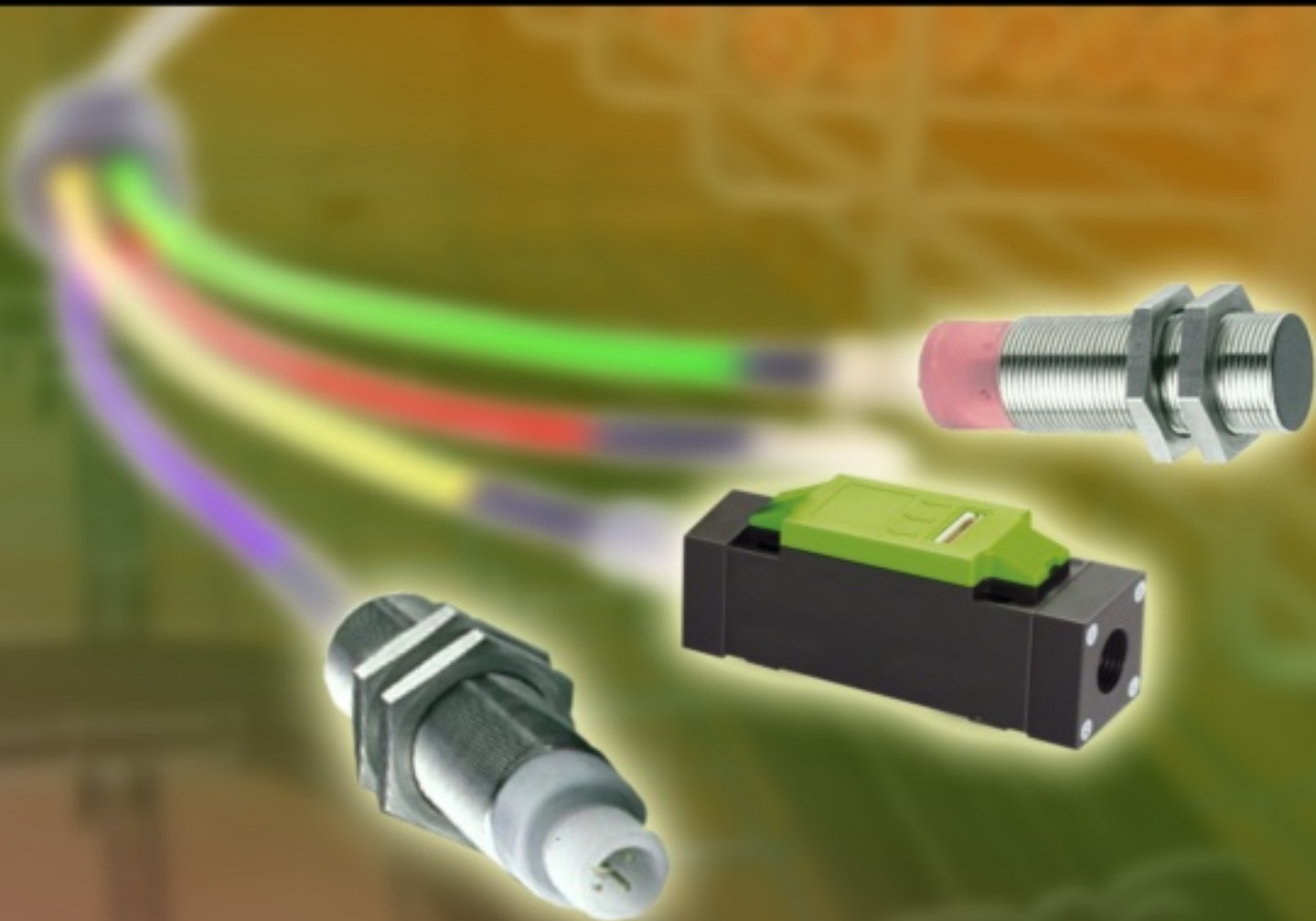


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SnO₂/PPy Screen-Printed Multilayer CO₂ Gas Sensor

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Abstract: Tin dioxide (SnO₂) plays a dominant role in solid state gas sensors and exhibit sensitivity towards oxidizing and reducing gases by a variation of its electrical properties. The electrical conducting polymer-polypyrrole (PPy) has high anisotropy of electrical conduction and used as a gas sensor. SnO₂/PPy multilayer, pure SnO₂, pure PPy sensors were prepared by screen-printing method on Al₂O₃ layer followed by glass substrate. The sensors were used for different concentration (ppm) of CO₂ gas investigation at room temperature (303 K). The sensitivity of SnO₂/PPy multilayer sensor was found to be higher, compared with pure SnO₂ and pure PPy sensors. The multilayer sensor exhibited improved stability. The response and recovery time of multilayer sensor were found to be ~2 min and ~10 min respectively. *Copyright © 2007 IFSA.*

Keywords: Tin dioxide, Polypyrrole, Multilayer sensor, CO₂

1. Introduction

Carbon dioxide (CO₂) is one of the most common greenhouse gases and is a primary component among corrosive gas species and affects corrosion rate of equipment used in chemical processing. There is essential need to control it. The control of CO₂ gas also plays an important role in agricultural sector [1, 2]. Literature survey reveals that SnO₂ is a promising material, used in different gas sensors. The reaction of SnO₂ in air with different test gases is relatively extensively studied. Generally CO, CH₄ and NO_x are the most used gases to characterize the performance of sensor, because of their different sensing mechanisms [3]. CO and CH₄ introduce electrons in the conduction band (decrease sensor resistance) by reacting with the SnO₂ surface, whereas NO_x removes electrons and increase sensor resistance [4].

SnO₂ based gas sensors are sensitive only at high temperature (>200⁰C), and less sensitive and selective for low gas concentration. Many researchers [5, 6] have developed conductive polymers for gas sensor applications, such as polyacetylene (PAC), polypyrrole (PPy), and polyaniline (PANI). The advantages of conducting polymers gas sensor are low cost, suitable for fabrication on various substrates, and near room temperature operation [7, 8]. PPy is highly sensitive to gases but it shows saturation effect at higher concentration of gases [9]. Zheng Jiao *et al* [10] reported that SnO₂/Fe₂O₃ multilayer thin film exhibited improved selectivity and stability, but slightly reduced sensitivity, compared with SnO₂ and Fe₂O₃ thin films.

The aim of this work is to study CO₂ gas sensing properties of SnO₂/PPy multilayer sensor, prepared by screen-printing method on Al₂O₃ layer followed by glass substrate. SnO₂ and PPy powder samples were analyzed by XRD.

2. Experimental

SnO₂ powder (AR grade) and Al₂O₃ powder (AR grade) were calcined at 800⁰C for 4h. Fine powder of SnO₂ and Al₂O₃ were formed in agate and mortar. PPy in powder form was prepared by using 4.290-weight ratio of pyrrole monomer to oxidant (FeCl₃) and methanol as a solvent. The reaction was carried out by procedure reported in reference [11]. The SnO₂ and PPy powder samples were characterized by XRD. The XRD pattern of powder samples was recorded on Philips-1730 (PANalytical) X-ray diffractometer using CuK α radiation ($\lambda = 1.54 \text{ \AA}$). The diffractogram was in the term of 2 θ in the range 5- 99⁰.

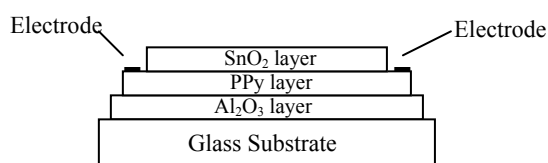


Fig. 1. The structure of SnO₂/PPy multilayer sensor.

The binder for screen-printing was prepared by thoroughly mixed 8 wt% butyl carbitol with 92wt% ethyl cellulose. Multilayer sensor was prepared in three steps. In first step, paste of Al₂O₃ powder was prepared and screen-printed onto a chemically cleaned glass substrate. The glass plate then dried at room temperature for 24h and heated at 150⁰C for 2h. The Al₂O₃ layer mainly provides the high thermal conductivity and mechanical support. In second, paste of PPy powder was screen printed onto Al₂O₃ layer, and dried at room temperature for 24h and heated at 150⁰C for 3h, so that removed the volatile organic solvent. In third step, paste of SnO₂ powder, screen printed onto PPy layer and dried at room temperature for 24h, heated at 200⁰C for 3h. For resistance measurement, electrical contacts of silver paint were deposited on adjacent sides of PPy layer. The structure of SnO₂/PPy multilayer sensor is shown in fig. 1. Thickness of PPy layer and SnO₂ layer was measured by Digimatic Outside Micrometer (series-293, Japan) having resolution $\pm 0.001\text{mm}$ and found to be 9 μm and 11 μm . The electrical resistance was measured by using voltage drop method adopted by Yawale *et al* [12].

3. Results and Discussion

3.1. XRD analysis

Figure 2 (a) and (b) shows XRD pattern of SnO₂ and PPy. From Figure 2 (a), it is observed that XRD pattern contain 8-10 peaks. These are prominent peaks of SnO₂. The (h k l) values are obtained by using 2θ and d-values from XRD pattern. SnO₂ has one stable state called as cassiterite or rutile. From Figure 2 (b), PPy is mainly amorphous in nature. A broad peak is observed at about 2θ = 12⁰, which is characteristic peak of amorphous PPy. However the peak obtained at 26⁰ matches with d-value (3.38Å) of FeCl₃. The average grain size, determined from XRD pattern using Scherrer formula [13] of these materials, is about 60 nm for SnO₂ and 73 nm for PPy respectively.

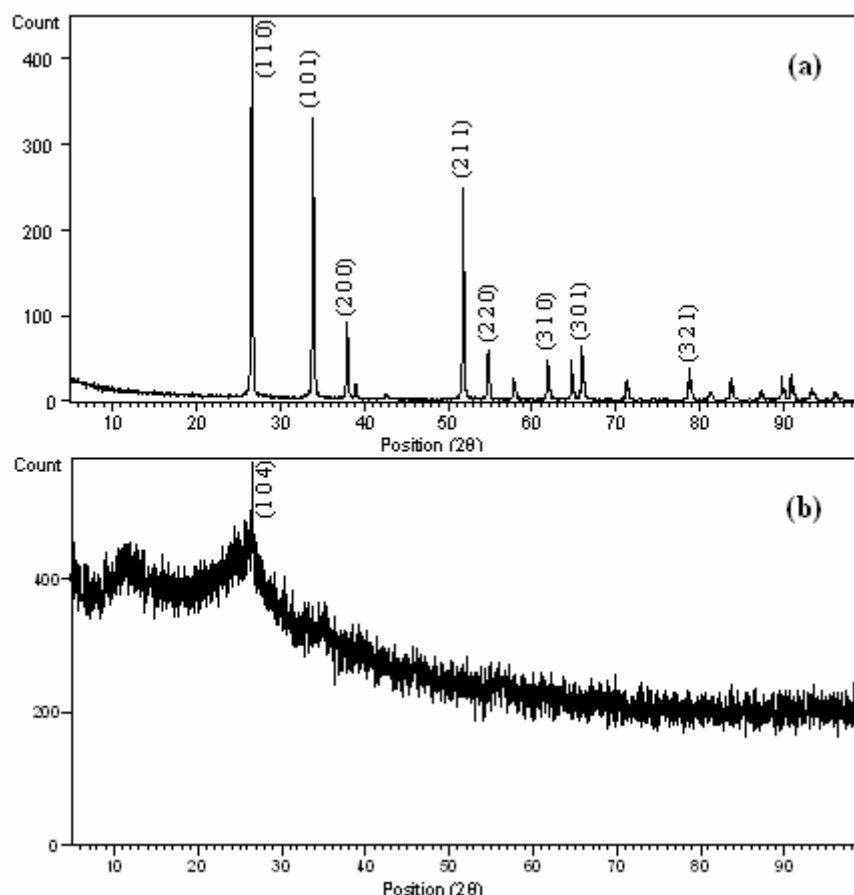


Fig. 2. XRD (a) SnO₂ calcined at 800⁰C for 4h and (b) Chemically synthesized PPy.

3.2. Sensitivity Measurement

The sensitivity is defined as

$$S = \frac{R_g - R_a}{R_a} = \frac{\Delta R}{R_a}, \quad (1)$$

where R_a and R_g are the resistance of sensor in air and the CO₂ gas respectively.

The resistance of SnO₂/PPy multilayer, pure SnO₂ and pure PPy sensors is increases with CO₂ gas concentration. The relationship between sensitivity and CO₂ gas concentration for SnO₂/PPy

multilayer, pure SnO₂ and pure PPy sensors at room temperature (303 K) displays in Figure 3. SnO₂/PPy multilayer sensor shows a high sensitivity to CO₂ gas, compared with pure SnO₂ and pure PPy sensors. SnO₂/PPy multilayer sensor exhibits a good dependence on CO₂ gas concentration up to 1100 ppm, where it reaches a saturation level. With a fixed surface area, a lower gas concentration implies a lower coverage of gas molecules on the surface. An increase in the gas concentration raises the surface coverage eventually leading to saturation level, thus determining the upper limit of detection.

3.3. Dynamic Response

The dynamic response of SnO₂/PPy multilayer sensor for 100 ppm, 300 ppm and 500 ppm CO₂ gas concentration at room temperature is shown in Figure 4. The response (τ_{res}) and recovery (τ_{rec}) time are two important parameters to characterize a sensor. The response time is defined as the time taken to reach 90% of the response when ppm of gas is changed. The recovery time is defined as the time taken to reach 90% of the recovery when gas is turned off. It is clearly shows that the response is fast (~ 2 min), but to recover the sensor takes longer time (~ 10 min). For a breath analysis sensor that requires high sensitivity and fast response, the slow recovery may not pose any practical challenge.

3.4. Stability

Stability is defined as the change of sensor resistance with time. The resistance change of SnO₂/PPy multilayer sensor in air is compared with that of pure SnO₂ sensor displays in Table 1. The resistance of SnO₂/PPy multilayer sensor is stable after 41h, while resistance of pure SnO₂ sensor continues to change even after 53 h. In other words, stability of the SnO₂/PPy multilayer sensor is better than that of pure SnO₂ sensor.

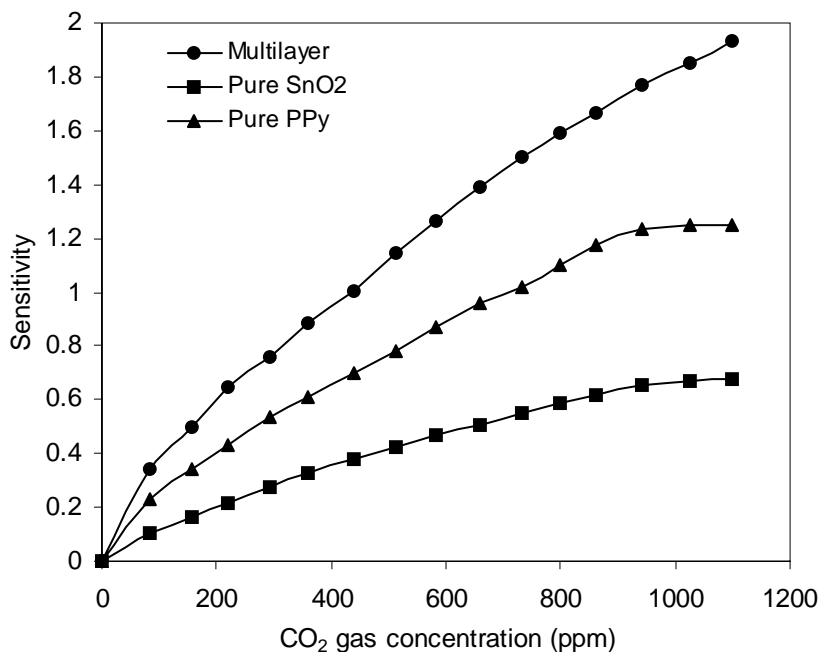


Fig. 3. Sensitivity of SnO₂/PPy multilayer, pure SnO₂ and pure PPy sensors as a function of CO₂ gas concentration at room temperature.

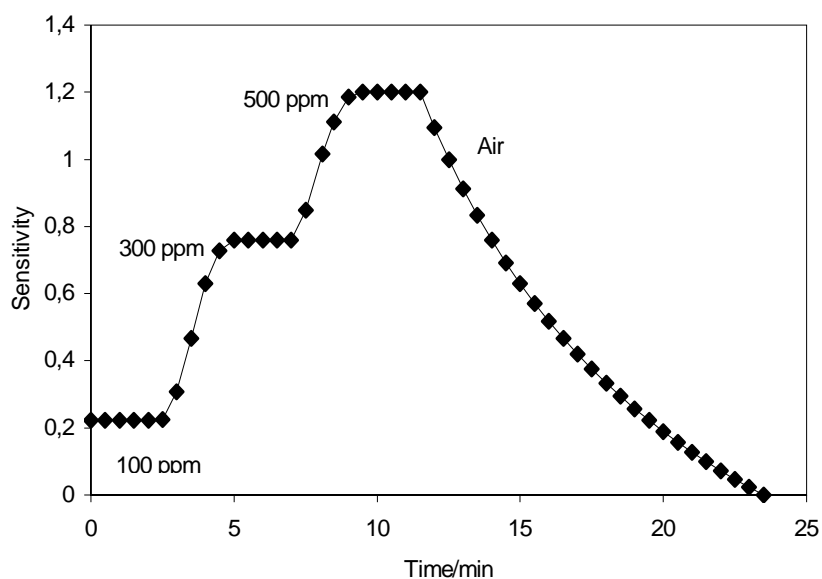


Fig. 4. Dynamic response of SnO₂/PPy multilayer sensor for 100, 300 and 500 ppm CO₂ gas concentration at room temperature

Table 1. The resistance change of SnO₂/PPy multilayer and pure SnO₂ sensor in air.

Time	0	5 h	10 h	21 h	29 h	37 h	41 h	45 h	49 h	53 h
R _M (10 ³ MΩ)	277	114	250	263	357	333	312	312	312	312
R _S (MΩ)	104	177	660	569	109	100	421	168	102	67

R_M = resistance of SnO₂/PPy multilayer sensor; R_S = resistance of pure SnO₂ sensor

There may be a possibility of current conduction part (PPy layer) and the gas sensitive part (SnO₂ layer) for obtaining better stability of SnO₂/PPy multilayer sensor. The current conduction part needs to be stable for long time. PPy is suitable for this purpose because of its current conduction stability [14]. The sensitive part needs to interact with selective gas.

The possible mechanism for CO₂ gas detection in SnO₂ material is based on reactions that occur at the sensor surface, resulting in a change in concentration of adsorbed oxygen. At lower temperature (<150°C), oxygen adsorption at the surface is mainly in the form of O₂⁻. Oxygen ions adsorb onto the surface of material removes electrons from the bulk and create a potential barrier that limits electron movement and resistivity. When exposed to an oxidizing gas such as CO₂ then it is chemisorbed on bridging oxygen atoms with the formation of a surface carbonate [15], subsequently increasing the barrier height and the resistivity. In case of PPy, response of sensor to CO₂ gas is due to small kinetic diameter of CO₂ molecule (0.33nm) [16]. The CO₂ molecule may form weak bond with π-electron of PPy surface, which results in a decrease of conductivity or an increase in sensor resistance [17].

4. Conclusions

From these studies the following conclusion can be drawn.

1. Screen-printing method is a simplest method for preparation of multilayer sensor.
2. Multilayer sensor gave high sensitivity to CO₂ gas, compared with pure SnO₂ and pure PPy sensors.

3. Multilayer sensor showed better stability than pure SnO₂ sensor.
4. Dynamic response of multilayer sensor showed ~ 2 min response time and ~ 10 min recovery time.

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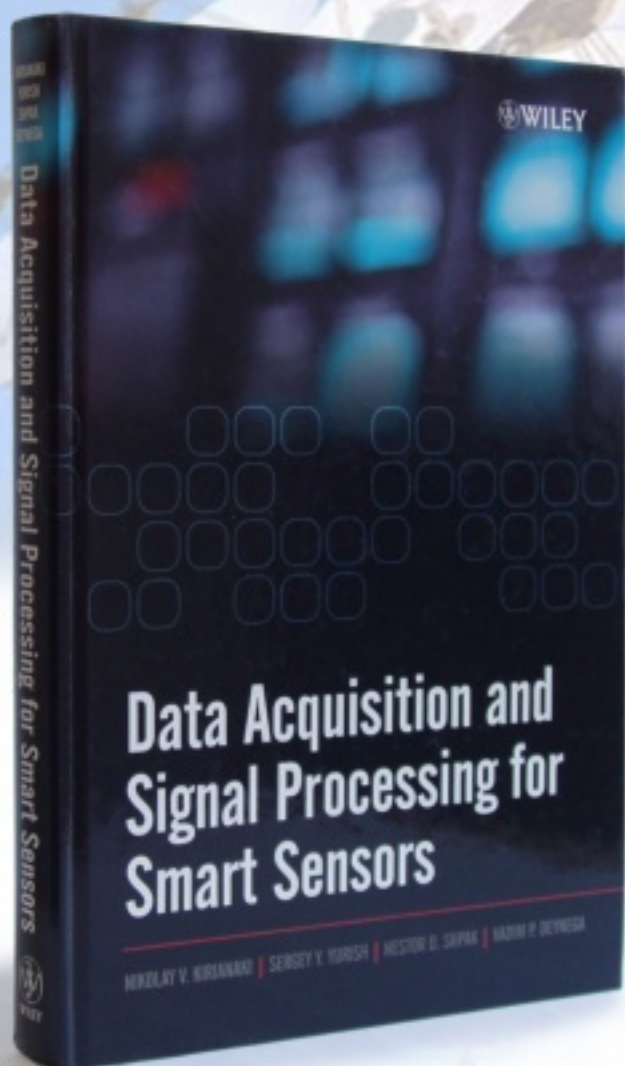
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