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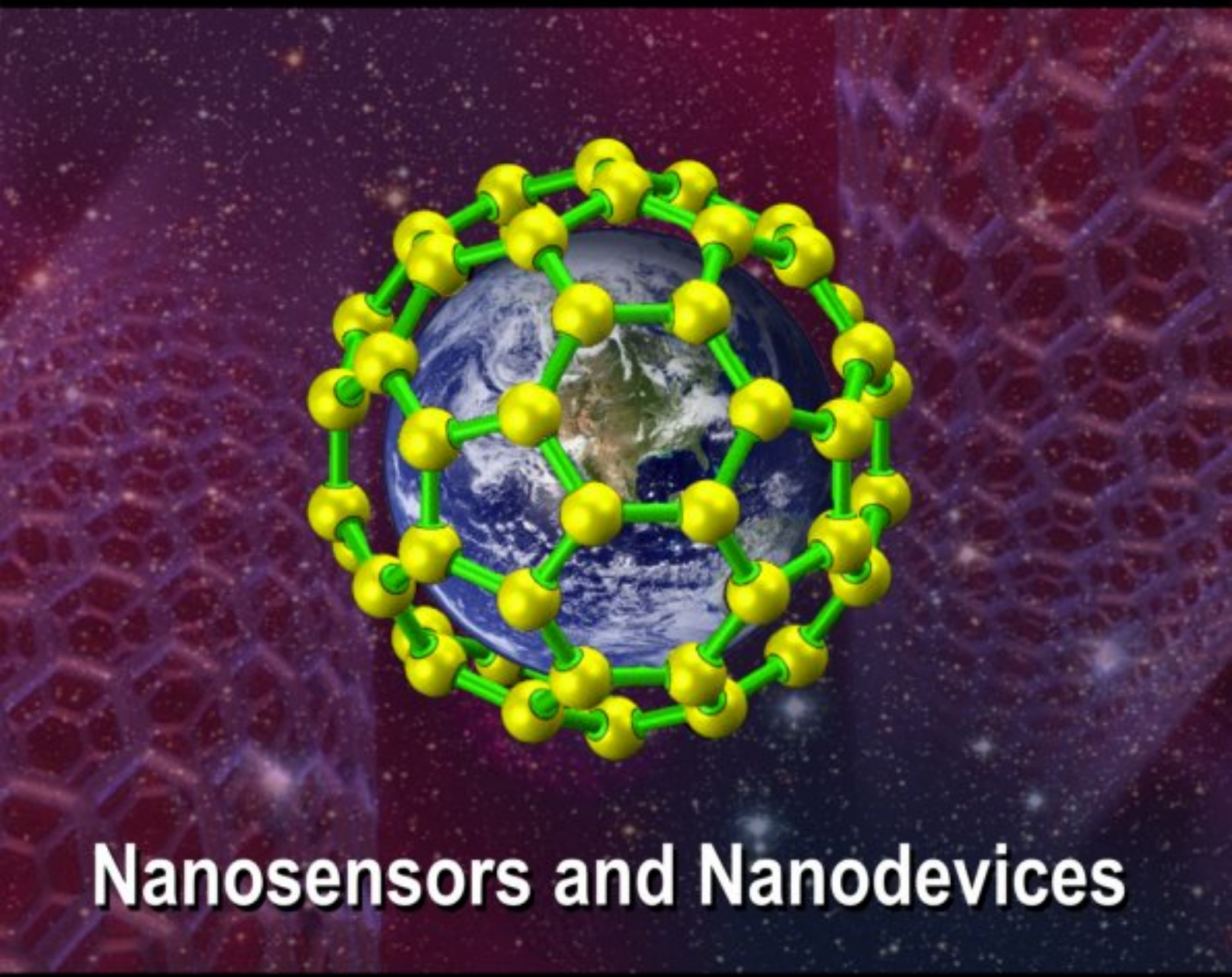

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Nanostructured Ferrite Based Electronic Nose Sensitive to Ammonia at Room Temperature

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Abstract: Manganese and Nickel doped Zinc Ferrite powder ($Mn_{0.3}Ni_{0.3}Zn_{0.4}Fe_2O_4$) was synthesized by autocatalytic thermal decomposition technique. The average crystallite size in the material powder was found to be of 10 – 13 nm. Characterization techniques such as X-Ray diffraction studies, Transmission electron microscopy, Infra-Red spectroscopy, etc, were employed to study the average particle size, phase and composition of the ferrite. Thick films of $Mn_{0.3}Ni_{0.3}Zn_{0.4}Fe_2O_4$ were prepared by screen printing technique. These films were observed to be sensitive to 10 ppm NH_3 at room temperature. The effects of surface microstructure, operating temperature, gas concentrations, etc., on the gas response, selectivity, response and recovery times of the sensor in the presence of NH_3 and other gases were studied and discussed. *Copyright © 2011 IFSA.*

Keywords: $Mn_{0.3}Ni_{0.3}Zn_{0.4}Fe_2O_4$, Ferrites, Room temperature, NH_3, Gas sensor.

1. Introduction

Recently, mankind along with the planet earth suffered from the serious natural disasters viz. Tsunami waves, cyclones, high tides, earthquakes, rainstorm, along with global diseases like chickun gunia, bird flu, swine flu, dengue, and many physiological and psychological hazards. Such natural disasters are the products of global warming. This ultimately depends upon environmental pollution. Pollution has raised its ugly head high in the global environment, in this century. To minimize the risks from environmental pollution, we should turn our attention towards the monitoring of air pollutants. Unwanted gases from industries and automobiles, smoke, hydrocarbons, particulate matters, etc., are the major air pollutants. Few natural activities like volcano eruption, accidental fires in forests, pollen

grains from plants [1], etc., also emit the pollutants in the environment. The gases which beyond the certain limit can cause undesirable and disastrous effects on human and environment are called as pollutants. Carbon monoxide, carbon dioxide, hydrogen sulfide, sulfur dioxide, sulfur trioxide, nitric oxide, nitrogen dioxide, chlorine, ethanol, ammonia, etc., are toxic and polluting gases; the leakages of which can reach to dangerous level up to 100 ppm or even lower than 100 ppm. It can cause the serious health hazards in all sense. Exposure of a few such gases up to 150 ppm and above can cause death. This article is specially prepared for ammonia gas sensing, as ammonia is toxic [2-6] in nature. The exposure of ammonia causes chronic lung disease, irritating and even burning the respiratory track, etc. Therefore, all industries working on and for ammonia should have an alarm system detecting and warning for dangerous ammonia concentration levels. It is therefore, necessary to monitor ammonia gas and to develop the sensors which could detect the ammonia gas at trace level.

Along with semiconducting materials, spinel ferrites [7-10] also exhibit this great property of detecting the gaseous species in the environment. Some well-known materials for NH_3 gas sensing are ZnO [11], modified-ZnO [12], iridium oxide [13], molybdenum oxide [14], polyaniline [15-17], polypyrrole [18], Au and MoO_3-modified WO_3 [19, 20], Pt and SiO_2-doped SnO_2 [21], etc. Various ammonia sensors reported basically work at higher temperature such as 300^{o}C - 350^{o}C, but it is not convenient to work at such high temperature. The room temperature sensors are therefore necessary. The electrolytic techniques using diaphragm electrodes are generally used for the detection of ammonia. However, this method is expensive and does not have sufficient response and selectivity for ammonia [22, 23]. Another technique utilizes a Pd-metal oxide semiconductor MOS device. This device is sensitive to ammonia but it suffers from poor selectivity. A few sensor models are also available for detecting ammonia gas. They are Figaro gas sensor models TGS 824 and TGS 826 (detection range 50-300 ppm) and Sierra gas monitor model CM 99-447 (electrochemical type, detection range 50-200 ppm). These models detect ammonia concentration in the range 50-200 ppm. However, threshold limit value (TLV) for NH_3 gas is 25 ppm. So, there is a need to develop the ammonia sensor which could detect the ammonia gas at trace level, i. e. below 25 ppm.

Also, the noble metal additives like Pt, Pd, Au, and Ag to a base material like ZnO, for the modification, increases the cost of the sensors. Therefore the applicability of these sensors remain limited. Hence the sensors operable at room temperature with low cost additives must be developed for larger applicability. In the present work, the efforts are made to develop a room temperature ammonia sensor with low cost ferrites.

2. Experimental Procedure

2.1. Synthesis of Powder and Thick Film Fabrication

A requisite quantity of sodium fumarate in aqueous medium was stirred with hydrazine hydrate (99-100 %) in an inert atmosphere for 2 h. In this solution, a stoichiometric amount of freshly prepared ferrous chloride solution mixed with manganous chloride, nickel chloride and zinc chloride was added drop wise with constant stirring in an inert atmosphere. The yellow colored precursor thus obtained was filtered, washed with ethanol and dried with diethyl ether using suction technique [24]. This dried precursor was then auto-catalytically decomposed to yield nano-size $Mn_{0.3}Ni_{0.3}Zn_{0.4}Fe_2O_4$ ferrite powder. For this, the precursor was first spread over a Petri dish and a burning splinter was brought near to it. When small portion of it caught fire, a red glow that formed spread over the entire bulk completing the total decomposition of the precursor in an ordinary atmosphere to form ‘as prepared’ nano-ferrite powder at lower temperature. The thixotropic paste was formulated by mixing the synthesized $Mn_{0.3}Ni_{0.3}Zn_{0.4}Fe_2O_4$ fine powder with a temporary binder as explained elsewhere [25, 26]. The thixotropic paste was screen printed on a glass substrate in desired patterns. The films prepared

were fired at 500 ^{o}C for 30 min. Silver contacts were made by vacuum evaporation for electrical measurements.

2.2. Details of the Gas Sensing System

Fig. 1 represents 'static gas sensing system' to examine the sensing performance of thick films. 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 with adjustable ON and 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 test gas using a gas-injecting syringe. A constant voltage was applied to the sensor, and current was measured by a digital pico-ammeter. Air was allowed to pass into the glass dome after every NH_3 gas exposure cycle.

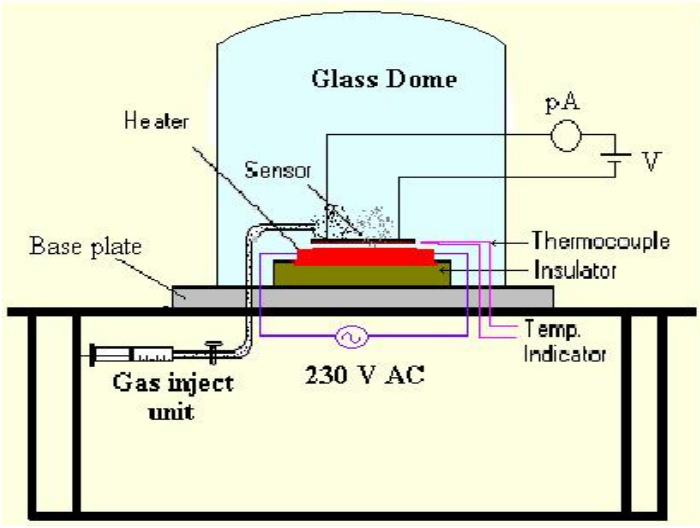

Fig. 1. Block diagram of Static gas sensing system.

3. Results and Discussion

3.1. Materials Characterizations

3.1.1. Structural Properties (XRD)

Fig. 2 depicts the X-ray diffractogram of as prepared $Mn_{0.3}Ni_{0.3}Zn_{0.4}Fe_2O_4$ ferrite powder. The observed peaks are matching well with JCPDS reported data of single phase spinel structure of $Mn_{0.3}Ni_{0.3}Zn_{0.4}Fe_2O_4$ ferrite. The sharp peaks of the XRD pattern correspond to $Mn_{0.3}Ni_{0.3}Zn_{0.4}Fe_2O_4$ and are observed to have high degree of crystallinity. The average grain size was determined using Scherer's formula and was estimated to be of ~ 13 nm. The crystals show anisotropy because different directions within the repeating pattern interact differently with incident radiations.

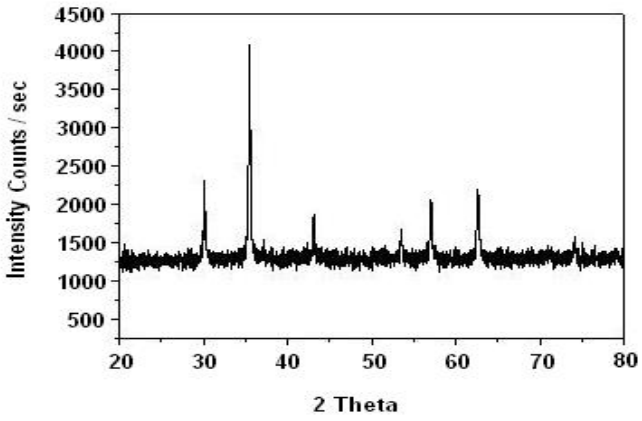

Fig. 2. XRD of $Mn_{0.3}Ni_{0.3}Zn_{0.4}Fe_2O_4$ powder.

3.1.2. Scanning Electron Micrographic Studies (SEM)

Fig. 3 depicts the micrograph of $Mn_{0.3}Ni_{0.3}Zn_{0.4}Fe_2O_4$ sample under test. All particles were observed to be distributed randomly. The film was observed to be porous. Porosity increases the effective surface area of the film. As, the gas sensing phenomenon is the surface phenomenon, the gas sensing performance was enhanced by such porous films. The maximum ammonia gas molecules may reach to interstitials of the films, which enhance the ammonia sensing even at room temperature.

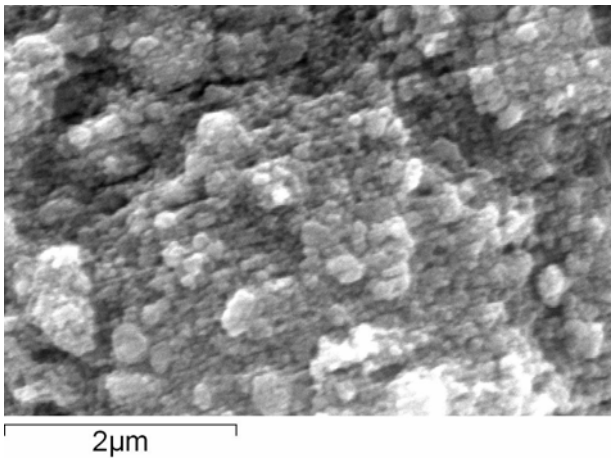

Fig. 3. SEM image of the Sample.

3.1.3. Transmission Electron Micrographic Studies (TEM)

Fig. 4 depicts the transmission electron micrograph of $Mn_{0.3}Ni_{0.3}Zn_{0.4}Fe_2O_4$ sample under test. The average crystallite size of $Mn_{0.3}Ni_{0.3}Zn_{0.4}Fe_2O_4$ ferrite powder was found to be of 10-13 nm. All the particles having identical shape and size with random distribution.

3.1.4. Infra-Red Spectroscopic Studies (IR)

IR spectra of as prepared $Mn_{0.3}Ni_{0.3}Zn_{0.4}Fe_2O_4$ is shown in Fig. 5, which shows two bands at 575 cm^{-1}, 394 cm^{-1} which are typical for spinel ferrites.

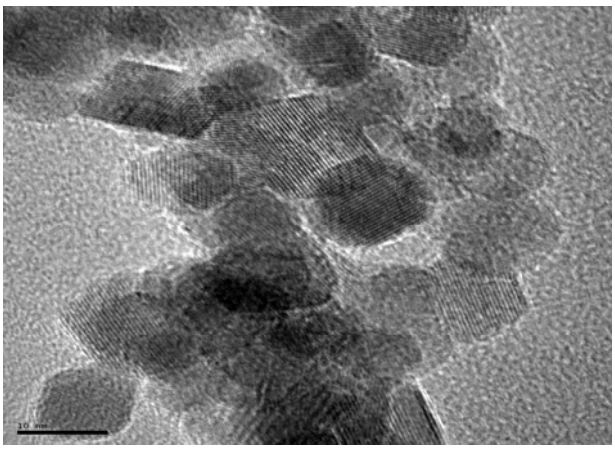

Fig. 4. TEM image of the Sample.

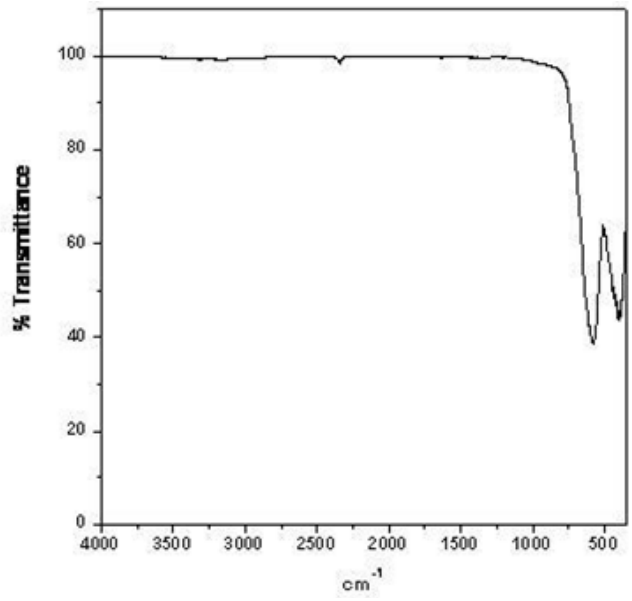

Fig. 5. IR of the Sample.

3.1.5. Thickness Measurement

The thicknesses of the ferrite films were observed to be in the range from 17 to 22 μm. The reproducibility of the film thickness was achieved by maintaining the proper rheology and thixotropy of the paste.

3.2. Electrical Conductivity of the Sensor

Fig. 6 shows the variation of log (conductivity) with reciprocal temperature of the $Mn_{0.3}Ni_{0.3}Zn_{0.4}Fe_2O_4$ film. The conductivity values of this ferrite sample increase with operating temperature. It is nearly linear to 1/T in the range from 75^{o}C to 400^{o}C. The increase in conductivity with increasing temperature could be attributed to the negative temperature coefficient of resistance and semiconducting nature of the $Mn_{0.3}Ni_{0.3}Zn_{0.4}Fe_2O_4$ film.

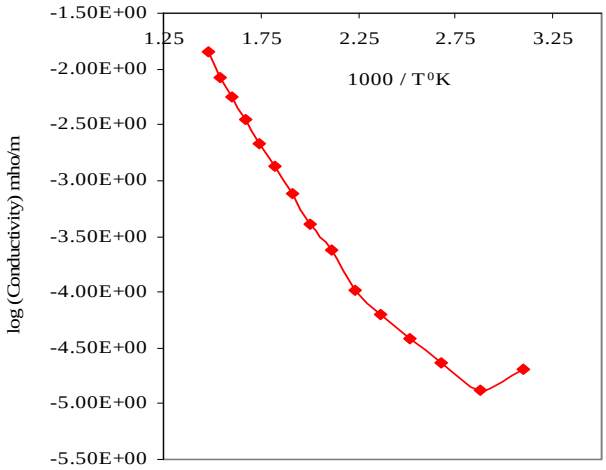

Fig. 6. Conductivity-Reciprocal Temperature profile of the Sensor.

3.3. Gas Sensing Performance of the Sensor

3.3.1. Measurement of Gas Response, Selectivity, Response and Recovery Time

The relative response of a sensor to a target gas can be defined as the ratio of the change in conductance of a sample before and after exposure of target gas to the conductance in air. The gas response can be defined as:

$$\text{Gas response} = \frac{G_g - G_a}{G_a} = \frac{\Delta G}{G_a}, \quad (1)$$

where G_a = conductance in air and G_g = conductance in a target gas.

Specificity or selectivity of the sensor can be defined as the ability of a sensor to respond to a certain gas in the presence of different gases. Response time (RST) is defined as the time required for a sensor to attain the 90% increase in the maximum conductance after exposure of the sensor surface to a test gas, while recovery time (RCT) is the time taken by the sensor to decrease the conductance up to 90% of the maximum conductance [25] in air.

3.3.2. Effect of Operating Temperature

Fig. 7 depicts the variation of NH_3 (10 ppm) response of $Mn_{0.3}Ni_{0.3}Zn_{0.4}Fe_2O_4$ ferrite thick film with operating temperature. The largest response of $Mn_{0.3}Ni_{0.3}Zn_{0.4}Fe_2O_4$ was observed to be 16.4 at room temperature. The ammonia response at room temperature is expected to be monitored by adsorption of moisture on the film. Upon exposure, ammonia interacts with adsorbed moisture on the film. The cumulative effect would decrease the film resistance, giving a response to ammonia gas at room temperature. There would be no oxygen adsorption on the film surface at room temperature. Therefore the oxygen adsorption-desorption mechanism is not employed to sense the NH_3 gas here. If the temperature of the film was raised above room temperature, the moisture from the film surface evaporates and hence the response would decrease further.

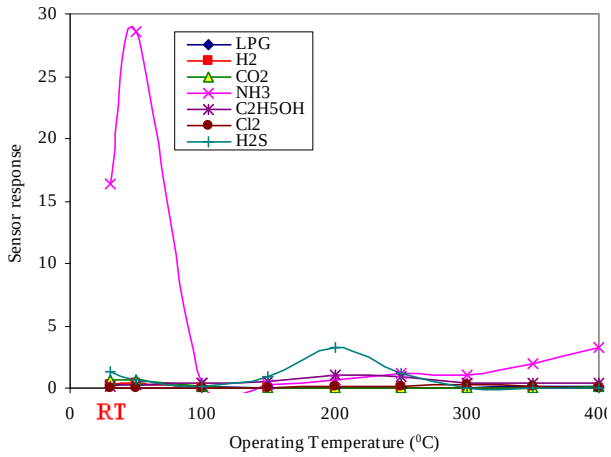

Fig. 7. Variation of Gas Response with Operating Temperature (^{o}C).

3.3.3 Active Region of the Sensor

The variation of gas response of the $Mn_{0.3}Ni_{0.3}Zn_{0.4}Fe_2O_4$ sample with NH_3 gas concentration at room temperature is represented in Fig. 8. This film was exposed to varying concentrations of NH_3. For the $Mn_{0.3}Ni_{0.3}Zn_{0.4}Fe_2O_4$ sample, the response values were observed to increase continuously with increasing the gas concentration up to 10 ppm at room temperature. The rate of increase in response was relatively larger up to 10 ppm, but smaller during 10 and 20 ppm. Thus, the active region of the sensor would be up to 10 ppm. At lower gas concentrations, the unimolecular layer of gas molecules would be formed on the surface of the sensor which could interact more actively giving larger response. The multilayers of gas molecules on the sensor surface, at the higher gas concentrations, would result into saturation in response beyond 10 ppm gas.

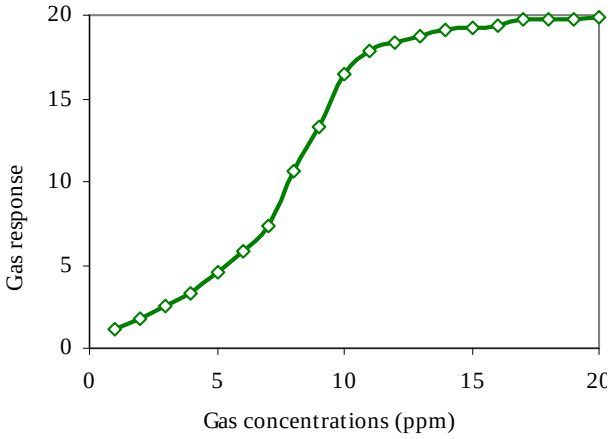

Fig. 8. Variation of Gas Response with Gas Concentration (ppm).

3.3.4. Selective Nature of the Sensor

Fig. 9 depicts the selectivity of $Mn_{0.3}Ni_{0.3}Zn_{0.4}Fe_2O_4$ sensor for NH_3 (10 ppm) gas at room temperature. The sensor showed high selectivity to NH_3 against LPG, CO_2, C_2H_5OH, H_2, H_2S and Cl_2 gases.

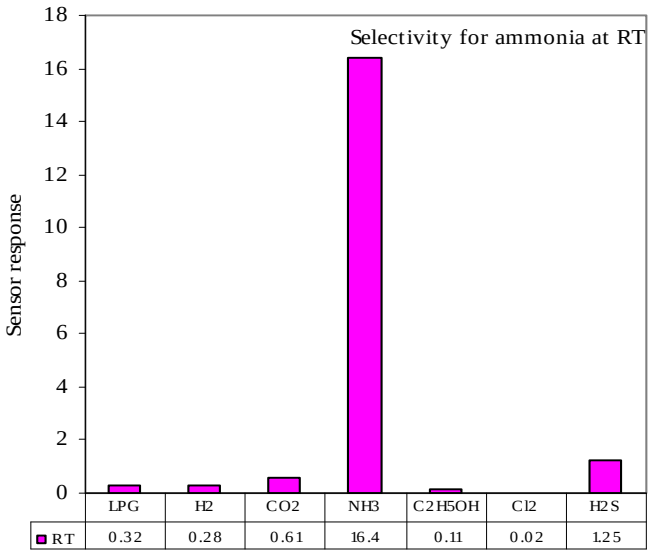

Fig. 9. Selectivity of the Sensor.

3.3.5. Response-recovery Profile

The response and recovery profiles of $Mn_{0.3}Ni_{0.3}Zn_{0.4}Fe_2O_4$ sensor are represented in Fig. 10. The response was quick (~ 13 s) to 10 ppm of NH_3, while the recovery was considerably fast (~ 22 s). A negligible quantity of the surface reaction product and its high volatility explain its quick response to ammonia and fast recovery to its initial chemical status.

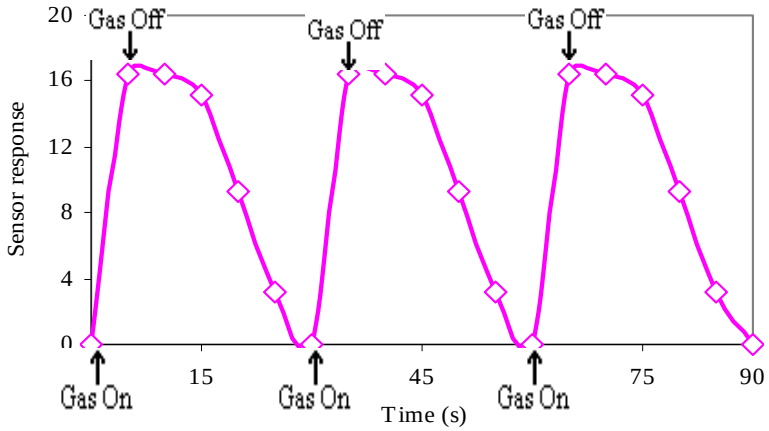

Fig. 10. Response-Recovery behavior of the Sensor.

4. Discussion

Gas sensing mechanism is generally explained in terms of conductance change either by adsorption of atmospheric oxygen on the surface and/or by direct reaction of lattice oxygen or interstitial oxygen with test gases. In the former case, the atmospheric oxygen adsorbs on the surface by extracting electrons from the conduction band to form superoxides or peroxides, which are mainly responsible for the detection of the test gases.

The selective ammonia response of the sensor at room temperature can be explained by the surface reaction processes. Few moles of H_2O from air (moisture) could be expected to adsorb on the surface of the film at room temperature. Upon exposure to ammonia (Fig. 11), a remarkable decrease in the resistance of the sensor was observed [11], which may be due to the surface reaction of ammonia with physisorbed H_2O or by proton conductivity via NH_4^+ cations. The solid acidity on the sensor surface would form NH_4^+ cations, which constitutes the proton conductivity leading to a crucial decrease of the resistance. This would decrease the barrier height among the $Mn_{0.3}Ni_{0.3}Zn_{0.4}Fe_2O_4$ grains.

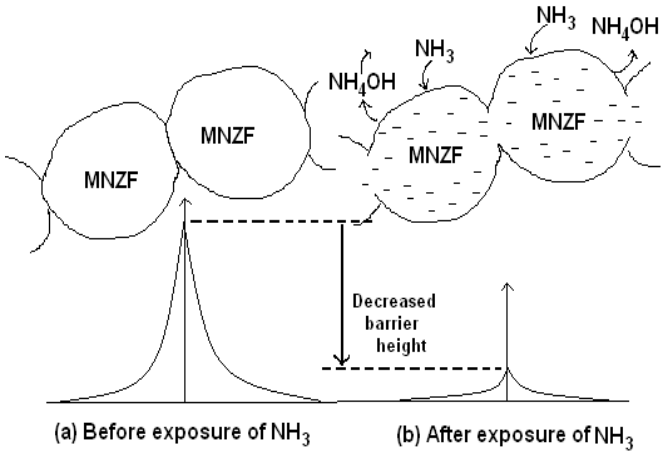

Fig. 11. Gas sensing mechanism for the Sensor material.

$$NH_{3(g)} + H_2O_{(surface)} \xrightarrow{RT} NH_4OH_{(g)} \quad (2)$$

Ammonium hydroxide NH_4OH produced during the surface reaction is volatile in nature. The high volatility of NH_4OH explains the quick response and fast recovery of the sensor.

5. Conclusions

From the results obtained, the following statements can be made for the sensing performance of $Mn_{0.3}Ni_{0.3}Zn_{0.4}Fe_2O_4$ sensors.

1. $Mn_{0.3}Ni_{0.3}Zn_{0.4}Fe_2O_4$ thick films were observed to be sensitive to 10 ppm NH_3 gas at room temperature.
2. The sensor was highly selective to 10 ppm NH_3 gas against other toxic gases of higher concentrations (> 1000 ppm).
3. The sensor showed very rapid response (~ 13 s) and recovery (~ 22 s) to NH_3 gas.
4. $Mn_{0.3}Ni_{0.3}Zn_{0.4}Fe_2O_4$ ferrite material has a potential to fabricate the room temperature NH_3 gas sensors at large scale.

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Sensors & Transducers Journal

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 of it is a peer reviewed international journal, papers rapidly published in *Sensors & Transducers Journal* will receive a very high publicity. The journal is published monthly as twelve issues per year by International Frequency Sensor 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. Since 2011 the journal is covered and indexed (including a Scopus, Embase, Engineering Village and Reaxys) in Elsevier products.

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