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Study of CO_2 gas detection by multilayer SnO_2-ZnO-PPy sensor

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ABSTRACT
The gas sensitivity response of SnO_2-ZnO composites in multilayer with PPy and Al_2O_3 was observed of good response. The chemicals used for the designing of gas sensor were first calcinated at 700^0C for 7 hr. Composites of SnO_2-ZnO were prepared and multilayer sensor was designed using screen printing technique with Al_2O_3 as substrate on glass plate and PPy in multilayer with SnO_2-ZnO. The surface morphologies of composites of SnO_2-ZnO and PPy were analyzed by FTIR and XRD. Sensitivity was found to be more for $80SnO_2$:20 ZnO/ PPy/Al_2O_3 multilayer sensor, supported by FTIR and XRD studies. It was found that response of multilayer sensor increases with increase in ppm concentration of CO_2 gas.

Keywords: SnO_2-ZnO; screen-printing technique; CO_2 gas sensor, sensitivity.

INTRODUCTION

It is well known that the sensing properties of SnO_2-based material depend on its chemical and physical characteristics, which are strongly dependent on the preparation conditions, dopant and grain size. This implies that the synthesis of the sensing material is a key step in the preparation of high-performance Metal oxide semiconductor (MOS) gas sensors.

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SnO_2 powders and films can be prepared by a variety of synthesis methods [1-4]. DC-electrical resistance of the films SnO_2 doped with ZnO and TiO_2 sensor was measured in presence of humidity and SnO_2–$5Al_2O_3$ and ZnO–$5Al_2O_3$ sensors are found to be good sensing materials for humidity [5]. The present investigation mainly deals with the preparation of CO_2 gas sensor in multi-layer pattern with SnO_2 doped as ZnO and polypyrrole layer. It was found that SnO_2-ZnO system in multilayer with polypyrrole shows more sensitivity to 80 ppm of carbon dioxide gas concentration even at room temperature.

A gas sensor is a device, which detects the presence of different gases in an atmosphere, especially those gases that might be harmful to living animals. The design of gas sensor technology has received considerable attention in recent years for monitoring environmental pollution. Tin dioxide (SnO2) based chemiresistors have high gas sensing response as compare to the chemiresistors based on conducting polymers but they are operated at high temperature (>200 °C). Whereas conducting polymer-polypyrrole (PPy) doped with metal oxides sensors have shown better sensing response at room temperature [6].

METHODOLOGY

Preparation of conducting Polypyrrole:

The method used for the preparation of polypyrrole is chemical polymerization. Powder polypyrrole was prepared with 4.290 (high) weight ratio of pyrrole (Py) monomer and oxidant ($FeCl_3$). During the synthesis, concentration of $FeCl_3$ was kept constant and methanol was used as a solvent. The Py monomer, anhydrous iron (III) chloride ($FeCl_3$) and methanol were used for synthesis of PPy. The solution of 7 ml methanol and 1.892 g $FeCl_3$ was first prepared in round bottom flask and 8.4 ml Py-monomer was added to ($FeCl_3$ + methanol) solution with constant stirring in absence of light. The amount of Py-monomer was added to the solution in such a way to get maximum yield. The polymerization of Py, which was suppressed in a solution, progressed rapidly due to an increase of oxidation potential caused by evaporation of solvent. In the polymerization reaction of Py, it was observed that as soon as the Py-monomer was added to the solution, the colour changed to dark green/black. There was an increase in temperature of the solution during the start of reaction, which showed that it is an exothermic reaction and it was carried out at room temperature for 4 hr. The final precipitated polymer was filtered by a conventional method. The polymer was washed with distilled water several times till the filtrate obtained was colourless. To remove last traces of un-reacted pyrrole and remaining ferric and ferrous chloride formed due to polymerization, it was then washed with methanol. The polymer, obtained in powder form was dried first at room temperature for a few hours and then finally dried in an oven kept at 80^0C for 5-6 hrs [6-9]. This polypyrrole is then used for active layers of Semiconductor Gas Sensors.

Sensor preparation:

SnO_2, ZnO and Al_2O_3 powders (AR grade) were calcinated at about 800 °C for 4-5 h and were crushed in mortal pestle to get fine powder of the samples. SnO_2, ZnO and PPy were characterized by XRD. XRD patterns of the samples were obtained using Diffractometer system from GVISH, Amravati. The diffraction pattern was in the terms of 2θ at continuous scan type at step size = 0.015^0.

SEM of the polypyrrolle:

The ink or paste of the sample was prepared by using screen-printing (thick film technique) technique. The binder for screen-printing was prepared by thoroughly mixing 8 wt% butyl carbitol with 92 wt% ethyl cellulose. On chemically cleaned glass plate, paste of Al_2O_3 was screen printed and it was kept for 24 hr to dry it at room temperature and then heated at 140^0C for 2.5 h to remove the binder. The Al_2O_3 layer provides mechanical support as well as high thermal conductivity. Paste of SnO_2 and ZnO mixed in proper stiochometry was then screen printed on Al_2O_3 layer. Again plate was dried at room temperature for 24 h and binder was removed by heating it at 150^0C for 2.5 h. Finally PPy layer was deposited on SnO_2 doped with ZnO layer by screen printing, whole plate was dried and again binder was removed as above. Fabrication of multilayer sensor is shown in following fig. (1).

Finally on the top surface of the sensor, interdigited electrodes [26] were fabricated using conducting silver paste as shown in the Fig.1 (b). Thickness of SnO_2 doped with ZnO layer and PPy layers were recorded with the help digital micrometer (series 293, Japan) having resolution of ± 0.001 mm and were found to be 10 µm and 7 µm respectively. To measure the sensitivity, electrical resistance was measured with the help of voltage drop method, best one.

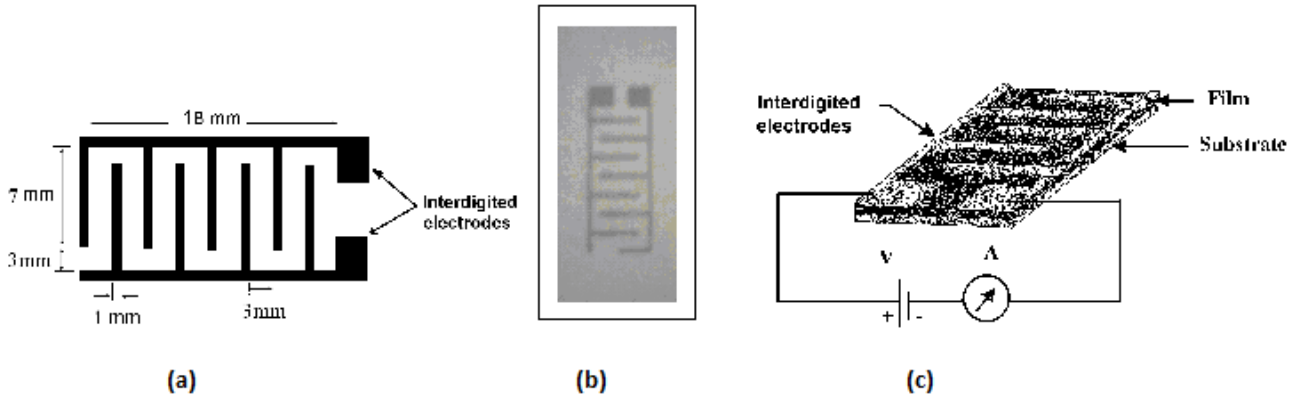
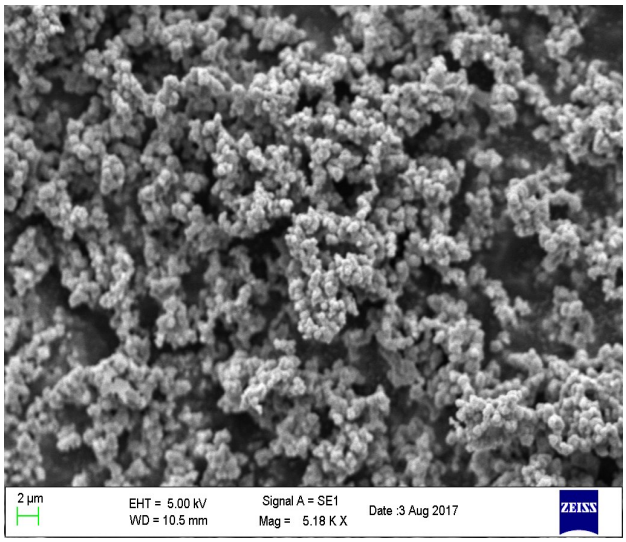


Fig. 1:(a) Fabrication of interdigited Electrodes (b) Actual photograph of interdigited electrodes (c) Circuit of resistance measurement using interdigited electrodes.

RESULTS AND DISCUSSION

XRD and SEM Analysis:



SEM were studied at department of physics, Rashtrasant Tukdoji Maharaj Nagpur University, Nagpur. XRD of PPy and $80SnO_2$:20ZnO [fig.2 (a) and (b)] showed that polypyrrole is amorphous in nature. A broad peak at $2\theta = 24^0$ was observed which gives the evidence for amorphous nature of polypyrrole. Broad peak is the characteristic of amorphous nature of polypyrrole and it is due to the scattering from PPy chains at the inter-planar spacing [28]. The average crystalline size of PPy was calculated by using Scherrer's formula given by equation (1),

$$D = \frac{K\lambda}{\beta\cos\theta} \qquad (1)$$

Where, D is the crystalline size, K is the shape factor and β is the full width at half maximum of diffraction angle in radians. The average crystallite size of PPy was found to be 101 nm. The sensor parameters are listed in the table (1)

Table1: sensitivity

Sr. No.	Composition (mol %)	crystallite size (nm)	Sensitivity (s) at 80 ppm at 313K	Response Time (sec)	
				ON time	OFF time
1	$80SnO_2$-20ZnO	85.09	0.56	59	101
2	Pure SnO_2	109.47	0.39	103	137
3	Pure ZnO	151.05	0.21	115	151

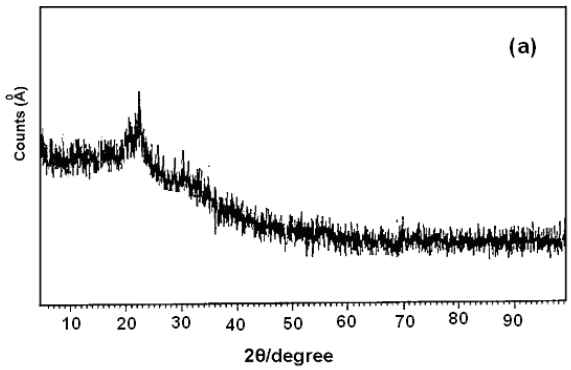


Fig 2(a): XRD of PPy

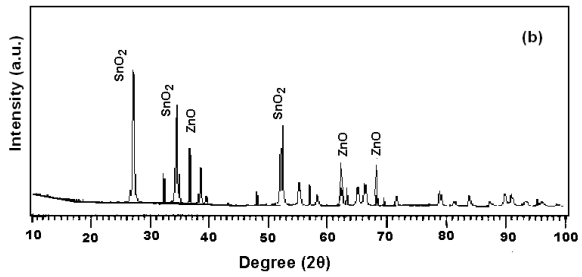


Fig 2(b): XRD of 80SnO_2:20ZnO

FTIR Spectroscopy

The Polypyrrole powders prepared were analyzed by FTIR. FTIR spectra showed the main characteristic peaks at 761cm−1 corresponding to C-N bond, 1271 cm−1 corresponding to C-H deformation,1533 cm−1 and 1459 cm−1 corresponding to the fundamental vibrations of polypyrrole . The peaks at 1640 cm−1 corresponding to C=C. The peak at 3411 cm−1 corresponds to the N-H bond . These peaks were observed in the present work for preparations using $FeCl_3$ as oxidants and various dopants such as SnO2 and ZnO. This is confirmed the formation of Polypyrrole[12]

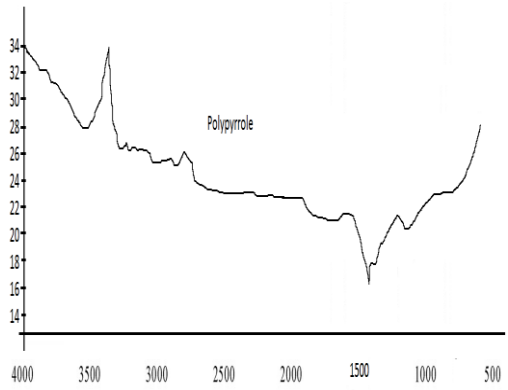


Fig. 3: FTIR of PPy

Sensitivity of sensor:

The sensitivity of the sensor is given by equation (2),

$$S = \left(\frac{R_{air} - R_{gas}}{R_{air}} \right) = \left(\frac{\Delta R}{R_{air}} \right) \quad (2)$$

Where, R_{air} and R_{gas} are the resistances of sensors in air and gas respectively.

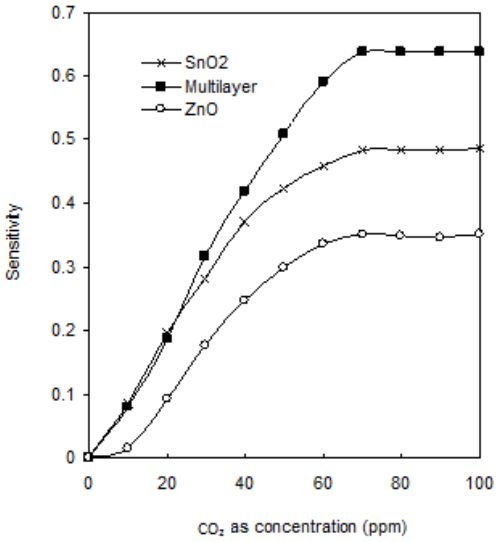


Fig.(4):Variation of sensitivity with of CO_2 gas concentration at room temperature.

From Fig. (4), multilayer structure of the sensor shows more sensitivity to Ammonia gas than that for pure ZnO and pure SnO_2. Resistance of multilayer sensor was found to be decreasing with increase of Ammonia gas concentration and thereby sensitivity was increasing[10]. Maximum sensitivity was recorded for multilayer sensor at 80 ppm concentration of CO_2.

Dynamic and Static responses:

Dynamic and static responses for pure SnO_2, pure ZnO and multilayer sensor for 40, 60 and 80 ppm are shown in figs. 5 and 6 respectively. The time taken to reach 85% of the response when ppm of gas is changed is known as response time and time taken to reach 85 % of recovery when gas is turned off is known as recovery time. Response time (t_{res}) and Recovery time (t_{rec}) are the two important parameters of the sensor. It was found that response time is 59 s and recovery time is 101s for multilayer sensor at 80 ppm of CO_2. Recovery time was found to be longer than response time, also t_{RES} and $t_{REC.}$ for multilayer sensor were found to be smaller than that for pure SnO_2 and pure ZnO, i.e. multilayer sensor is fast.

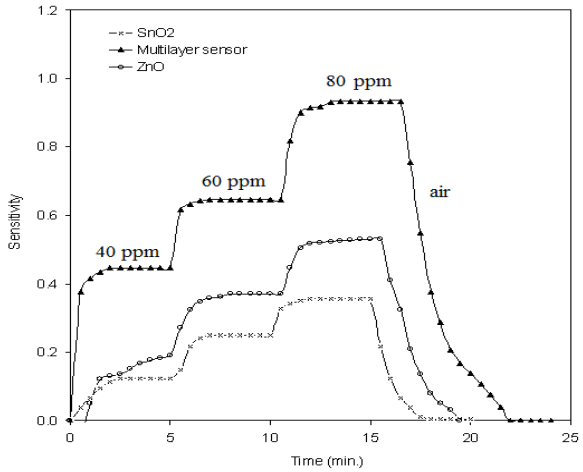


Fig.(5):Dynamic response of pure SnO_2, pure ZnO and Multilayer sensor at room temperature.

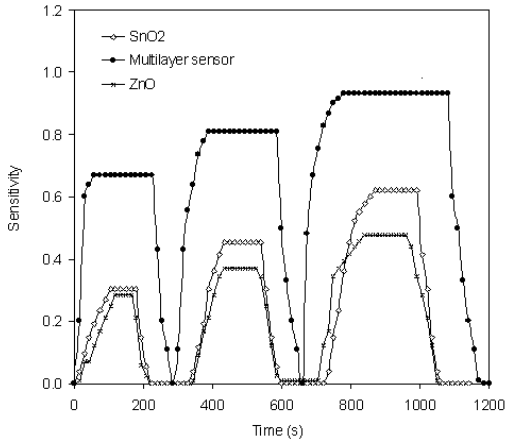


Fig.(6): Static response of pure SnO_2, pure ZnO and Multilayer sensor at room temperature

Stability of sensor:

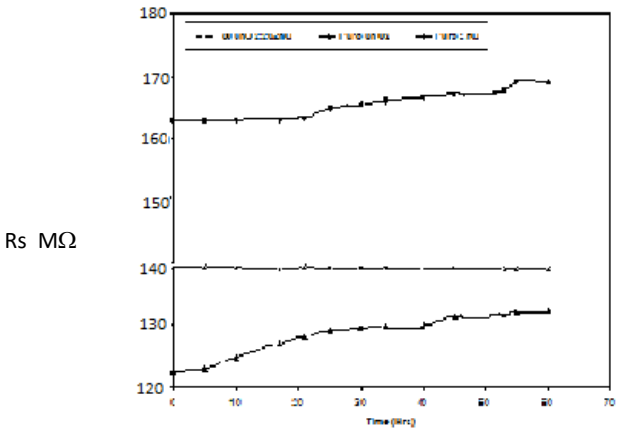


Fig. (7): Stability of the sensor

Rate of change of resistance of the sensor with respect to time defines the stability of the sensor. A sensor should be more stable for its better response. The changes in the resistance for multilayer sensor ($80SnO_2$:20ZnO) [13-14] and pure samples are shown in the fig. (7).

From fig. (7), it is observed that resistance of multilayer sensor does not change drastically as that in case of pure samples. This shows that multilayer sensor is more stable than other.

CONCLUSION

From XRD and SEM characterization it is concluded that the crystallite size of $80SnO_2$:20ZnO/PPy/Al_2O_3 multilayer is smaller and it is more porus and hence has greater surface area and therefore shows greater response to CO_2 gas. Screen printing technique is the easiest for the preparation of sensor. $80SnO_2$:20ZnO/PPy/Al_2O_3 multilayer sensor shows good stability than pure samples and dynamic response is also fast.

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Conflicts of interest: The authors stated that no conflicts of interest.

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