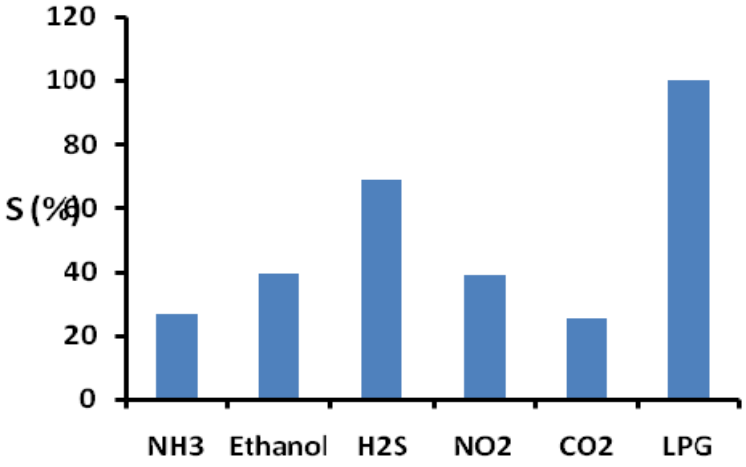

Fig. 4. Selectivity of 5wt. % Cu: ZnO film for different gases against LPG.

Fig. 5 shows the variation of sensitivity of the 5 % Cu doped ZnO film with LPG concentrations (in ppm) at 300 ^{o}C temperature. The response and recovery times of 5wt. % Cu doped ZnO thick films are represented in Fig. 6. The response was quick (~ 08 sec) to 1000 ppm of LPG while the recovery time was fast (~ 17 sec). The response/recovery time is an important parameter, used for characterizing sensors. It is defined as the time required to reach 90 % of the final change in voltage or resistance, when the gas is turned on or off, respectively.

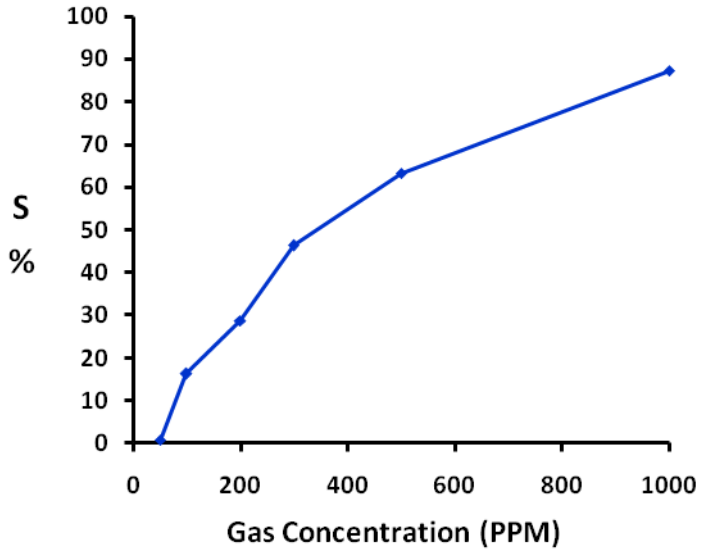

Fig. 5. Variation of gas Sensitivity with gas concentration.

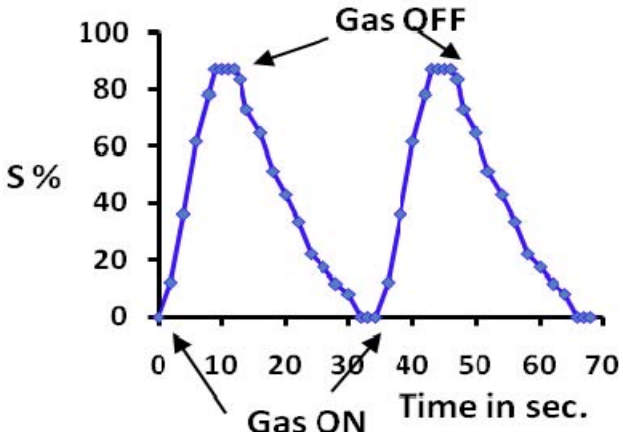

Fig. 6. Response and recovery time of 5 % Cu doped ZnO film.

The higher response may be attributed to the optimum porosity and largest effective surface area available to react with the gas. The response could be attributed to the adsorption–desorption type of sensing mechanism. The amount of oxygen adsorbed on the surface would depend on the number of Cu misfits to adsorb the oxygen which in turn would oxidize the exposed gas.

When the optimum amount of Cu (5 wt. %) is incorporated on the surface of the ZnO film, Cu species would be distributed uniformly throughout the surface of the film. As a result the initial resistance of the film is high and this amount would also be sufficient to promote the catalytic reaction effectively and the overall change in the resistance on the exposure of LPG leading to an increase in the

sensitivity. When the amount of Cu on the surface of the film is less than the optimum, the surface dispersion may be poor and the sensitivity of the film is observed to be decreased since the amount may not be sufficient to promote the reaction more effectively. On the other hand, as the amount of Cu on the surface is more than the optimum, the Cu atoms would be distributed more densely. Therefore the initial resistance of the film would decrease and the overall change in the resistance on the exposure of LPG would be smaller leading to lower response [8].

These films were exposed to different gas concentrations of LPG. The sensitivity values were observed to have increased continuously by increasing the gas concentration up to 1000 ppm. The response was highest for 1000 ppm of LPG. The monolayer of the gas molecules formed on the surface could cover the whole surface of the film. The excess gas molecules would remain idle and would not reach the surface active sites of the film. So, the response at higher concentrations of the gas was not expected to increase in large extent [29].

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. The negligible quantity of the surface reaction products and their high volatility explain the quick response to LPG and fast recovery to its initial chemical status.

3.5. Sensor Mechanism

Gas sensing mechanism is generally explained in terms of conductance either by adsorption of atmospheric oxygen on the surface and/or by direct reaction of lattice oxygen or interstitial oxygen with the test gases. In case of former, the atmospheric oxygen adsorbs on the surface by extracting an electron from conduction band, in the form of super-oxides or peroxides, which are mainly responsible for the detection of the test gases. At higher temperature, the adsorbed oxygen captures the electrons from conduction band as:

$$O_{2(air)} + 2e^{-}{}_{(cond.band)} \rightarrow 2O^{-}{}_{(filmsurface)} \quad (3)$$

It would result in decreasing conductivity of the film. When LPG reacts with the adsorbed oxygen on the surface of the film, it gets oxidized to CO_2 and H_2O by following series of intermediate stages. This liberates free electrons in the conduction band. The final reaction takes place as:

$$C_4H_{10(gas)} + 13O^{-}{}_{(filmsurface)} \rightarrow 4CO_{2(gas)} + 5H_2O_{(gas)} + 13e^{-}{}_{(cond.band)} \quad (4)$$

This shows n-type conduction mechanism. Thus generated electrons contribute to a sudden increase in conductance of the thick film. The Cu misfit regions dispersed on the surface would enhance the ability of base material to adsorb more oxygen species giving high resistance in air ambient.

The mass% of Zn and O in each samples were not as per the stoichiometric proportion and all samples were observed to be the oxygen deficient (Table 1). This deficiency gets reduced (though in less extent) due to adsorption of atmospheric/molecular oxygen.

This helps in decreasing electronic conductivity of the film. Upon exposure, LPG molecules got oxidized with the adsorbed oxygen ions by following the series of intermediate stages, producing CO_2 and H_2O. This results in evolving oxygen as electrically neutral atoms trapping behind the negative charges (electrons). Upon exposure, the energy released in decomposition of LPG molecules, would be sufficient for trapped electrons to jump into the conduction band of activated ZnO, resulting in increase in the conductivity of the film. The drastic increase in conductivity of the sensor could be attributed to the adsorption–desorption mechanism resulted from the electronic defects [26].

4. Conclusions

From the results obtained, it has become possible to make thick film gas sensors using screen printing method. Pure ZnO thick films showed low response to LPG. 5wt. % Cu doped ZnO thick films showed highest response to LPG at 300 ^{0}C.The sensitivity increases in proportion to the test gas concentration. The sensor has good selectivity to LPG against NH3, H_2S, Ethanol, CO_2 and NO_2 at 300 ^{0}C. The sensor showed very rapid response and quick recovery to LPG. Over long exposure to LPG, it was observed that sensor exhibited a good stability and repeatability as gas sensor with consistent pattern and response magnitude.

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The Second International Conference
on Sensor Device Technologies and Applications

SENSORDEVICES 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:
- Sensor devices
- Photonics
- Infrared
- Ultrasonic and Piezosensors
- Sensor device technologies
- Sensors signal conditioning and interfacing circuits
- Medical devices and sensors applications
- Sensors domain-oriented devices, technologies, and applications
- 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

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

CENICS 2011

August 21-27, 2011 - French Riviera, France

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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

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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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