iJRASET
International Journal For Research in Applied Science and Engineering Technology

INTERNATIONAL JOURNAL FOR RESEARCH

IN APPLIED SCIENCE & ENGINEERING TECHNOLOGY

Volume: 9 **Issue:** I **Month of publication:** January 2021

DOI: https://doi.org/10.22214/ijraset.2021.32812

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International Journal for Research in Applied Science & Engineering Technology (IJRASET)
ISSN: 2321-9653; IC Value: 45.98; SJ Impact Factor: 7.429
Volume 9 Issue I Jan 2021- Available at www.ijraset.com

SnO_2 Substituted In_2O_3 Thick Films as PPM Level NH_3 Gas Sensors

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Abstract: ***The SnO_2 and In_2O_3 powder mixed with different ratio and heated at 900^0C about 5 hrs and then this powder is used to prepared thick films by a screen – printing technique on glass substrate. The NH_3 gas sensing properties, particularly the rate of response of SnO_2-In_2O_3 sensors are studied at room temperature. SEM and EDAX analysis showed that crystallite size is small (86.43 nm) for $65SnO_2$-$45In_2O_3$ composition. The Sensitivity increases drastically as the expose of NH_3 gas for $65SnO_2$-$45In_2O_3$ sample. This sample is found to be better sensing material as regards to other in all respect.***
Keywords: Ammonia Gas; SnO_2; In_2O_3; SEM; EDAX.

I. INTRODUCTION

Harmful gases are detected by gas sensors and protect from the harmful gases by fabrication of sensor. Due to modern lifestyle industrial society and older population many chronic diseases are affected . Human diseases are monitoring by the breath are studied in many of the peoples. For analysis breathe that can detect in stomach lung or in other body parts very highly sensitive sensors for particular gases are needed. Strong correlation between exhaled breath and specific diseases many people's studies have been presented . Ammonia gas NH_3 has also been recognized as one of the markers for hepatic or kidney diseases [1]. Ammonia gas (NH_3), a toxic and corrosive indoor air pollutant, is affect to human skin, eye and respiratory system [2, 3]. Various industries and other sources is emitted Ammonia gas (NH_3) into the atmosphere and causes environmental pollution. Considering the danger of ammonia, the application of sensors for detecting leakage from vessels and pipes. Therefore, the development of NH_3 gas sensors are importance to researchers and regulators. For detecting NH_3 gas the use of metal oxide sensors (MOS), such as Tin oxide (SnO_2) and Indium Oxide(In_2O_3), has been develop to detect NH_3 gas, even at low concentrations.

In this paper is focused on the fabrication of Ammonia gas NH_3 sensor based on SnO_2-In_2O_3 annealing route to enhance the NH_3 sensing performance at room temperature. To the best of our knowledge, SnO_2-In_2O_3 gas sensor has not yet been reported in the literature. The fabricated sensors were evaluated systematically in terms of their response, response/recovery times and Sensitivity toward NH_3. The main target of this work is to optimise samples of SnO_2:In_2O_3 and promote the gas sensing performance toward NH_3 at room temperature. The fabricated SnO_2:In_2O_3 gas sensor successfully showed a response several times higher than that of a pure mixed oxide gas sensor.

II. MATERIALS AND METHODS

A. Preparation of Materials and Fabrication Technique

The powders of $SnO_2$and In_2O_3 were calcinated at 800^0C in an automatically temperature-controlled muffle furnace for 6 to 7 hrs. The powders of same were crushed in pestle before after the calcination to get the homogeneity in the powders.

The mass percentage of same were mixed in the ratios of 100:00, 70:30, 65:45, 30:70 and 00:100 in acetone to form the homogeneous mixture of both. After mixing, the mixtures were kept for heating at 800^0C in a furnace for half an hour. In this way, the powders of pure material and composite of SnO_2:In_2O_3 were prepared for active sensing layer of Gas Sensors.

The paste of the material was prepared by using screen-printing technique. Butyl Carbitol Acetate were used for the screen-printing process [4-7], as a binders. The active powder and Ethyl cellulose were mixed thoroughly. During this mixing process, the Butyl Carbitol Acetate was added drop by drop to obtain the proper paste.

The glass substrate of size 7.5 x 2.5 cm^2 was used. The substrate is an important part of any thick-film process. For normal electronic purpose, the substrates structure should be rectangle. And before used it cleaned by using distilled water and acetone.

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The screen-printing of paste of active powder mixture was done in different steps as shown in Fig. 1.1 (A to I)

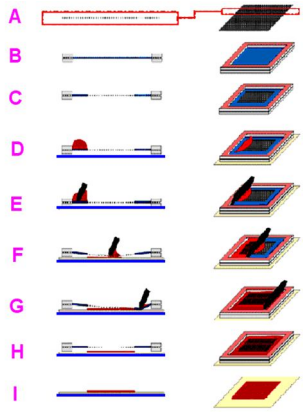

Fig. 1.1 Different steps involved in screen-printing

These samples were further heated at 150°C for 2h with a heating and cooling rate of 20°C/min to remove binder. These samples were used to analyze the gas sensing properties in air ambient and with Ammonia gas atmosphere.

B. Electrical Properties Measurement

The conductivity properties were studied between resistance and temperature by static gas characterization system shown in Fig. 1.2.

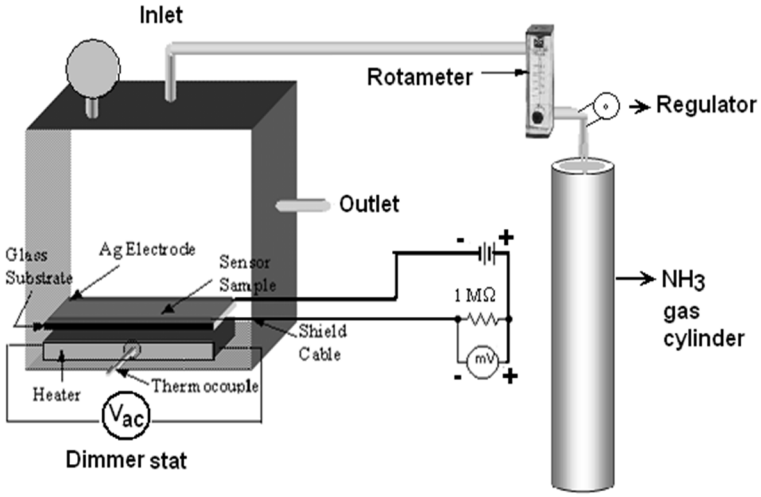

Fig. 1.2 Schematic diagram of Gas Sensing System

The sensing performance of the sample/material prepared under the optimized processing parameters were studied by using the various measurements i.e. sensitivity (S), stability, selectivity, response and recovery time of the sensor, lower detection limit (LDL), dynamic response & static response.

Resistance of the sample was measured in the air atmosphere as well as in the presence of testing gas at room temperature. In this method, resistance of R = 1MΩ was connected in series with the material and used DC supply (0-5V).(Fig 1.3)

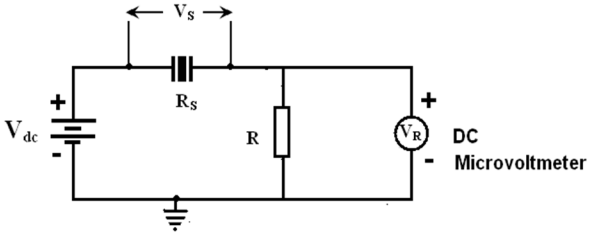

Fig. 1.3 Measurement of sample resistance

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Sample resistance was calculated

$$R_S = \left(\frac{V_{dc}}{V_R} - 1 \right) R_{Eff}$$

Where, R_{Eff} is the effective resistance of parallel combination of R and internal impedance of DC microvoltmeter.
The characteristics of the device to perceive the change in the properties of the sensing material when exposed to gas is referred as sensitivity and it is generally denoted by 'S' and is expressed as

$$S = \left(\frac{R_{air} - R_{gas}}{R_{air}} \right) = \left(\frac{\Delta R}{R_{air}} \right)$$

III. RESULTS AND DISCUSSIONS

A. SEM Analysis

The surface morphology of SnO_2, In_2O_3, $65SnO_2{:}45In_2O_3$ material was studied by SEM and its picture is shown in the Fig. 1.4 to1.6.

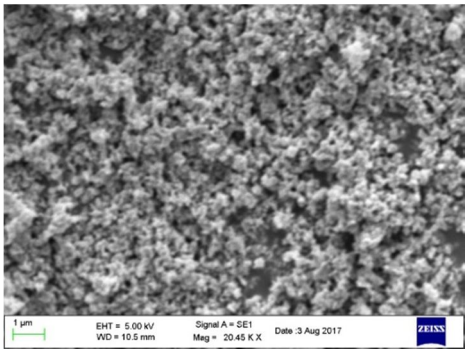

Fig.1.4 SEM picture of SnO_2

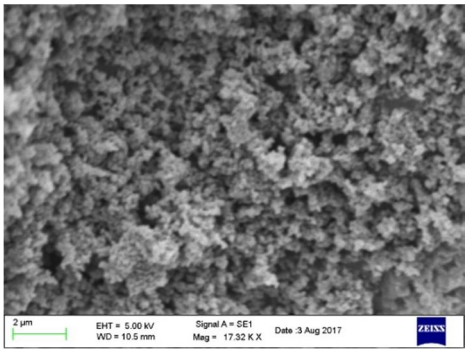

Fig.1.5 SEM picture of In_2O_3

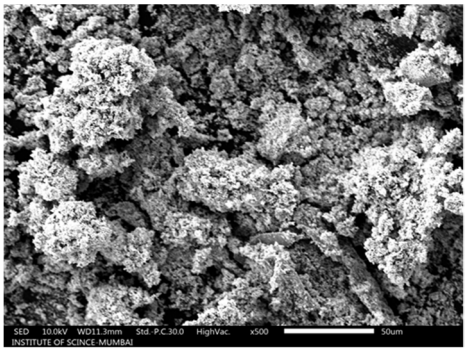

Fig.1.6 SEM picture of $65SnO_2{:}45\ In_2O_3$

From above SEM images it is also observed that $65SnO_2{:}45In_2O_3$ is porous in nature. In Some section of SEM images shows some rods with fine voids over them which helps to enhance gas sensing properties.

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Table 1.1 Average diameter of pore and number of pores per inch of pure samples and their compositions

Sr. No.	Pure sample and their compositions (mole %)	Average diameter of pore (nm)
1	SnO_2	564
2	In2O3	653
3	*$65SnO_2$:45In2O3*	234

It is also found that average diameter of pore in case of these composition is small as compared to pure metal oxides(Table 1.1). This also tends to exhibit large surface area and high response of the sample.

B. EDAX Analysis

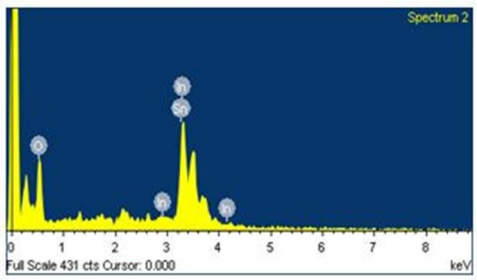

Fig.1.7 EDAX picture of $65SnO_2$:45 In_2O_3

Table 1.2 Data for EDAX

Element	Weight%	Atomic%
O K	23.98	69.72
In L	55.33	22.28
Sn L	20.69	8
Totals	100.00	

The presence of elemental composition of oxygen, Indium and tin were confirmed by the analysis through EADX spectrometers (Fig1.7). The horizontal axis displays energy in KeV and vertical axis displays the number of X-ray counts.(Table 1.2)

C. Gas Sensing Properties

The variations of sensitivity of Pure SnO_2 ,Pure In_2O_3 and SnO_2: In_2O_3 compositions with concentration of ammonia gas at room temperature are shown in Fig.1.8.

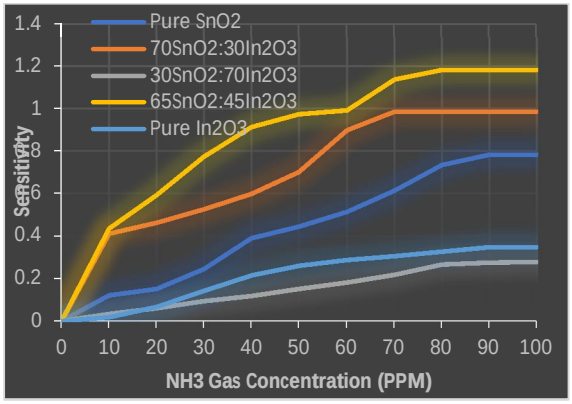

Fig. 1.8 Variation of sensitivity of SnO_2:In_2O_3 system with NH_3 gas concentration (ppm) at room temperature (303 K).

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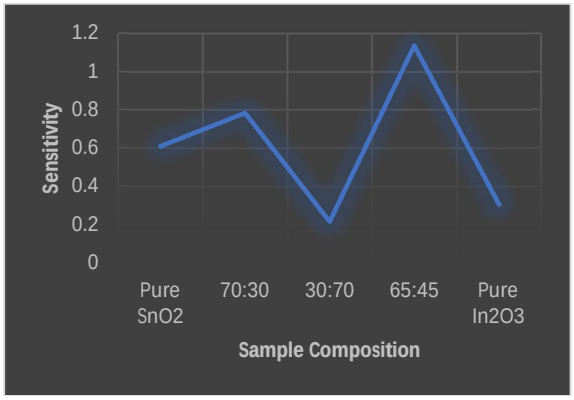

Fig. 1.9 Variation of sensitivity with SnO_2:In_2O_3 (mole %) at 70 ppm of NH_3 gas at 303 K

From Fig. 1.9, it is observed that for Pure Oxide Samples like Pure SnO_2 and Pure In_2O_3 sensitivity is less. It is observed from the cure sensitivity increases for composite samples and becomes maximum for $65SnO_2$:$45In_2O_3$ composition. From SEM picture, it is found that porosity of $65SnO_2$:$45In_2O_3$ composition is large as compared to other Pure SnO_2 and Pure In_2O_3, thus active surface area is more. Also the average crystallite size of $65SnO_2$:$45In_2O_3$ composition is small and it means large active surface area. That's why sensitivity of $65SnO_2$:$45In_2O_3$composition is large as compared to other compositions and pure samples.

D. Static Response

Fig 1.10 Static response under static condition, it is observed that response is fast for $65SnO_2$:$45In_2O_3$. It is also observed that recovery time for all sensors is very slow than the response time. The response and recovery time for all sensors for 70 ppm Ammonia gas concentration are calculated. Response time for optimize sensor i.e $65SnO_2$:$45In_2O_3$ is 90 S and recovery time is 170S

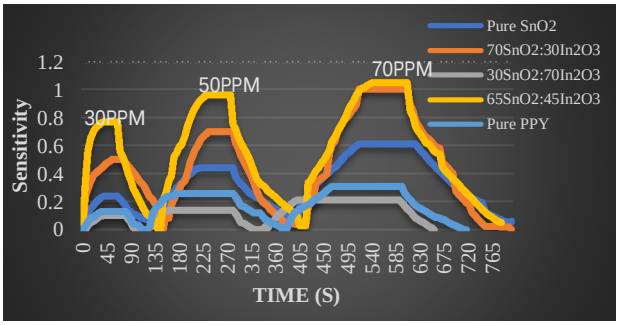

Fig.1.10 Step response of SnO_2:In_2O_3 series

E. Stability of Sensor

Sensor stability is expressed in terms of measurement of resistance with time. It is defined as the change in resistance of sensor with time [8,9].The resistance values of optimize sensors, measured with time at room temperatures it gives stable response from fig 1.11.

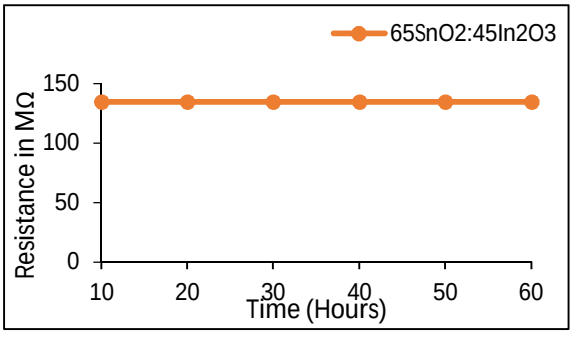

Fig. 1.11 Variation of resistance of sensors with time in air

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F. Lower Detection Limit (LDL)

It is the minimum concentration of the gas that the sensor can detect. It is observed that $65SnO_2$:$45In_2O_3$ composition sample is detected 7PPM concentration of gas.

IV. CONCLUSION

The gas-sensing properties of SnO_2:In_2O_3 Screen printing thick films towards Ammonia Gas have been investigated and compared to those of single oxide In_2O_3 and SnO_2. The pure thick film of SnO_2 and In_2O_3 sensors showed low response than the composite films to NH_3.In general, the best performances in terms of response, recovery, sensitivity and low detection limit were found in $65SnO_2$:$45In_2O_3$ sensor. This sensor showed higher sensitivity than pure In_2O_3 and SnO_2, due a *n*-doping of Sn cations in In_2O_3 lattice, and higher defectiveness than single oxides. The NH_3-sensing of the sensors were also discussed in function of the gas/surface interaction processes and different detection mechanisms were proposed for the two oxides.

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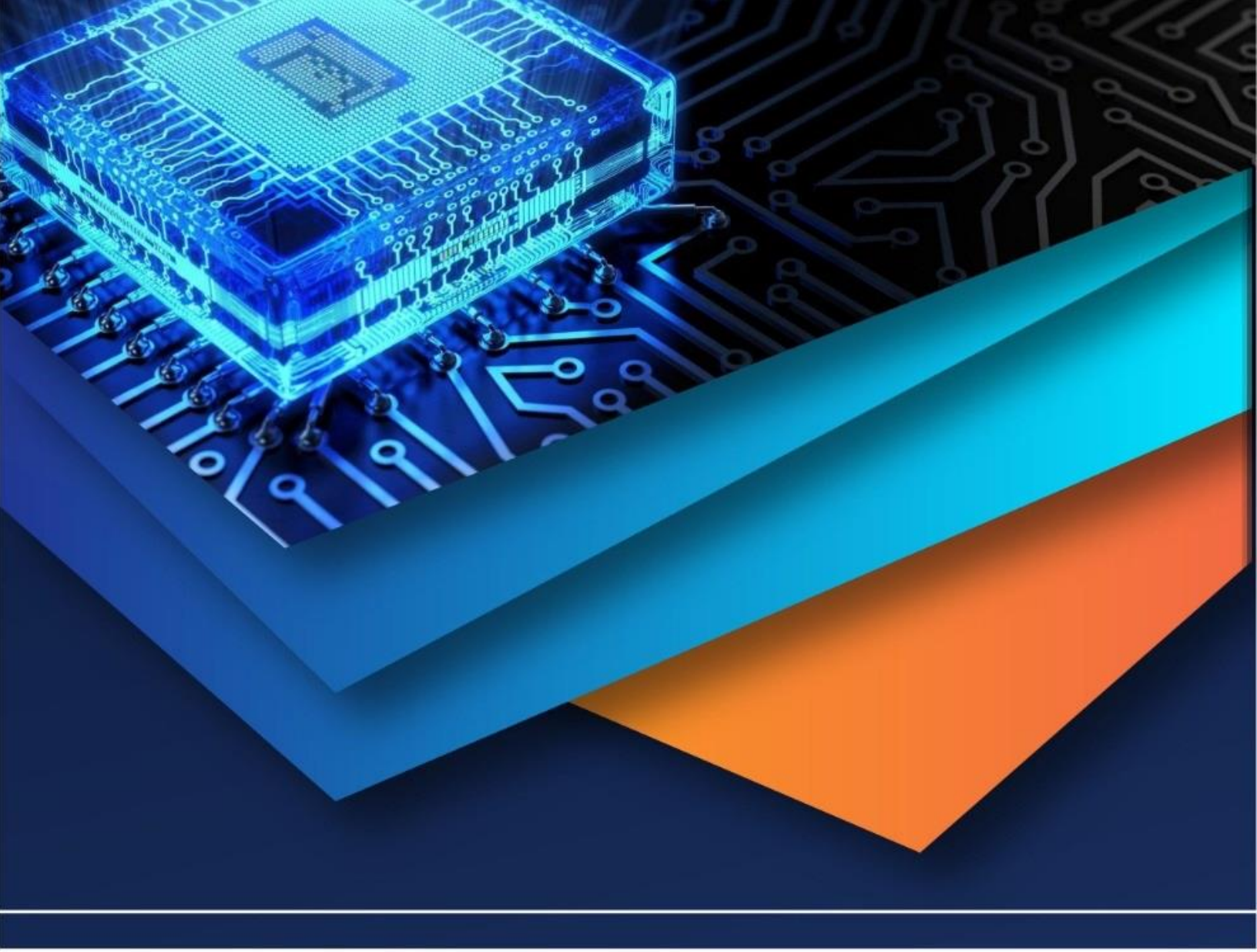

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