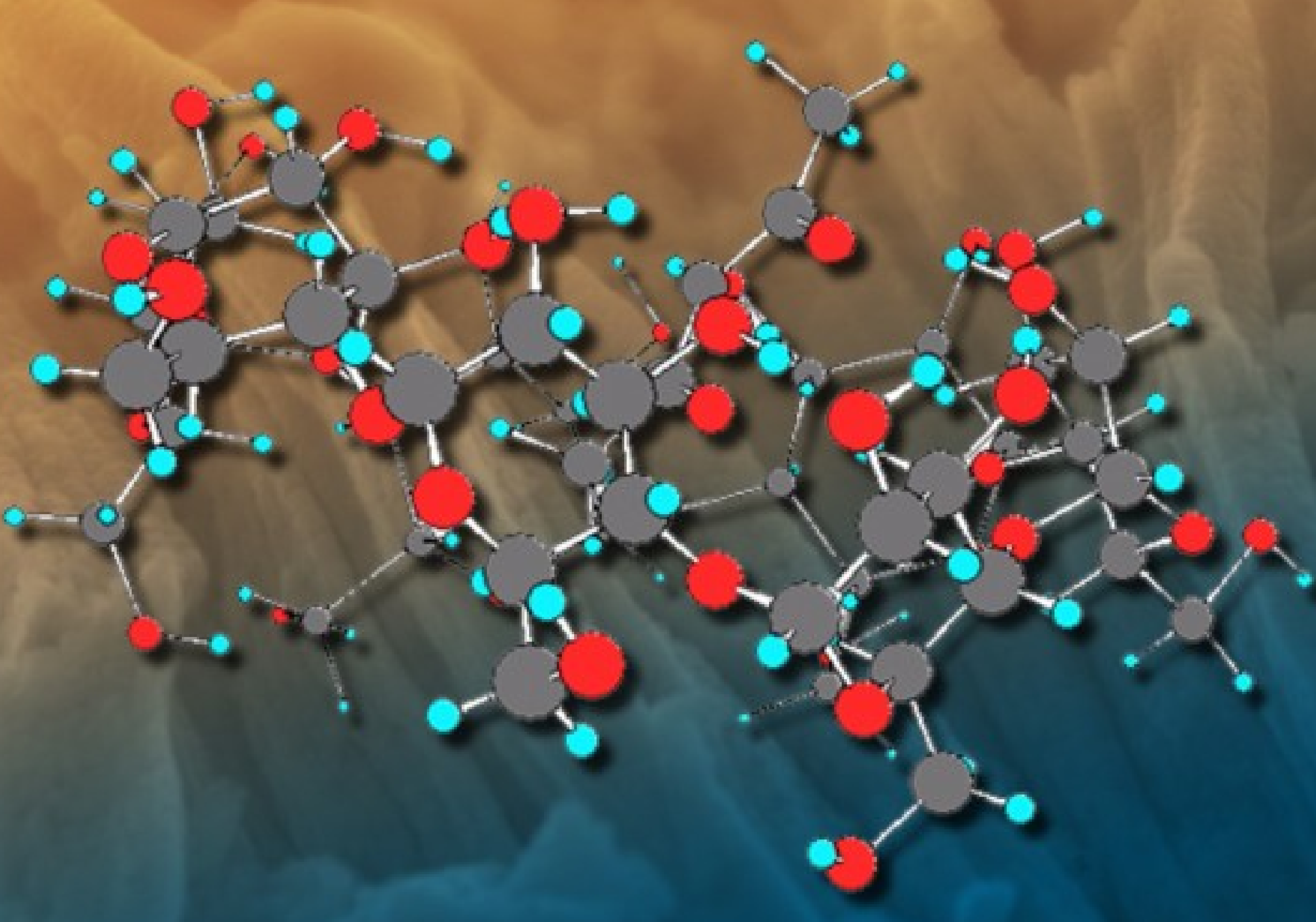
ISSN 1726-5479

SENSORS & TRANSDUCERS

11/12
vol. 146



Nanosensors and Nanodevices

International Frequency Sensor Association Publishing

IFSA

Sensors & Transducers

Volume 146, Issue 11
November 2012

www.sensorsportal.com

ISSN 1726-5479

Sensors & Transducers Journal

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Volume 146
Issue 11
November 2012

www.sensorsportal.com

ISSN 1726-5479

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Sensors & Transducers Journal, Vol. 146, Issue 11, November 2012, pp. 145-155

Sensors & Transducers

ISSN 1726-5479

http://www.sensorsportal.com

A Novel Combustion Route for the Preparation of Nanocrystalline $LaAlO_3$ Oxide Based Electronic Nose Sensitive to NH_3 at Room Temperature

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Received: 26 May 2012 /Accepted: 23 November 2012 /Published: 30 November 2012

Abstract: Semiconductive nanoparticles of $LaAlO_3$ were synthesized by a solution combustion technique. This process is a convenient, environment friendly, inexpensive and efficient for the preparation of $LaAlO_3$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 $LaAlO_3$ pellet is measured by exposing the pellet to reducing gases like ammonia, ethanol, acetone. The sensor exhibited a fast response and a good recovery. The results demonstrated that $LaAlO_3$ can be used as a gas-sensing material which has a high sensitivity and good selectivity 80% to ammonia gas at 50 ppm. *Copyright © 2012 IFSA.*

Keywords: $LaAlO_3$, ammonia gas sensor, XRD, TEM, gas response.

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1. Introduction

Ammonia is produced and utilized extensively in many chemical industries, fertilizer factories, refrigeration systems, food processing, medical diagnosis, fire power plants etc. A leak in the system can result the health hazards. Ammonia is harmful and toxic [1-5] in nature. Therefore all industries working on and for ammonia should have an alarm system detecting and warning for dangerous ammonia concentration levels. Environmental pollution is a burning global issue; pollution has raised its ugly head high in the global environment. Various polluting and toxic gases result in crucial pollution. Perovskite materials have attracted a great deal of attention because of their electronic, ferroelectric, catalytic, and magnetic properties which are useful for applications in ferroelectric random access memory, magnetic data storage, catalysts. Gas sensing and photocatalysts, photovoltaic cells, solid oxide fuel cells, and electromechanical transducers [6, 7]. Many methods were developed to prepare perovskites, such as solid-state reaction [8], sol–gel process [9], reverse microemulsion synthesis [10], co-precipitation [11], hydrothermal synthesis [12] etc. Lanthanum ferrite ($LaFeO_3$), a typical perovskite material, has been also prepared through the above methods. Semiconductor oxides such as SnO_2 and WO_3 have been reported for detection of NH_3. Some well-known materials for NH_3 gas sensing are ZnO [13], modified-ZnO (viz. Fe–ZnO and Ru–ZnO) [14-15], iridium oxide [16], molybdenum oxide, polyaniline, Au and MoO_3-modifiedWO_3 [17-18], Pt- and SiO_2-doped SnO_2 [19-20], etc. With the rising tide of nanotechnology, researchers find that the sensing properties of nanostructured metal oxide materials differ substantially from bulk materials of the same element and composition, and may lead to sensors with high sensitivity, fast response/recovery time, low energy consumption and small in size making it ideal for portable devices.

The adsorption of the gas species on the surface of the metal oxide results in the change in surface coverage, which is responsible for the change in the surface electrical resistance. Thus the surface morphology has an essential role on the sensitivity of solid state sensors. Films are usually compact and nonporous, causing the limited interaction of the external surface of the sensitive layer with gas. The advantage of the pellet formation is that it increases surface area; the increase in response is attributed to the smaller crystalline size. It results in greater oxygen adsorption on the pellet surface because of the larger specific surface area. These adsorbed oxygen species react most effectively with ammonia at room temperature. The pellet shows fast response and recovery to ammonia at room temperature.

Herein, we present a novel and facile synthesis of porous $LaAlO_3$ nanocrystals based on the self-combustion, were prepared from Al $(NO_3)_3 \cdot 9H_2O$, $La(NO_3)_3 \cdot 6H_2O$, and glycine source of the cation, but also the fuels. Homogeneity is achieved primarily because it is mixed on an ionic scale, which ensures the formation of nanopowders. To our knowledge, this is the first report of formation of perovskites through the self-combustion, which is a high yield, solvent less route and helps to develop the new field of $LaAlO_3$ in synthesis of inorganic materials. The gas sensitivity studies have been performed on the $LaAlO_3$ nanocrystalliine powder possible application on gas sensors.

2. Materials and Methods

2.1. Materials

All the reagents are of analytical grade and are used as received without further purification. Zinc nitrate [$La(NO_3)_3.6H_2O$], chromium nitrate [$Al(NO_3)_3.9H_2O$], glycine are purchased from Sigma-Aldrich chemical reagents Co. (USA).

2.2. Methods

Polycrystalline $LaAlO_3$ was synthesized by the combustion synthesis method [21-23] using glycine as fuel (organic fuel). All chemical reagents were analytical grade and used without further purification. Stoichiometric quantity of solid mixture of one mole reagents i.e. La $(NO_3)_3$ nH_2O (purity 98.5 %), Al

$(NO_3)_3$ $9H_2O$ (purity 98.5 %) and two mole of glycine (purity 99.5 %) were mixed together in a flat Pyrex disc. The solid were stirred for five minutes (clear solution was obtained). The solution formed was evaporated on hot plate in temperature range 80-90 ^{0}C which give thick gel. The gel was kept on a hot plate for auto combustion and heated in the temperature range of 160-180 ^{0}C.The nanocrystalline $LaAlO_3$ powder was formed within five minutes. This powder was grind in the mortal pestle and used for the characterization and white colour of synthesized $LaAlO_3$ powder.

2.3. Pellet Formation

The crystalline powder after phase formation at 850 ^{0}C was crushed in to fine powder using a pestle and mortar. A quantity of 2 gm powder was taken with 0.05 ml (25mg) of an organic binder, polyvinyl alcohol (PVA) and it was again ground to mix PVA uniformly in the powder. This powder was used to make a dense pellet with a die of diameter 6 mm. A hydraulic press machine was used to apply pressure of about 10 tones on the die. The pellets prepared were then fired at 500 ^{0}C for 2 h to remove the organic binder.

2.4. 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 was constant 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/OFF time intervals. A Cr-Al thermocouple was used to sense the operating temperature of the sensors. The output of the thermocouple was connected to digital temperature indicators. A 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. A 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.

3. Result and Discussion

3.1. Structure and Formation Analysis

TG and DTA measurements were performed to study the thermal behavior of sol Fig. 1. At the beginning synthesized powder was stable up to the temperature 450 ^{0}C. This indicates that no loss of any material but beyond that temperature there is slight weight gained and this is due to the adsorption of nitrogen gas supplied by the instrument. The slight weight gained was occur up to the temperature 750 ^{0}C.After this temperature the weight loss take place due to the desorption of nitrogen gas. At 850 ^{0}C temperature the powder is stable. This weight loss and weight gained is very negligible. This weight change is in the range of 0.002 % only. This indicates that the synthesized powder was almost stable from the beginning.

3.2. X-Ray Diffraction Studies

X-ray diffraction XRD measurements were performed on Philips Analytic X-ray B.V. (PW-3710 Based Model) Advanced X-ray diffraction using Cu Kα 1.54056 radiation. The XRD pattern shows that the product is pure perovskite oxide $LaAlO_3$ with an orthorhombic structure. The diffraction data is good agreement with JCPD card of $LaAlO_3$ (JCPDS No.33-0699). The average crystalline size of $LaAlO_3$ perovskite powder is determine with the help of Scherrer's equation $t=0.9\lambda/\beta\cos\theta$, where t is the average size of the particles, λ is wavelength of X-ray radiation, β the full width at half maximum of the diffracted peak and θ is the angle of diffraction. The average crystalline size obtained for nanocrystalline $LaAlO_3$ calcinated at 850 ^{0}C is found to be 45 nm. $LaAlO_3$ nanocrystals are more attractive in the field of catalytic application.

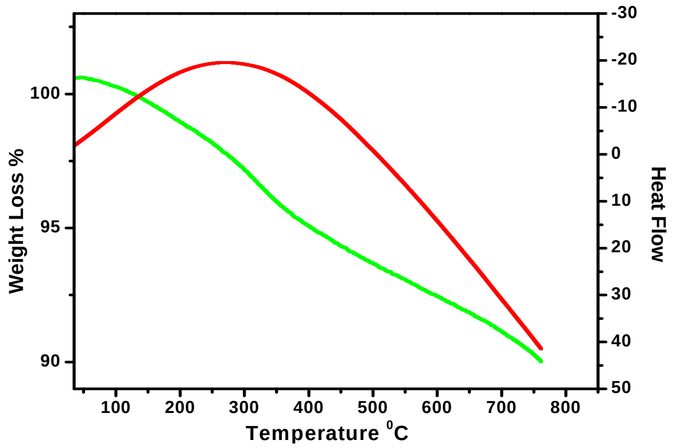


Fig. 1. TG-DTA curve of mixed precursor $LaAlO_3$.

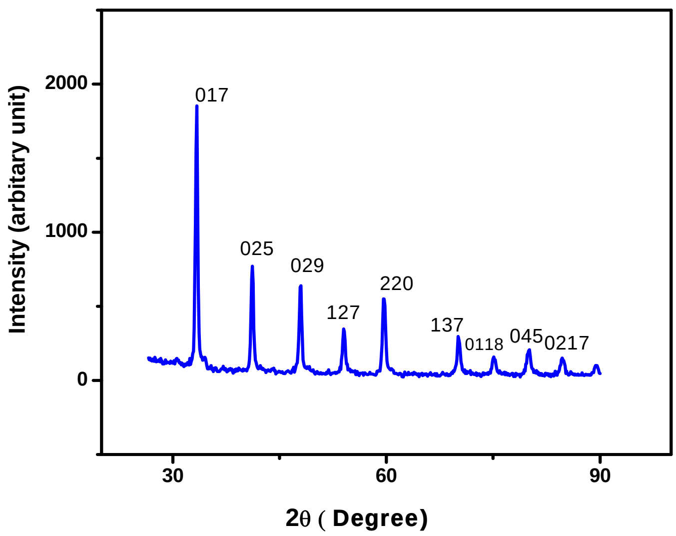


Fig. 2. XRD pattern of calcinied mixed precursor $LaAlO_3$ at 850 ^{0}C in air for 4 h.

3.3. EDX Analysis

The elemental analysis of the sample was carried out by using energy dispersive X-ray spectrometer (EDS) and is shown in Fig. 3.The EDS result clearly shows that $LaAlO_3$ contains La, Al, and O without any impurity. Quantitative EDX analysis verified that doping with lanthanum is close to the expected concentration.

3.4. SEM Analysis

The surface morphology is studied by using scanning electron microscopy (SEM) Model No. JEOL, JED-2300 analysis station. The SEM-EDX image of $LaAlO_3$ is shown in Fig. 4(a, b). The SEM picture of pure $LaAlO_3$ shows that the size of $LaAlO_3$ particles are not uniform, agglomeration (particle-particle interactions) is observed. SEM images reveal the low density product, loose and porous material that is favourable to a gas sensing application.

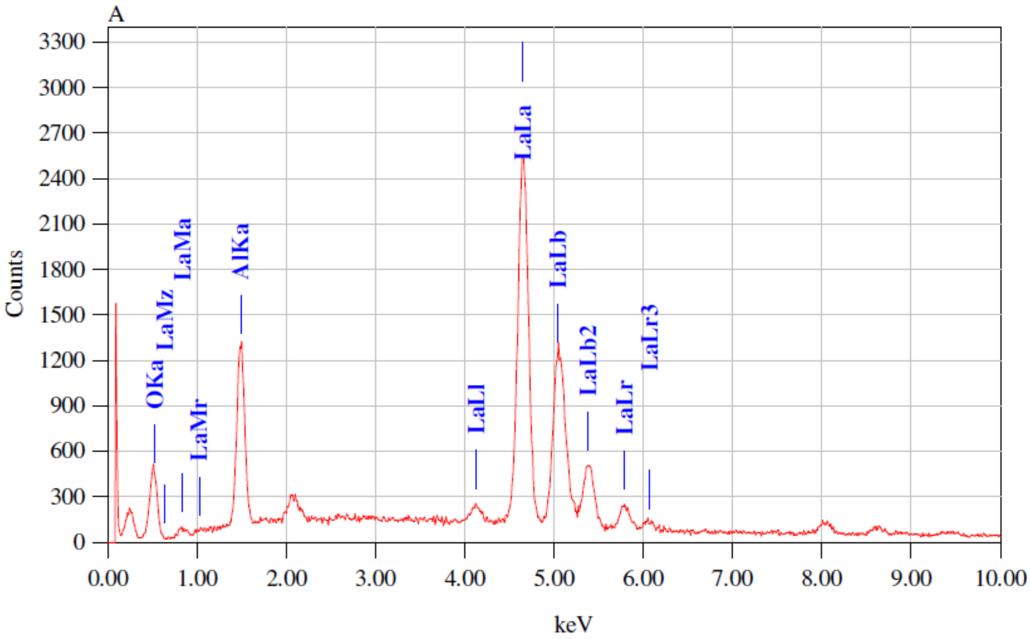
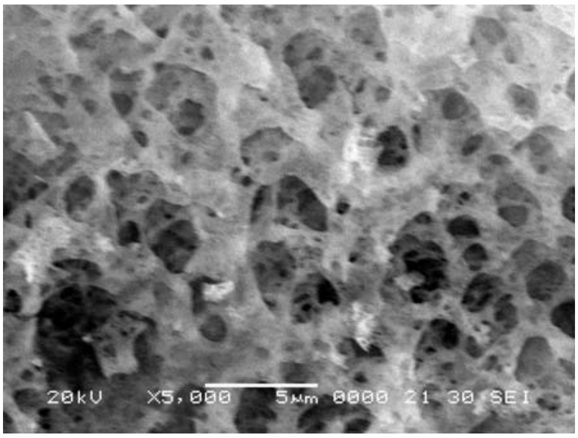
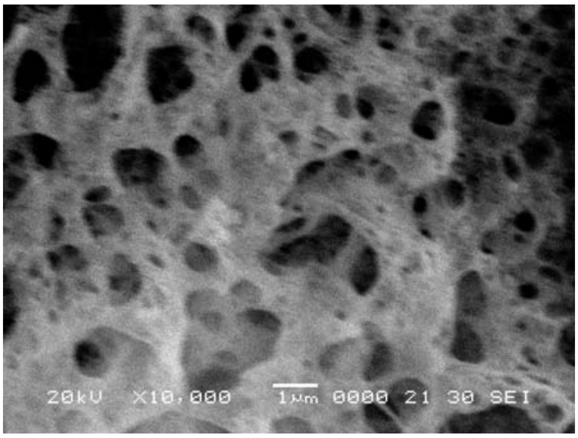


Fig. 3. EDX pattern of mixed precursor $LaAlO_3$at 850 ^{0}C in air for 4 h.



(a)

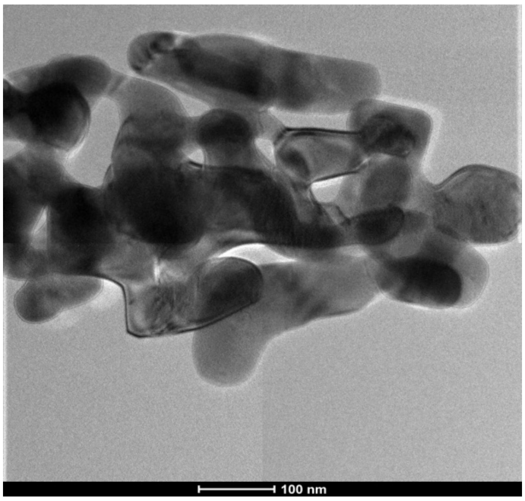


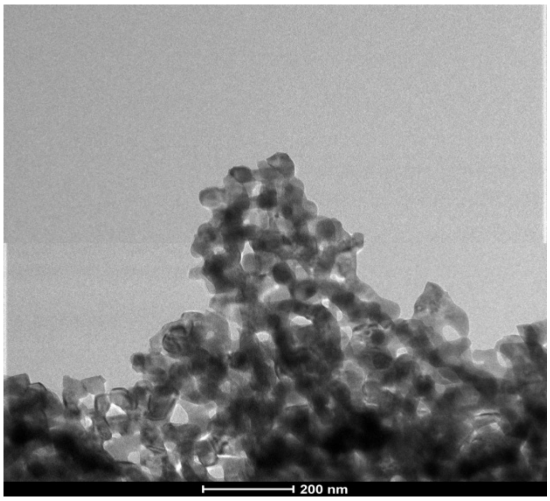
(b)

Fig. 4. SEM images of $LaAlO_3$ powder.

3.5. TEM Analysis

The TEM specimens were prepared by placing microdrops of colloid solutions on a carbon film supported by a copper grid. The TEM images of the nanocrystalline $LaAlO_3$ calcinated at 850 ^{0}C in air for 5 h are shown in Fig. 5 (a, b). It indicates the presence of $LaAlO_3$ nanoparticles with 40-70 nm size which form spherical type of oriental aggregation, agglomeration and polymeric linkage throughout the region. The pores are well developed spherical shape with diameter from 40-70 nm size. The results are correlated with the XRD.





(a) (b)

Fig. 5. Transmission electron microscopy image of $LaAlO_3$ calcinated at 850 ^{0}C.

4. Electrical Properties

4.1. I–V Characteristics

Fig. 6 depicts the I–V characteristics of the $LaAlO_3$ pellet at room temperature and at 300 ^{0}C 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.

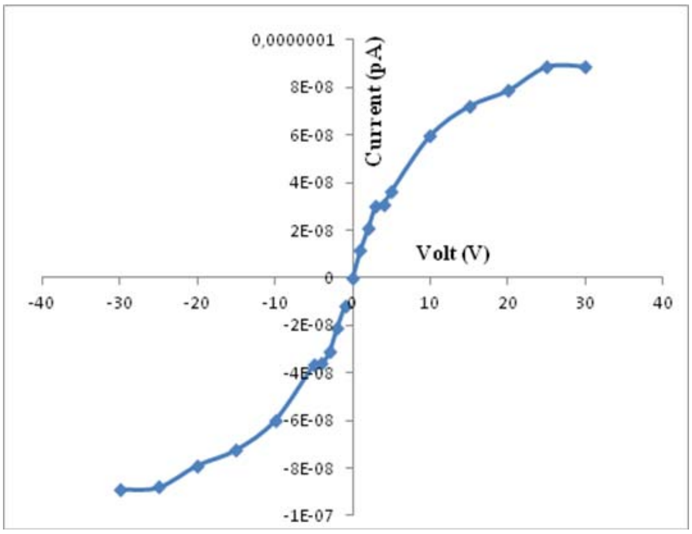


Fig. 6. I-V characteristics of the sensor.

4.2. Electrical Resistivity

The semiconducting nature of $LaAlO_3$ is observed from the measurements of resistivity with temperature. The semiconductivity in $LaAlO_3$ 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 $LaAlO_3$. It is observed that the electrical conductivity of the $LaAlO_3$ is nearly linear in the temperature range from 300–650°K in air ambient.

5. Sensing Performance of the Sensor

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

Gas response (S) is defined as the ratio of the change in conductance of the sensor on exposure at the target gas to the original conductance in air. The relation for S is as:

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

where G_a and G_g are the conductance of sensor in air and in target gas medium, respectively.

Selectivity or specificity is defined as the ability of a sensor to respond to certain gas in the presence of other gases. The time taken for the sensors to attain 90 % of the original conductance act as the recovery time.

5.2. Sensing Performance of Pure $LaAlO_3$ Pallet

5.2.1. Effect of Operating Temperatures

Fig. 7 depict the variation of response to NH_3 (50 ppm) with operating temperature of $LaAlO_3$ pellet. The largest response of $LaAlO_3$ was observed to be at 80 % at room temperature. The ammonia response at room temperature is expected to be monitored by adsorption of moisture on the $LaAlO_3$ pellet. The cumulative effect would decrease the pellet resistance, giving a response to ammonia gas at room temperature. At room temperature, there would be no oxygen adsorption. Therefore, the oxygen adsorption-desorption mechanism is not employed to sense the NH_3 gas.

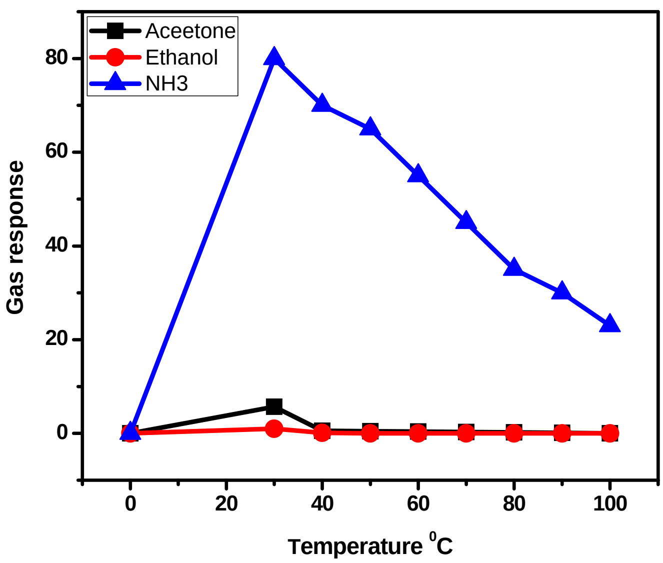


Fig. 7. Variation of different gas responses with operating temperatures.

5.2.2. Effect of NH_3 gas Concentration at Room Temperature (Active Region)

The variation of gas response of the $LaAlO_3$ sample with NH_3 gas concentration at room temperature is represented in Fig. 8, this pellet was exposed to varying concentrations of NH_3 gas for $LaAlO_3$ samples. The response values were observed to increase continuously with increasing the gas concentration up to 50 ppm at room temperature. The rate of increase in responses was relatively larger up to 50 ppm, but smaller during 50 and 50 ppm. Thus, the active region of the sensors would be up to 50 ppm. At lower gas concentration, the unimolecular layer of gas molecules would be formed on the surface of the sensor which could interact more actively giving larger response. The multilayer of the gas molecules on the sensor surface, at the higher gas concentration, would result into saturation in response beyond 50 ppm gas.

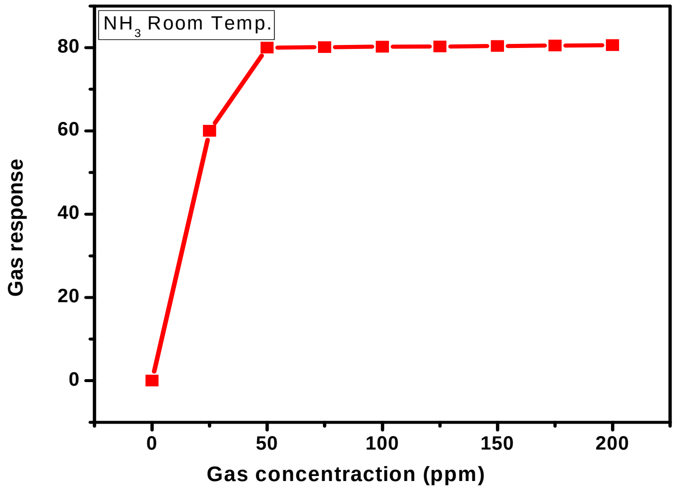


Fig. 8. Variation of gas response with gas concentration.

5.2.3 Selectivity for NH_3 Against Various Gases

Fig. 9 depicts the selectivity of the $LaAlO_3$ sensor for NH_3 (50 ppm) gas at room temperature. The sensor showed high selectivity to NH_3 against other gases (400 ppm) ethanol, acetone.

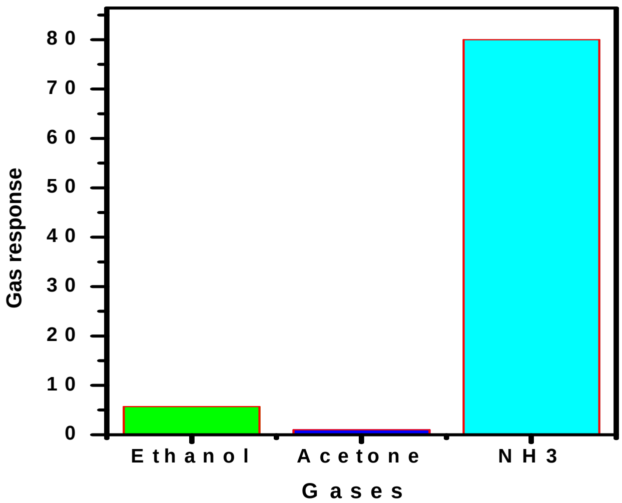


Fig. 9. Selectivity factor of sensor for various gases.

5.2.4. Response and Recovery of the Sensor

Fig. 10 depicts the response and recovery of the $LaAlO_3$ sensor. The response was quick (~ 10s) to 50 ppm of NH_3, while the recovery was considerably fast (~ 60 s). A negligible quantity of the surface reaction product and its volatility explain its quick response to ammonia and fast recovery in its initial chemical status.

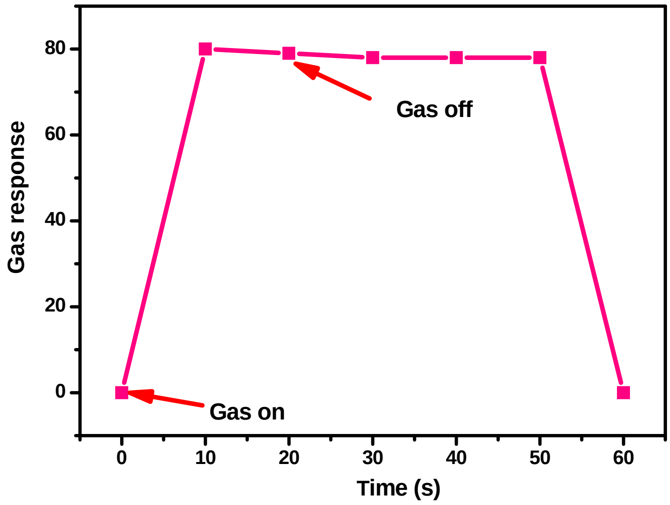


Fig. 10. Response and recovery time of sensors.

6. Discussion

Gas sensing mechanism is generally explained in terms of conductance change either by adsorption of atmospheric oxygen on the surface at higher temperature 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 surface reaction processes can explain the selective ammonia response of the sensor at room temperature.

$$NH_{3(g)} + H_2O_{(Surface)} \xrightarrow{RT} NH_4OH_{(g)}$$

The $LaAlO_3$ pellet causes the formation of intergrain boundaries of $LaAlO_3$- $LaAlO_3$ grains. The exposed NH_3 gas molecule captures the lattice oxygen from the surface of the pellet at room temperature. This would result the oxygen deficiency in the bulk of the material preferably at the surface. The semiconductivity in $LaAlO_3$ may be due to large oxygen deficiency. The increase in the conductivity of $LaAlO_3$ pellet could be attributed to the charge-carrier generation mechanism resulted from the electronic defects due to nanostructured size of the grains. These generated electrons and the donor level in the energy band gap of $LaAlO_3$ will contribute to increase in conductivity. This results in increasing the conductance of the pellet at room temperature.

7. Conclusions

(I) A novel combustion method was developed for preparation of pervoskite –type oxygen permeable materials. The method offer the advantages of facilitating the crystallization of pervoskite phase structure and easy handling of product.

(II) The phase formation of the $LaAlO_3$ is investigated by TG-DTA and XRD techniques. The synthesized product shows single phase of inverse Perovskite structure with an average diameter 30-70 nm.
(III) These nanoparticles with good I-V characteristics show ideal semiconducting nature at room temperature.
(IV) Among all other additives tested, $LaAlO_3$ is outstanding in promoting the ammonia gas.
(V) $LaAlO_3$ to be optimum and showed highest response to ammonia at room temperature.
(VI) The sensor showed very rapid response (~10 s) and recovery (~ 60 s) to ammonia gas. The sensor has good selectivity to ammonia against ethanol, acetone.

Acknowledgement

The authors acknowledge Principal Dr. Patangrao Kadam Mahavidyalaya, Sangli for providing the laboratory generous help on sensor measurement. The authors also like to express their thanks to Dr. S.S. Kadam Vice Chancellor Bharti Vidyapeeth Deemed University Pune & Prof. K. D. Jadhav, Principal Y. M. Collоge for constant encouragement and helpful discussion.

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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.

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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