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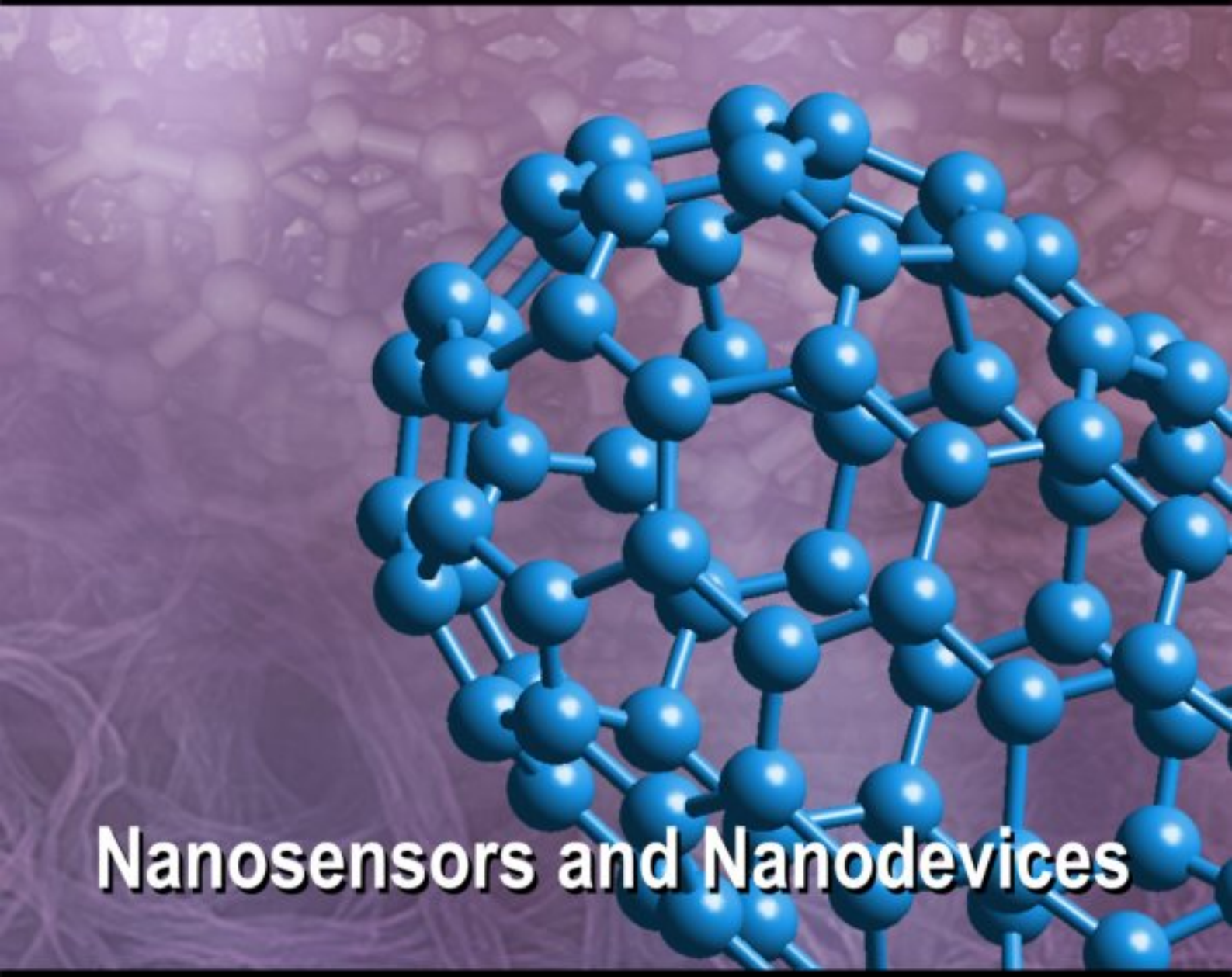

Nanosensors and Nanodevices

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Ammonia Gas Sensing Properties of Nanocrystalline $Zn_{1-x}Cu_xFe_2O_4$ Doped with Noble Metal

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Abstract: The sensors are required basically for monitoring of trace gases in environment. In order to detect, measure and control these gases; one should know the amount and type of gases present in the environment. Among the most toxic and hazardous gases, it is necessary to detect and monitor the ammonia gas because this is enhance in the agricultural sector by the addition of large amounts of NH_3 to cultivated farmland in the form of fertilizers. Nanocrystalline spinel type $Zn_{1-x}Cu_xFe_2O_4$ (x=0, 0.2, 0.4 0.6 & 0.8) has been synthesized by sol-gel citrate method. The synthesized powders were characterized by XRD and SEM. The results revealed that the particle size is in the range of 40–45 nm for Cu–Zn ferrite with good crystallinity. The gas sensing properties were studied towards reducing gases like CO, LPG, NH_3 and H_2S and it is observed that Cu–Zn ferrite shows high response to ammonia gas at relatively lower operating temperature. The $Zn_{0.6}Cu_{0.4}Fe_2O_4$ nanomaterial shows better sensitivity towards NH_3 gas at an operating temperature 300 ^{0}C. Incorporation of Pd improved the sensitivity, selectivity, response time and reduced the operating temperature from 300 ^{0}C to 250 ^{0}C for NH_3 sensor. *Copyright © 2010 IFSA.*

Keywords: NH_3 sensor, Cu–Zn ferrite, Sensitivity, Selectivity, Palladium.

1. Introduction

The increasing concern over environmental monitoring and safety demand in industry and home has generated great interest in developing gas sensors [1]. A large number of metal oxides, i.e. SnO_2, ZnO, WO_3, TiO_2, Fe_2O_3 and mixed oxides, were reported to be sensitive to certain gas species. Now-a-days, the atmospheric pollution has become a global issue. The gases from auto and industrial exhausts are polluting the environment. The need to detect, measure and control these gases has led to the research and development of a wide variety of sensors using different materials and techniques.

Recently, the need of ammonia sensor has greatly increased in many fields of technological importance, such as food technology, chemical engineering, medical diagnosis, environmental protection, monitoring of car interiors and industrial processes [2]. Semiconductor metal oxide chemical sensors have shown advantages in commercialization prospect and market potential. These advantages include long lifetime, fast response and recovery time, low cost, simple electronic structure, and low maintenance [3]. Hence, the metal oxide gas-sensing materials have been widely investigated for a long time [4–6]. Recently, some composite oxides such as spinel [7] and perovskite [8] were found to be more attractive than single-metal oxides for their better selectivity and/or sensitivity to certain gases. Spinel-type oxides with a general formula of AB_2O_4 are important mixed oxides in gas sensors, and have been investigated for the detection of both oxidizing and reducing gases.

Other earlier reports about the application study of $ZnFe_2O_4$ as a gas sensing material include $ZnFe_2O_4$ particles to H_2S [9], a directly deposited film to CO [10], ultrafine powder to Cl_2 [11], CdO-doped $ZnFe_2O_4$ to ethanol [12], a $ZnO/ZnFe_2O_4$ thick film to propanol [13]. Duan *et al.* [14–16] have designed a novel synthetic route of pure MFe_2O_4 (M = Mg, Co, and Ni) spinel ferrites using a single molecular precursor.

In present work, the Cu doped $ZnFe_2O_4$ nanopowders were synthesized by sol-gel citrate method. The $Zn_{0.6}Cu_{0.4}Fe_2O_4$ shows high sensing response to ammonia at an operating temperature 300 ^{0}C and negligible sensing response to other test gases like LPG, CO and H_2S. Dispersion of noble metal such as Pd further enhanced the sensing response and reduced the operating temperature up to 250 ^{0}C for ammonia gas. The structural and microstructural characteristics of $ZnFe_2O_4$ have been carried out with the help of XRD and SEM.

2. Experimental Details

2.1. Material Synthesis

The $Zn_{1-x}Cu_xFe_2O_4$ (x=0.2, 0.4, 0.6 and 0.8) were synthesized by sol-gel method using ethylene glycol as a solvent. All reagents were of analytical grade were used. Firstly, the analytically pure grade $[Fe(NO_3)_3.9H_2O]$, $[Zn(NO_3)_2.6H_2O]$, $[Cu(NO_3)_2{\cdot}3H_2O]$ and citric acid were weighed and dissolved in ion-free water at 80 ^{0}C for 2 h. Then ethylene glycol was added under constant stirring to obtain a homogeneous and stable sol. The solution was further heated in pressure vessel at about 130 ^{0}C for 12 h. During this reaction transparent solution was transform into a gel state with very high viscosity. The material was then heated in a furnace at 350 ^{0}C for 3 h and a violent combustion was occurs which spontaneously propagates until all the gel was burnt out to form a loose powder. The powder was then calcined at 650 ^{0}C for 6 h in order to improve the crystallinity of materials. Samples of resulting $Zn_{1-x}Cu_xFe_2O_4$ with varied weight percentage of Pd were prepared by the impregnation technique.

2.2. Characterization of Samples

The synthesized samples were characterized by powder X-ray diffraction (XRD) using a Siemens D 5000 diffractometer .The XRD data were recorded by using Cu Kα radiation (1.5406 Å). The intensity data were collected over a 2θ range of 10–70 ^{0} C. The average crystallite size of the samples was estimated with the help of Scherrer equation using the diffraction intensity of all prominent lines. The microstructure of the materials was evaluated by scanning electron microscopy (SEM).

3. Results and Discussion

3.1. X-ray Diffraction Study

The XRD patterns of the compositions $ZnFe_2O_4$ and $Zn_{0.6}Cu_{0.4}Fe_2O_4$ prepared by sol-gel citrate method calcined at 650 ^{0}C are presented in Fig. 1. Fig. 1 (a), shows the XRD pattern, which is in good agreement with XRD results previously reported in the literature for $ZnFe_2O_4$ [17]. Further confirmation of this result was found using the JCPDS card No: 77-0011 [18]. The diffraction lines are found to be sharp and no other phase has been detected for all the samples, which are consistent with face centered cubic spinel structure. Fig. 1 (b), shows the XRD pattern of $Zn_{0.6}Cu_{0.4}Fe_2O_4$ nanomaterial, which is a spinel- type oxide [19].The XRD peaks correspond to the cubic structure of the spinel ferrite and no other peaks observed are indicating the absence of any other phase or impurities. Extremely broad reflections are observed indicating nanosized particle nature of the material obtained. The average particle size of the nanocrystalline $ZnFe_2O_4$ and $Zn_{0.6}Cu_{0.4}Fe_2O_4$ according to the scherrer formula were in the range of 40–45 nm.

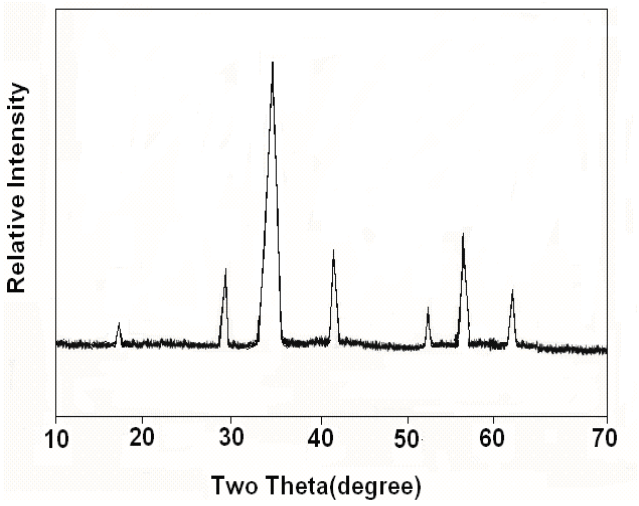

Fig. 1 (a). X-ray pattern of $ZnFe_2O_4$ calcined at 650 ^{0}C.

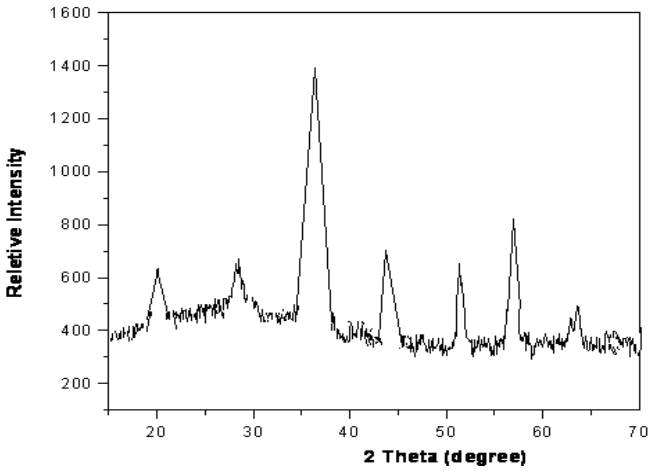

Fig. 1 (b). X-ray pattern of $Zn_{0.6}Cu_{0.4}Fe_2O_4$ calcined at 650 ^{0}C.

3.2. SEM analysis

The microstructure of the samples can be visualized from scanning electron micrograph of the synthesized material as in Fig. 2. One can notice the presence of irregular-shaped aggregates is formed by aggregation of very fine particles. Many large and small pores are present in the whole material. We assumed that the pores are mainly intergranular because intragranular pores are not observed on the SEM photograph.

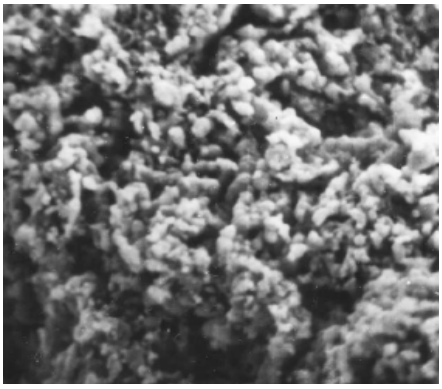

Fig. 2. SEM micrograph of $Zn_{0.6}Cu_{0.4}Fe_2O_4$ calcined at 650 ^{0}C.

3.3. Gas Sensing Properties

3.3.1. Fabrication and Analysis of Gas Sensors

The prepared powders were mixed with PVA (polyvinyl alcohol) to form paste, and then the paste was coated onto an Al_2O_3 tube on which two platinum wires had been installed at each end. A small Ni–Cr alloy coil was placed through the tube as a heater, which provided operating temperature at 50–350 ^{o}C. The temperature was controlled by adjusting the heating power. Gas response is defined as the ratio of change in resistance of the sample on exposure to a test gas to the resistance in the presence of air.

$$S = (R_a - R_g)/R_a = \Delta R/R_a\,, \tag{1}$$

where R_a is the resistance in air, R_g the resistance in a sample gas, and ΔR the change in resistance. The gas sensing properties of reducing gases such as LPG, H_2S, CO and NH_3 were measured. The test temperature range is from 50-300 ^{0}C and the gas concentration tested for 350 ppm.

3.3.2. Sensitivity of $Zn_{1-x}Cu_xFe_2O_4$

Fig. 3. shows the response as a function of operating temperatures for undoped $ZnFe_2O_4$ nanopowder calcined at 650 ^{0}C for 6 h for NH_3 gas. The sensor element shows the high response 0.22 towards NH_3 at an operating temperature 300 ^{0}C. In order to promote gas response, dopants are shown to effectively influence the semiconductive properties of sensor materials. For the mixed ferrites such as $Zn_{1-x}Cu_xFe_2O_4$ (x=0.2 to 0.8), the gas sensitivity depends on the operating temperature and Cu content. The sensitivity increases with increasing temperature and reaches a maximum value corresponding to an optimum operating temperature. For the $Zn_{0.8}Cu_{0.2}Fe_2O_4$ sample, there is a slow increase in the sensitivity to the maximum value of 0.44 at optimum operating temperature of 300 ^{o}C, whereas for $Zn_{0.6}Cu_{0.4}Fe_2O_4$sample, the sensitivity to the maximum value of 0.75 at 300 ^{o}C, then sensitivity decreases. Compared with other samples, $Zn_{0.6}Cu_{0.4}Fe_2O_4$ showed the large response to NH_3. The

reason may be that the partial replacement of Zn ions by Cu ions at the A-sites is advantageous to adsorption by creating active sites and oxidization for NH_3. In addition, the partial replacement results in a decrease of grain size and hence in an increase of surface area. Since small grains have relatively large grain boundary areas, the adsorption of NH_3 molecules is relatively high.

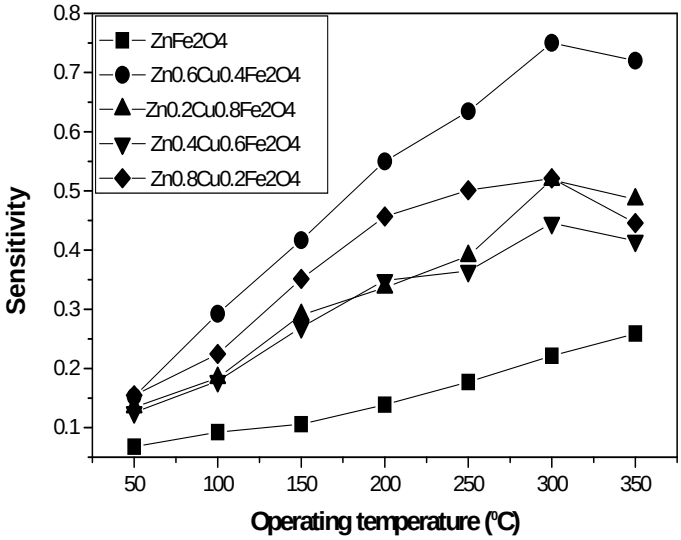

Fig. 3. Sensitivity vs. operating temperature for studied samples calcined at 650°C for NH_3 gas.

3.3.3. Selectivity Test

The sensitivity of the sample $Zn_{0.6}Cu_{0.4}Fe_2O_4$ was also investigated for different reducing gases such as LPG, NH_3, H_2S and CO at different operating temperatures. Fig. 4, shows the sensitivity of the $Zn_{0.6}Cu_{0.4}Fe_2O_4$ sample to various test gases as a function of operating temperature. It is seen that the sensor exhibits high sensitivity to NH_3 gas at an operating temperature of 300 ^{0}C and other reducing gases like LPG, H_2S and CO shows less response.

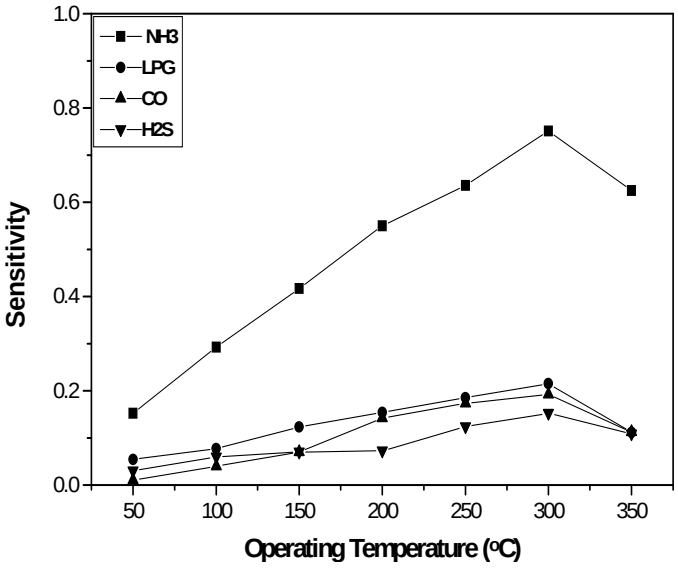

Fig. 4. Sensitivity vs. operating temperature of $Zn_{0.6}Cu_{0.4}Fe_2O_4$ calcined at 650 ^{0}C for various reducing gases.

3.3.4. Sensing behavior of Pd Loaded

In order to improve the response, Pd was incorporated to $Zn_{0.6}Cu_{0.4}Fe_2O_4$. Pd is proved to be highly effective in improving the sensitivity of semiconducting oxides for reducing gases [20]. The Pd is known to have a catalytic effect due to its excellent oxidation capability [21]. Fig. 5, shows sensitivity versus operating temperature of different wt. % Pd doped $Zn_{0.6}Cu_{0.4}Fe_2O_4$,. It is evident from the figure that 1.0 wt.% Pd doped $Zn_{0.6}Cu_{0.4}Fe_2O_4$ shows the maximum sensitivity and then further increase in Pd concentration, sensitivity decreased drastically towards NH_3 gas, that suggests the importance of the dispersion of Pd on the semiconductor surface [22]. The incorporation of Pd results in a drastic decrease in the operating temperature for maximum response for NH_3 from 300 to 250 ^{0}C.

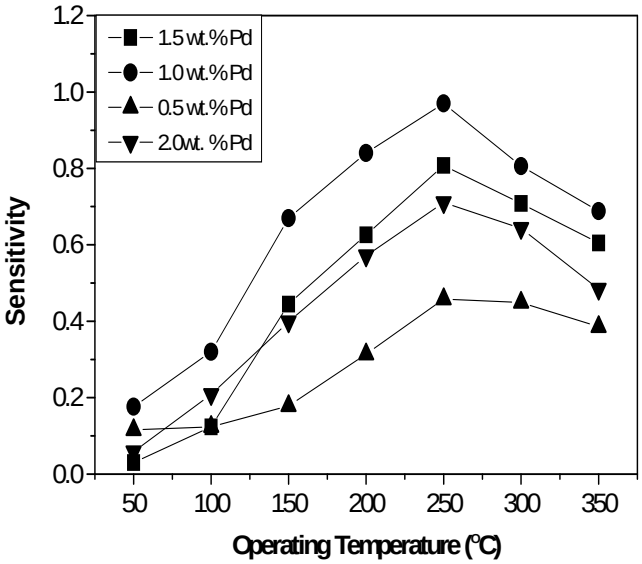

Fig. 5. Sensitivity vs. operating temperature with different concentration of Pd doped $Zn_{0.6}Cu_{0.4}Fe_2O_4$ calcined at 650 ^{o}C for NH_3 gas sensor.

3.3.5. Effect of Concentration on Gas Sensitivity

The sensitivity with the NH_3 gas concentration of the 1.0 wt. % Pd doped $Zn_{0.6}Cu_{0.4}Fe_2O_4$ calcined at an operating temperature 650 ^{0}C as shown in Fig. 6. It is observed that the sensitivity increases linearly up to 250 ppm of NH_3 gas concentration and after that it saturates. The response of a sensor depends on removal of adsorbed oxygen molecules by reaction with a target gas and generation of electrons. For a small concentration of gas, exposed on a fixed surface area of a sample, there is a lower coverage of gas molecules on the surface and hence lower surface reaction occurred. An increase in gas concentration increases the surface reaction due to a larger surface coverage. A further increase in surface reaction will be gradual when the saturation point on the coverage of molecules is reached. After 250 ppm level of NH_3 gas the curve flattens because there would not be enough ionosorbed oxygen species to contribute to detecting mechanisms. The linear behavior of the curve may be extended to some extent by taking longer area of the sensor.

3.3.6. Response and Recovery of the Sensor

Fig. 7 shows the response characteristics for the $Zn_{0.6}Cu_{0.4}Fe_2O_4$ at 300 ^{o}C and 1.0 wt. % Pd doped $Zn_{0.6}Cu_{0.4}Fe_2O_4$ at 250 ^{o}C to 250 ppm NH_3 gas. The 1.0 wt. % Pd doped $Zn_{0.6}Cu_{0.4}Fe_2O_4$ sensor exhibits shorter response and recovery times (10 and 25 s respectively) than pure $Zn_{0.6}Cu_{0.4}Fe_2O_4$.

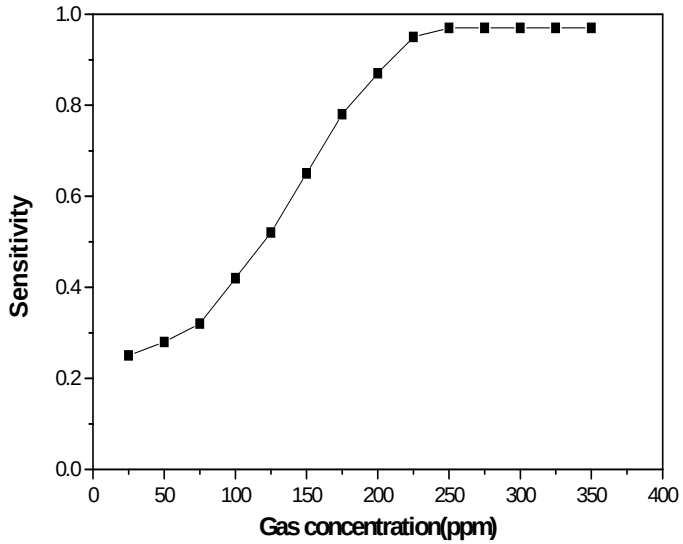

Fig. 6. Sensitivity to ethanol gas concentration for 1.0 wt. % Pd doped $Zn_{0.6}Cu_{0.4}Fe_2O_4$ calcined at 650 ^{0}C.

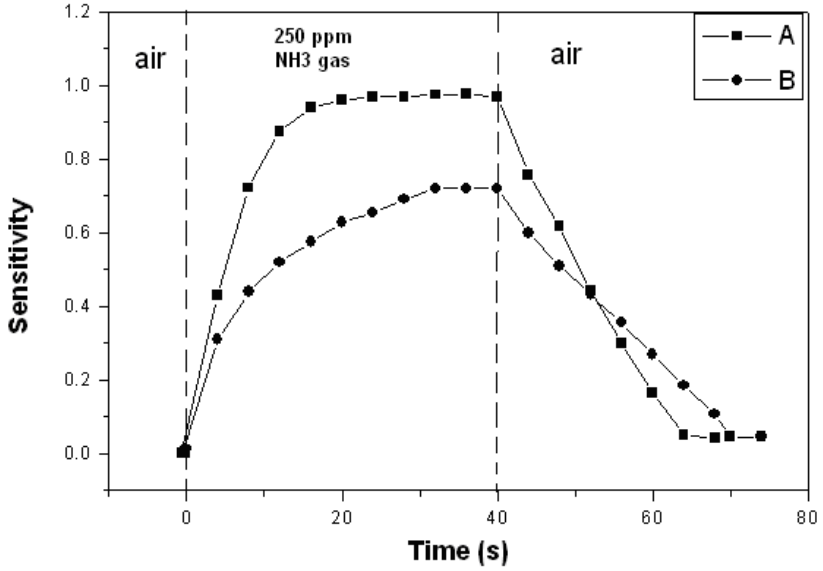

Fig. 7. The response and recovery characteristics to 250 ppm NH_3 gas for (A)1.0 wt. % Pd doped $Zn_{0.6}Cu_{0.4}Fe_2O_4$ and (B) $Zn_{0.6}Cu_{0.4}Fe_2O_4$

4. Concussion

Nanocrystalline Cu–Zn ferrite has been synthesized by a convenient sol–gel citrate route. The XRD pattern of $Zn_{0.6}Cu_{0.4}Fe_2O_4$ shows spinel structure. The results revealed that the particle size is in the range of 40–45 nm for $Zn_{0.6}Cu_{0.4}Fe_2O_4$ with good crystallinity. The $Zn_{0.6}Cu_{0.4}Fe_2O_4$ sensor shows excellent sensing characteristics towards NH_3 gas at operating temperature 300 ^{0}C The 1.0 wt% Pd incorporation lowers the operating temperature from 300 to 250 ^{0}C and exhibited the highest response value, excellent selectivity and quick response behavior to NH_3 gas. The sensor is very promising for NH_3 detection in the range 0-250 ppm with a response time in second range.

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

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

Guide for Contributors

Aims and Scope

Sensors & Transducers Journal (ISSN 1726-5479) provides an advanced forum for the science and technology of physical, chemical sensors and biosensors. It publishes state-of-the-art reviews, regular research and application specific papers, short notes, letters to Editor and sensors related books reviews as well as academic, practical and commercial information of interest to its readership. Because it is an open access, peer review international journal, papers rapidly published in *Sensors & Transducers Journal* will receive a very high publicity. The journal is published monthly as twelve issues per annual by International Frequency Association (IFSA). In additional, some special sponsored and conference issues published annually. *Sensors & Transducers Journal* is indexed and abstracted very quickly by Chemical Abstracts, IndexCopernicus Journals Master List, Open J-Gate, Google Scholar, etc.

Topics Covered

Contributions are invited on all aspects of research, development and application of the science and technology of sensors, transducers and sensor instrumentations. Topics include, but are not restricted to:

- Physical, chemical and biosensors;
- Digital, frequency, period, duty-cycle, time interval, PWM, pulse number output sensors and transducers;
- Theory, principles, effects, design, standardization and modeling;
- Smart sensors and systems;
- Sensor instrumentation;
- Virtual instruments;
- Sensors interfaces, buses and networks;
- Signal processing;
- Frequency (period, duty-cycle)-to-digital converters, ADC;
- Technologies and materials;
- Nanosensors;
- Microsystems;
- Applications.

Submission of papers

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

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