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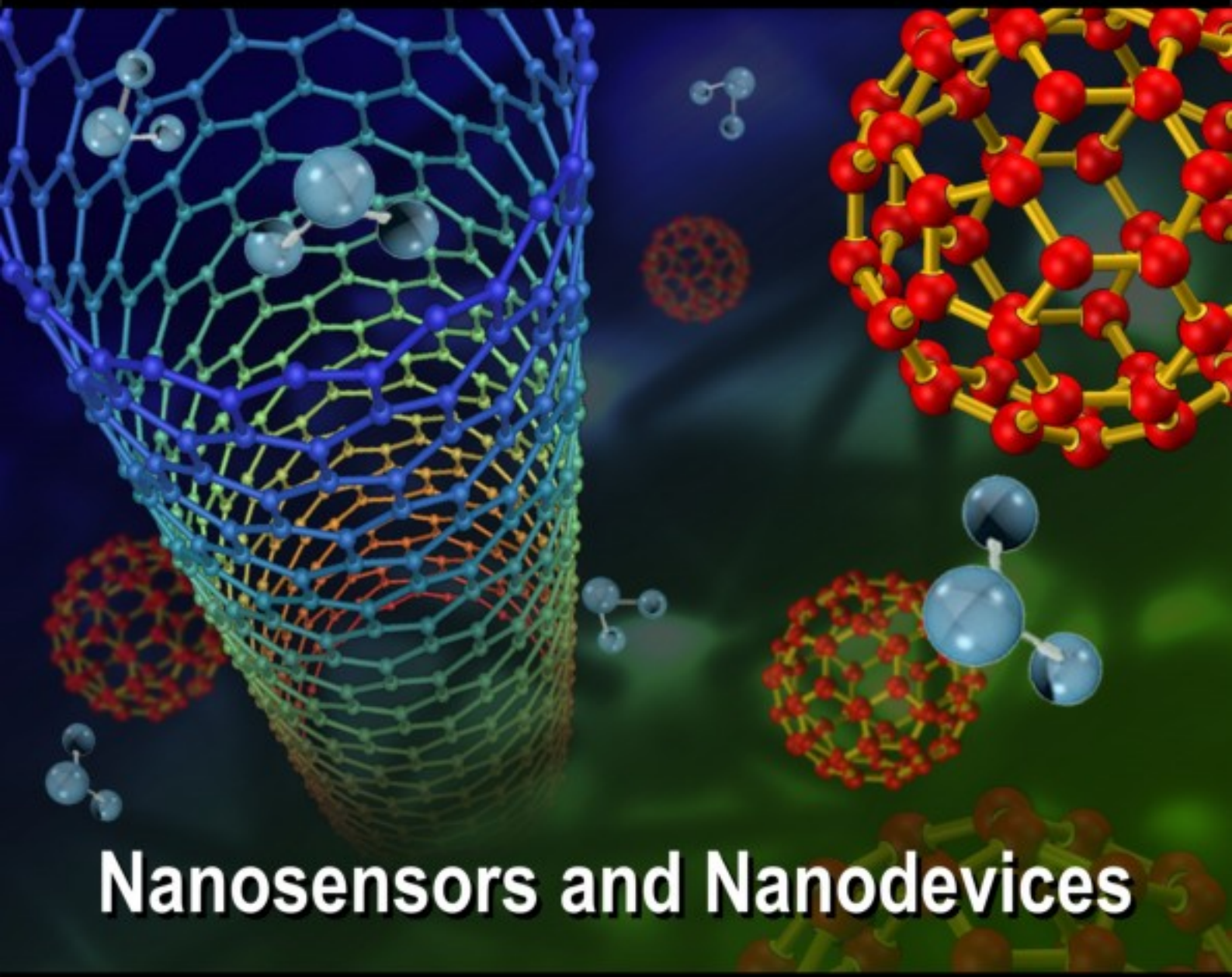

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Synthesis of Nanocrystalline SnO_2 Modified TiO_2: a Material for Carbon Monoxide Gas Sensor

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Abstract: Nanocrystalline SnO_2 doped TiO_2 having average crystallite size of 45-50 nm were synthesized by the sol-gel method and studied for gas sensing behavior to reducing gases like CO, liquefied petroleum gas (LPG), NH_3 and H_2. The material characterization was done by using X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FT-IR) and scanning electron microscope (SEM). The sensitivity measurements were carried out as a function of different operating temperature in SnO_2 doped TiO_2. The 15 wt.% SnO_2 doped TiO_2 based CO sensor shows better sensitivity at an operating temperature 240°C Incorporation of 0.5 wt% Pd improved the sensitivity, selectivity, response time and reduced the operating temperature from 240°C to 200°C for CO sensor.

Keywords: TiO_2, SnO_2, CO sensor, Selectivity, Response time

1. Introduction

Nanometer sized materials, which have high surface activity due to their small particle size and enormous surface area, have been widely studied in the field of gas sensors in recent years [1-3]. A large number of metal oxides, i.e. SnO_2, ZnO, WO_3, TiO_2, Fe_2O_3, etc were reported to be sensitive to certain gas species. Semiconductor metal oxides as gas sensing materials have been extensively studied for a long time due to their advantageous features, such as good sensitivity to the ambient conditions and simplicity in fabrication [4-6]. The electrical conductivity of a semiconducting metal oxide can be altered by adsorption of gases from the ambient. This property of semiconducting oxides

has been exploited and used in sensors fabricated for detection of inflammable and toxic gases such as hydrocarbons, H_2, CO and NO_X [7-9].

The gas-sensing mechanism of metal oxide materials is based on the reaction between the adsorbed oxygen on the surface of the materials and the gas molecules to be detected. The state and the amount of oxygen on the surface of materials are strongly dependent on the microstructure of the materials, such as specific area, particle size, as well as the film thickness of the sensing film. In order to obtain gas sensors with good performance, the recent research works [10-12] were devoted to nano-materials because they have high specific area and contain more grain boundaries. Many approaches have been made to modify the sensing properties of these semiconductor oxide gas sensors in order to achieve high sensitivity and selectivity. In this direction different approaches are adopted such as addition of metal oxides and noble metals are well known for enhancing the rate of response and raising selectivity to a single gas [13-15].

Tin titanate crystallizes in a rutile structure showing n-type semiconductivity [16]. Tang *et al*. [17-19] have studied the behaviour of anatase TiO_2 thin film sensors and found their properties quite different from those of the extensively studied and used rutile phase. TiO_2 crystllizes in three modifications of rutile, anatase and brookite. Out of these, films of rutile phase of TiO_2 are used in gas sensing applications. TiO_2 and SnO_2 have a wide range of applications in gas sensors [20-26]. It is shown that surface modification of semiconducting materials like SnO_2 and additives incorporated [27-29] enhanced the gas sensing properties largely.

Carbon monoxide (CO) is one of the most dangerous gases in air pollution and human life. CO is produced by incomplete combustion of fuels and commonly found in the emission of automobile exhausts, the burning of domestic fuels, etc. It is highly toxic and extremely dangerous because it is colorless and odorless. CO sensors are, therefore, required in various situations including the detection of smoldering fires. CO gas sensors have already been developed [30-33].

In this paper, we report for the first time CO gas sensing phenomena observed in SnO_2 doped TiO_2 thick films prepared by sol-gel method. It has been observed that there is an increase in gas response when exposed to CO than other reducing gases like liquid petroleum gas (LPG), hydrogen (H_2), and ammonia (NH_3). We have also tried to improve the response for CO gas by modifying by adding noble metals Pd in order to improve the performance.

2. Experimental Details

Nanostructured undoped and SnO_2 doped TiO_2 was synthesized by the modified sol-gel technique using citric acid as the chelating agent. Briefly, an aqueous solution containing citric acid and ethylene glycol (mass ratio 60:40) was prepared. A known amount $TiC!_4$ and $SnC!_2$ of were added in this solution. This solution was then heated to evaporate water slowly; the residual viscous oily mass was then heated at a temperature 80°C, to polymerize citric acid and ethylene glycol by polycondensation resulting into a solid resin. The resin formed was then heated to burn off the polymer matrix. The resulting resin was treated in a pressure vessel at a calcinations temperature 130°C over 12 h to fully evaporate highly combustible species in the glassy mass and to burn down most of the organic constituents. The resulting material had the appearance of a dark brown sticky ash, which was slightly ground into a powder by a Teflon rod. The ash-obtained powder is referred to as the "precursor" hereafter and was heat-treated 650°C for 6 h.

An appropriate quantity of $PdCI_2$ was added to SnO_2 modified TiO_2 and dissolved in deionizer water. This mixture was vigorously stirred and slowly dried on a water bath. The dried compound was ground to a fine powder and calcined at 200°C for 1 h to decompose the chloride.

The powder constituents were characterized by X-ray diffraction (XRD) using a Siemens D5000 differactometer operating with CuKα radiation. The crystallite sizes of powders were calculated according to Scherrer's equation,

$$d = k\lambda / \beta \cos \theta , \quad (1)$$

where, k is 0.9, λ is X-ray wavelength, β is full width at half-maximum in radians and 9 is the diffraction peak position.

The samples calcinied at 650°C was analyzed by infrared spectroscopy (FT-IR) (Magna- IR 760 spectrometer, Nicolet). The optimum synthesizing condition was established based on the changing IR spectra. The fine powder was observed under a JEOL, JSM-5600 N scanning electron microscope (SEM) by dispersion it on a carbon paste to determine the particle size and morphology.

For gas sensing properties, the calcinated powder was then mixed with 2% PVA (polyvinyl alcohol) as a binder and 5% ethanol as a solvent, the resulting paste was coated over a cylindrical alumina tube of length 15mm and diameter 5 mm. A small Ni-Cr alloy coil was placed through the tube as a heater, which provided operating temperature at 50-350°C. The temperature was controlled by adjusting the heating power.

The gas sensitivity (S) was defined.

$$S = (Ra-Rg) / R_a = \Delta R/R_a , \quad (2)$$

where R_a and R_g are the resistance of sensor in air and the test gas respectively. The sample gas concentration is 1000 ppm.

3. Result and Discussion

3.1. Structural Characteristics

Fig. 1 (a) shows the XRD patterns nanocrystalline TiO_2 calcinated at 650°C. All prominent peak corresponds to (d= 3.53, 2.431, 2.378 and 1.699) and their corresponding planes (101), (103), (004) and (105) respectively, Small peaks observed at 55° (d=1.66), 62.75° (d= 1.4808) and 75° (d=1.26) correspond to (211), (204) and (215) plans are observed which shows good crystalline quality and corresponds to anatase structure of TiO_2. Hayakawa *et. al.* [21] are also suggested anatase phase of TiO_2 prepared by precursor method. Fig. 1 (b) shows 15 wt. % SnO_2 doped in TiO_2 and calcinated at 650°C shows good crystalline quality and complete phase formation was observed. The crystallite size was calculated from Scherrer formula applied to major peak and was found to be about 45-50 nm. No extra peaks are observed due to addition of 15 wt. % addition of SnO_2.

Fig. 2 (a, b) shows the FTIR absorption spectra of TiO_2 and 15% SnO_2 doped TiO_2 samples respectively calcinied at 650°C. The broad absorption peak observed in the region of 500-800 cm^{-1} fall in the region corresponding to the vibrations of the type Ti-O-M (M=Ti or in fig 2 (b) M=Sn) ,stretching and deformation modes, respectively. The broad band approximately 3400 cm^{-1} is attributed to the O-H stretching and the band at 1622 cm^{-1} is attributed to adsorbed water. The absorption bands were observed approximately at 2900 cm^{-1}. As can be observed in both the samples, indicates the presence of hydrocarbon species, which could come from remnant alkoxy groups.

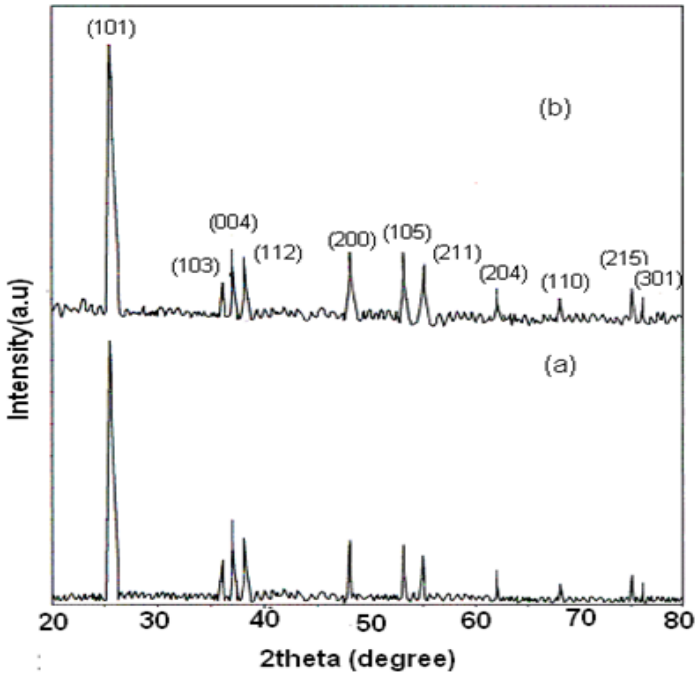

Fig. 1. XRD pattern of (a) undoped TiO_2, (b) 15 wt.% SnO_2 doped TiO_2 calcined at 650°C for 6 h.

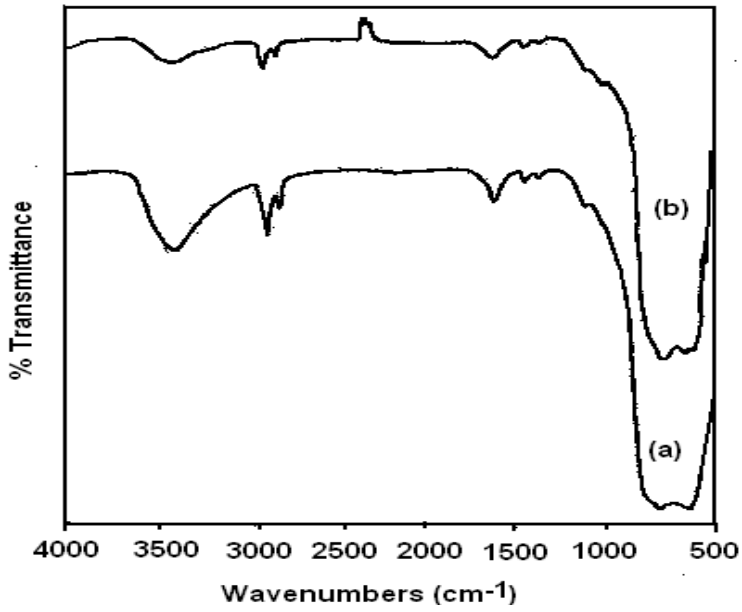

Fig. 2. IR study of (a) TiO_2 and (b) 15 wt.% SnO_2 doped TiO_2, calcined at 650°C for 6 h.

Fig. 3 shows the SEM micrograph of 15 wt.% SnO_2 doped TiO_2 nanoparticle calcined at 650°C. SEM pattern shows spherical particles with uniform grain size distribution. The measured particle sizes are consistent with the sizes calculated by using Scherrer formula of XRD patterns. The particle size is estimated to be between 45 and 50 nm for 15 wt.% SnO_2 doped TiO_2.

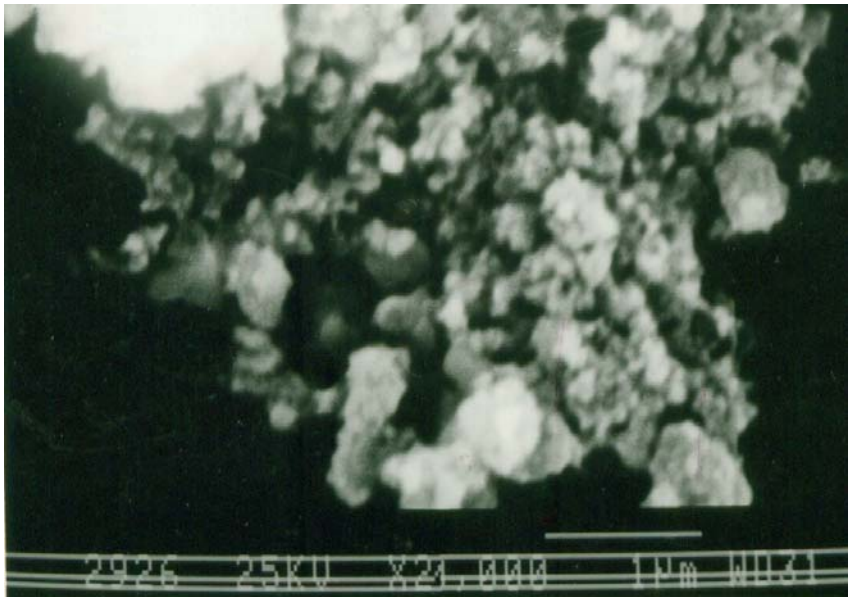

Fig. 3. SEM images of 15 wt.% SnO_2 doped TiO_2, calcined at 650°C for 6 h.

3.2. Gas sensing Characteristics

Fig. 4 shows the response as a function of operating temperature for different wt. % SnO_2 doped TiO_2 for 1000 ppm of CO gas. It is seen from figure that for each sample, the response increased temperature up to 240°C and then decreased at higher temperature. The response of all the samples does not exhibit any significant difference at lower temperatures. At a low operation temperature, the low response can be expected because the gas molecules do not have enough thermal energy to react with the surface adsorbed oxygen species. At higher temperatures the thermal energy obtained was high enough to overcome the potential barrier, and a significant increase in electron concentration resulted from the sensing reaction. The response of semiconductor oxide gas sensor to the presence of a given gas depends on the speed of the chemical reaction on the surface of the grains and the speed of diffusion of the gas molecules to that surface which are activation processes, and the activation energy of the chemical reaction is higher. In this case, at low temperatures the sensor response is restricted by the speed of the chemical reaction, and at higher temperatures it is restricted by the speed of diffusion of gas molecules. At some intermediate temperature, the speed , values of the two processes become equal, and at that point the sensor response reaches its maximum [34], Furthermore, it can be evidenced from the figure that for sample 15 wt.% SnO_2 doped TiO_2, the response was maximum at 240°C, and it was higher than other samples . It is well known that the gas response of the metal-oxide semiconductor sensors is mainly determined by the interactions between a target gas and the surface of the sensors. So it is obvious that for the greater surface area of the materials, the interaction between the adsorbed gases and the sensor surface are stronger, i.e., the gas response is higher [35].

Fig. 5 shows the sensitivity of the 15 wt. % SnO_2 doped TiO_2 sensor element to various test gases as a function of operating temperature. It is seen that the sensor exhibits a very high degree of selectivity by sensing only CO at an operating temperature of 240°C even in the presence of other interfering gases like liquefied petroleum gas (LPG), NH_3 and H_2. For further improvement in the sensitivity and impart selectivity of 15 wt.% SnO_2 doped TiO_2 by adding small quantity of noble metal Pd.

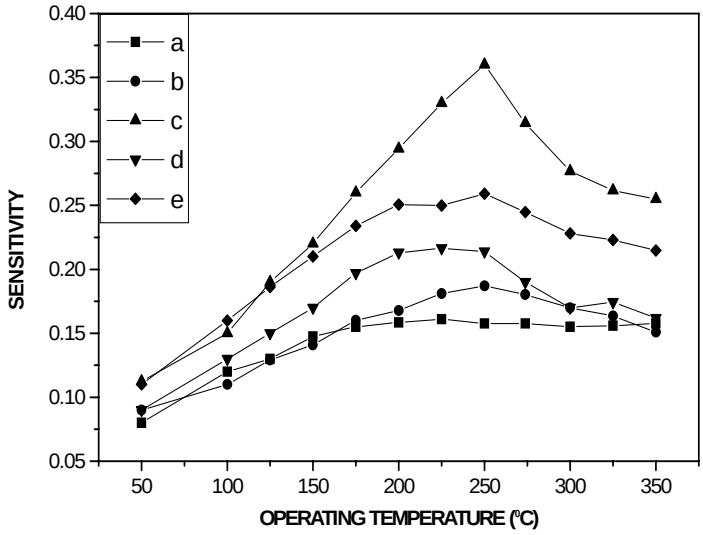

Fig. 4. Response as a function of operating temperature for undoped TiO_2 and different wt.% of SnO_2 - (a) 0 (b) 5 (c) 15 (c) 10 and (d) 20.

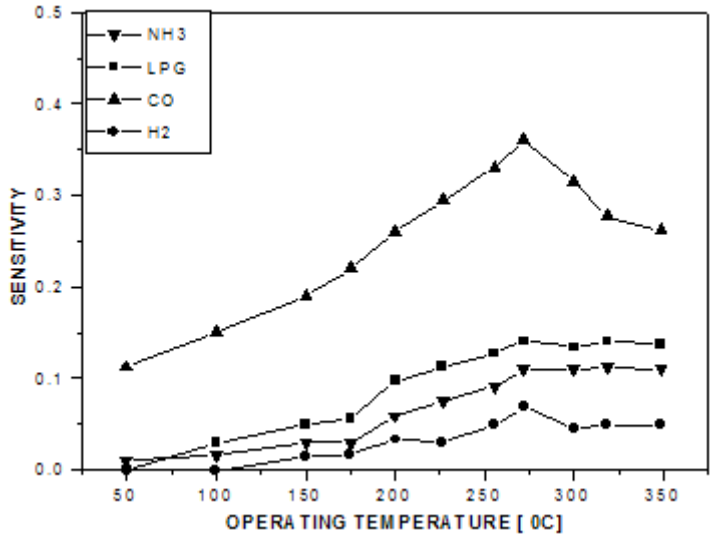

Fig. 5. Response as a function of operating temperature for 15 wt.% SnO_2 doped TiO_2 for CO, LPG, H_2 and NH_3.

Fig. 6 shows the sensitivity versus operating temperature of 0.5 wt.% Pd incorporated to 15 wt.% SnO_2 doped TiO_2 for different gases. It shows highest sensitivity and selectivity at about 0.95 for an operating temperature 200°C. A rapid increase in the sensitivity was observed as the operating temperature was increased to 200°C, and reached a maximum and decreased thereafter with further increase in operation temperature. At a low operating on temperature, the low sensitivity can be expected because the CO molecules do not have enough thermal energy to react with the surface adsorbed oxygen species, O_2^-, i.e. reaction rate of Eq. (3) is essentially low [36].

$$2CO_2 + O_2^- = 2CO_2 + e^- \quad (3)$$

While as T_{op} was increased to 200°C , the adsorbed oxygen converted from O_2^- to O^- and the CO sensing reaction was then expressed as Eq. (4)

$$CO + O^- = CO_2 + e^- \quad (4)$$

The increase in sensitivity for T_{op} above 200°C can be attributed to the fact that the thermal energy obtained was high enough to overcome the activation energy barrier to there action and a significant increase in electron concentration resulted from the sensing reaction as shown in Eq. (4), in which the maximum sensitivity of 0.95 was found at 200^0C. While the reduction in sensitivity above 200°C was due to the difficulty in exothermic CO gas adsorption, therefore, an optimum operating temperature should be considered for obtaining a high sensitivity.

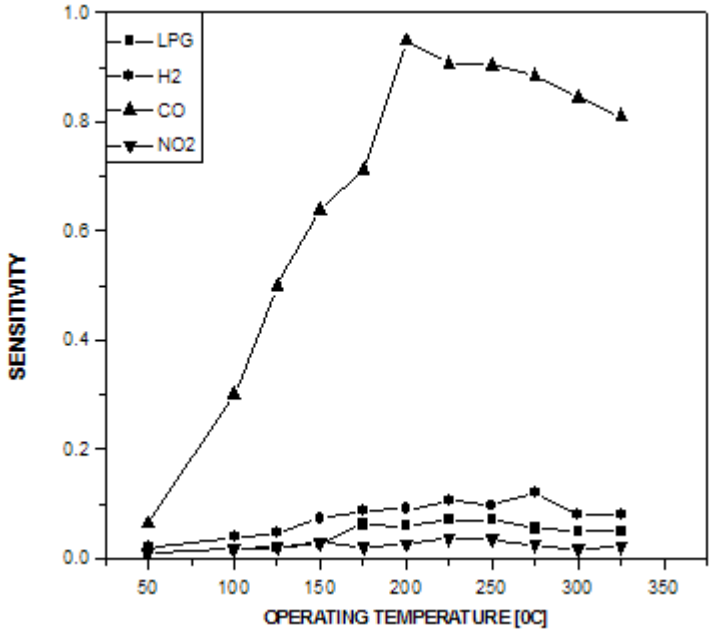

Fig. 6. Response as a function of operating temperature for Pd incorporation in 15 wt.% SnO_2 doped TiO_2 for CO, LPG, H_2 and NH_3.

Fig. 7 (a and b) shows the response characteristics of 15 wt. % SnO_2 doped TiO_2 and 0.5 wt.% Pd incorporated to 15 wt. % SnO_2 doped TiO_2 systems respectively. It has clearly seen that the sensor element reaches to its maximum value of sensitivity in less than 1 min. apart for the low value of rise time and full time the maximum value of the sensitivity i.e. the saturation value is also much higher in 0.5 wt. % Pd incorporated in 15 wt.% SnO_2 doped TiO_2 as compared to that 15 wt.% SnO_2 doped TiO_2.

The effect of Pd is seen not only in increasing the sensitivity of CO considerably but also the rate of response. The variation in sensitivity of 0.5 wt.% Pd incorporated to 15 wt.% SnO_2 doped TiO_2 toward CO concentration (in ppm) at 200°C is shown in Fig.8. In general, the sensitivity of the gas sensor increases with the increase of the concentration of sensing gas in air. For the gas sensor investigated, the magnitude of sensor response increased linearly with CO concentration from 50 to 200 ppm and it is observed that the sensor have tendency to saturated at gas concentration above 200 ppm. The highest sensitivity was observed at 200 ppm.

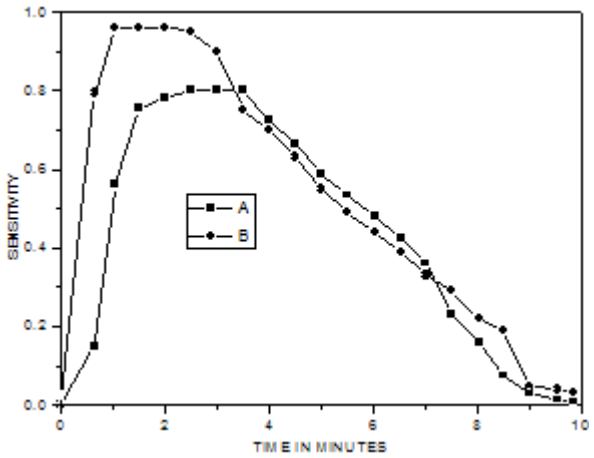

Fig. 7. Response characteristics of (a) 0.5 wt.% Pd incorporated in 15 wt.% SnO_2 doped TiO_2 and (b) 15 wt.% SnO_2 doped TiO_2 at 200°C.

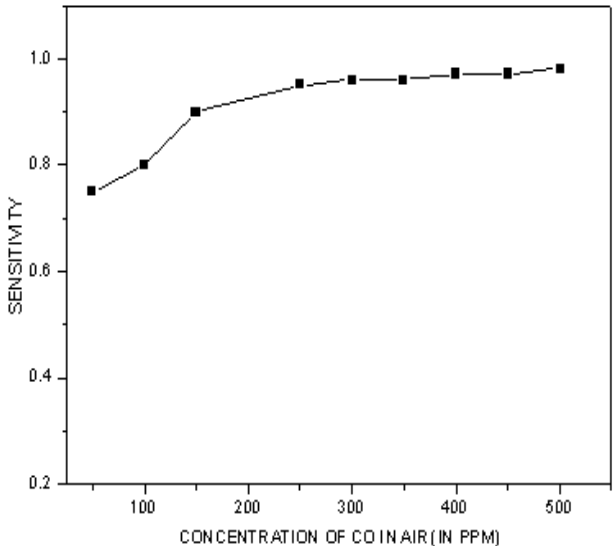

Fig. 8. Variation in response for 0.5 wt.% Pd incorporated in 15 wt.% SnO_2 doped TiO_2 gas sensor as a function of gas concentration for CO in ppm.

4. Conclusions

We have studied the sensitivity and selectivity of undoped and SnO_2 doped TiO_2 for CO sensor by adding small concentration of noble metal Pd. 15 wt.% SnO_2 doped TiO_2 shows high response and selectivity for CO gas at an operating temperature of 240°C. XRD, SEM and FTIR revealed the phase composition of anatase structure of TiO_2 shows good crystalline quality with a grain size 45-50 nm. Incorporation of 0.5 wt% Pd to15 wt% SnO_2 doped TiO_2 improves the gas response, response time and reduced the operating temperature for CO sensor at an operating temperature 200°C.

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Guide for Contributors

Aims and Scope

Sensors & Transducers Journal (ISSN 1726-5479) provides an advanced forum for the science and technology of physical, chemical sensors and biosensors. It publishes state-of-the-art reviews, regular research and application specific papers, short notes, letters to Editor and sensors related books reviews as well as academic, practical and commercial information of interest to its readership. Because it is an open access, peer review international journal, papers rapidly published in *Sensors & Transducers Journal* will receive a very high publicity. The journal is published monthly as twelve issues per annual by International Frequency Association (IFSA). In additional, some special sponsored and conference issues published annually.

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Contributions are invited on all aspects of research, development and application of the science and technology of sensors, transducers and sensor instrumentations. Topics include, but are not restricted to:

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- Smart sensors and systems;
- Sensor instrumentation;
- Virtual instruments;
- Sensors interfaces, buses and networks;
- Signal processing;
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