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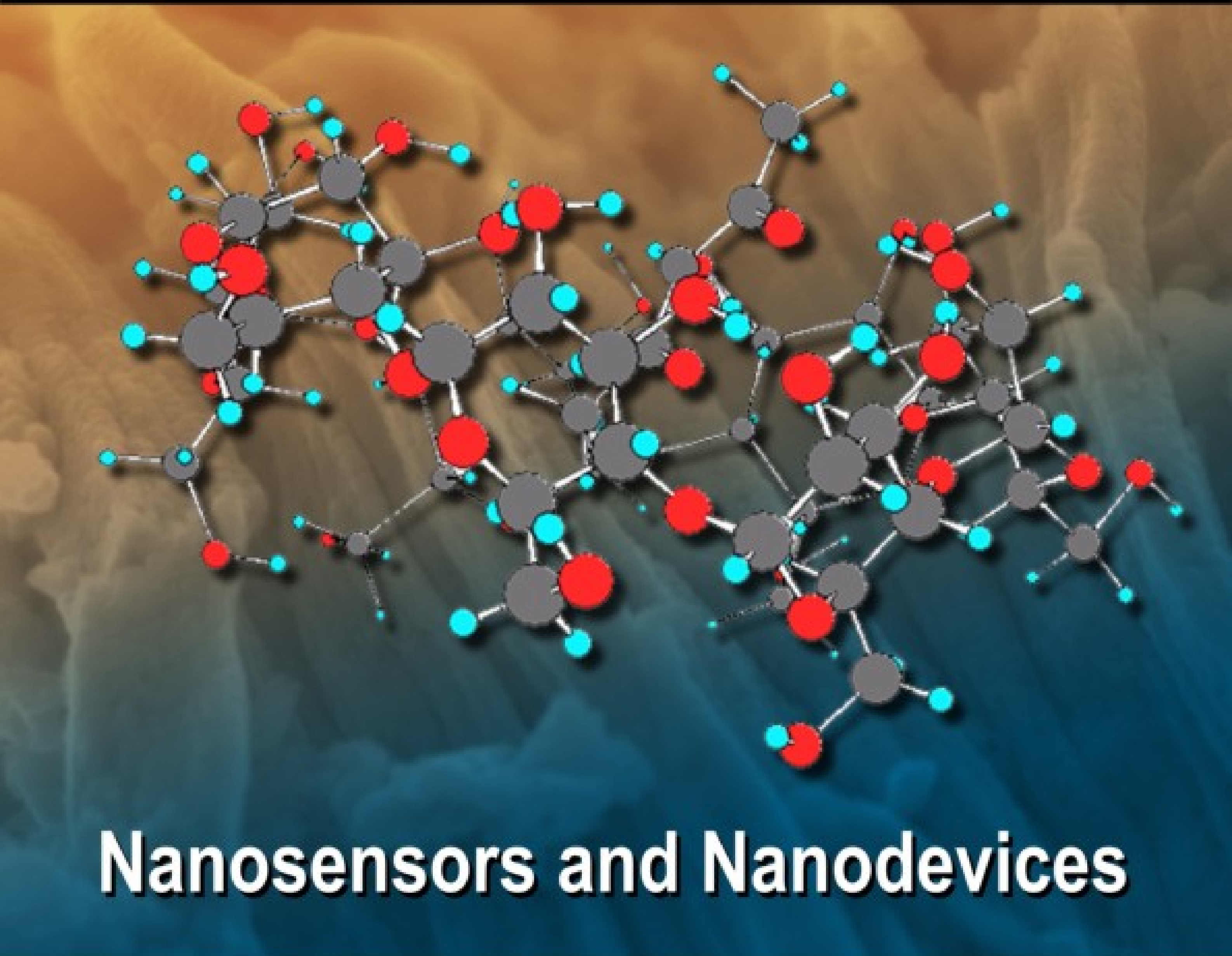

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Gas Sensing Characteristics of ZnO Nanowires Fabricated by Carbothermal Evaporation Method

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Abstract: Zinc Oxide nanowires were synthesized on the Si substrate using carbothermal evaporation of ZnO+C at elevated temperatures. The samples were characterized by scanning electron microscopy (SEM), X-ray diffraction (XRD), and Energy Dispersive X-ray (EDAX) methods. Resistive gas sensor was fabricated by providing ohmic contacts and micro-heater to the sample. The gas detection response of the fabricated sensor was obtained for different concentrations of methanol and ethanol vapors at different temperatures. In comparison with other resistive sensors, this sensor demonstrated higher response, and its maximum response occurred at lower operating temperature. By recording the response variations as a function of temperature, at different gas concentrations, it was demonstrated for the first time that by increasing the gas concentration, the maximum response of the device shifted to lower temperatures. Investigating the response variations in the equal gas concentrations showed that the response to ethanol, as compared to methanol, was five times higher. *Copyright © 2012 IFSA.*

Keywords: Gas sensor, Carbothermal evaporation, ZnO, Nanowire.

1. Introduction

Improvement of the sensing performance of Resistive Gas Sensors (RGSs), such as response time, gas detection response ($R=r_a/r_g$, see below), and selectivity have been the subject of the experimental [1-3] and theoretical [4-6] investigations. In order to increase the response of the RGSs, several methods were developed, such as the addition of doping impurity to semiconductor [7], employment of new methods for sensitive layer synthesis [8, 9] and deposition of catalysts onto sensitive layer surface [10, 11]. On the other hand, several studies have shown that the response of these sensors is closely related to the morphology, or particularly the effective porosity of sensing layer [12]. This behavior arises from the fact that the major interaction of the Target Gas (TG) and the sensitive layer takes place at the actual surface of it, and the effective gas-solid interface is larger in the sensing layer with more porosity [6, 13]. In other words, sensor response (R) is profoundly higher in the RGSs fabricated of more porous films [14-15]. Therefore, the scientific efforts for fabricating the higher porosity layers are one of the best approaches for improving the response of the RGS.

In addition to the response, it was also approved that the porosity is an important factor in controlling the response time of RGSs [16]. This is mainly due to the fact that the response time is related to the transmission of TG molecules from the environment to the grain boundary of sensing element; which is faster in the layers with more porosity [17]. Dependence of the sensor performance to porosity is of prime technical importance in the RGS fabrication process. For example, a sensor designer should be able to predict the value of the R and response time for a certain sensitive film before the fabrication of the sensor.

One-dimensional nanostructures, or nanowires, of Zinc Oxide (ZnO) have been suggested as the high porosity sensing layers for fabrication of gas sensors [8, 12, 18]. Fabricating such devices in specified micro-sized physical dimensions are of importance for special applications [19, 20]. ZnO is an n-type semiconductor with hexagonal wurtzite structure with a wide energy gap (Eg = 3.37 eV) at room temperature [21]. It is widely employed for fabrication of RGSs. Production of ZnO layers for gas sensing applications were achieved by various methods, such as the Physical Vapor Deposition (PVD) [22], Electrophoretic Deposition (EPD) [23], sol-gel and the powder pressing methods [24]. Among these methods, the highest porosity was about 60% and belonged to the layers produced by the EPD method [4].

In this paper, we show that the porosity of layers formed by ZnO nanowires is more than 90 %, which is higher than those fabricated by the EPD method. Thus these nanowires can be considered as appropriate candidates for fabrication of sensing elements for RGSs with improved specifications. This hypothesis was confirmed by fabricating of thick layers from ZnO nanowires and then comparing their morphological variables with the layers fabricated by EPD method.

For the growth of ZnO nanowires on Si substrates, several methods have been proposed [25-26]. But the most common ones are based on the vapor transmission process, which is called Vapor - Liquid - Solid (VLS) [27]. In this method, the target is evaporated and transmitted into a substrate and then deposited onto it as nanowires. Also there are several methods for evaporation of the target. In the present research the carbothermal evaporation method was applied, which is simple and suitable for mass production. The resultant layers then employed as the sensing element for fabrication of RGS. Finally, by measuring the device response, advantage of ZnO nanowires for production of gas sensor in comparison to other ZnO layers was demonstrated.

2. Experimental

2.1. Nanowire Synthesis

The schematic presentation of the system employed for the growth of ZnO nanowires is depicted in Fig. 1. It consists of a deposition chamber which is a conventional 80 cm long and 5 cm in diameter quartz tube surrounded by a cylindrical controllable heating element which is located at its center. This cylindrical heater acted as an electric furnace and its temperature can be increased up to 1500 ^{o}C using an electric current control. One side of the chamber was attached to the nitrogen gas source through the gas control facilities, and the other side was released free for exhaust. Heating of the furnace created a temperature gradient along the tube. Considering this gradient and the required temperature for source materials and substrates, they are arranged at the predetermined positions inside the tube.

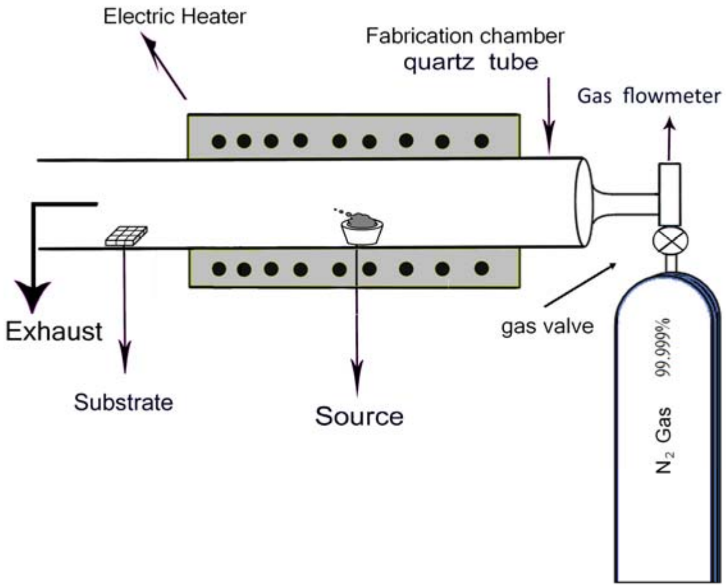


Fig. 1. Schematic presentation of system employed for growth of ZnO nanowires.

For precursor, high purity ZnO (99.999 %, Alfa Aesar) and the graphite (C) (99.999 %, Alfa Aesar) powders were mixed in a 1:1 weight ratio and 4 cm^3 of the mixture was loaded into a crucible to be located at the center of the deposition tube.

For the substrates, a p-type silicon (111) wafer was cut and polished into 10×10 mm^2 dices, cleaned, and washed by DI water. After drying, they were coated with 7 nm-thick Au catalyst film and annealed at 700 ^oC for half an hour. This heating procedure caused the Au islands to form nano-scale gold particles on the Si surface.

During the deposition phase, these particles serve as the preferred sites for the deposition of ZnO seeds and the diameter of nanowires is dictated by the diameter of these gold particles.

The details about the application of the proposed system for the synthesis of nanostructures have already been reported by in the reference [28]; in which the main steps of the process can be summarized as follows:

1). Cleaning the internal wall of the chamber to prevent any undesirable contaminations.
2). Positioning of precursor and substrates at the predetermined place inside the chamber.
3). Heating of the chamber to 1050 ^oC. In this condition, the substrates temperature was increased up to 800 ^oC, which is the optimum temperature for synthesis of ZnO nanowires.
4). Opening the control valve and flowing of the carrier gas through the chamber.

The deposition runs prolonged about 60 minutes, during which, all the parameters of the system, including temperature of the precursor and substrates as well as carrier gas flow rate were kept constant. The experiments showed that nanowires of different morphology can be deposited by altering the deposition parameters such as time, substrate temperature, catalyst material, and also gas flow rate. Details about the effect of catalyst material and deposition parameters on the morphology of the grown nanowires will be reported elsewhere.

2.2. Characterization

After cooling and bringing the sample out of the chamber, their morphology was examined by the Scanning Electron Microscope (SEM) method. In the next step, structural properties of the samples were characterized by recording their X-Ray Diffraction (XRD) patterns. A Cu target (CuKα radiation) was used as X-ray source. To investigate their chemical composition and stoichiometry, the Energy Dispersive X-ray Spectroscopy (EDAX) technique employed.

2.3. Sensor Fabrication

Fabrication of the sensor is similar to what has already been reported in the previous research [29] and schematic illustration of the device structure is presented in Fig. 2. It comprises of a gas sensitive layer with a predefined apparent surface area, two ohmic contacts for connecting the sensing layer to the external measurement circuit and also a micro-heater for biasing the sensor at the desired operating temperatures.

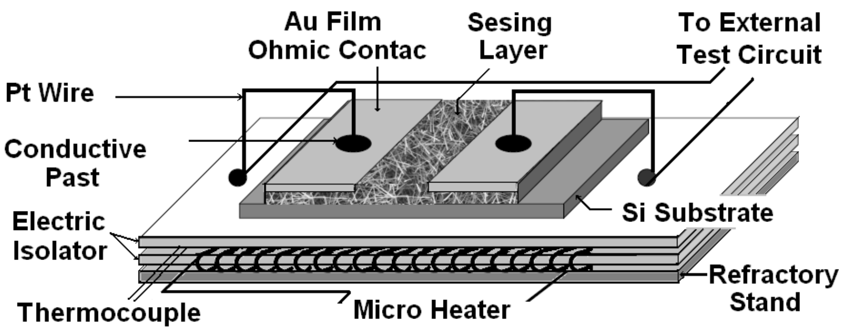


Fig. 2. Schematic illustration of the fabricated gas sensor.

Construction of the sensor was initiated by the formation of ohmic contact on the sensitive layer. For this purpose, area-selective metal contact was created by patterning of 300 nm-thick Au conductive films on the ZnO layer using the conventional sputtering method. Two gold electrodes of 10×4 mm^2 were deposited onto each sample. The typical gap between two Au electrodes was about 2 mm. Thin platinum wires were subsequently cemented to the metalized area by Ag conductive adhesives. After metallization and wiring, a micro-heater was devised beneath the substrate, and the resultant device was mounted on a refractory stand. Using this heater, the temperature of the sensor can be controlled from room temperature up to 500 ^oC. For monitoring of the actual temperature of the sensor a thin thermocouple was embedded on it. This capability is necessary for assessing the gas detection response of the device at the elevated temperature.

To facilitate employment of sensor, a sensor probe similar to what reported in [29] was formed by mounting the sample on the thick-walled borosilicate glass tubing. Through this tube three insulated connection cables were guided towards the temperature control unit, thermocouple and the resistance measurement device, respectively.

2.4. Response Measurement

Different definitions of the response (R) of a gas sensor to a particular gas have been presented in the background literatures [29-31]. However, most recently the authors of the related technical papers

have almost unanimously employed $R = r_a/r_g$ [30], in which r_a and r_g are the steady state resistances of the sensor in the pure and the gas contaminated air, respectively. According to this definition, both r_g and r_a should be measured at the same operating temperature.
Measurement of the device's response was performed at the following stages:

1. Heating of the sensor to be set at various operating temperatures and measurement of r_a against temperature.
2. Injection of TG to the test chamber for providing the gas contaminated environment.
3. Insertion of the sensor probe into the test chamber and sealing off its opening.
4. Measurement of r_g after the transient time.
5. Variation of operating temperature for the measurement of r_g at other temperatures.

The measurement of resistance was performed by applying of a constant AC voltage (10 V, 80 Hz) to the sensing layer and recording of AC current. Application of the AC voltage will prevent ionic migration and electrode instability, which is an inherent result of DC voltage.

The test chamber was a 2-liter borosilicate glass tank which was equipped with a small lid to isolate it from the outside environment and prevent the gas leakage. The lid is opened only for sensor and TG insertion; so the TG concentration inside the chamber was expected to be constant during the test period. On the other hand, the sensor insertion takes a short time, and the gas leakage during this time is less than the system errors and can be neglected.

After sensor insertion into the chamber, its current was recorded through a data acquisition system to obtain its resistance variations against the time (t). This information can be employed to obtain the transient response of sensor. The steady state value of r_g was calculated following of the transient time (t_r). Due to high porosity of sensing layer, the senor response is very fast (i.e. t_r <1.5 second); but for assurance, we defined t=15 second as stabilization time of r_g.

In order to evaluate the effect of temperature on the device response, the corresponding values of r_g and r_a were calculated at different temperatures. Using these values, the temperature dependence of the response, R(T), was calculated and recorded. For investigating the mutual effects of concentration and temperature on the sensor behavior, the above experiment was repeated for different value of TG concentration, and R(T) was calculated many times.

3. Results and Discussion

3.1. Morphology and Structure of the Layers

Fig. 3 shows a SEM micrograph of synthesized ZnO layers, which consist of randomly oriented nanowires. The wires are almost uniform with average diameter and length of 50 nm and 6-7 μm, respectively. The porosity of the sample was approximately estimated by random line measurement on the SEM micrograph to be more than 90 % (in this technique a random line is drown within a specified region of the micrograph and its crossing over pores and wires are linearly compared). Based on our previous reports, porosity of the sensitive film is a key factor in defining of the response and the response time of a gas sensor [6]. So, the sensors fabricated by these layers are expected to have a better response than the layers fabricated by other methods.

To study the crystal structure of the nanowires, they were analyzed by X-Ray Diffraction, XRD system (Philips PW1880). A typical XRD pattern for one of the samples is presented in Fig. 4. Analysis of diffraction peaks indicated that the fabricated nanowires were highly crystalline with hexagonal Wurtzite structure and their lattice constant were a = 3.249 ˚A and c = 5.205 ˚A. These values are in good agreement with lattice constant of an ideal ZnO crystal. Regardless of Si (111) peak in the spectrum which belongs to the substrate, the second major peak relates to the ZnO(002) direction which indicates that the dominant direction of synthesized nanowires is along the c-axis.

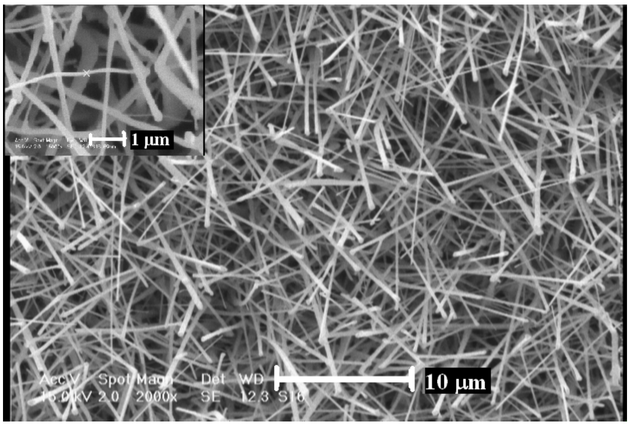


Fig. 3. A SEM micrograph of the synthesized ZnO layer grown by VLS method.

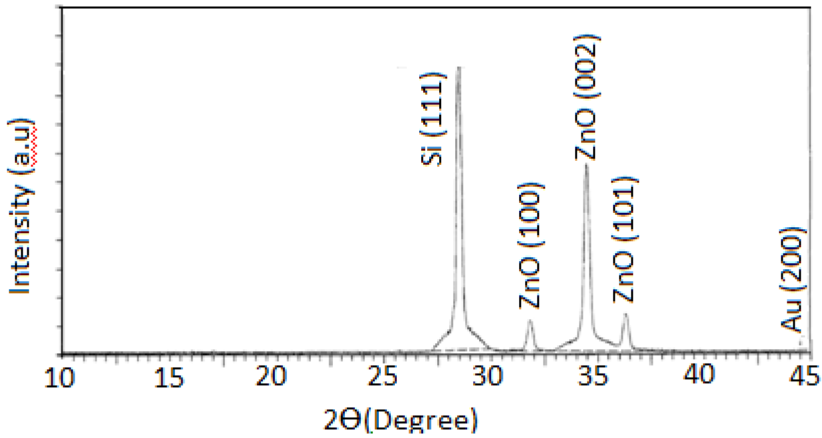


Fig. 4. XRD pattern for one sample of ZnO layer grown by VLS method.

Fig. 5 presents a typical EDAX spectrum of the sample in which the main peaks correspond to Si, Au and Zn elements. The peaks of Si and Au are contributions of substrate and catalyst, respectively and the peaks of oxygen are omitted because of instrument limitation. Except for the above mentioned elements, there are no other peaks related to the impurity elements; so it can be concluded that the produced nanowires are of highest purity, except for the end containing Au catalyst as an alloy particle.

3.2. Gas Sensing Properties of Layers

In the present study, methanol and ethanol in the concentrations range of 250-1000 ppm were considered as the TGs. Fig. 6 presents the gas detection response of the fabricated sensor at different operating temperatures and TG concentrations. At a constant concentration level, according to Fig. 6, the sensor response increases with temperature at the lower temperature range, reaching a maximum response level at a specific temperature (T_{opt}). At temperature above T_{opt}, the response descends by further elevation of the temperature.

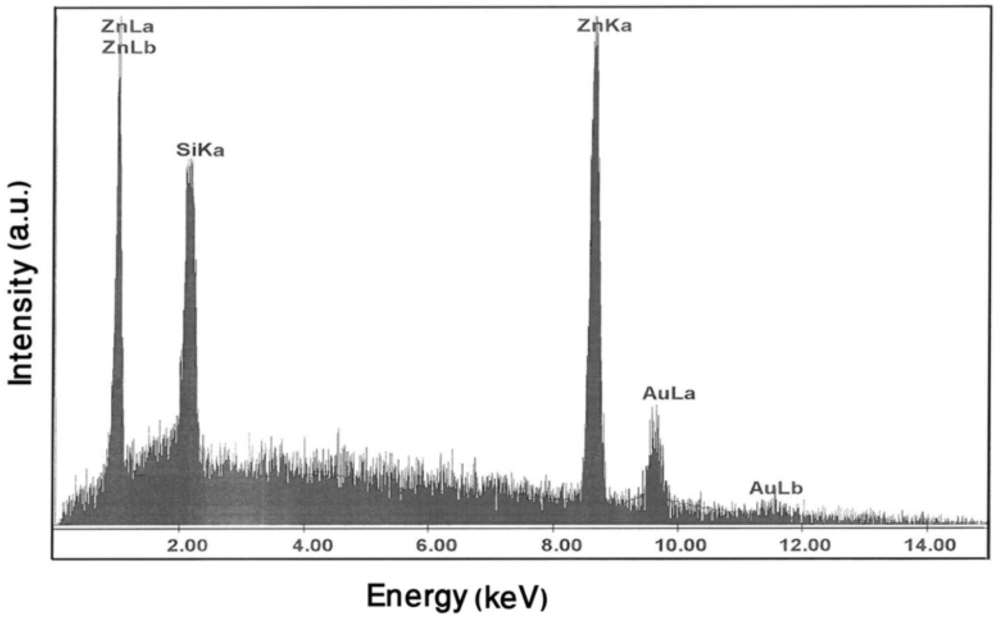
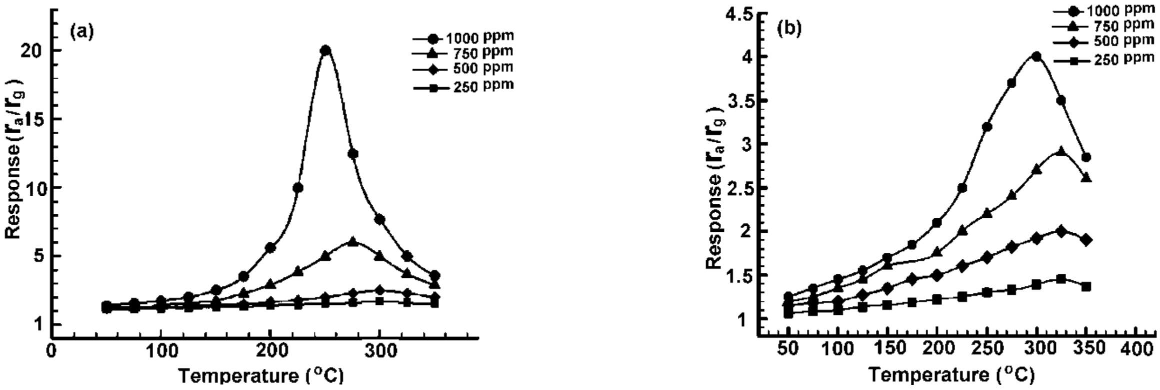


Fig. 5. EDAX spectrum of ZnO layer grown by VLS method.

Such a "volcano-shaped" temperature dependence of the response is commonly observed in metal oxide sensors [32-33]; and T_{opt} is usually chosen as the optimum operating temperature for these devices. This behavior of metal oxide gas sensors is attributed to a competition between the two main carrier generation mechanisms taking place in the oxide semiconductor. The first mechanism is due to the reaction of oxygen adsorbates with the TG, which takes place at the surface of the sensitive material. The second mechanism is owing to the thermal generation of carriers which occurs naturally in the semiconductor materials. The rate of the first mechanism increases with temperature as the thermal decomposition of the TG molecules at the oxide surface along with the subsequent removal of the oxygen species from the metal oxide surface increase rapidly with the temperature. This drives the sensor's response up, as the caused rate of carrier generation surpasses the second mechanism. At $T > T_{opt}$ temperature range, however, the sensor surface is too hot and some of the TG molecules are decomposed, oxidized, and diffused outwards before reaching the sensitive surface. This reduces the relative concentration of the TG on the most sensitive region in the sensor and the gas-solid interaction rate. Meanwhile, the rate of carrier generation due to the thermal excitation increases exponentially at this temperature range, which reduces the resistance level in the clean air, r_a, resulting a further response reduction. Moreover, these high temperatures accelerate re-adsorption of chemisorbed oxygen species to the oxide surface, irrespective of the consumption of oxygen adsorbates by the reaction with the TG molecules. As a result of this additional process, a lower decrease in equilibrium surface coverage of oxygen and lower changes in carrier concentration induced by the reaction of oxygen adsorbates with TG will prevail. All these major mechanisms, along with a number of other minor ones, are effective in the formation of the volcano shaped temperature dependence of response, which is not quantitatively analyzed yet.

The temperature corresponding to the maximum response, T_{opt}, is of prime technical importance. This temperature can be obtained by recording the response of the device at various operating temperatures. Comparison of temperature dependence of response between different gas concentrations in Fig. 6 indicates that the T_{opt} depends on TG nature and for ethanol and methanol occurs at 250–300 ^{o}C which is lower than the sensors fabricated by other methods. For instance, optimum temperature of ZnO sensor fabricated by low-heating solid-state chemical reaction is about 350 ^{o}C [34]. Another remarkable result in this figure is the reduction of T_{opt} by an increment of TG concentration.



(a) (b)

Fig. 6. The experimental relationship between the gas detection response and the operating temperature in the gas sensors fabricated exposure (a) ethanol, (b) methanol, with different concentrations.

At 1000 ppm of ethanol vapor, the device exhibits the maximum response of 20 at the operating temperature of 250 °C. In same concentration of methanol vapor, the highest response is about 4 at the working temperature of 300 ˚C. These values of responses are higher than the similar measurements reported for Zinc Oxide sensors fabricated by other techniques [35]. Lower sensitivity of RGSs to ethanol, as compared to the methanol, is a usual phenomenon [34] and we have already observed it in commercial gas sensors [19]. In that research, the response of the device to 2000 ppm of ethanol, R_e, was about 10; whiles, in a same concentration and operating temperature, the response to methanol (R_m) was about 6.4. Such difference of responses was reported to be due to dissociation (pyrolysis) energy (or temperature) of gases [36]. As a matter of fact, dissociation energy of ethanol is lower than that of methanol [37]. Difference in dissociation energy of gases has already been employed as a diagnosis basis for development of selective gas sensors [36].

By interpolation, the response of above commercial sensor to 1000 ppm of ethanol and methanol was estimated about 6.2 and 4, respectively. This result indicates that the fabricated sensor, in comparison with the commercial one, has a better response to ethanol. This higher response to ethanol can be related to high porosity and Au catalyst as well [7]. However, the Au-doped aria is a small part of effective surface in the sensing layer; so, its action on whole device response can be neglected. Regarding to methanol, both of the sensors have a similar response.

The result of comparison indicates the superiority of fabricated sensor for ethanol detection applications. Improvement of the response to methanol can be considered as another research subject matter, which can be followed after the present research. One of the best methods for such improvement is the addition of some dopants to as sensitive material [38]. The necessary additives can be loaded on the precursor crucible before growth of nanowires.

Fig. 7 represents T_{opt} of the device to ethanol and methanol vapor at concentrations of 250, 500, 750, and 1000 ppm. It can be seen that T_{opt} decreases by increasing of the gas concentration. This behavior is likely due to the limitation of absorbent surface on sensing layer. This limitation causes the absorbent surface to be saturate at high TG concentration and reduce the contribution of gas–surface interaction on T_{opt}. However, this is an initial interpretation, and more investigations are recommended to find the exact cause of this phenomenon. A more significant result of the Fig. 7 is that, relation between T_{opt} and concentration depends on the nature of TG. Therefore, investigating the effects of concentration on T_{opt} can be considered as a new line of research on selective RGSs.

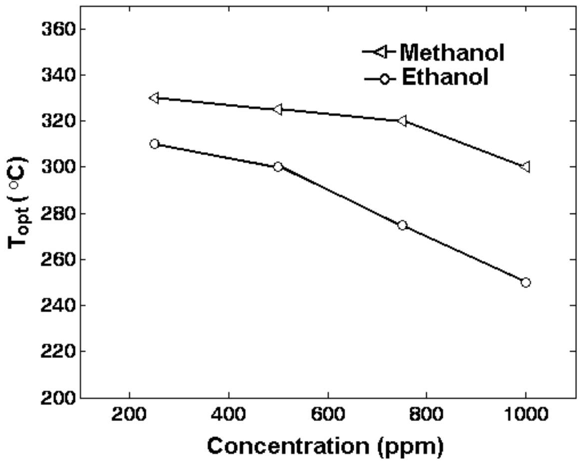


Fig. 7. Variations of optimum temperature, T_{opt}, by TG concentration at the fabricated device.

4. Conclusion

Zinc Oxide nanowires were synthesized on the silicon substrates using a simple and cost-effective carbothermal evaporation method. Characterization of layers indicates that they are pure ZnO crystals with hexagonal structure growing about the c axis. The SEM micrograph of the layers formed by these nanowires showed that, their porosities are considerably more than the layers fabricated by other deposition methods such as EPD. So, the gas detection response of sensors fabricated by these layers is expected to be higher than that of other sensors. A prototype gas sensor was fabricated using a sample layer, and its sensing characteristics were tested for different concentrations of methanol and ethanol vapors. By evaluating of device response at different operating temperatures, it was demonstrated that the sensor optimum temperature is in the range of 250-300 ^{o}C, which is lower than that of other sensors. On the other hand, in equal gas concentration, the maximum response to ethanol was higher than the response to the ethanol and also occurs at a lower temperature. By recording optimum temperature at different gas concentrations, it was concluded that the temperature related to the maximum response of the device, T_{opt}, depends on the TG nature and also the gas concentration in which a higher concentration results in a lower T_{opt}. Dependence of T_{opt} to the gas nature and its concentration value has technical importance and can be considered as a new basis for the development of the selective gas sensors.

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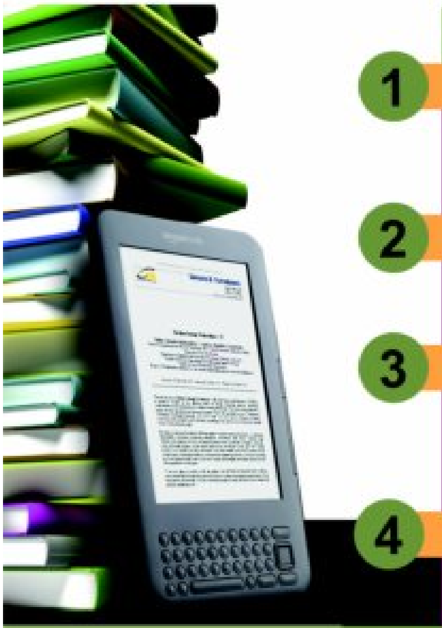

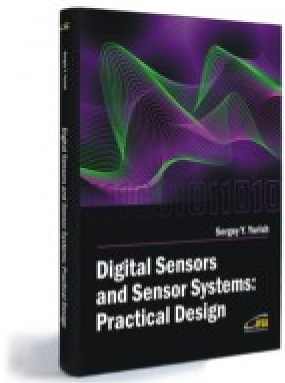
Sergey Y. Yurish
Digital Sensors and Sensor Systems: Practical Design

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 of it is a peer reviewed international journal, papers rapidly published in *Sensors & Transducers Journal* will receive a very high publicity. The journal is published monthly as twelve issues per year by International Frequency Sensor Association (IFSA). In additional, some special sponsored and conference issues published annually. *Sensors & Transducers Journal* is indexed and abstracted very quickly by Chemical Abstracts, IndexCopernicus Journals Master List, Open J-Gate, Google Scholar, etc. Since 2011 the journal is covered and indexed (including a Scopus, Embase, Engineering Village and Reaxys) in Elsevier products.

Topics Covered

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:

- Physical, chemical and biosensors;
- Digital, frequency, period, duty-cycle, time interval, PWM, pulse number output sensors and transducers;
- Theory, principles, effects, design, standardization and modeling;
- Smart sensors and systems;
- Sensor instrumentation;
- Virtual instruments;
- Sensors interfaces, buses and networks;
- Signal processing;
- Frequency (period, duty-cycle)-to-digital converters, ADC;
- Technologies and materials;
- Nanosensors;
- Microsystems;
- Applications.

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