ISSN 1726-5479

SENSORS & TRANSDUCERS

vol. 139
4/12

MEMS and Modern Technologies

International Frequency Sensor Association Publishing

IFSA

Sensors & Transducers

Volume 139, Issue 4, April 2012 **www.sensorsportal.com** **ISSN 1726-5479**

Sensors & Transducers Journal (ISSN 1726-5479) is a peer review international journal published monthly online by International Frequency Sensor Association (IFSA).
Available in electronic and on CD.

Sensors & Transducers Journal

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Volume 139
Issue 4
April 2012

www.sensorsportal.com

ISSN 1726-5479

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International Frequency Sensor Association (IFSA).

Sensors & Transducers Journal, Vol. 139, Issue 4, April 2012, pp. 109-121

Sensors & Transducers

ISSN 1726-5479

http://www.sensorsportal.com

Nanostructured $Ni_{0.5}Zn_{0.5}Ce_3O_5$ Oxide Based Electronic Nose Sensitive to Ammonia at Operable Temperature

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Received: 22 March 2012 /Accepted: 23 April 2012 /Published: 30 April 2012

Abstract: Semiconductive nanoparticles of $Ni_{0.5}Zn_{0.5}Ce_3O_5$ were synthesized by a solution combustion technique. This process is a convenient, environment friendly, inexpensive and efficient for the preparation of $Ni_{0.5}Zn_{0.5}Ce_3O_5$ nanomaterial. The synthesized material is characterized by Thermo gravimetric Differential analysis (TG/DTA), X-ray Diffraction studied (XRD), Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM) techniques. Conductance response of the nanocrystalline $Ni_{0.5}Zn_{0.5}Ce_3O_5$ thick film is measured by exposing the film to reducing gases like ammonia, liquefied petroleum gas (LPG), hydrogen (H_2), acetone, carbon dioxide (CO_2) and ethanol. The sensor exhibited a fast response and a good recovery. The results demonstrated that $Ni_{0.5}Zn_{0.5}Ce_3O_5$ can be used as a gas-sensing material which has a high sensitivity and good selectivity to ammonia gas at 100 ppm. *Copyright © 2012 IFSA.*

Keyword: Nanostructured $Ni_{0.5}Zn_{0.5}Ce_3O_5$, XRD, TEM, Ammonia gas sensors.

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1. Introduction

Rare earth mixed metal oxides has an unusual variety of magnetic properties and structural changes [1-3]. Nanocrystalline $Ni_{0.5}Zn_{0.5}Ce_3O_5$ is chemically stable in oxidizing atmosphere. The doped nanocrystalline $Ni_{0.5}Zn_{0.5}Ce_3O_5$ shows high electrical conductivity, stability, dielectric constant, low dielectric loss, moderate permittivity, susceptibility, polarizability, ferroelectricity, and piezoelectricity. Catalytic activity in the decomposition of hydrogen peroxide as well as being used as sensors, can utilized in microwave dielectrics and as solid electrolytes. Ammonia is produced and utilized extensively in many chemical industries, fertilizer factories, refrigeration system, food processing, medical diagnosis, fire power plant, etc. A leak in the system can result the health hazards. Ammonia is harmful and toxic in nature [4-8]. The exposure of ammonia causes chronic lung disease, irritating and even burning the respiratory track etc. Environmental pollution is a burning global issue, pollution has raised its ugly head high in the global environment. Therefore, all industries working on and for ammonia should have an alarm system detecting and working for dangerous ammonia concentration levels. Detection of low concentration of ammonia is not only important from the points discussed above, but also it is very important from the view of chemical pollution in the production of silicon devices in clean rooms. It is therefore necessary to monitor ammonia gas and to develop the ammonia gas sensor. Efforts are made to develop the $Ni_{0.5}Zn_{0.5}Ce_3O_5$ gas sensor which should detect ammonia temperature dependent. Among various materials Cr_2O_3-activated ZnO thick film [9], some well known materials for NH_3 gas sensing are ZnO [10], modified –ZnO (viz. Fe-ZnO and Ru-ZnO), iridium oxide [11], molybdenum oxide [12], polyaniline [13], Au and MoO_3-modified WO_3 [14], Pt-and SiO_2-doped SnO_2 [15-17], etc.

The project describes a simple and cost-effective way of preparing of $Ni_{0.5}Zn_{0.5}Ce_3O_5$ nanoparticles by auto combustion method using nickel nitrate, zinc nitrate, cerium nitrate, glycine as fuel. Further it illustrates the structural, chemical composition, thermal morphology and gas sensing study of the samples using Thermo gravimetric Differential analysis (TG/DTA), X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FT-IR), high-resolution scanning electron microscopy (HR-TEM), transmission electron microscopy (TEM), particle size analyzer and static gas sensing units to studied sensing properties.

2. Experimental

2.1. Sample Preparation

Nanocrystalline powders of $Ni_{0.5}Zn_{0.5}Ce_3O_5$ compounds were synthesized by the combustion synthesis using urea as fuel (organic fuel). All chemical reagents were analytical grade and used without further purification. Stiochiometric quantities of reagent grade Nikel nitrate $Ni(NO_3)_2.6H_2O$, Zinc nitrate $Zn(NO_3)_2{\cdot}6H_2O$ and Ceric ammonium nitrate $(NH_4)_2.[Ce(NO_3)_6]$ and urea $CO(NH_2)_2$ (organic fuel) were taken in pyrex dish. One mole of metal nitrates and two moles of organic fuel were mixed together in a flat pyrex dish. The solution was stirred and evaporated on water bath at the temperature 80-90 ^{0}C. A thick gel formed finally was kept on hot plate for auto combustion at the temperature of 100-120 ^{0}C. The powder formed within five minutes (as- synthesized compound) was further annealed in air at different temperatures in the range of 250-850 ^{0}C for 8 h each. Nanocrystalline $Ni_{0.5}Zn_{0.5}Ce_3O_5$ powder synthesized by combustion route and gray colour powder.

2.2 Powder and Paste Preparation

Synthesized powder was ground in an agate pastel motor to ensure sufficiently fine particle size. The fine powder was calcined at 950 ^{0}C for 8 h, in air and re-ground. The thixotropic paste was formulated by mixing the resulting nanocrystalline $Ni_{0.5}Zn_{0.5}Ce_3O_5$ fine powder with a solution of ethyl cellulose (binder) in a mixture of organic solvents such as butyl cellulose, butyl carbitol acetate and turpineol. The ratio of inorganic to organic part was kept as 75:25 in formulating the paste. The paste was then used to prepare thick films.

2.3 Thick Film Preparation

The thixotropic paste was screen printed on a glass substrate in desired patterns. The films prepared were fired at 500 ^{0}C for 24 h. Silver contacts were made by vacuum evaporation for electrical measurements.

2.4 Thickness Measurement (Talystep Method)

Film thickness was observed to be in the range from 20 to 30 μm. The reproducibility of film thickness was achieved by maintaining the proper rheology and thixotropy of the paste.

2.5. Fabrication and Analysis of Gas Sensors

The sensing performance of the sensors was examined using a “static gas-sensing system. There were electrical feeds through the base plate. The heating wire was fixed on the base plate to heat the sample under test up to the required operating temperatures. The current passing through the heating element was monitored using a relay with adjustable ON and OFF time intervals. Cr-Al thermocouple was used to sense the operating temperature of the sensors. The output of the thermocouple was connected to digital temperature indicators. Gas inlet valve was fitted at one port 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. Constant voltage was applied to the sensors, and current was measured by a digital Pico-ammeter. Air was allowed to pass into the glass dome after every gases exposure cycle as shown in Fig. 1.

2.6. Characterization Techniques

X-ray diffraction (CuK_{α}, Bruker D 8 Advance) was used for confirming the phase and structural study. The surface morphological features were observed by using scanning electron microscopy (JSM-6360A). Transmission electron microscopy (TEM) of the powder was carried out by Make Philips CM-200 superwinster spectrometer at 200 kV. The DC resistivity was measured by the two probe method as a function of temperature (room temperature to 550 °C.)

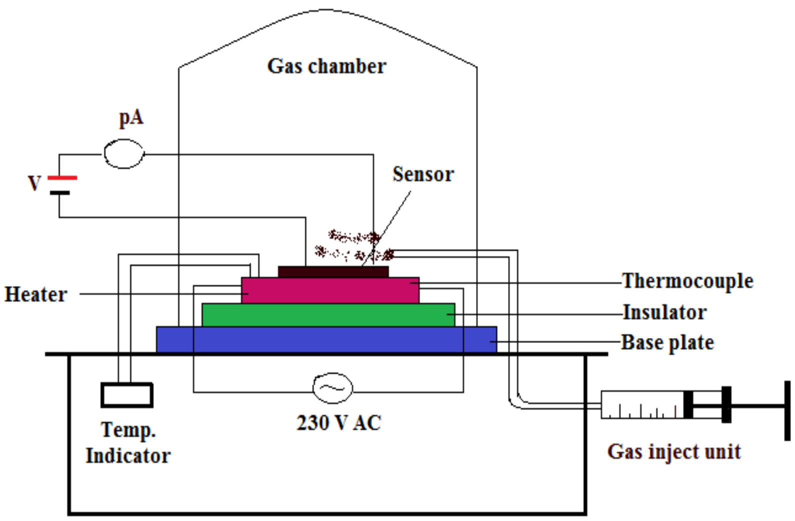

Fig. 1. Block diagram of static gas sensing setup.

3. Results and Discussion

3.1. TG-DTA Analysis

When reactants were heated at 180^0C the reaction proceeds by the mechanism indicated by equation (1) and (2) give the final product nanocrystalline $Cu_x Zn_{(1-x)}Ce_3O_5$.

$$2CO(NH_2)_2 + (3/2)O_2 \rightarrow N_2\uparrow + 2CO_2\uparrow + H_2O\uparrow + 2NH_3\uparrow \tag{1}$$

$$\begin{gathered} Zn(NO_3)_2.6H_2O + Ni(NO_3)_2.6H_2O + 3[(NH_4)_2.Ce(NO_3)_6] + CO(NH_2)_2 \rightarrow \\ Ni\,Zn\,Ce_3O_5 + CO_2\uparrow + 26H_2O\uparrow + 15N_2\uparrow + 23O_2\uparrow \end{gathered} \tag{2}$$

The TG curve recorded for thermal decomposition of nanocrystalline $Cu_{0.25}Zn_{0.75}Ce_3O_5$ is shown in Fig. 2. The curve indicates that the slight weight loss in nanocrystalline $Ni_{0.5}Zn_{0.5}Ce_3O_5$ powder at 120 ^{0}C due to little loss of moisture present in the material, weight loss at 200-500^{0}C was due to carbon dioxide, ammonia and nitrogen gas in the nanocrystalline $Ni_{0.5}\,Zn_{0.5}Ce_3O_5$. The DTA curve of nanocrystalline $Ni_{0.5}\,Zn_{0.5}Ce_3O_5$ recorded in static air, curve shows that nanocrystalline $Ni_{0.5}\,Zn_{0.5}Ce_3O_5$ did not decompose, but weight loss was due to dehydrogenation decarboxylation and denitration and yield final product at 950 ^{0}C. This weight loss was very negligible. This weight change was in the range of 2 % only. These indicate that the synthesized powder was almost stable. DTA curve showed the single phase material.

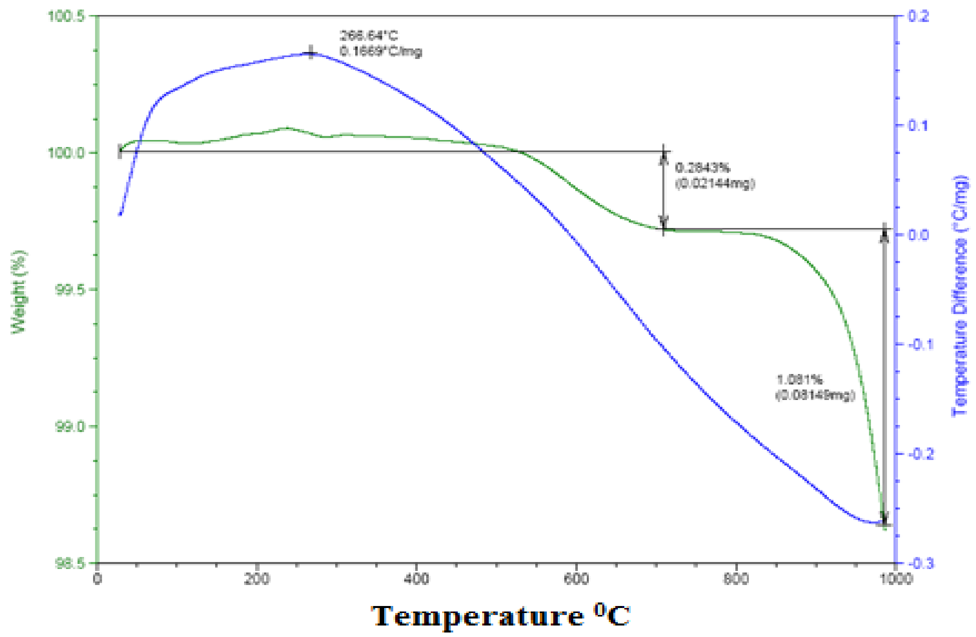

Fig. 2. TG - TDA curves of nanocrystalline $Ni_{0.5}\,Zn_{0.5}Ce_3O_5$ as synthesized powder.

3.2. XRD Analysis

Formation of the desired $Ni_{0.5}Zn_{0.5}Ce_3O_5$ perovskite was confirmed by X-ray diffraction analysis. The XRD pattern shown in Fig. 3 depicts broad peaks due to the nanosized of particles and matches with the reported JCPDS data. The intense peaks at 2θ, 33, 47, 56, 59, 69, 76, 79, 88 degrees which conveniently be assigned to reflections corresponding to (200), (102), (110), (222), (400), (331), (220) and (422) planes of $Ni_{0.5}Zn_{0.5}Ce_3O_5$ respectively. It is well known that, nanocrystalline

$Ni_{0.5}Zn_{0.5}Ce_3O_5$ (102) reflection is more intense in the cubic perovskite structures. No unidentified peaks are observed. The average crystalline size (t) was calculated using Scherrer's equation $t = 0.9\,\lambda / \beta \cos\theta$ where, t is the average crystalline size of the particles [18-23], assuming particles to be spherical K= 0.9, λ the wavelength of X-ray radiation, β the full width at half maximum (FWHM). The average crystalline size obtained for nanocrystalline $Ni_{0.5}Zn_{0.5}Ce_3O_5$ calcinated at 950 ^{0}C was found to be ~15 nm.

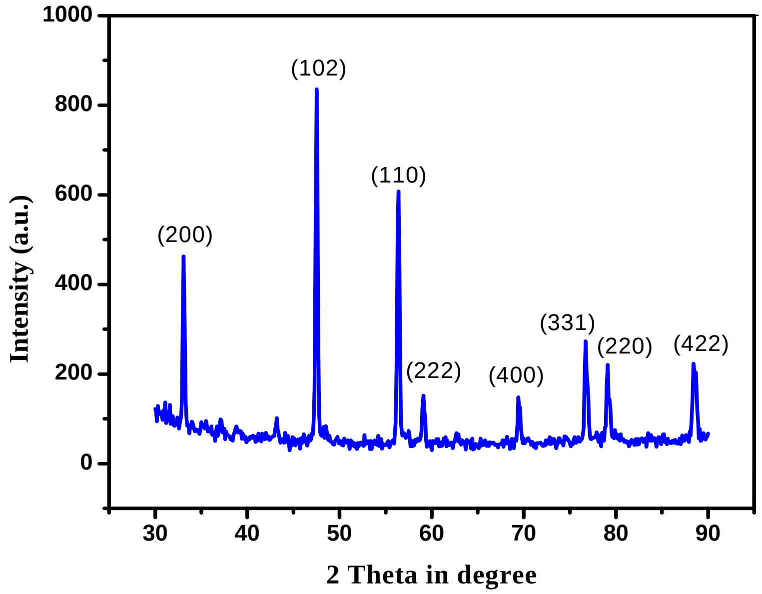

Fig. 3. X-ray diffraction patterns of nanocrystalline $Ni_{0.5}\,Zn_{0.5}Ce_3O_5$ as synthesized powder.

3.3. Particle Size Analysis

Particle size distribution studies have been carried out by using dynamic light scattering techniques (DLS) Via Laser input energy of 632 nm). It was observed that $Ni_{0.5}Zn_{0.5}Ce_3O_5$ oxide nanoparticles have narrow size distribution within the range of about 30-50 nm. Fig. 4. which are similar to the values calculated from Scherrer equation.

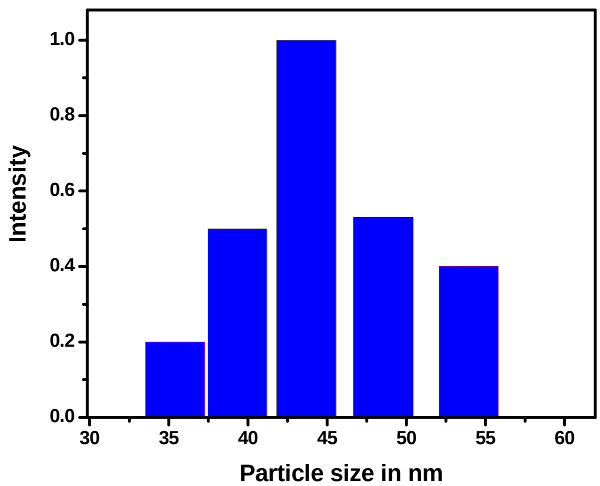

Fig. 4. Particle size distribution studies.

3.4. EDX Analysis

The elemental analysis of the sample was carried out by using energy dispersive X-ray (EDX) and is shown in Fig. 5. EDX result clearly shows that nanocrystalline $Ni_{0.5}Zn_{0.5}Ce_3O_5$ contains only Ni, Zn, Ce and O without any impurity.

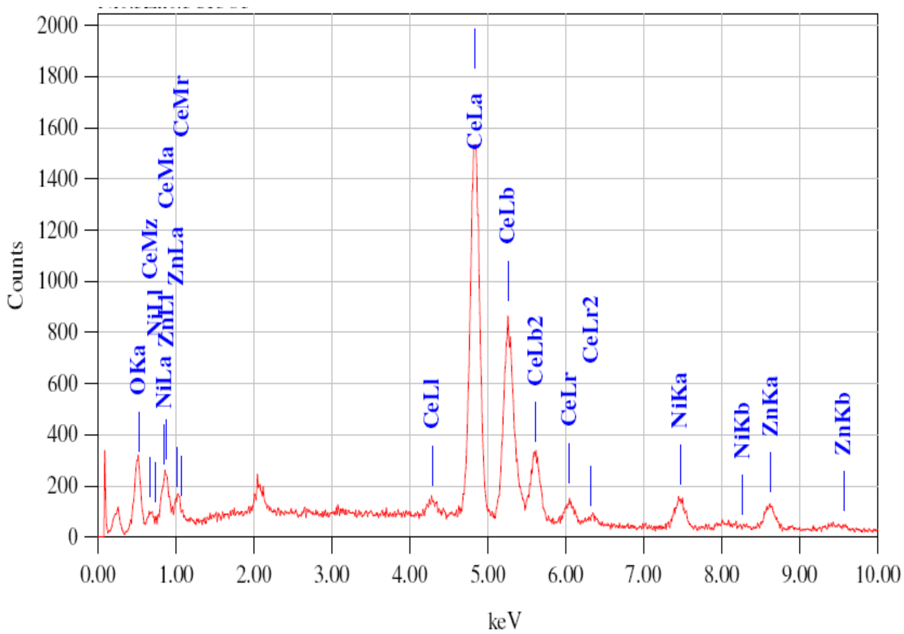

Fig. 5. Energy Dispersive X-ray (EDX) of $Ni_{0.5}Zn_{0.5}Ce_3O_5$ powder.

3.5. SEM Analysis

The SEM technique was employed for finding morphology of nanocrystalline powder as synthesized powder and heated at 950 ^{0}C Fig. 6. The initial powders exhibit fluffy bubble-like, micro agglomeration very fine particles under the electron microscope Fig. 6 (a). This can be attributed to the organic impurities and the large amount of gas produced during the violent combustion process. In order to remove organic impurities, the initial powders were calcined at 500 ºC in air. Fig. 6 (b) presents the SEM image of the calcined powders. After calcination, the particle size has reached the nano-meter level. It was also observed that the higher the content of combustion improver is, the more intense combustion will be, and the smaller particle size of the powders will become. presents the SEM image of nanocrystalline $Ni_{0.5}Zn_{0.5}Ce_3O_5$ powder which was pressed into wafer then sintered for 3 h at 950 ºC,. It can be seen that the particles was growing up.

3.6. TEM Analysis

The TEM specimen was prepared by placing microdrops of colloid solution on a carbon film supported by a copper grid. To get better understanding and clarity of the morphology the TEM image was taken which validate nearly uniform spherical particles with average size of 13-21 nm.The agglomeration, as seen in the SEM image was not observed here; on the contrary the image shows distinct nanoparticles of nearly spherical structure and clear boundaries Fig. 7 (c-d). The HRTEM image Fig. 7 (b) shows well developed lattice fringes, which are correlated well with the XRD results. The selected area diffraction (SAED) pattern Fig. 7 (a) shows the spot type pattern which is indicative of single crystalline particles. No evidence was found for more than one pattern, suggesting the single phase nature of the material.

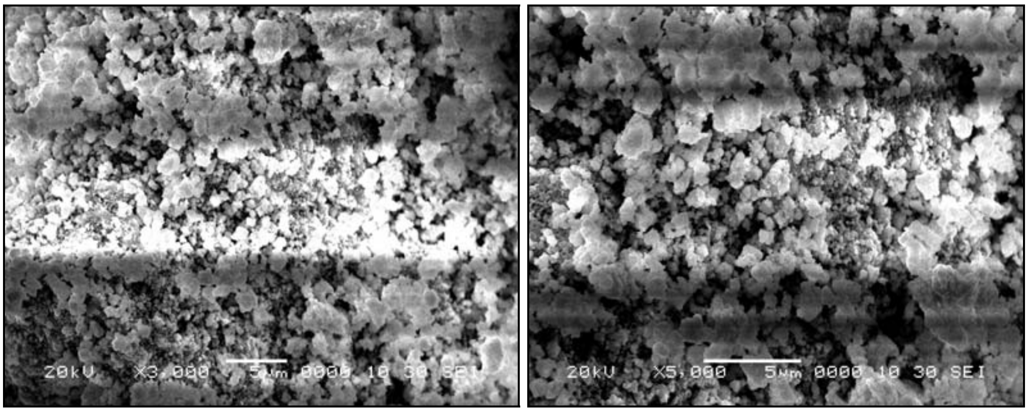

Fig. 6. SEM micrographs for nanocrystalline $Ni_{0.5}Zn_{0.5}Ce_3O_5$ powder.

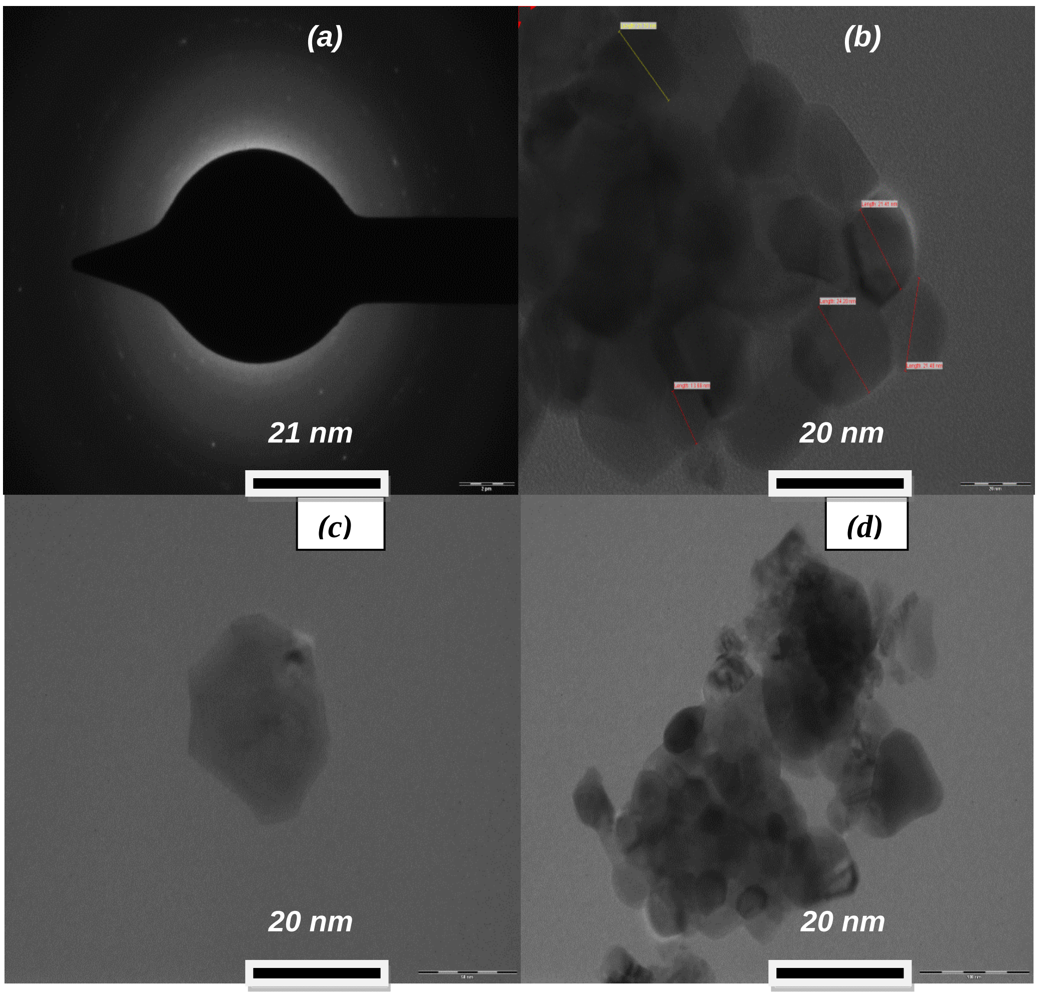

Fig. 7. Selected area electron diffraction pattern (a): high resolution transmission electron microscopy image of nanocrystalline $Ni_{0.5}Zn_{0.5}Ce_3O_5$ (b, c, d) transmission electron microscopy images of $Ni_{0.5}Zn_{0.5}Ce_3O_5$ calcinated at 850 ^{0}C.

4. Electrical Properties

4.1. I–V Characteristics

Fig. 8 depicts the I–V characteristics of the nanocrystalline $Ni_{0.5}Zn_{0.5}Ce_3O_5$ films at room temperature. It was clear from the I–V characteristics that the sliver contacts fabricated on the film were ohmic in nature. The voltage applied was in the range 1–30 V.

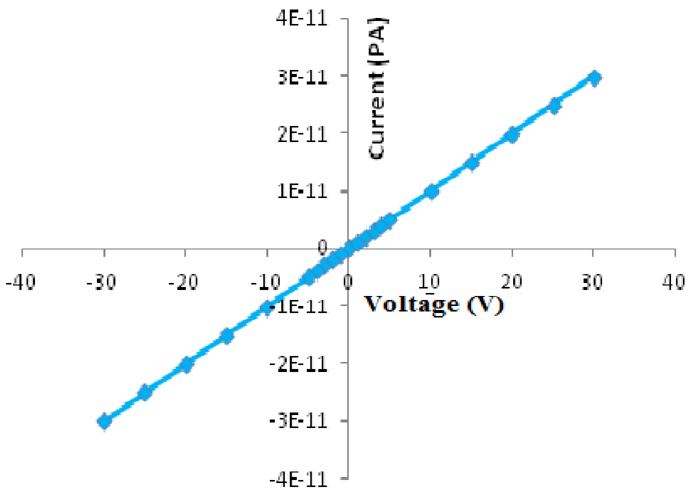

Fig. 8. I-V characteristics of nanocrystalline $Ni_{0.5}Zn_{0.5}Ce_3O_5$ thick film.

4.2. Electrical Resistivity Measurement

The direct current (dc) electrical resistivity measurements of annealed (660 ^{0}C) thick films were done by using dc two probe method. The area of the film was defined and silver paste was applied to ensure good electrical contact to the film. The working temperature was recorded using Chromal - Alumel thermocouple. The potential drop across the film was measured with the help of Meco-801 digital multimeter and current passed through the sample was recorded with a sensitive four digit Picoammeter (Model Roorkee DPM 111). The semiconducting nature of nanocrystalline $Ni_{0.5}Zn_{0.5}Ce_3O_5$ is observed from the measurements of resistivity with temperature.

The semiconductivity in nanocrystalline $Ni_{0.5}Zn_{0.5}Ce_3O_5$ must be due to large oxygen deficiency in it. The material would then adsorb the oxygen species at higher temperatures ($O_2^- \rightarrow 2O^- \rightarrow O^{2-}$). The increase in conductivity with increasing temperature could be attributed to negative temperature coefficient of resistance and semiconducting nature of the nanocrystalline $Ni_{0.5}Zn_{0.5}Ce_3O_5$. It is observed from Fig. 9 that the electrical conductivity of the nanocrystalline $Ni_{0.5}Zn_{0.5}Ce_3O_5$ is nearly linear in the temperature range from 300–675 K in air ambient.

5. Sensing Performance of Sensor

5.1. Gas Response, Selectivity, Response and Recovery Time

The relative response (S) to a target gas is defined as the ratio of the change in conductance of a sample upon exposure of the gas to the original conductance in air, which can be calculated by fallowing equation.

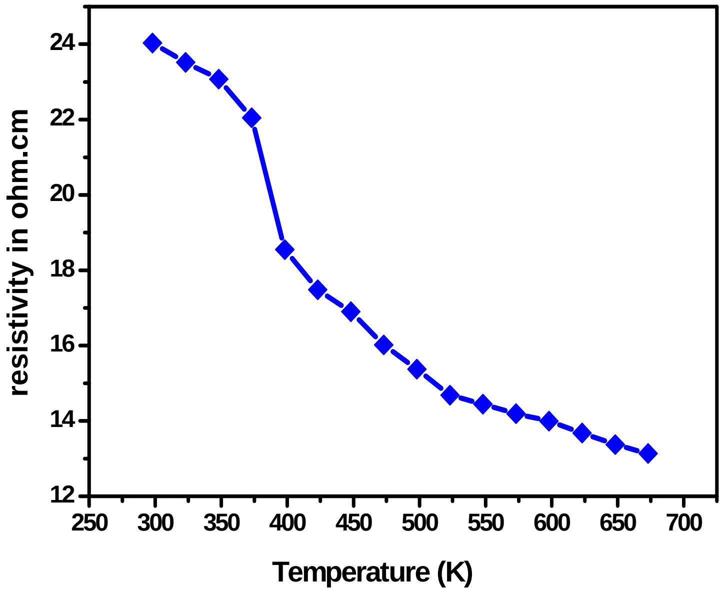

Fig. 9. Resistivity–temperature profiles of nanocrystalline $Ni_{0.5}Zn_{0.5}Ce_3O_5$ oxide.

$$S = \frac{G_g - G_a}{G_a},$$

where G_a is the conductance in air, and G_g is the conductance in a sample gas. Specificity or selectivity is 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 requires for a sensor to attain 90 % of the maximum increases in conductance on exposure to the target gas. Recovery time (RCT) is the time taken to get back 90 % of the original conductance in air.

5.2. Sensing Performance of $ZnCr_2O_4$ Thick Films

5.2.1. Response of Sensors to Various Gases

The response of $Ni_{0.5}Zn_{0.5}Ce_3O_5$ sample variation for different gases with operating temperature is represented in Fig. 10. It is clear from the figure that the gas responses goes on increasing and attain to their respective maxima and decreased further with increase in operating temperatures. It is clear from the figure that the $Ni_{0.5}Zn_{0.5}Ce_3O_5$shows the largest response to ammonia gas 200 ^{0}C.

5.2.2. Active Region of Sensor

Fig. 11 depicts the variation of gas response of $Ni_{0.5}Zn_{0.5}Ce_3O_5$ sample with ammonia gas concentrations. It is clear from the figure that the gas response goes on increasing linearly with gas concentration up to 100 ppm and saturated beyond it. The rate of increase in gas response was relatively larger up to 100 ppm. The monolayer of the gas molecules formed on the surface could cover the whole surface of the film. The gas molecules from that layer would reach the surface active sites of the film. The excess gas molecules would remain idle and would not reach surface active site of the sensor. So, the gas response at higher concentrations of the gas is not expected to increase further in large extent. Thus, the active region of the sensor would be up to 100 ppm.

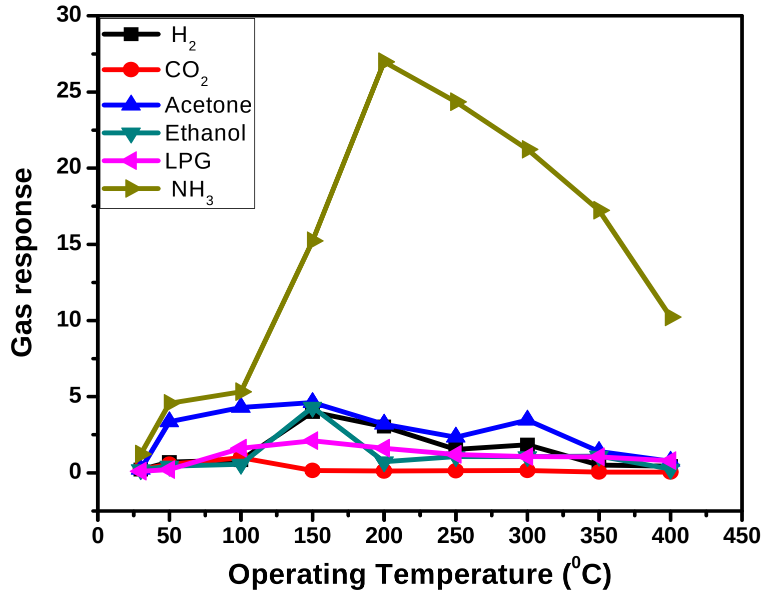

Fig. 10. Variation of gas response of $Ni_{0.5}Zn_{0.5}Ce_3O_5$ *i*th operating temperature.

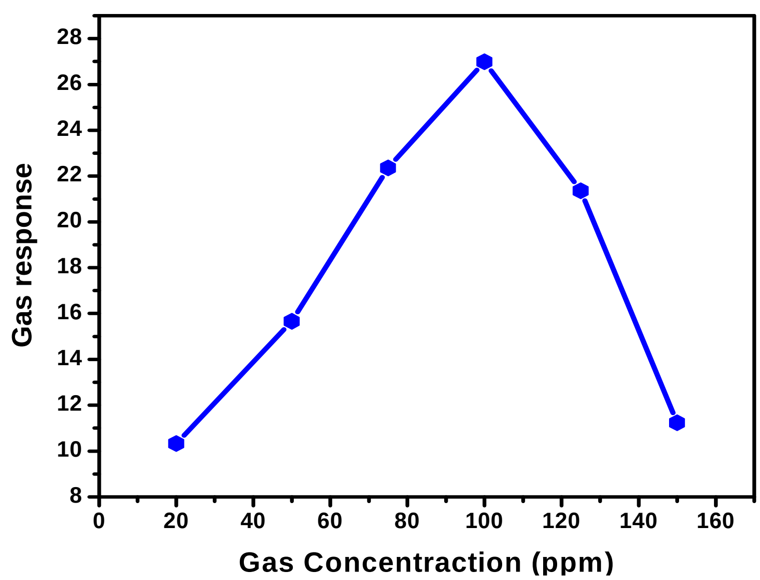

Fig. 11. Variation of gas response with gas concentration.

5.2.3. Selectivity Factor of $Ni_{0.5}$ $Zn_{0.5}Ce_3O_5$ for Various Gases

It is observed from Fig. 12. That the $Ni_{0.5}$ $Zn_{0.5}Ce_3O_5$ sensors give maximum response to ethanol vapours (100 ppm) at 200 ^{0}C. The sensors showed highest selectivity for ammonia gas against all other tested gases, LPG, Hydrogen, Acetone, Carbon dioxide and ethanol.

5.2.4. Response and Recovery Time

The response and recovery of $Ni_{0.5}$ $Zn_{0.5}Ce_3O_5$ sensor are the response was quick (~ 10 s) to 300 ppm of ethanol, while the recovery was fast (~ 20 s) .The quick response may be due to faster oxidation of gas. The negligible quantity of the surface reaction product and its high volatility explains its quick response and fast recovery to its initial chemical status.

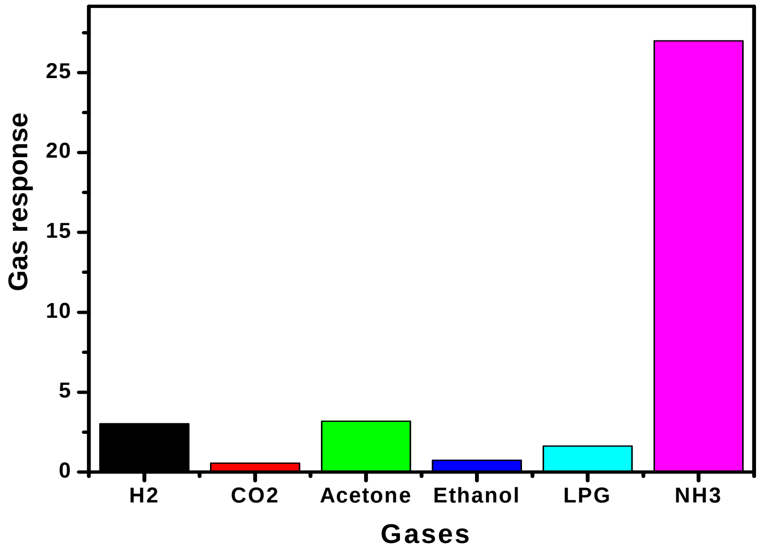

Fig. 12. Selectivity of $Ni_{0.5}Zn_{0.5}Ce_3O_5$ thick film among various gases.

6. 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 super oxides or peroxides, which are mainly responsible for the detection of the test gases.

The selective ammonia response of the sensor at high 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 high temperature. Upon exposure to ammonia a remarkable increase in the resistance of the sensor was observed 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 $NH4^+$ cations, which constitutes the proton conductivity leading to a crucial increase of the resistance. This would decrease the barrier height among the $Ni_{0.5}Zn_{0.5}Ce_3O_5$ grains. Ammonium hydroxide NH_4OH produced during the surface reaction is volatile in nature. The highvolatility of NH_4OH explains the quick response and fast recovery of the sensor [24-25].

7. Conclusion

(I) A nitrate gel synthesized from metal nitrate and glycine by sol-gel process with molar ratio of 1:1 exhibits auto combustion behavior. The auto combustion method proves good for the synthesis of nano-sized magnesium ferrites.

(II) The phage formation of the $Ni_{0.5}Zn_{0.5}Ce_3O_5$ is investigated by TG-DTA, XRD, FT-IR techniques. The synthesized product shows single phage of inverse spinel structure with an average diameter 10-15 nm.

(III) The sensor was highly selective to ammonia gas (100 ppm) against other toxic gases of higher concentrations.

(IV) The sensor showed very rapid response (~ 10 s) and recovery (~ 20 s) to ammonia gas.

(V) The sensor has good selectivity to ammonia gas against LPG, Hydrogen, Acetone, Carbon dioxide and ethanol.

Acknowledgement

The author S. V. Bangale is thankful to IIT Bombay for providing the TEM facility during this research work.

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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 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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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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- 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;
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- Technologies and materials;
- Nanosensors;
- Microsystems;
- Applications.

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