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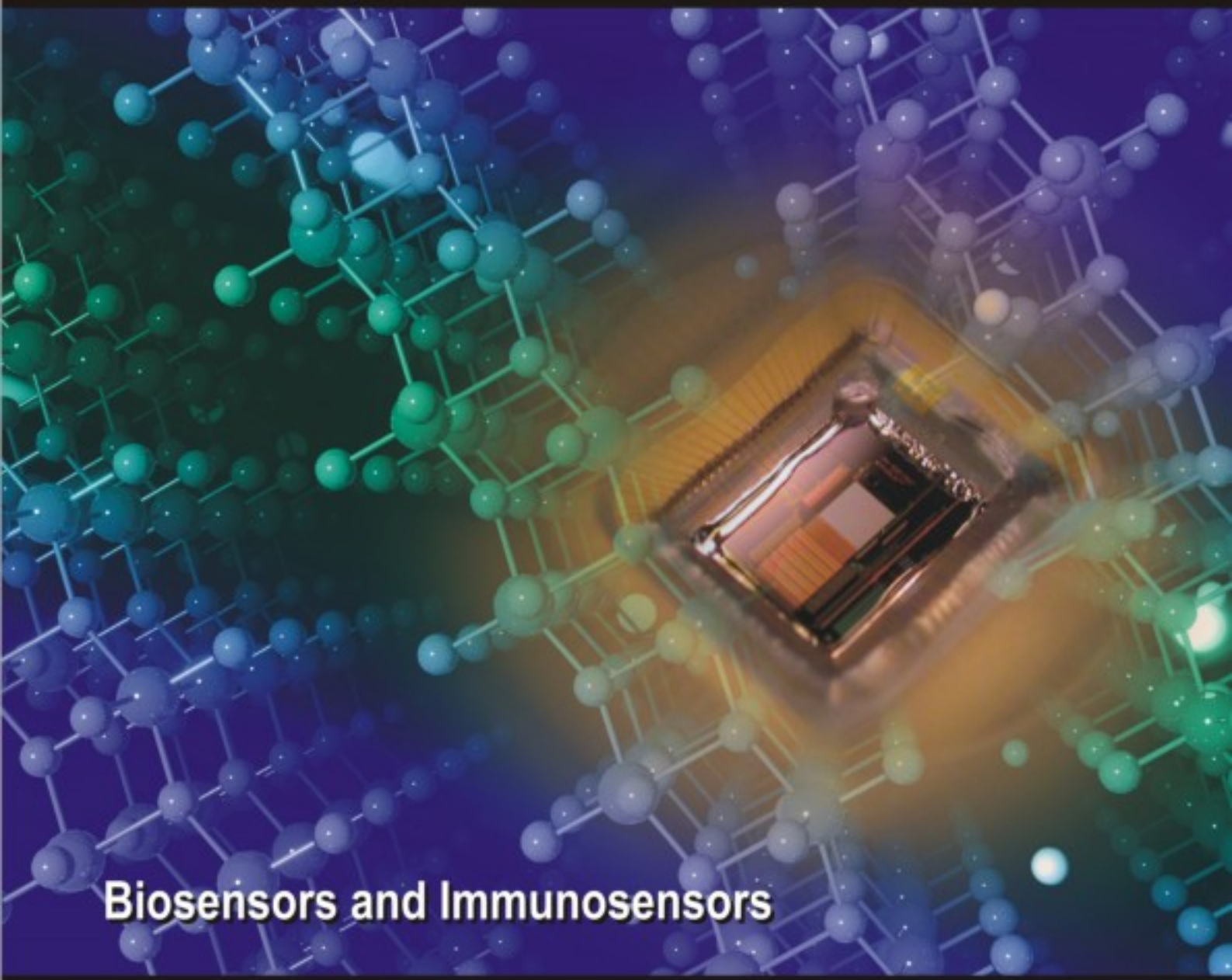

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Molybdenum Doped SnO_2 Thin Films as a Methanol Vapor Sensor

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Abstract: The molybdenum doped SnO_2 thin films were synthesized by conventional spray pyrolysis route and has been investigated for the methanol vapor sensing. The structural and elemental composition analysis of thin films was carried out by X- ray diffraction and Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray spectroscopy (EDAX).The XRD spectrum revealed that the thin films have the polycrystalline nature with a mixed phase comprising of SnO_2 and MoO_3. The scanning Electron Microscopy (SEM) clears that the surface morphology observed to be granular, uniformly covering the entire surface area of the thin film. The methanol vapor sensing studies were performed in dry air at the different temperatures. The influence of the concentration of Molybdenum and operating temperature on the sensor performance has been investigated.
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Keywords: Gas sensor, Spray pyrolysis, Methanol, Tin oxide, Molybdenum trioxide.

1. Introduction

Tin dioxide is an n-type semiconductor with a band gap of approximately 3.6 eV. When the SnO_2 is exposed to a reducing atmosphere, the interaction between the gas and the adsorbed oxygen result in oxidation at the surface and in a decrease of chemisorbed oxygen concentration and the electrons that have been trapped as negatively charged ions are released. Due to the n-type behavior of SnO_2, the electrical conductivity increases when in contact with a reducing gas like CO and / or CH_4, on the contrary decreases in the presence of an oxidizing gas like NO_2. [1, 2].

The foreign metal atoms on SnO_2 serve as adsorption sites with ionic bond strength depending on their electronegativity. Therefore in order to enhance the sensing properties of the tin dioxide material, introduction of noble metal additives is usually performed. The dopant, not only act as a catalyst, but also modifies the electrical transport properties of the sensor by introducing new states in the band structure of the active materials and the surface morphology of the material and the size of the crystalline grains. The most important effects of noble metal addition are the increase of the maximum sensitivity, rate of response and lowering the operating temperature of the sensor. [3-11]. In any case, the contact of the additive with the semiconducting oxide creates a barrier that is fully characterized by the electron affinity of the semiconductor, the work function of the metal and

the density of surface states of the semiconductor that are located inside the energy band gap. This contribution creates a Schottky barrier through the formation of a depletion region in the semiconductor surface in contact with the cluster. As a result the surface states created by the presence of the additives can shift the Fermi level of the semiconductor to that of additive.

In late 1990s, SnO_2 and MoO_3 mixtures were shown to have excellent catalytic properties for selective oxidation of methanol and other organic compounds [12]. It is well known that the addition of metals or metal oxides with catalytic properties can influence on the gas sensing behavior of the SnO_2 material [13-21]. In this case, the addition of MoO_3 to SnO_2 has been proposed as an outstanding alternative to modify the sensor response to certain gas species as the presence of Mo atoms at the surface of SnO_2 changes the acidity performances which varies mainly its reactivity with alcohols, ammonia or amine groups.

Recently, nanocomposites of SnO_2 and MoO_3 have received technological importance for the development of resistive gas sensors as the catalytic characteristics in this nano-binary system are enhanced [22-26], presenting a very high active surface value. SnO_2/MoO_3 composites are n-type semiconductors, just as the constituent oxides [23-25]. The introduction of molybdenum notably reduces the electrical conductivity of SnO_2 in air and according to the previous literature, it may be associated either with the transfer of electrons trapped at oxygen vacancies ($V^{0\bullet\bullet}$ and $V_0^{\bullet}$) to Mo^{6+} ions [25, 26],

$$Mo^{6+} + V_0^{\bullet} = Mo^{5+} + V_0^{\bullet\bullet} \quad (1)$$

$$Mo^{6+} + 2\,V_0^{\bullet} = Mo4^{+} + 2\,V_0^{\bullet\bullet\,\bullet\bullet} \quad (2)$$

or with the formation of Moi^{5+} interstitials in the structure of SnO_2 by the quasi-chemical reaction [23, 24].

$$Mo_{Sn}^{6+} + e' = Moi \quad (3)$$

Thus, it is assumed SnO_2 could contain Mo ions in three different oxidation states [22-24]: Mo^{6+}, Mo^{5+}, and Mo^{4+}. Ivanovskaya et al. [23] from electron spin resonance (ESR) measurement have corroborated that some Mo atoms present Moi^{5+} state in enough concentration to justify the conductivity decrease.

Various researchers have attempted detection of volatile organic compounds (VOCs) [27-29]. Fabrication of SnO_2 based sensor array for recognition of VOCs has been attempted by Dae-Sik Lee and co-workers [28]. The authors have studied the effect of various additives such as Pd, Pt, La_2O_3, CuO, Sc_2O_3, TiO_2, WO_3, ZnO and V_2O_5 on sensitivity and selectivity of SnO_2 sensor array. The authors have observed high and selective sensitivity to the VOCs at 400 ^oC.

Spray pyrolysis is a method for thin film fabrication, which is applicable to almost any inorganic system. Does not require vacuum, which is a great advantage if the technique is to be scaled up for industrial applications, Operates at moderate temperatures in any desirable ambient, thus being a low-cost, fast and environmentally wise technique. Doping with most elements can be conveniently achieved by introducing them in the precursor solution, which can be a simple aqueous solution. Deposition rate and film thickness can be easily controlled over a wide range and film morphology to some extent as well. Virtually any type of substrate can be used, even those unsuitable for most high-energy deposition methods. Unlike the high-energy methods, spray pyrolysis does not causes local overheating that can be harmful for some materials, which conduct heat poorly and melt incongruently. There are no restrictions on substrate dimensions or its surface profile. Multi-layered or patterned film deposition is feasible.

2. Experimental

For the preparation of precursor solution stannic chloride ($SnCl_4$: $5H_2O$) and molybdenum trioxide (MoO_3) were used as source materials. First 1.75 g of stannic chloride powder was dissolved in 10 ml solvent (a mixture of methanol and distilled water in a volume ratio of 4:1) so as to prepare 0.5 M solution. Similarly an aqueous solution of molybdenum trioxide was prepared using solvent (a mixture of methanol and distilled water in a volume ratio of 4:1). In order to achieve chemical doping of molybdenum into the SnO_2 thin film, molybdenum trioxide aqueous solution was mixed with the stannic chloride solution in equal volume ratio i.e. 5 ml each. This 10 ml solution thus prepared was used for spraying.

It is well known that the dopant concentration affects the physical and chemical properties of the thin film. In this regard, to achieve variation in the dopant concentration, instead of increasing the volume ratio of molybdenum trioxide solution (of a given molarity) in the precursor solution, higher molar molybdenum trioxide solution with the same volume ratio was used to make the precursor solution. Thus by dissolving 0.288, 0.576 and 0.864 g powder of molybdenum trioxide in 10 ml solvent, 0.2, 0.4 and 0.6 M solutions were prepared.

The glass substrates were cleaned by chromic acid solution followed by rinsing with double distilled water. The substrates were heated to temperature ~ 400 ^oC and 5 ml of the precursor solution (4 ml of stannic chloride solution and 1 ml of molybdenum trioxide solution) was sprayed over the hot substrates using compressed air as carrier gas. The spray rate was monitored at ~ 2 ml per minute.

The structural and morphological characterizations of the molybdenum doped SnO_2 thin films were performed on X-ray Diffractometer

(D-8 Advance Brucker, Germany) and Scanning Electron Microscope (JEOL JSM-6360) respectively. The SEM images were recorded with accelerating voltage ~ 20 KV and filament current ~ 60 mA.

The sensing characteristics measurements were carried out under controlled ambient of the test gas in a specially designed glass jar. The bell jar was mounted onto a base plate equipped with electrical feed through and a gas inlet port. Fixed quantity of vapor was injected into the jar through a side port using micro-syringe. The thin film resistance was measured using two-probe method, by putting two ohmic contacts (separation ~ 1 cm) on the film surface using conducting silver paste. The sensor element was mounted on a resistive heater, which facilitated variation in film temperature. A tiny chromel-alumel thermocouple was used to measure the film temperature, which was monitored by a temperature controller. Enough time was given for temperature stabilization, before introducing the test gas.

3. Results and Discussion

The X-ray diffraction spectra of the cobalt doped SnO_2 thin films synthesized at 400 ^oC shows well defined diffraction peaks indicating formation of polycrystalline films. A typical X-ray diffraction spectrum of the spray deposited molybdenum doped tin oxide thin film synthesized on glass substrate is depicted in Fig. 1. The XRD spectrum is identical to that reported by various researchers [28, 29]. The diffraction peak indexing was done by comparing the observed 'd' values with the standard data base (JCPD card no. 21-1250).

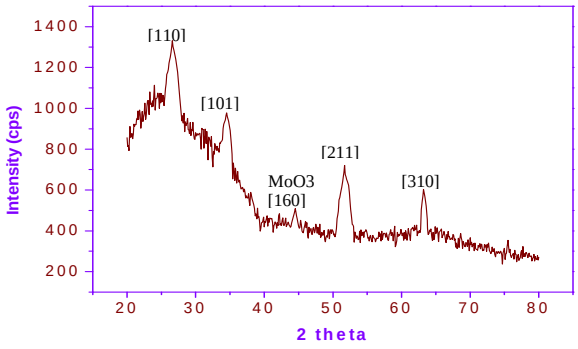

Fig. 1. X-ray diffraction spectrum of Mo doped SnO_2 thin film.

The XRD patterns show a couple of well defined diffraction peaks indicating formation of polycrystalline thin films under the employed experimental conditions. The XRD patterns are identical to those reported in the literature [30, 31]. The diffraction peaks indexing done by comparing the observed d values with the standard JCPD data cards of SnO_2 and MoO_3, clearly revealed formation of the SnO_2 phase with tetragonal structure [32, 33]. The major diffraction planes of SnO2 are found to be (110), (200), (220) and (310), In addition to this a low intense diffraction peak corresponding to MoO_3 (160) is also noticed.

The surface morphology of molybdenum doped SnO_2 thin films was studied using Scanning Electron Microscope. All these films exhibit uniform and granular morphology, covering the entire substrate area. A typical SEM image is shown in Fig. 2. The average grain size derived from the SEM image is found to be ~ 200 nm. The elemental composition obtained from the EDAX spectrum showed presence of MoO_3, tin and oxygen. Atomic weight percentage of molybdenum was found to increase with increasing molarity of molybdenum trioxide.

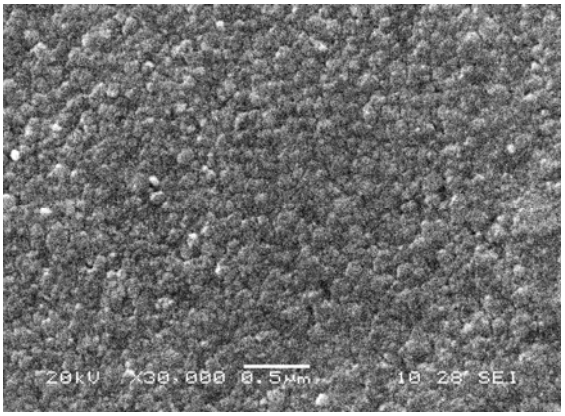

Fig. 2. Scanning Electron Microscope of Mo doped SnO_2 thin film.

The gas sensing characteristics of the molybdenum doped SnO_2 thin films were investigated as a function of operating temperature and methanol vapor concentration. In the present studies the "sensitivity" S_R is defined as

$$S_R = (R_a - R_g) / R_a,$$

where R_g is the film resistance in presence of the methanol vapor and R_a is the film resistance in air, measured at the respective temperatures In the present studies, the sensor response was studied as a function of operating temperature and it is found that the sensitivity is 35 % at operating temperature of 245 ^oC.

Fig. 3 shows the methanol vapor sensing characteristics of the molybdenum doped SnO_2 thin films measured at different temperatures when exposed to controlled amount of methanol vapor. It is known that as the temperature increases, the diffusion rate increases Therefore, as the temperature increases, the number of methanol vapor molecules diffusing to the surface increases resulting in a higher sensitivity The conductivity peak showed a maximum at the temperature where it is most sensitive to air. [19].

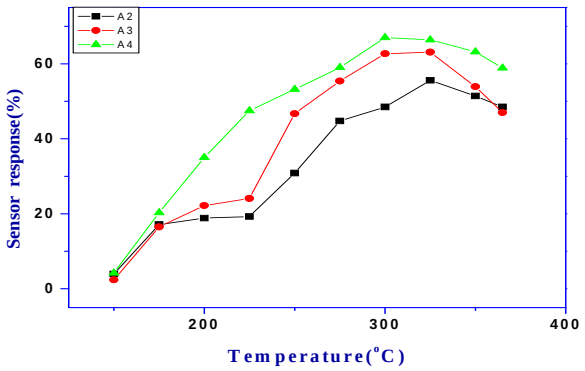

Fig. 3. Sensitivity – temperature characteristics of methanol vapor (4 ml).

The variation of sensor response S_R with molybdenum concentration measured at different temperatures when exposed to a fixed amount of methanol vapor (injecting 0.04 ml methanol into the sensor chamber) is seen Fig. 3. The sensing characteristics were studied in the operating temperature range from 100 °C to 370 °C. Upon exposure to the methanol vapors the film resistance is seen to decrease. As seen from the figure, the sensor response S_R shows dependence on both the operating temperature and molarity of the molybdenum trioxide solution. The sensor response characteristics exhibit identical behavior for the films synthesized using different molar molybdenum trioxide solutions; however the values of sensor response are different for each film, particularly at lower operating temperatures. With increasing temperature, the sensor response is found to improve exhibiting saturation in the high temperature range, above 300 °C. The sensor response is found to be very good for the 0.4 M and 0.6 M films.

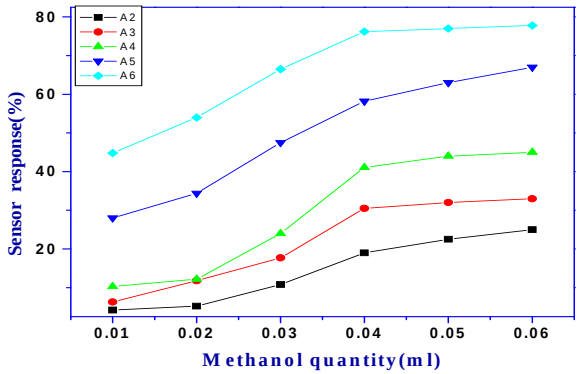

Fig. 4. Sensitivity –concentration behavior of methanol.

The sensor response was also investigated at different methanol vapor concentrations by injecting 0.01, 0.02, 0.03, 0.04, 0.05 and 0.06 ml of methanol into the sensor chamber. Fig. 4 depicts the variation of the sensor response as a function of methanol vapor concentrations measured at different temperatures of the film synthesized using 0.6 M molybdenum trioxide solution. It is interesting to note that, for all temperatures in the range from 150 °C to 350 °C, variation of the sensor response as a function of methanol vapor concentration showed same behavior, initially increasing with concentration and finally leading to saturation at higher concentrations. The saturation of sensor response S_R is due to total coverage of the film surface by the adsorbed methanol vapor molecules. Saturation of the sensitivity at higher vapor concentrations for volatile organic vapor detection using SnO_2 array sensor has been observed by various researchers

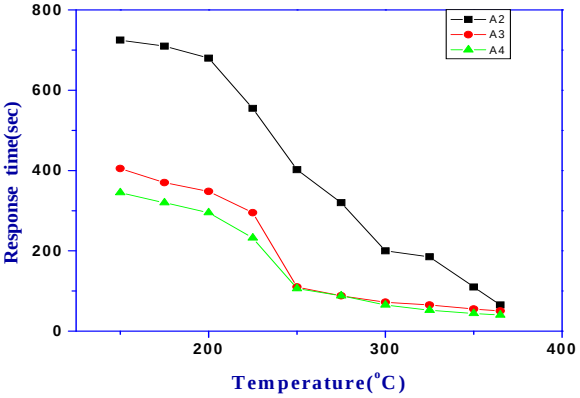

Fig. 5. Response time-temperature characteristics

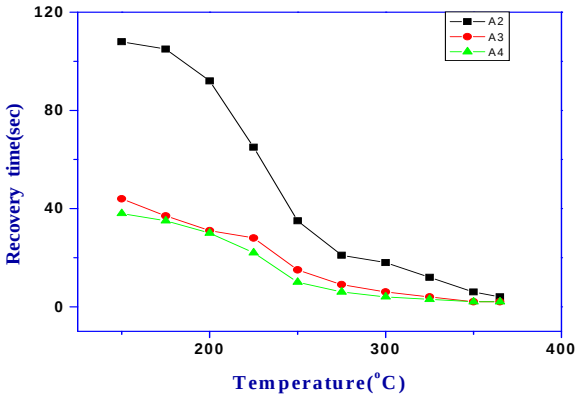

Fig. 6. Recovery time-temperature characteristics.

The response and recovery times versus temperature characteristics of the molybdenum doped tin oxide thin films recorded at fixed methanol vapor concentration (injecting 0.04 ml) are shown in Figs. 5 and 6 respectively. The observations were recorded in the operating temperature range from 150 °C to 350 °C. The response time and recovery time are found to be dependent on the molybdenum concentration and the film synthesized using 0.6 M molybdenum trioxide solution exhibits smallest values of response and recovery times, at each temperature in the operating range. In addition to the molybdenum concentration (present in the films), the

response time and recovery time are found to depend on the operating temperature.

At the lower operating temperatures the response and recovery times are seen to be larger and with increasing temperature they decrease in non-linear manner. It is interesting to note that, at higher temperature, films with various molybdenum concentrations have the response time in the neighborhood of 100 seconds and the recovery time of the order of 10 second. The above results show that molybdenum doped tin oxide thin film deposited at 425 ^{o}C using 0.6 M molybdenum trioxide solution has potential to be used as methanol vapor sensor.

Temperature strongly affects processes occurring at the surface of the sensor. The adsorption and desorption processes are temperature activated. Also the surface coverage by molecular and ionic species, chemical decomposition and reactive sites are all temperature dependent. This means that, dynamic properties of the sensors such as response and recovery time and the static characteristics of the sensor depend on the temperature. And there is always a temperature for which the sensitivity of a sensor is maximum. This may be attributed to the saturation time and mean residence period of the methanol vapor molecules on film surface.

4. Conclusion

The spray deposited molybdenum doped SnO_2 thin film on glass substrate exhibits good sensing capability for detection of methanol vapor. The sensing characteristics such as sensitivity, response and recovery times are observed to depend on the operating temperature and at higher methanol vapor concentrations the sensor response is seen to reach a saturation value of ~ 90 %. The optimum response and recovery times, each of ½ (half) minute with 90 % value of the senor response, observed at 250 ^{o}C, suggest that the spray deposited cobalt doped SnO_2 thin film is a promising sensing material for practical application as methanol vapor detector.

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Sergey Y. Yurish
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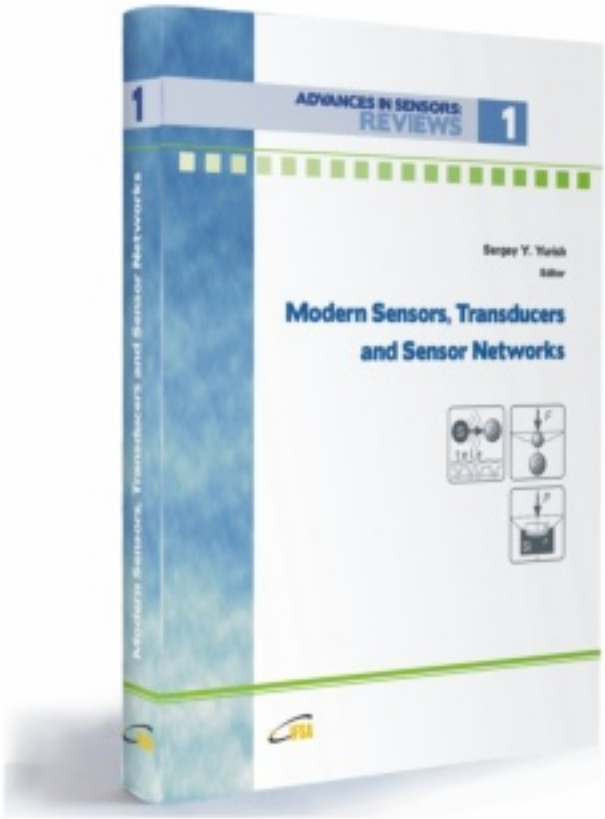

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