Sensors & Transducers, Vol. 250, Issue 3, March 2021, pp. 18-25

Sensors & Transducers
Published by IFSA Publishing, S. L., 2021
http://www.sensorsportal.com

Highly Sensitive Formaldehyde Based on Hierarchical ZnO Nanostructure to Defeat Sick Building Syndrome

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Received: 4 February 2021 /Accepted: 17 March 2021 /Published: 31 March 2021

Abstract: Zinc oxide (ZnO) hierarchical Nanofoam structure on ZnO nanofibers grown SiO_2 coated p-Si substrate were successfully constructed under chemical deposition technique by forming a homogeneous solution of Sodium Zincate (Na_2ZnO_2) in Teflon-lined Autoclave at 120 °C. The seed layer of hierarchical ZnO nanostructure was formed by Sol-gel technique followed by CBD (Chemical bath deposition) technique to grow ZnO nanofibers. The surface morphology and crystallinity of the thin film was characterized by scanning electron microscope (SEM) and X-ray diffraction (XRD) and controlled by adjusting the pH of the reaction solution. The hierarchical ZnO Nanostructure based Formaldehyde (HCHO) Sensor was tested in the resistive mode at dynamic range of 190 ppm to 2020 ppm formaldehyde vapour to defeat sick building syndrome. It was found to offer high response magnitude 94.8 % at 2020 ppm at very low optimum operating temperature 100 °C with fast response time (20 s). Finally, the possible mechanism of decomposition of formaldehyde sensor has been discussed with detailed explanation of higher response magnitude on exposure of formaldehyde vapour with energy band diagram.

Keywords: ZnO hierarchical nanostructure, ZnO nanofibers, Teflon-lined Autoclave, Formaldehyde sensor, Sensing mechanism, Energy band diagram.

1. Introduction

Hierarchical nanostructure has ushered with immense possibilities as there are much scope to explore the opportunities with the use of it [1-3]. By controlling their physical, chemical properties with modifying their shapes and morphology the effectiveness of this hierarchical nanostructure can be varied in a broad way. There are many reports till date with 3-D nanostructures [2-4]. But lower surface area with limited gas interaction facets, these types of nanostructures cannot compete as a next generation gas sensor. Researchers are looking for some new developments as next generation gas sensors are already in the market. Synthesis of these hierarchical nanostructures with prescribed dimension is quite tricky as it requires some improved technology to perk up their physical properties which will positively enlarge their area of application. There are several methods to available to fabricate hierarchical nanostructures. Many of them require sophisticated hi-end equipment. Moreover, control of morphology, thickness is getting restricted several times. To circumvent the above mentioned problem, hydrothermal synthesis is one of the best possible methods of producing hierarchical nanostructures owing to its ease of production, lower cost and scalability [2-4]. Moreover the structure offers high purity with narrow particle size distribution. Metal oxide semiconductor based gas sensors are there in the market since last two decades as the band gap can be manipulated judiciously to obtain the optimum output. It is heavily used in optoelectronics, sensors, nanopiezotronics and catalysis [1-8].

Among different metal oxides Zinc oxides, (ZnO) is one of the proven material in gas sensor family. As it has got unique properties like higher band gap, direct band gap, porosity, smaller grazing size etc., the morphology and structuring of ZnO is multidimensional.

A fast detection sensor is required to continuously monitor the ambience around us and alert us about the presence of toxic gases in extremely low concentrations even in the indoor places where common people are unaware about it.

Among different silent diseases, sick building syndrome is one of the silent killer that cannot be detected at an early stage sometime. This disease is caused by harmful volatile organic compounds (VOCs) formaldehyde. Formaldehyde is found frequently in acrylic paint, adhesive resins that bind powdered wood products like particle board etc. Formaldehyde is also used in the textile manufacturing industry. Prolonged exposure of it may cause headaches, fatigue, nausea, and many more. As it is toxic, it may cause leukemia after prolonged exposure even where the concentration is low, *i.e.*, ppb level [3-5].

Not only that this hazardous gas is also formed during ozonation which one of the part of water treatment processes, and water animals are highlyaffected. As per the standard, formaldehyde levels of greater than 1 ppm grounds cause problem to different organs and above it may affect the human lives precariously.

As per the existing safety standards, a human can withstand maximum exposure of 2 ppm for 8-hour, while inside room levels should not go beyond 0.08 ppm (80 ppb) over 30 minutes. The Chinese Environmental Protection Agency limits exposure to 0.06 ppm over 30 minutes [7, 8]. So, formaldehyde detection is very much needed to save the life of common people across the globe. Functional material synthesis is quite a challenging task that can be eliminated by the use of hierarchical nanostructures. The way the formaldehyde is detected should be competent as very low level detection is required.

Several metal oxide based formaldehyde sensor have been reported by the researchers [1-3, 7, 8] and compared with the present work. Li, Cheng and Wang, *et al.*, have been successfully synthesized low temperature (120 °C) hierarchical Sn/SnO_2 based formaldehyde gas sensor using hetero-junction at1ppb concentration [3]. A hierarchical Co_3O_4 nanostructure has been made-up by calcinating Co_5-based metal organic structure by Zhou, Wu and Zhao, *et al.* at 170°C to detect formaldehyde maintaining long term stability [1]. Y-doped SnO_2 hierarchical flower-like nanostructures was made via one-step hydrothermal method for improving the formaldehyde sensing at 180°C has been successfully prepared by Zhu, Ma, Tie, Zhang, *et al.* [2]. Deng, Li and Wang, *et al.* [7] reported a hierarchical flower-like CuO nanostructure based formaldehyde gas sensor with a high response of 1.378 to 50 ppb formaldehyde at 250 °C (11.9 sec response time and 8.4 sec recovery time). Li, Guoans Su, *et al.* [8] have reported Hierarchical morphology-dependent gas-sensing performances of 3D SnO_2 nanostructures to detect formaldehyde with comparison of other VOCs at 200 ppm at optimum temperature of 325 °C.

Table 1. Comparison of different electrochemical exfoliation technique for synthesis of graphene with the present work.

Material	**Testing temp.**	**Testing conc.**	**Response magnitude***	**Response time ****	**Recovery time ****	**Range of gas concentration (ppm)**	**Ref.**
ZnO	100 °C	1000 ppm	90 %	29 s	76 s	190 - 2020	Present work
Co_3O_4	170 °C	200 ppm	93 %	8 s	11 s	10 - 500	[1]
SnO_2	180 °C	50 ppm	94.4 %	16 s	20 s	1 - 100	[2]
Sn/SnO_2	120 °C	50 ppm	93 %	60 s	75 s	1 - 50	[3]
CuO	250 °C	1 ppm	57.9 %	10 s	17 s	0.05 - 1	[7]
SnO_2	325 °C	100 ppm	95.6 %	15 s	20 s	5 - 200	[8]

* All sensitivity/response magnitude of all the references were calculated as S = ((Ra-Rg) / Ra) ×100 and compared with the present work.

** Response & recovery time were calculated as time taken to reach 90 % of the saturation value and to reach back to the original value respective.

So, developing a fabrication-friendly method to synthesize ZnO hierarchical nanostructure with distinct, definite morphology is still a major concern. This paper, presents a simple hydrothermal method for the fabrication of foam like ZnO hierarchical nanostructure using twin method of CBD and chemical deposition technique at very low temperature (100°C). The gas sensing capability of this structure was checked by the incorporation of formaldehyde in the test chamber.

2. Experimental

2.1. Materials

All the chemicals were of analytical reagent grade and used as-received, without further purification. Zinc Acetate Dihydrate (Merck, 99.9 %), 2 Propanol (25 ml), Diethanolmine, Hexamethylenetetramine $[(CH_2)_6N_4,]$ Deionized Water (Resistivity ~18.2 MΩ-cm).

2.2. Sample Preparation and Gas Measurement Set-up

The schematic of the hierarchical ZnO nanostructured Formaldehyde sensor is shown in Fig. 1. The base (seed layer) of ZnO hierarchical Nanofoam structure on ZnO nanofibers is formed by Sol-gel technique by reacting Zinc Acetate Dihydrate ((Merck, 99.9 %), 3 gm) and 2 Propanol (25 ml) on SiO_2 coated the p type silicon (Si<100>) substrate.

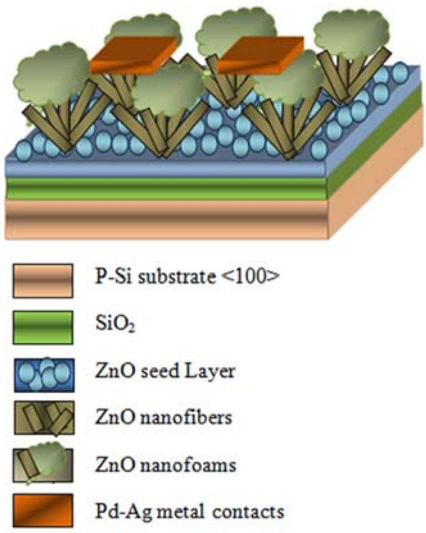

Fig. 1. Schematic diagram of formaldehyde sensor.

3 ml Diethanolmine was also then added drop by drop to get a crystal clear solution followed by 24 hours aging. The samples were spin coated (1000 r.p.m.) for 25 s and put in an annealing furnace at 450 °C for 1 hours and subsequently ZnO seed layer was formed by thermal decomposition of Zinc Acetate Dihydrates. To prepare ZnO nanofibers, a 50 ml solution was prepared by mixing Zinc Acetate Dihydrate and HMT [Hexamethylenetetramine $[(CH_2)_6N_4,]$ in deionized water (resistivity ~18.2 MΩ-cm) solution in the mass ratio of 1:3. The substrates were then immersed into the solution and after removal kept in a baking oven for 1 hr at 100 °C. Samples were allowed to cool down gradually.

For getting ZnO hierarchical nanostructure, ZnO nanofoam layer was formed by a chemical deposition technique by preparing a homogeneous solution of Sodium Zincate (Na_2ZnO_2) by mixing Zinc Nitrate ($Zn(NO_3)_2$, $6H_2O$) and Sodium Hydroxide (NaOH) in the ratio (wt.%) of (1:3) and was magnetically stirred at room temperature for 20 minutes.

$$Zn(NO_3)_2+2NaOH = Zn(OH)_2+2NaNO_3, \quad (1)$$

$$Zn(OH)_2+2NaOH = Na_2ZnO_2+2H_2O, \quad (2)$$

$$Zn(NO_3)_2+4NaOH=Na_2ZnO_2+2NaNO_3+2H_2O \quad (3)$$

After stirring, the mixture was then transferred to a 50 mL Teflon-lined autoclave, sealed, and kept at 120 °C for 12 h and finally cooled to room temperature. Two metal electrodes (Pd-Ag) were then deposited by e-beam vacuum evaporation technique on top of the sensing layer using Al shadow mask. The device dimension was [5 mm × 5 mm] having an electrode area of [2 mm × 2 mm] (each electrode area having 1 mm × 1 mm).

The details of vapor measurement set-up have already been reported earlier [5, 6. 10]. The formaldehyde vapors were bring in the compartment by passing IOLAR grade N_2 (carrier gas) through the formaldehyde kept in a conical bottle (Fig. 2).

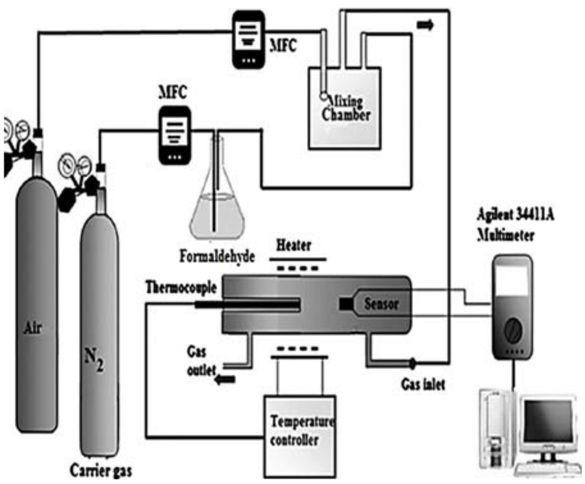

Fig. 2. Gas sensor measurement set-up.

Assuming that carrier got satisfied with the formaldehyde, the desired concentrations of the vapor were obtained in the sensing chamber by controlling the flow rate of N_2 through formaldehyde and adding

IOLAR grade N_2 (diluents) through a separate gas flow line. The mixture that is consistent in nature carrying the sufficient target vapor was fed into the chamber with a bendable PVC pipe. To measure the sensor resistance, electrodes are connected to an Agilent U34411A Multimeter with Agilent GUI Data logging software (v2.0). The final concentration of formaldehyde vapors was calculated using this formula [9, 10].

$$C = \frac{\frac{P \times L}{760 - L}}{\frac{P \times L}{760 - L} + L + L'} \times 10^{6} \quad (4)$$

2.3. Characterization of the Sensor

A comprehensive study of shapes and structural characterization of the ZnO hierarchical layer has been reported here. XRD (Fig. 3) (using ULTIMA-III, CuKα target, X-ray Diffractometer, λ=1.54A°) confirmed the strongest (002) preferential c-axis growth (at 34.44°) with 5.12 A° interplanner spacing. The other diffraction peaks positioned at 2θ values of 31.76° and 36.21° can be indexed to the hexagonal wurtzite phase of ZnO (JCPDS card no: 79-2205) with crystallinity of (100) and (101) planes respectively.

The surface morphology of the hierarchical ZnO was characterized by atomic force microscopy. Fig. 4 show a typical height profile of ZnO nanostructure on SiO_2 substrate. AFM micrograph of nearly 50 × 50 µm area of Si/SiO_2 substrate supporting hierarchical ZnO nanostructure is presented here. The thickness was calculated from the height difference between the surface of ZnO and that of the substrate and has been recorded as ~300 nm.

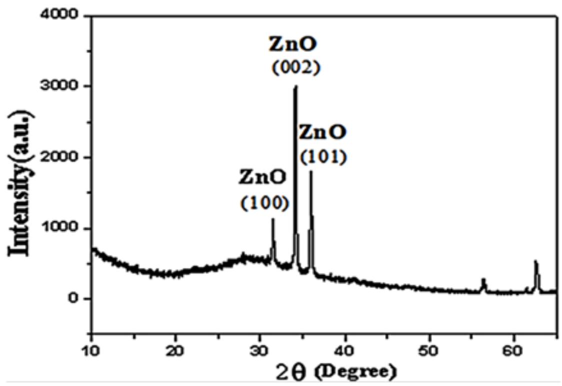

Fig. 3. XRD pattern of ZnO hierarchical formaldehyde sensor.

The average lateral size of these electro-chemically hierarchical ZnO nanostructure ranges from 300 to 350 nm.

FESEM (Fig. 5) result depicts the uniformly distributed ZnO nanofoam having average dimension of ~300 nm with average fiber diameter of ~20 nm and length 400 nm.

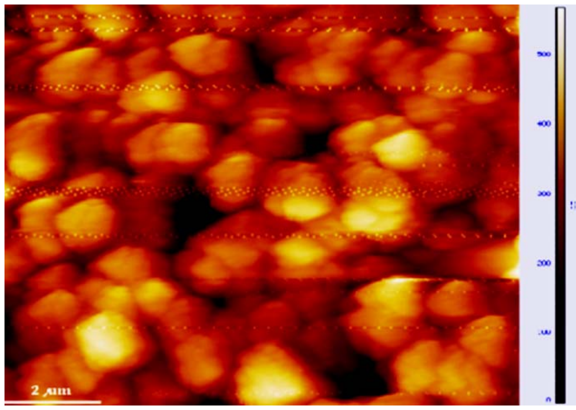

Fig. 4. AFM image of ZnO hierarchical formaldehyde sensor.

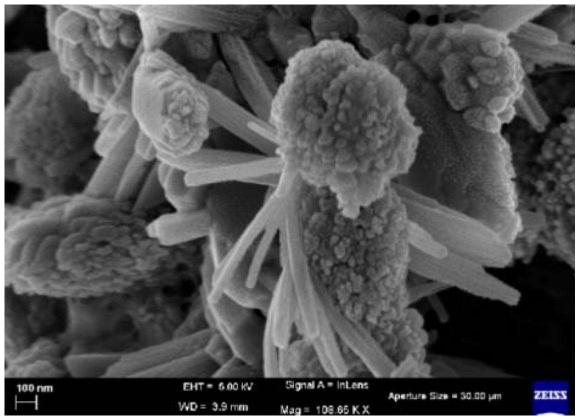

Fig. 5. FESEM image of ZnO hierarchical formaldehyde sensor.

3. Results and Discussions

3.1. Sensor Study

To report the optimum operating temperature of sensing, sensor resistance was calculated in presence of air and formaldehyde vapor and the equivalent response magnitude was calculated as a function of temperature. Response Magnitude (RM) of the sensor was considered with this formula:

$$RM = \frac{Ra - Rg}{Ra} X 100\%,$$

where R_g is the sensor resistance in the test vapor (formaldehyde vapor diluted with air) and R_a is the sensor resistance in air. In this study, response time is defined as the time required for the sensor to reach 90 % of the saturation value and the recovery time is defined as 90 % of the time required to come back to the original value from saturation value [11].

Before testing with gas I-V characteristics of the Pd-Ag contact on aligned ZnO nanofoams were studied at different temperatures. The I-V characteristics (Fig. 6) indicated a good ohmic nature of the Pd-Ag contacts for all three temperatures.

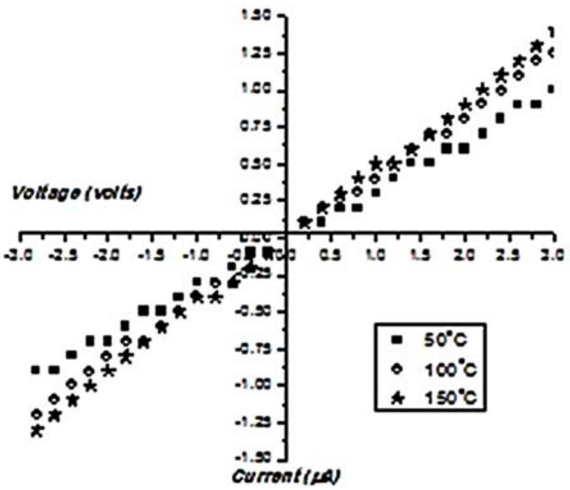

Fig. 6. I-V characteristic of Pd-Ag contact on ZnO sample.

The change in resistance of the resistive sensor was measured in order to get the optimum operating temperature using 1000 ppm formaldehyde vapor (in air) as shown in Fig. 7. Transient response characteristics of ZnO hierarchical Nanofoam structure on ZnO nanofibers resistive sensor at it's respective optimum operating temperature (100 °C) for different formaldehyde vapor concentrations (190-2020 ppm) are shown in the Fig. 8.

It can be envisaged from the graph (Fig. 8) that the sensor resistance was initially high due to absence of free electrons on the ZnO sensing surface. After introducing the formaldehyde vapor, the sensor resistance decreased due to release of free electrons; on cutting off the vapor supply, the sensor resistance increased due recapturing of the adsorption site by oxygen species (from air) causing a reduction in the available surface electron concentrations. At 2020 ppm concentration of the formaldehyde sensor shows high response magnitude (94.8 %) with 20 s response time and 88s recovery time but at lower concentration (190 ppm) the sensor offers 70.4 % response magnitude with high response (45 s) time. As a result response time decreases to the higher concentration but the recovery time increases due to slow desorption rates of the formaldehyde molecules from ZnO surface. At higher concentration, formaldehyde sensor acquires a tendency towards saturation due to occupancy of all the adsorbent sites of the surface. It is also noticed that there are minimal baseline shift due to the fact of some target vapor species remained adsorbed even after the vapor pulse is cut off and the amount of such adsorbed species increased as the vapor concentration increased. The variation of response magnitudes as a function of formaldehyde concentrations (190, 380, 570, 760, 950, 1260, 1530 and 2020 ppm) at particular optimum operating temperature (100 °C) shows in Fig. 9 by the bar chart representation.

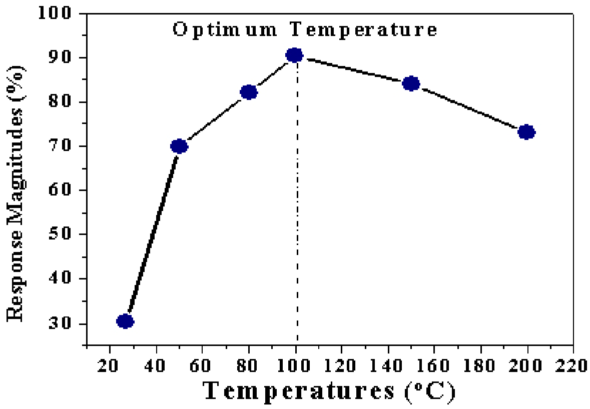

Fig. 7. Response magnitude (function of temperature) for 1000 ppm formaldehyde concentration.

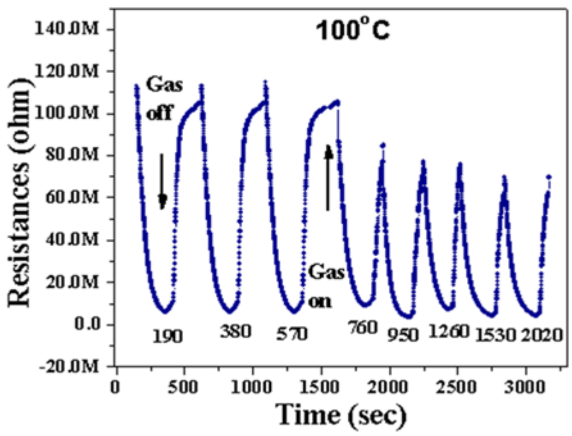

Fig. 8. Transient response characteristics of the Sensor at 100 °C.

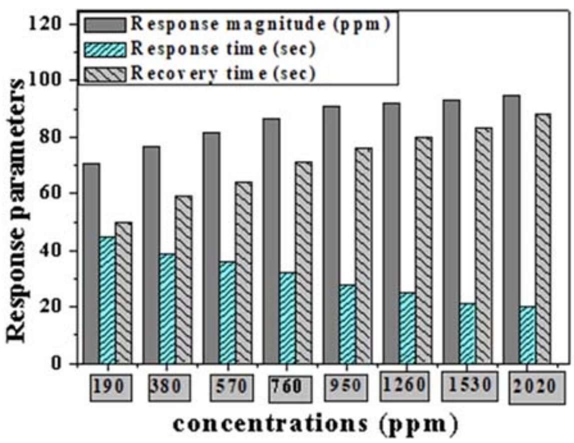

Fig. 9. Comparison charts for response magnitude, response and recovery time of the sensor for different concentrations of gas.

Overall results (different sensing parameters in a concentration range of 190-2020 ppm at 100 °C) for the formaldehyde sensor with Pd-Ag contact is represented in a tabular form below (Table 2).

Table 2. Response parameters in the range of (190-2020) ppm of Formaldehyde sensor.

ZnO hierarchical nanofoam based formaldehyde sensor (100 °C)			
Concentrations (ppm)	**Response magnitude (%)**	**Response time (s)**	**Recovery time (s)**
190	70.4	45	50
380	76.6	39	59
570	81.8	36	64
760	86.4	32	71
950	90.8	28	76
1260	91.9	25	80
1530	93.2	21	83
2020	94.8	20	88

3.2. Formaldehyde Sensing Mechanism

Like other gas/vapor sensing mechanism, for formaldehyde gas sensing also, initial oxygen adsorption plays a pivotal role in the sensing mechanism of Zinc oxide (ZnO) hierarchical Nanofoam sensor. Oxygen ion adsorption process (on ZnO surface) reduces conduction electron concentrations resulting in an increase of ZnO film resistance. Reactive oxygen species such as O_2^-, O^{2-} and O^- are adsorbed on the ZnO surface. Therefore during the chemisorption, atmospheric oxygen forms ionic species such as O_2^- and O^- by taking electrons from the conduction band of ZnO surface. The reaction kinetics by chemisorption of oxygen on ZnO surface is shown below [15-17]:

$$O_2\,(gas) \leftrightarrow O_2\,(ads)$$

$$O_2\,(ads) + e^- \leftrightarrow O_2^- \quad (5)$$

$$O_2\,(ads) + e^- \leftrightarrow O_2^- \quad (6)$$

$$O_2^-\,(ads) + e^- \leftrightarrow 2O^-\,(ads)$$

The overall formaldehyde sensing mechanism of Zinc oxide (ZnO) hierarchical nanofoam structure is shown in Fig. 10 (a and b).When the ZnO sensor is exposed to formaldehyde vapor, after thermal decomposition of formaldehyde, the activated fragments of formaldehyde (which are mainly H) spill over onto the ZnO surface, then water vapour (H_2O) and carbon-di-oxide (CO_2) are formed by reacting the products (H_2 and CO) of formaldehyde with chemisorbed oxygen of ZnO surface and release electrons into the conduction band of ZnO resulting in a decrease in the ZnO film resistance. The combined vapours exposure (water vapour and CO_2 vapour) after formaldehyde dissociation offers higher response magnitude of the sensor.

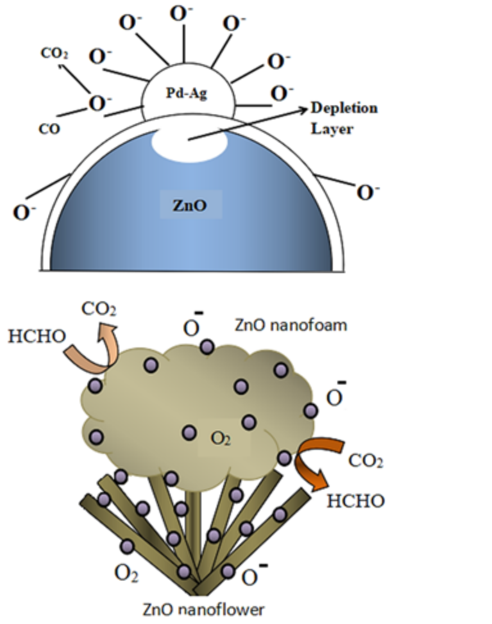

Fig. 10. Formaldehyde sensing mechanism of Zinc oxide (ZnO) hierarchical nanofoam structure.

3.3. Energy Band Diagram

Formaldehyde sensing mechanism of ZnO hierarchical nanostructure can also be reinforced by the corresponding energy band diagram of inter-granular junction and Pd-Ag with ZnO (Metal-Semiconductor junction) in presence and in absence of formaldehyde vapour shown in Fig. 11.

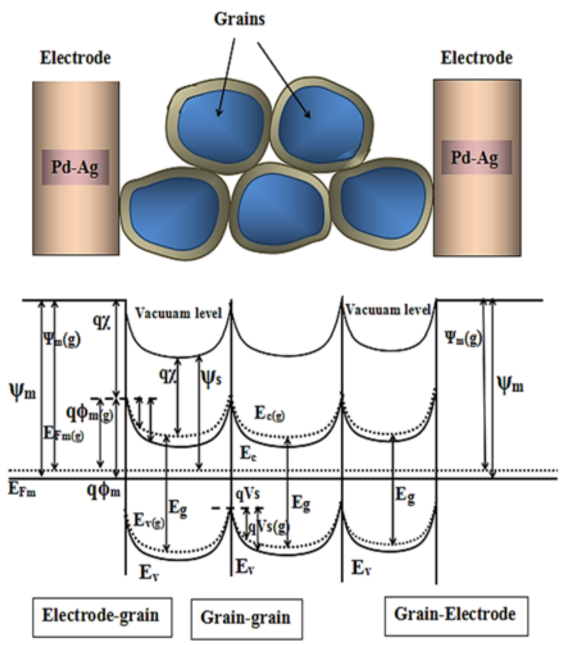

Fig. 11. Geometry of the hierarchical nanostructured ZnO thin film with Pd-Ag electrodes and it's corresponding energy banddiagram.

In the present case, the work function (ψ_m) of metal [Pd: 5.12] is greater than the work function of ZnO

[ψ_s: 4.45]. Initially, Pd attracts atmospheric oxygen to form weakly bonded Pd:O which eventually increases the work function of Pd. The increase of Pd work function decreases the barrier height of the Pd-ZnO contact and accordingly increasing the resistance of the sensor. But when some formaldehyde vapor is introduced, it (vapor) reacts with adsorbed oxygen (O^-) of ZnO surface and decreases the work function of metal due to reduction of O^- from surface of metals thereby increasing the barrier height. Therefore the total majority carrier (electrons) increases in the conduction band of ZnO (with respect to that in air) which reduces the resistance of formaldehyde sensor.

3.4. Stability Study

Stability study of the sensor was carried out with IOLAR grade N_2 and 1 % HCHO in N_2 for 24 h with a duration of 6 h per day in 4 days, at the optimum temperature (~100ºC) (Fig. 12). It was observed that the sensor exhibits quite linear response for both N_2 and 1 % HCHO in N_2 with nominal variation of sensor resistance from its reference baseline value (3.35 MΩ in N_2 and 2.2 MΩ for 1 % formaldehyde in N_2).

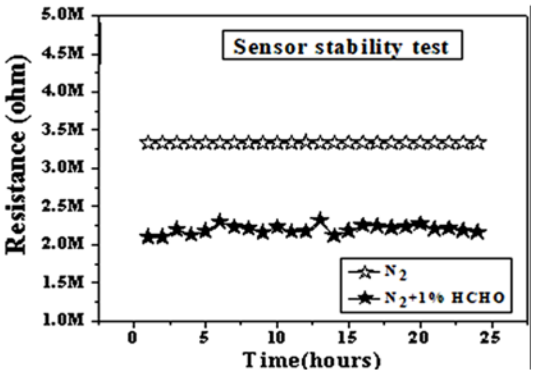

Fig. 12. Stability study of sensor with N_2 and with 1 % HCHO in N_2 at 100ºC.

4. Conclusion

This paper focuses on the formaldehyde sensing characteristics, corresponding sensing mechanism with energy band diagram, electrical characteristics and stability study of ZnO hierarchical nanofoam based resistive sensors. Autoclave played a crucial role for the formation of this hierarchical ZnO nanostructure. After detailed structural characterizations (XRD, AFM etc.), the sensor was employed to detect formaldehyde. Catalytic effects, to improve the sensing performance, were incorporated by Pd-Ag catalytic contact on ZnO hierarchical nanostructure. The sensor was tested in the range of 190 to 2020 ppm by offering much higher response magnitude (94.8 %) at very low operating temperature (100 ºC). The mechanism of decomposition of formaldehyde sensor has also been explained with energy band diagram.

Acknowledgments

Authors thankfully acknowledge DST, Govt of India (FIST-level 0, Sanction letter no:SR/FST/COLLEGE-466/2018) for providing laboratory and equipment facility partially.

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