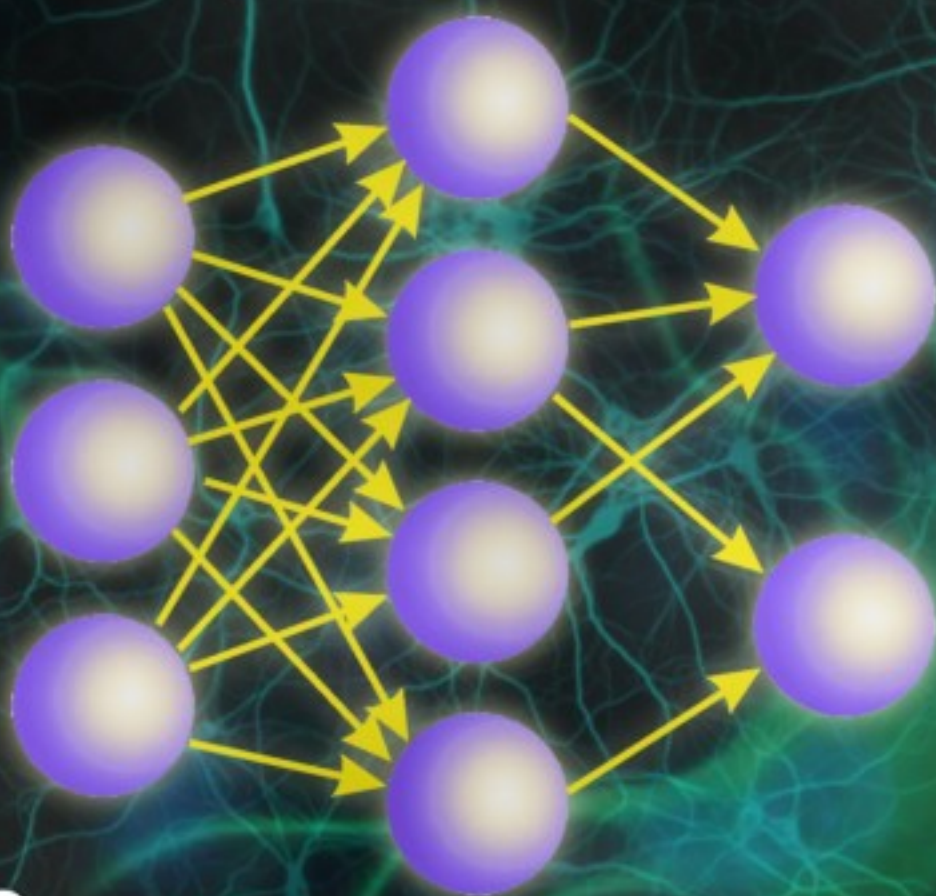


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Contents

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Research Articles

Aspects of Gas Sensor's Modeling and Implementation in a Dynamic Environment <i>Hakim Baha, Zohir Dibi</i>	1
A Fuzzy-Based Tactile Sensor for Implementation in Specialized Motion of Land Rover <i>Badal Chakraborty, K. Rajanna</i>	13
Neuro and Fuzzy Computing Approach for the Flow Sensorless Measurement <i>R. Kumar, and P. Sivashanmugam</i>	21
Fuzzy Logic Based Set-Point Weighting Controller Tuning for an Internal Model Control Based Pid Controller <i>Maruthai Suresh, Ranganathan Rani Hemamalini, Gunna Jeersamy Srinivasan</i>	29
Adaptive PI Controller for a Nonlinear System <i>D. Rathikarani, D. Sivakumar</i>	43
Integration of Fault Detection and Isolation with Control Using Neuro-fuzzy Scheme <i>A. Asokan, D. Sivakumar</i>	59
Control Scheme of a Designed Step Climbing Wheeled Robot <i>Srijan Bhattacharya, Sagarika Pal, Subrata Chattopadhyay</i>	70
Accurate Fluid Level Measurement in Dynamic Environment Using Ultrasonic Sensor and v-SVM <i>Jenny Terzic, Romesh Nagarajah, Muhammad Alamgir</i>	76
Design of a MEMS Capacitive Comb-drive Micro-accelerometer with Sag Optimization <i>B. D. Pant, Lokesh Dhakar, P. J. George and S. Ahmad</i>	92
Remarkable Electromechanical Coupling in the 2–2 Composite Based on Single-domain PMN–0.33PT Crystal <i>Vitaly Yu. Topolov, Sergei V. Glushanin, Christopher R. Bowen and Anatoly E. Panich</i>	108
Effect on H₂S Gas Sensing Performance of Nb₂O₅ Addition to TiO₂ Thick Films <i>C. G. Dighavkar, A. V. Patil, S. J. Patil and R. Y. Borse</i>	117
Al₂O₃- BSST Based Chemical Sensors for Ammonia Gas Sensing <i>L. A. Patil, H. M. Baviskar</i>	126
Wireless Telemetry 2.4 GHz for Measurement Occupational Vibration <i>Diogo Koenig and Alexandre Balbinot</i>	137
pH Homeostasis Linked with Capacitance Relaxation Phenomena and Electrostrictive Energy in Cancer Cells <i>T. K. Basak, T. Ramanujam, J. C. Kavitha, Poonam Goyal, Deepali Garg, Arpita Gupta, Suman Halder</i>	147

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Al₂O₃- BSST Based Chemical Sensors for Ammonia Gas Sensing

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Abstract: Gas sensing behaviour of pure and modified (Ba_{0.9}Sr_{0.1})(Sn_{0.5}Ti_{0.5})O₃ (BSST) thick films is reported in this article. The surface of the BSST thick film was modified by dipping it into aqueous solution of AlCl₃, for different intervals of time. These films were then dried at 500 °C for 24 hours in air ambient for transformation of AlCl₃ into Al₂O₃, for the evaporation of organic binders and also to improve the texture of the film. The gas response, selectivity, response and recovery time of the sensors were measured and presented. The role played by the aluminium species to improve the gas sensing performance of the sensors is discussed. *Copyright © 2009 IFSA.*

Keywords: BSST, Thick film, Surface modification, Al₂O₃ modified films, Ammonia sensor

1. Introduction

The gas sensors using metal oxide semiconductors were first reported by Seiyama et al and Taguchi [1, 2]. Nowadays, increased awareness over the need to monitor the toxic gases for environmental pollution and efficiency in a combustion process has stimulated substantial research and development in the field of gas sensors. Such sensors are n-type semiconductors where loss of oxygen on heating results in the generation of oxygen vacancies, leading to electron donor states.

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 [3-9] in nature. The exposure of ammonia causes chronic lung disease, irritating and even burning the respiratory track, etc. Environmental pollution [10-13] 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 warning for dangerous ammonia concentrations. It is therefore, necessary to monitor ammonia gas and to develop the ammonia gas sensor. Efforts are made to develop the BSST based NH₃ gas sensors.

2. Experimental

2.1. Powder and Paste Preparation

The (Ba_{0.9}Sr_{0.1})(Sn_{0.5}Ti_{0.5})O₃ powder was synthesized by mechanochemical process. The AR grade powders of Ba(OH)₂.8H₂O and Sr(OH)₂ in the ratio 9:1 and SnO₂ and TiO₂ in the ratio 1:1 are mechanochemically mixed. The mixture was ball milled to mix thoroughly, followed by sintering at 800 °C for 12 h. The resulted white powder was termed as Barium Strontium Tin Titanate (BSST). The BSST thick films were printed on a glass substrates using screen printing technique [14-21]. The films were fired at 550 °C for 30 min.

2.2. Surface Modification of Thick Films

Thick films prepared were surface modified by dipping them into a 0.01 M aqueous solution of AlCl₃ for different intervals of time and were dried at 90 °C under an IR lamp, followed by firing at 500 °C for 24 h in air ambient. The AlCl₃ dispersed on the films would be oxidized in firing process, and sensor elements with different mass % of Al₂O₃ were obtained. Silver contacts were made by vacuum evaporation for electrical measurements.

2.3. Thickness Measurement

Thickness measurements were carried out using a Taylor-Hobson (Talystep, UK) system. The thicknesses of the films were observed to be in the range from 40 to 45 μm. The reproducibility of the film thickness was achieved by maintaining the proper rheology and thixotropy of the paste.

2.4. Details of Gas Sensing

Fig. 1 represents the schematic diagram of the system to test the sensing performance of the sensors. There are electrical feeds through the base plate. Heater is fixed on the base plate. A sample under test can be mounted on the heater. The Cr-Al thermocouple is mounted to measure the operating temperature. The output of the thermocouple is connected to a temperature indicator. A gas inlet valve is fitted at one of the ports of the base plate. The gas concentration inside the static system is achieved by injecting a known volume of a test gas with a gas – injecting syringe. Constant voltage is applied to the sensor and the current is measured by a current meter.

3. Results

3.1. X-Ray Diffraction Studies

The structure of the films was analyzed with X-ray diffractogram (RIGAKU DMAX 2500) using CuK_α radiation with a wavelength 1.5418 Å. Figs. 2 (a-c) show the X-ray diffractograms of the pure and Al₂O₃-modified BSST films (30 and 60 min). The observed peaks are matching well with ASTM

reported data of BSST. The sharp peaks of the XRD pattern correspond to BSST material and are observed to be microcrystalline in nature. No peaks corresponding to Al_2O_3 are observed. It may be due to its very small mass % dispersed on the surface of BSST. The average grain size was determined using Scherer's formula and was estimated to be of 272 nm.

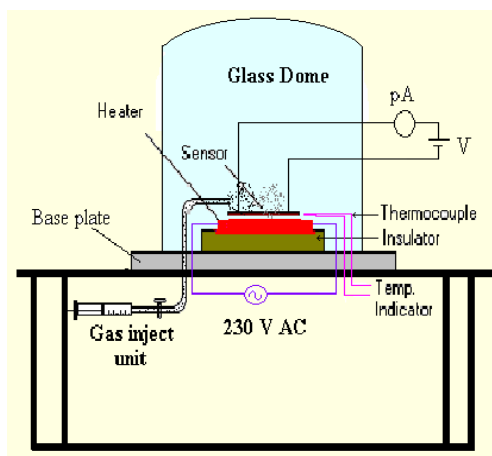


Fig. 1. Schematic diagram of the system to test the sensing performance of the sensors.

3.2. Microstructure-SEM

The microstructure and chemical composition of the films were analyzed using a scanning electron microscope (JOEL JED 2300) coupled with an energy dispersive spectrometer (6360 LA). Fig. 3 depicts the SEM images of pure BSST and Al_2O_3 -modified BSST thick films. Unmodified BSST film (Fig. 3a) consists of randomly distributed grains with broader size and shape distribution. Fig. 3 (b) depicts the microstructure of a most sensitive Al_2O_3 -modified BSST film. It consists of grains of uniform size with narrow size distribution. This film is more porous, giving largest effective surface area. This enables larger surface for the gas to react giving higher response. Fig. 3 (c) consists of non uniform grain size and relatively broader particle size distribution of grains as compared to the grains associated with Fig. 3 (b). The porosity of this film is also smaller. Due to larger dipping time interval, larger amount of Al_2O_3 would be expected to percolate up to deeper portion of films. The effective surface area to react the gas would therefore be smaller. It may therefore give smaller gas response.

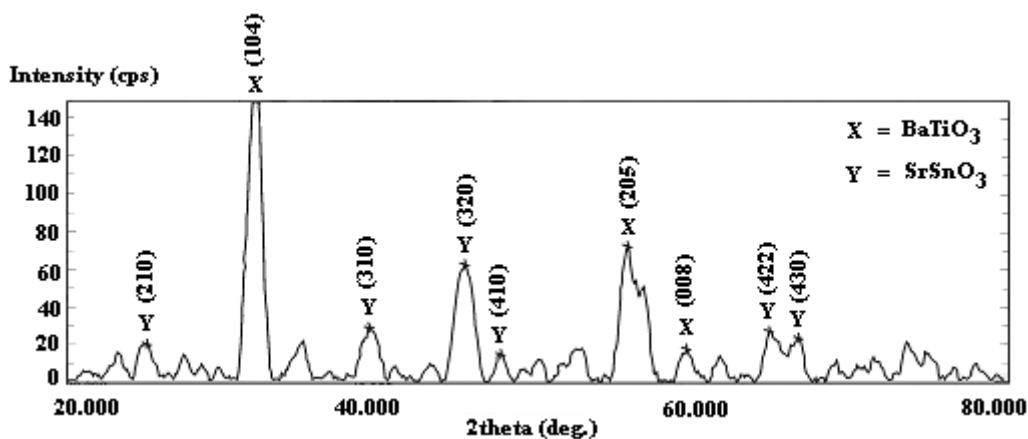
4. Gas Sensing Performance

4.1 Definition of Sensing Characteristics

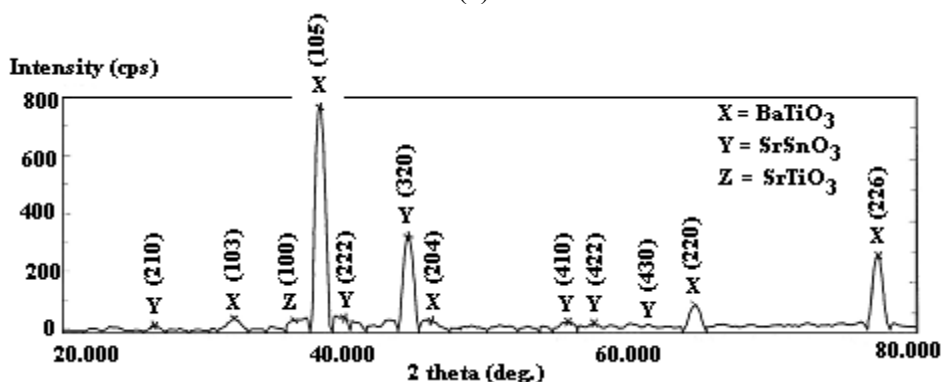
Gas response of a sensor is defined as the ratio of the conductance change upon exposure to a test gas to the conductance in air.

$$\text{Gas response} = (G_g - G_a) / G_a,$$

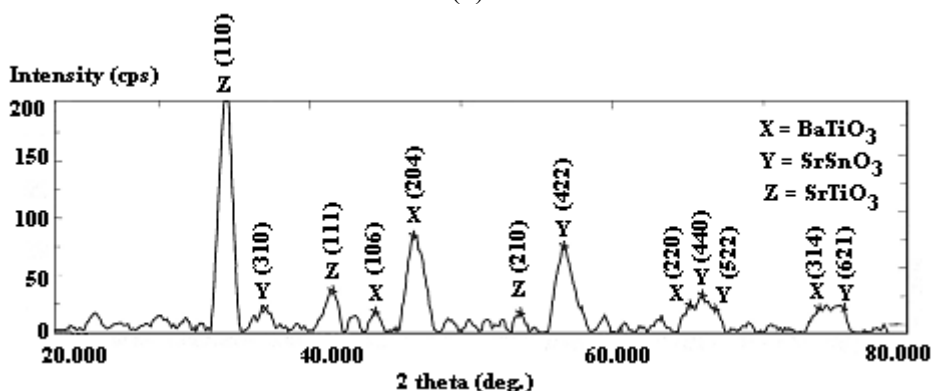
where G_g and G_a are the conductance of a sample in the presence and absence of a test gas respectively.



(a)



(b)



(c)

Fig. 2. XRD of (a) Pure, (b) Al_2O_3 -modified (30 min) and (c) Al_2O_3 -modified (60 min) BSST.

4.2. Unmodified BSST: NH_3 Sensor

4.2.1. Gas Response and Operating Temperature

Fig. 4 shows the variation of gas responses of unmodified BSST thick films to 1000 ppm of various gases such as LPG, NH_3 , CO_2 , $\text{C}_2\text{H}_5\text{OH}$, H_2 , Cl_2 and H_2S with operating temperatures. Pure BSST shows highest sensitivity to NH_3 as compared to other gases at 300°C . The response of each gas goes on increasing with operating temperature, reaches to maximum at 300°C and then decreases with further increase in temperature.

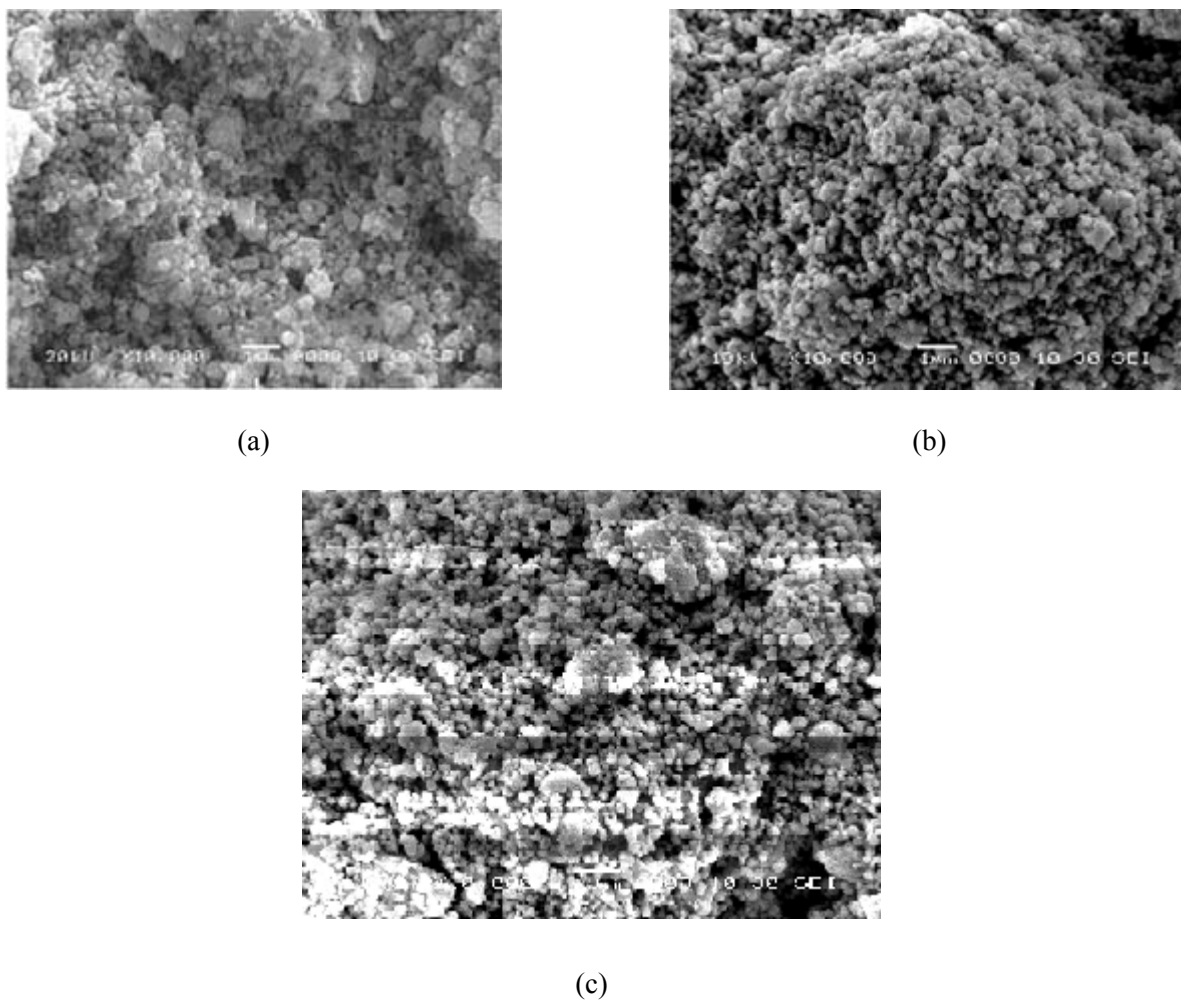


Fig. 3. Micrographs of (a) Pure, (b) Al₂O₃-BSST (30 min), (c) Al₂O₃-BSST (60 min) thick films.

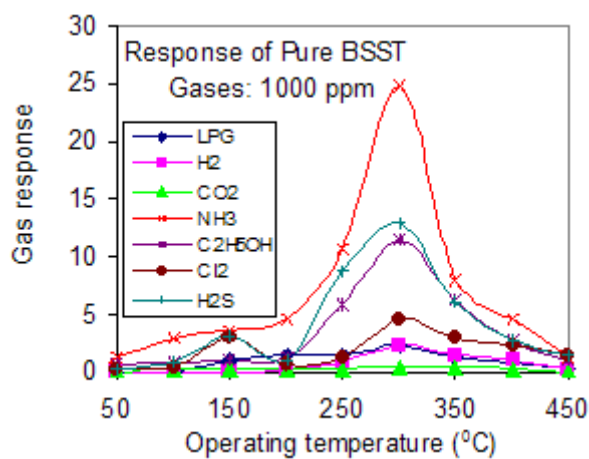


Fig. 4. Gas responses-operating temperature.

4.2.2. Selectivity

It is observed from the Fig. 5 that the pure BSST is most sensitive to NH₃ gas (1000 ppm) at 300 °C. It is clear from histogram that the sensor is selective to NH₃ in presence of other gas.

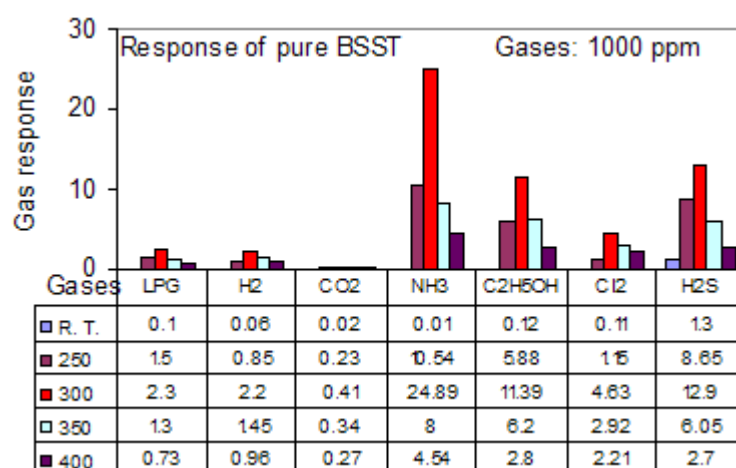


Fig. 5. Selectivity of pure BSST thick film.

4.3. Al₂O₃-modified BSST: NH₃ Sensor

4.3.1. Active Region of the Sensor

The variation of gas response of Al₂O₃-modified (30 min) BSST sample with NH₃ gas concentration is represented in Fig. 6. It is clear from figure that the gas responses go on increasing linearly with gas concentration up to 300 ppm and then saturate. Thus, the active region of the sensor would be up to 300 ppm.

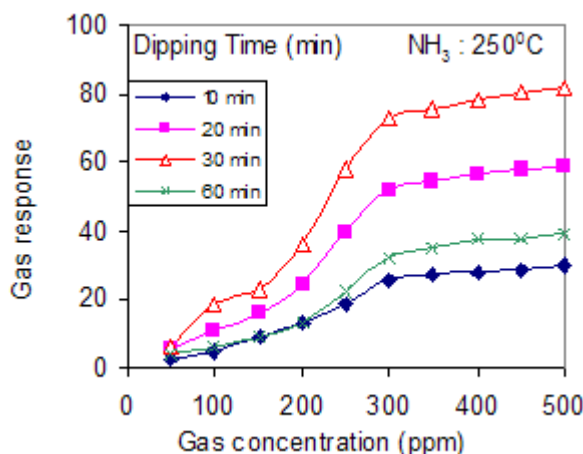


Fig. 6. Variation of gas response with NH₃ gas concentration.

4.3.2. Response and Operating Temperature

Figs. 7(a) through 7(g) depict the variation of modified (5, 10, 20, 30, 60) films to responses of LPG, NH₃, CO₂, C₂H₅OH, H₂, Cl₂ and H₂S with operating temperatures. Al₂O₃ modified film (for the interval of 30 min) is most sensitive to NH₃ (S=73) at 250 °C.

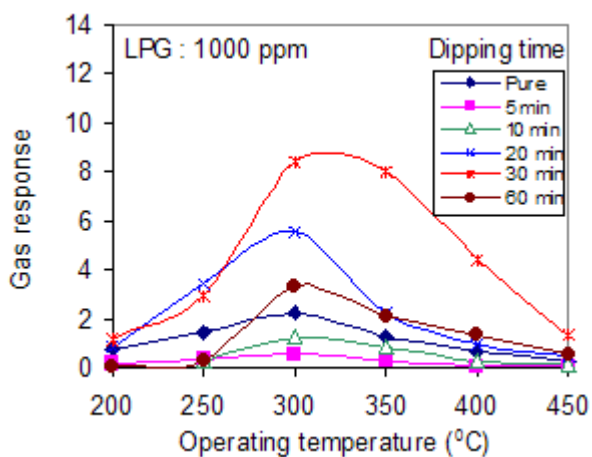


Fig. 7 (a). LPG response-operating temperature.

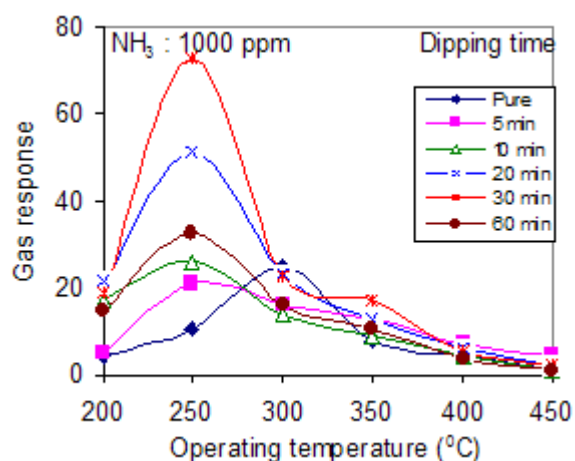


Fig. 7 (b). NH₃ response-operating temperature.

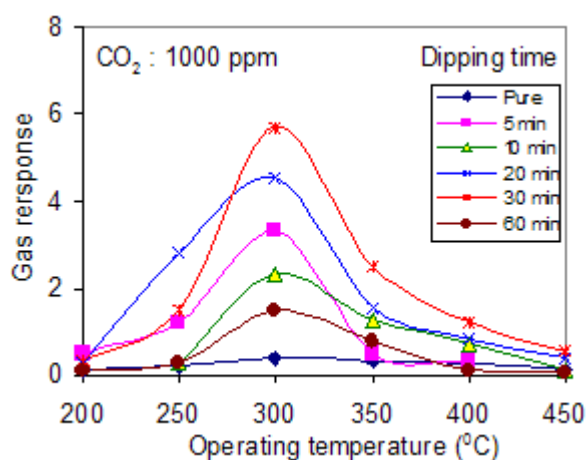


Fig. 7 (c). CO₂ response-operating temperature.

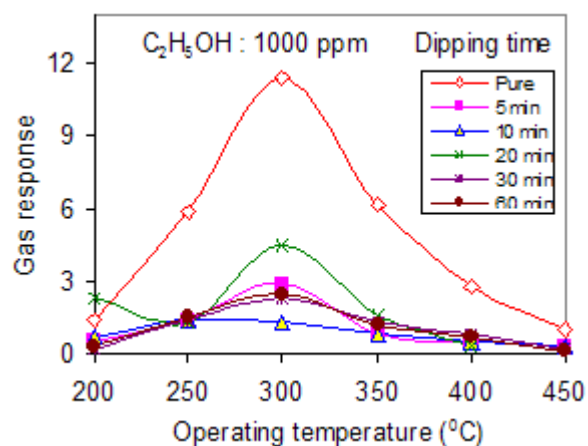


Fig. 7 (d). C₂H₅OH response-operating temperature.

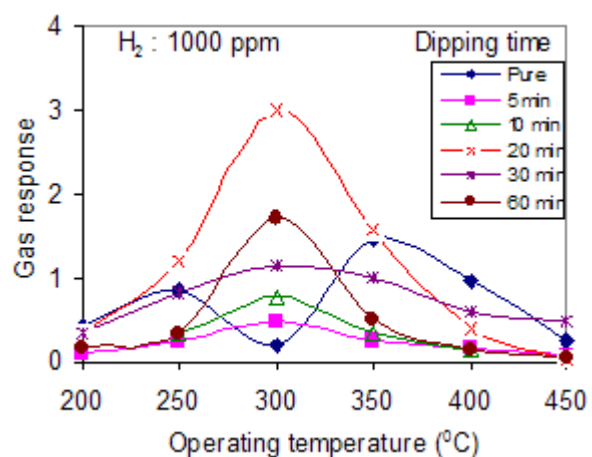


Fig. 7 (e). H₂ response-operating temperature.

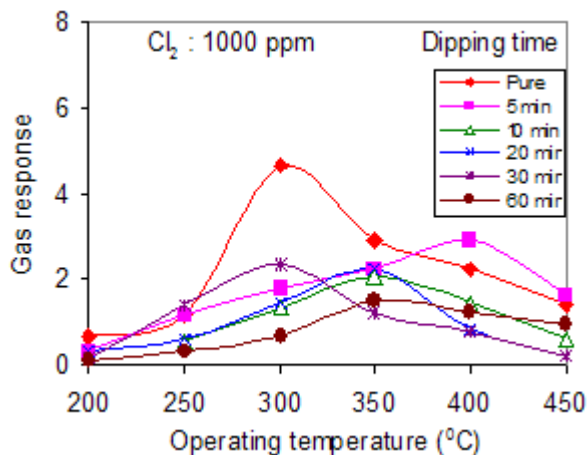


Fig. 7 (f). Cl₂ response-operating temperature.

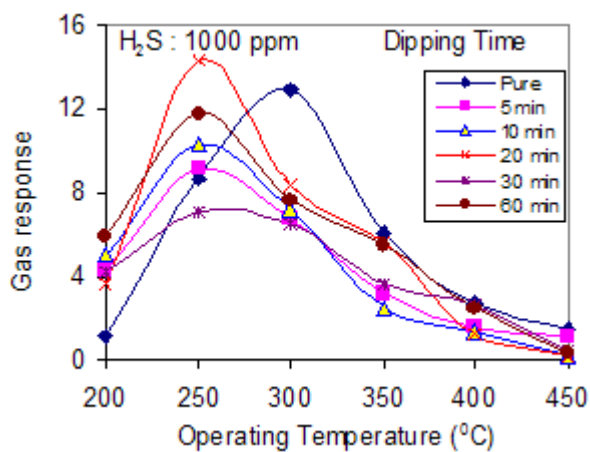


Fig. 7 (g). H₂S response-operating temperature.

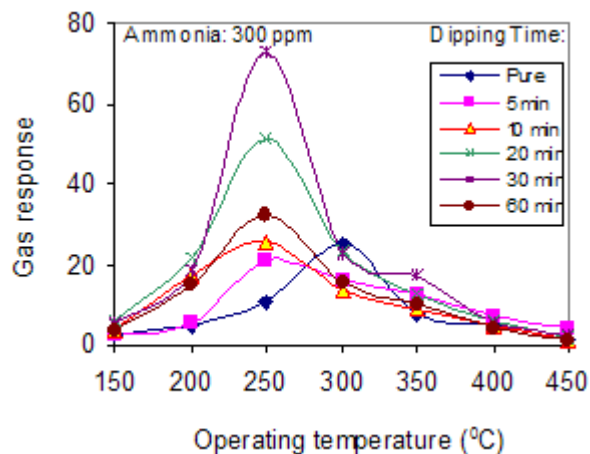


Fig. 7 (h). Gas response-operating temperature.

4.3.3 Effect of Dipping Time

Fig. 8 is the histogram indicating the NH₃ gas (300 ppm) response as a function of dipping time. The sensor with 30 min dipped in AlCl₃ was observed to be most sensitive at 300 °C.

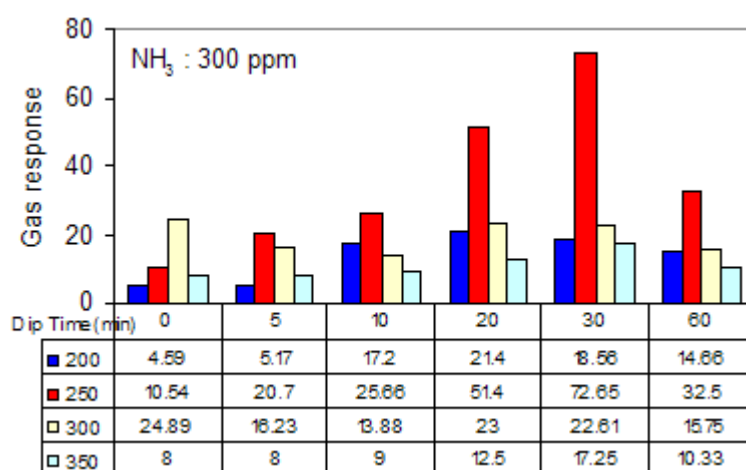


Fig. 8. Response values of different Al₂O₃-modified samples.

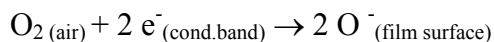
4.3.4. Selectivity for NH₃ Against Various Gases

Fig. 9 shows the histogram indicating the gas response of various samples tested at 250 °C. The sensor showed high selectivity for NH₃ and could distinguish the NH₃ among all the gases, such as LPG, CO₂, C₂H₅OH, NH₃, H₂S, H₂ and Cl₂.

5. Discussion

Gas sensing mechanism is generally explained in terms of change in conductance (Fig. 10) 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 the electrons 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, it captures the electrons from conduction band as:



It would result in decreasing conductivity of the film. When ammonia reacts with adsorbed oxygen on the surface of the film, it gets oxidized to nitrogen oxide gas and water vapors as the products liberating free electrons in the conduction band. The following possible reaction would take place.

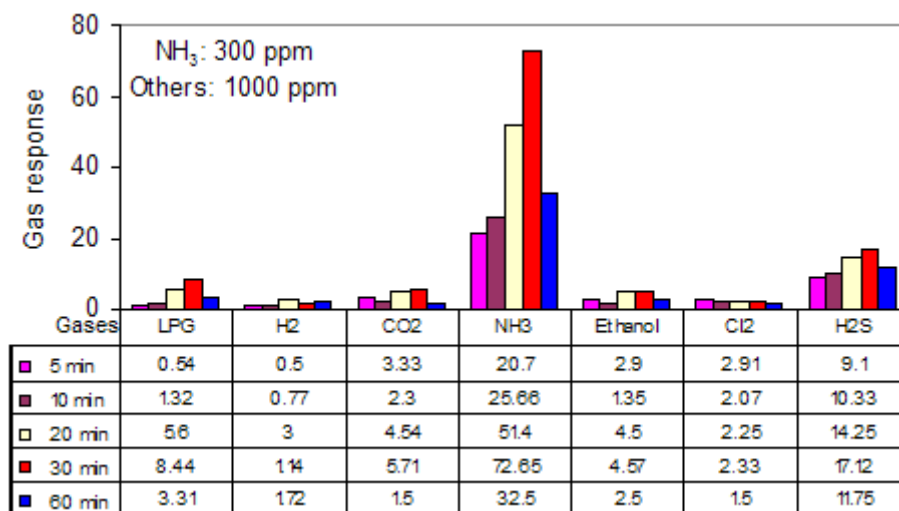
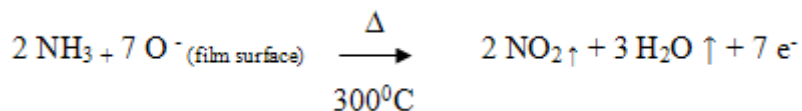


Fig. 9. Selectivity of NH₃S gas from mixture of gases.

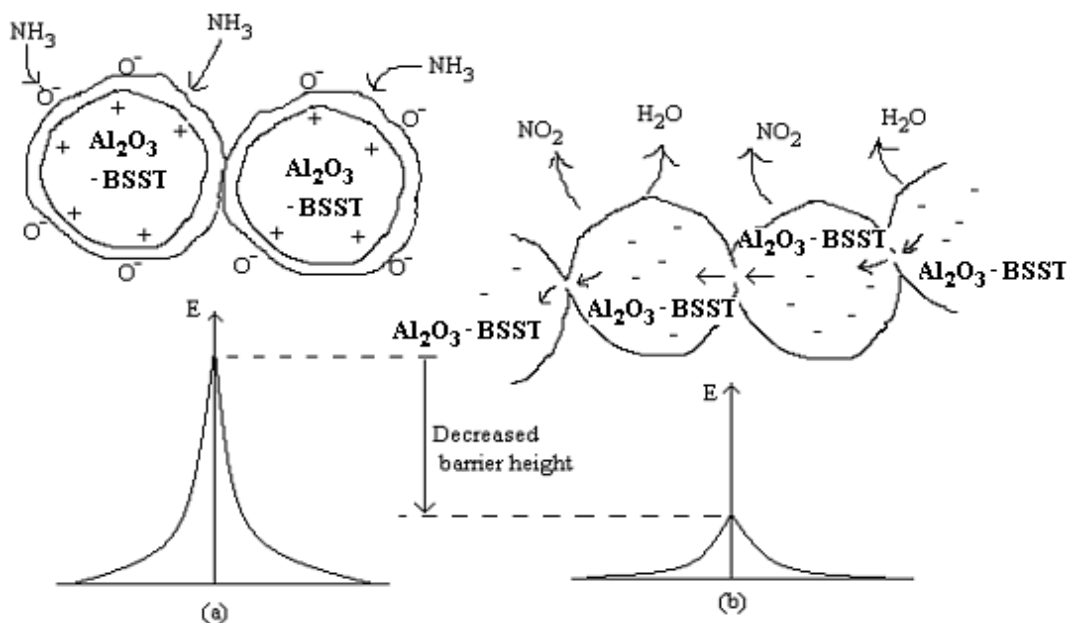
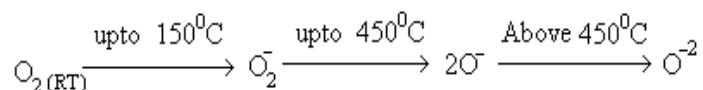


Fig. 10. NH₃ gas sensing mechanism of ironated BSST films at 350 °C.

This shows n-type conduction mechanism. Thus generated electrons contribute to sudden increase in conductance of the thick film. The Al₂O₃ misfit regions dispersed on the surface of the BSST film would enhance the ability of the material to adsorb more oxygen species giving high resistance in air ambient. On exposure to the NH₃ containing atmosphere, the resistance was observed to decrease in large extent. Therefore, the high response was obtained to 300 ppm NH₃ gas. As NO₂ and water vapors are released from the film surface, the sensor recovers back to its original chemical status, which would result in fast recovery.

The surface is the region where periodicity of the crystal is interrupted. Because of this, localized energy levels are formed in the forbidden gap. Such energy levels can either capture electrons or give up electrons. In case of semiconductor like pure or Al₂O₃-modified BSST, the surface oxygen ions give up electrons (act as donors). The donor levels are completely ionized if they are near the conduction band; however, if the donor levels are little below the conduction band, then these levels are not completely ionized at room temperature but ionized at higher temperature.

The response could be attributed to the adsorption-desorption type sensing mechanism. The largest response may be due to the higher amount of oxygen adsorption occurred at higher temperature. Oxidation of gas depends upon the amount of oxygen species (O₂⁻ → 2O⁻ → O²⁻) adsorbed on the surface. It is observed that the oxygen adsorption on the BSST surface occurs at different temperature ranges. The conversion is as follows:



During oxidation of the gas, the electrons are released soon and become free to carry the current. At lower temperature, the smaller amount of oxygen would be adsorbed causes weak oxidation of target gas, resulting in smaller gas response. At higher temperature (> 300 °C), the adsorbed oxygen species may start to desorb from the surface of the film, causing the less oxidation of ammonia gas, which in turn decreases the ammonia response of the sensor. Another reason of decreasing the ammonia response at higher temperatures (> 300 °C) may be attributed to the oxidation of ammonia before reaching the surface at higher temperatures (> 300 °C). Therefore, the gas response decreases further with increasing temperature.

6. Summary

From the results obtained, following statements can be made for the sensing performance of γ-Al₂O₃-modified BSST sensors.

1. Pure BSST thick films showed poor response to NH₃ gas.
2. Surface properties of the films were conveniently customized (without affecting bulk properties) by dipping BSST thick films in aluminium precursor.
3. γ-Al₂O₃-modified BSST (30 min) sensors showed better response to NH₃ gas at 250 °C than unmodified films.
4. The sensor was highly selective to NH₃ gas (300 ppm) from other toxic gases of higher concentrations (1000 ppm).
5. The sensor showed very rapid response (~ 4 s) and recovery (~ 12 s) to NH₃ gas.

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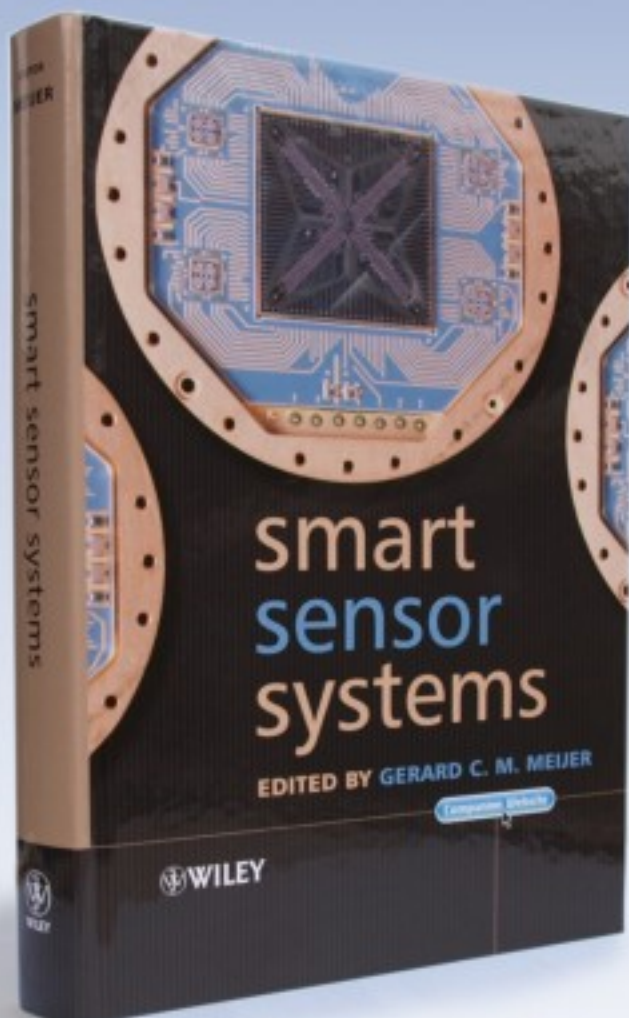
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