

Reliable Gas Sensors using ZNO Nanostructures

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Abstract: Gas sensors are devices that can convert the concentration of an analyte gas into an electronic signal. Zinc oxide (ZnO) is an important n-type metal oxide semiconductor which has been utilized as sensor for several decades. In recent years, there have been extensive investigations of nanoscale semiconductor gas sensors. The size reduction of ZnO sensors to nanometer scale provides a good opportunity to dramatically increase their sensing properties in comparison with their macro scale counterparts. Among the semiconductor metal oxides, zinc oxide (ZnO) is one of the most widely used gas sensing material. Before making any gas sensor, it is very much necessary to know the sensitivity, selectivity of the sensor and their optimization. In this paper, we present the growth of ZnO nanostructures by thermal evaporation technique and investigation of their gas sensing properties. It is observed that the sensing characteristics of single nanowires and films made using nanowires to clearly differentiate the intra grain and grain boundary contributions as well as to develop sensors with better sensitivity/ selectivity. This paper is very much useful for those who would like work on gas sensors for better gas sensing performances.

Keywords: Gas sensor, Nanowires, ZnO

I. INTRODUCTION

Zinc oxide (ZnO), as an important semiconductor material, has been widely applied to the fields of optics, photocatalysis, biomedical sciences and sensors [1–3]. ZnO is an interesting sensing material due to its high mobility of conduction electrons and good chemical and thermal stability under the operating conditions of sensors. Although there are many reports on the gas sensing properties of ZnO, most studies focused on nanoparticles based gas sensors because of their large specific surface and quantum size effects. For example, Nath et al. reported sensing properties of ZnO quantum dots for acetone [4]. Forleo et al. synthesized ZnO nanoparticles with sizes ranging from 2.5 to 4.5 nm, which showed remarkable response to NO₂, acetone and methanol [5]. In addition, our previous research also demonstrated that sensors based on 7–8 nm ZnO nanoparticles exhibit a high response to NO₂ and high selectivity to CO and CH₄ at operating temperature of 290°C [6]. Recently, many interesting nanostructures of 1D ZnO have attracted much attention due to their well defined morphology, high surface to volume ratio and efficient transport of electrons and excitons. Many methods have been used to synthesize 1D ZnO nanostructures. Among them, the hydrothermal method is recognized to be the most promising one for preparing well-crystallized products under relatively mild conditions and it has been successfully used to grow many 1D metal oxides such as nanobelts, nanobridges, nanorods and nanoribbons. Moulahiet al. successfully synthesized nanostructured ZnO with different morphologies by parameter controlling in hydrothermal process [7].

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Du et al. reported on synthesis of ZnO architectures by hydrothermal method [8]. In this work, we reported a one-step hydrothermal method to synthesize flower-like ZnO nanostructures using low-cost raw materials without any surfactant or organic solvent. The ZnO nanocrystals with well-defined morphology and high crystallinity were obtained, and it is favorable for large-scale producing with a relatively low cost. The product obtained not only exhibits high response to NO₂ but also high selectivity to CO and CH₄ gases at lower work temperature. It is known that the sensing mechanism of n-type semiconductor ZnO based sensor belongs to the surface controlled type in which the grain sizes, morphology and surface states play important roles in its gas sensing properties [9,10]. The gas sensing mechanism of these sensors involves adsorption of atmospheric oxygen on the oxide surface that extracts electrons from the semiconducting material leading to change in carrier density and conductivity. On interaction with oxidizing or reducing gases, adsorbed oxygen concentration and thereby conductivity changes. The change in conductivity is a measure of gas concentration. For reducing gases such as H₂S and NH₃ the conductivity increases for n-type materials (as SnO₂ and ZnO) and reduces for p-type materials (as Te). The effect of oxidizing gases is opposite to that of reducing gases. Since the gas sensing mechanism is a surface reaction, use of nanostructured materials is expected to improve gas sensing characteristics. Among the semiconductor metal oxides, zinc oxide (ZnO) is one of the most widely used gas sensing material. [3–6] In this they have reported the growth of ZnO nanostructures by thermal evaporation technique and investigation of their gas sensing properties. The studies aim at (a) development of sensors with better sensitivity and room temperature operation provided by increased surface area, (b) single nanowire sensors with a view to miniaturize them, (c) comparison of the sensing characteristics of single nanowires and films made using nanowires to clearly differentiate the intragrain and grain boundary contributions as well as to develop sensors with better sensitivity/ selectivity. It is observed that (i) both intragrain and grain boundary regions contribute to sensitivity, (ii) for different gases either of the two responses may be higher leading to possibility of improved selectivity and (iii) depending on growth conditions, changes in crystalline defects may improve sensitivity.

II. ZINC OXIDE SEMICONDUCTOR

Zinc oxide is an important semiconductor which has been studied for many decades. It has a wide band-gap of 3.37 eV and a large exciton binding energy of 60 meV at room temperature. [11] Polycrystalline ZnO has attracted intensive research effort for its unique properties and versatile applications in ultraviolet (UV) light emitters, short-wavelength nano-lasers, piezoelectric devices, ultrasensitive, spin electronics, field-effect transistors, and field emitters. [12] ZnO, like ZnS, has the wurtzite structure type,

which is the thermodynamically stable phase under ambient conditions. The ZnO wurtzite structure has a hexagonal closed-packed unit cell, with dimensions $a = 3.25 \text{ \AA}$ and $c = 5.12 \text{ \AA}$. (Fig.1).

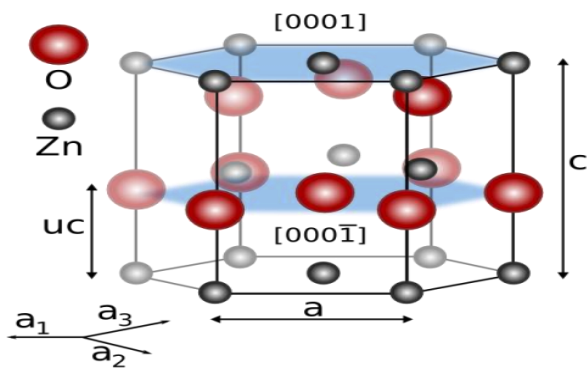


Fig. 1 Structure of Zinc Oxide

The structure can be visualized as Zn^{2+} ions in half of the tetrahedral holes of a hexagonal close-packed oxide lattice; the structure with the longitudinal axis (c -axis). In this crystal structure, both zinc and oxygen ions are coordinated with four ions of the opposite charge with strong ionic binding. Due to the size differential of the ions, the ions fill about 44% of the total volume in a ZnO crystal, and leaving a relatively large free volume. [13-15]

III. RESPONSE MECHANISM

The sensing mechanism of a sensor is that their properties vary with the environment changes, and it is easier to us to monitor the property varies than the concentration of the gases. To the semiconductor, the electronic property changes in the oxidation and reduction gases. The process of gas sensing with a metal oxide semiconductor involves two key functions: one is the recognition of a target gas through a gas-solid interaction, which induces an electronic change of the oxide surface, commonly referred to as a receptor function; the other is transduction of the surface phenomenon into an electrical resistance change of the sensor, which is referred to as a transducer function. When the solid state gas sensor is exposed to air at elevated temperatures, the surface is covered by oxygen anions and a space-charge layer is formed on the surface of the metal oxide, as shown in Figure 1.1. [16] The space-charge layer is a region that has deficient carriers due to electron trapping by the chemisorbed oxygen. A typical value of the space-charge thickness (Debye thickness, or Debye length) is 30 nm for undoped ZnO. When n-type semiconductor gas sensors are exposed to air, the resistance of the sensors increases with respect to the vacuum reference value, as the high resistance at grain boundaries leads to a lower conductivity through the whole oxide.

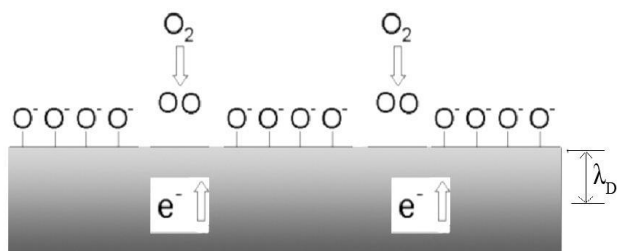


Fig. 2 Schematic Description of the Chemisorption of O2 on the Surface of a Metal Oxide Gas Sensor. [16]

H2O vapour can be adsorbed on metal oxide surfaces in both molecular and hydroxyl forms, and thus competes with O2 for oxygen vacancy sites on the oxide surface, as shown in Figure 1.2. This competition for oxygen vacancy sites will also lead to changes the conductivity of the metal oxide sensor. When ZnO sensors are operating in humid conditions, the H2O molecules occupy some of the O vacancies, leading to a lower chemisorption of O2 on the oxide surface; thus, fewer electrons are trapped by oxygen anions. This leads to an increase in the conductivity of ZnO sensors resulting from a decrease in the energy barriers between metal oxide grains. [17]



Fig. 3 Schematic Description of the Sensing Mechanism of Water Vapour on the Surface of a Metal Oxide Gas Sensor

Figure 3 shows the structure of the surface species formed. The reaction of CO with adjacent pairs of oxygen anions produced surface bidentate carbonate. The bidentate carbonate species subsequently transforms into surface unidentate carbonate. The adsorption of CO at a single oxygen anion site then yields surface carboxylate groups. During the formation of all these surface species, there is no electron transfer to the bulk metal oxide sensor, thus no conductance change. However desorption of CO2 provides opportunities for the electrons to return to the sensor solid. CO2 desorption is favoured at higher temperatures, but not favoured at low temperatures. [18][19]

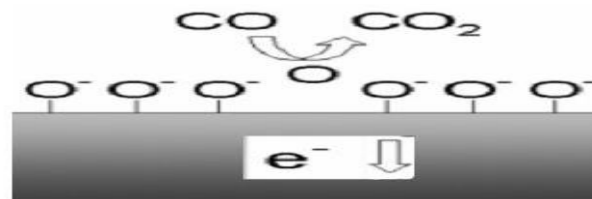


Fig. 4 Schematic Description of the Sensing Mechanism of Carbon Monoxide over the Surface of Metal Oxide Gas Sensor. [19]

In the past several years, although many studies have been carried out the sensing of hazardous gases like CO, there is a common problem in the area, which is the poor selectivity to specific gas. Common metal oxide sensors show responses for all three gases discussed above (water, CO, ethanol). This would be problematic, e.g. because CO detectors may show false signals due to high humidity and/or ethanol vapour. Thus sensors that have strong specific responses to one gas are highly desirable

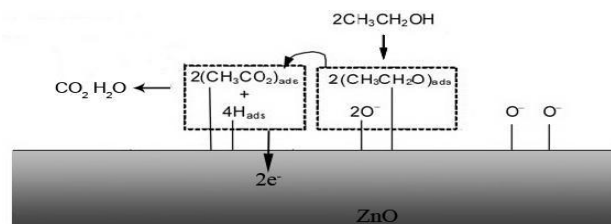


Fig. 5 Schematic Description of Sensing Mechanism of Ethanol over the Surface of a Metal Oxide Gas Sensor

IV. GROWTH OF NANOSTRUCTURES

ZnO nanostructures were prepared in a horizontal tube furnace having facility for introducing gases at controlled rates. In the initial experiments, pure Zn metal powder was heated to temperatures between 600 and 900°C under argon atmosphere (flow rate 500 cc/min) and on stabilizing the temperature at desired value, the gas atmosphere was switched to 95% argon and 5% oxygen. ZnO nanowires (<100 nm diameter) and thicker nanowires called nanorods (~500 nm dia) were obtained at source itself at temperatures of 700 and 800°C respectively. At source temperature of 900°C, nanowires of 100–500 nm diameter were deposited on substrates placed in downward gas flow direction at temperature of 200°C. For growth of tetrapods and long nanowires required for isolated nanowire experiments, a mixture of ZnO and graphite in 3 : 1 ratio (by weight) was heated to 1050°C (at a rate of 6°C/min) under argon atmosphere. While heating, 2–5% oxygen was added to argon gas at temperatures between 800 and 1050°C. Nature of nanostructures mainly depended on the temperature at which oxygen was introduced (which controls the nature of nuclei formed). Nanowires and nanobelts were obtained in case of oxygen introduction at 800–950°C and tetrapods in case of oxygen introduction at 950–1050°C. All nanostructures were deposited on substrates maintained at temperatures from 200 to 450°C.

V. PREPARATION OF SENSORS

Two different type of sensors were prepared, (a) thick films of nanostructures (NS) and (b) isolated nanowires/belts. For thick films, the nanostructures were dispersed in suitable organic solvents and deposited on alumina substrates. This was followed by heating to temperatures of 200–400°C and deposition of gold contacts. In some cases nanowires were directly grown on substrates with predeposited thick film gold contacts. Isolated nanowires/belts based sensors were prepared by dielectrophoresis [22] technique, where, gold electrodes with a spacing of 25 µm were deposited on alumina substrates by thermal evaporation. An ac voltage of 20 V (peak to peak) at 100 kHz was applied to the electrodes and a suspension of nanowires/nanobelts (NS) in methanol (0.1 mg NS in 10 ml of methanol) was put dropwise between them.

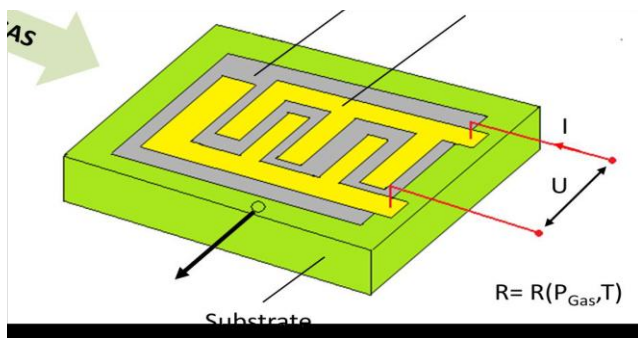


Fig. 6 Typical Diagram of Gas Sensing

Typically, 5–10 drops of suspension were put on a substrate over a period of 20 min. The samples were examined using SEM and those consisting of one or few NS between two electrodes were selected for measurement of response to gases.



Fig. 7 Microstructure of an Interdigitated Array Electrode

S. K Gupta et.al [23] have considered three different kind of nanostructures, i.e. tetrapods, nanowires and nanobelts as shown in figure 1 were used for preparation of sensors. Nanowires and nanobelts were usually obtained as a mixture. Diameter of different nanowires and legs of tetrapods varies from 50 to 150 nm range.

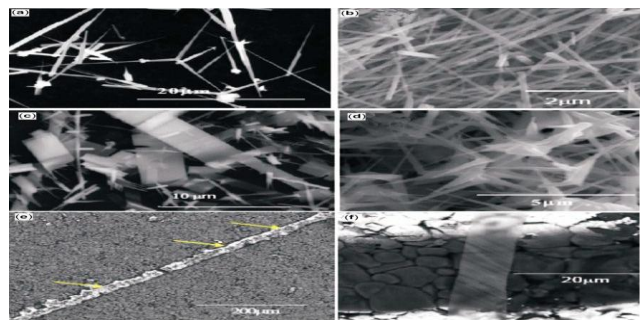


Fig. 8 Scanning Electron Micrographs of (A) ZnO nanotetrapods, (B) Nanowires, (C) Nanobelts and (D) Tetrapods Film Deposited on Alumina Substrate (Zn1 Sensors). Isolated Nanostructures Aligned between Gold Electrodes with 25 µm Separation: (E) Three Nanowires/Belts shown by Arrows and (F) a Nanobelt. All of the Structures shown in this Figure have been Prepared by using ZnO and Graphite Source [23]

Five different kind of sensors (Zn1 to Zn5) that were fabricated are: (a) thick film tetrapods (Zn1) and nanowires (Zn2) directly deposited on alumina substrates, (b) nanowires dispersed on substrates to yield thick film (Zn3) followed by annealing, (c) isolated few (2 to 8) nanowires or nanobelts aligned between two electrodes (Zn4) using dielectrophoresis technique and (d) using polycrystalline ZnO powder sensors (Zn5).

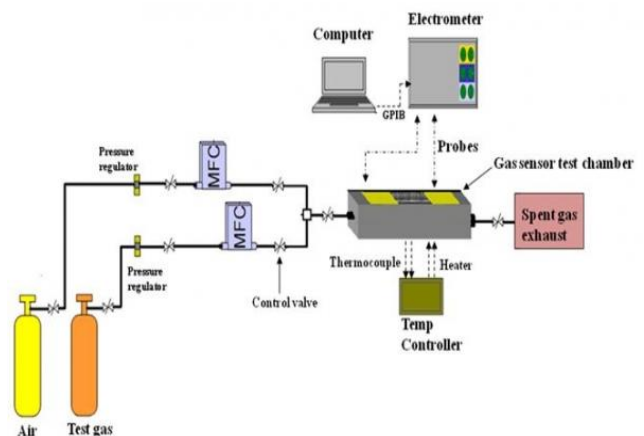


Fig. 9 Schematic of Gas Sensor Test Bench for Characterization of Gas Sensor

VI. VLSSENSITIVITY, SELECTIVITY AND RESPONSE TIME OF ZnO GAS SENSOR

The key characteristics of semiconductor gas sensor performance are sensitivity, selectivity, response time and recovery time. For metal oxide semiconductors, the measurable parameter is the resistance of the metal oxide, thus the sensitivity of the semiconductor is defined as follow

$$sensitivity(s_g) = \frac{R_{gas}}{R_{air}} \text{-----1.1}$$

where R_{air} the resistance of the sensor in air; and R_{gas} is the resistance of the sensor after exposure to the analyte gas. Using this definition of sensitivity, the sensitivity is easily calculated from the measured resistance values. Thus, it is very convenient to compare the sensitivities in different substrate gas environments. The sensitivity of semiconductor gas sensors can also be empirically represented as,[20]

$$S_g = A \times P_g^\beta \text{----- (1.2)}$$

where P_g is partial pressure, which is directly proportional to its concentration of the target gas. The sensitivity is characterized by the pre-exponential factor A and the exponent .may have some rational fraction value (usually 1 or 1/2), depending on the charge of the surface species and the stoichiometry of the elementary reactions on the surface. The selectivity of metal oxide semiconductors is defined as the ratio of the sensitivity of one analyte gas relative to another analyte gas under same conditions. Semiconductor gas sensors often exhibit poor selectivity since their operation depends, in most cases, on the reaction of reducing gases with adsorbed oxygen (see next section for mechanistic discussion).

$$sensitivityS_{(p)} = \left| \frac{S_{gas(a)} - S_{gas(b)}}{S_{gas(a)}} \right| \text{----- (1.3)}$$

The response time and recovery time are usually defined as the time taken to achieve 90% of the final change in resistance following the change of gas concentration (τ_{90}). However, as the gas sensor response is often very fast initially, followed by a long drawn out tail before reaching a steady value, response times are alternatively often expressed as 50% or 70% of the final time [21]. The response time is an important parameter since it can determine the commercial applicability of the sensor; unfortunately, it is probably the most difficult parameter to measure in a reproducible manner. It requires special gas flow systems which are designed to ensure that step changes in gas concentration are faster than the response time of the sensor, especially when dealing with highly adsorptive and reactive gases. The commercial usage of a gas sensor is highly dependent on its recovery time; a gas sensor that has a short recovery time will have greater applications in the commercial market than one with a long recovery time. The gas sensing properties of ZnO nanowires can be highly affected by the diameter of the nanowires. As the diameter of the nanowires become smaller, and the dimension of the nanowires approaches the Debye length of ZnO (~ 30 nm), all the ZnO atoms can be treated as surface atoms and the gas sensor shows the best sensor performance. Typical response of ZnO nanowires (Zn2), tetrapods (Zn1) and polycrystalline material (Zn5) to show that tetrapods have

maximum while polycrystalline material has minimum sensitivity to H2S. Response of tetrapods to different concentration of the gas [23] shows that the films fully recover to original resistance after exposure to the gas. The films were found to be sensitive to 1 ppm and lower concentration of H2S. Relative response of different type of materials may be understood with reference to the PL data. Oxygen vacancies and defects in lattice act as adsorption sites for oxygen. As the detection of gases originates from interaction of these adsorbed oxygen atoms with reducing gases such as H2S, maximum (minimum) defect density in tetrapods (polycrystalline material) yields maximum (minimum) response. Smaller size of nanowires and tetrapods may also contribute to their better response. These thick films had typical room temperature resistance (in air) of 500 MΩ. Comparison with Zn1 and Zn2 sensors shows that (a) sensitivity or relative change in resistance on exposure to same concentration of gas is significantly higher and (b) initial resistance in air is higher. From the results[23] It can be understood as in this case the nanowires have been dispersed from organic solution leading to poorer connectivity between nanowires. This leads to higher initial resistance as well as increased contribution to response from grain boundary region. As seen later, response to H2S arises only from grainboundary resistance. It is seen that dispersed nanowires have good response to both H2S and NO gases. Response of Zn4 samples (isolated NS) was tested to different gases. The results showed (a) the samples having only nanowires had no response to NO,H2S, NH3, CO and CH4 gases and (b) samples having one or more nanobelts had good response to NO but less than 1% change in resistance on exposure to other gases including H2S. High sensitivity of nanobelts (for NO) compared to nanowires may partly arise due to their small thickness (<20 nm) in comparison to (~ 100 nm) diameter of nanowires that yields larger surface to volume ratio. Effect of exposure to different concentration of NO and response versus concentration characteristics for a typical sensor are shown in reference[23]. The sensitivity of these sensors to NO is quite similar to that of Zn3 sensors. The results show that the response of ZnO to H2S arises only from change in resistance of grain boundaries (that are absent in Zn4 sensors) while that to NO is contributed both by intragrain and grain boundary resistances. Impedance spectroscopy measurements to confirm these findings are being carried out. The results may be compared with those reported by us on gas sensing characteristics of tellurium(a p-type semiconductor), where oxidizing gas chlorine has larger influence on grain boundary resistance and reducing gas H2S has higher contribution from intragrain regions . Further studies are necessary to understand detailed mechanism of these differences in response from intragrain and grain boundary regions. From the results[23] the lattice defect as oxygen vacancies influence the response of ZnO sensors to H2S. It is seen that the response of ZnO to H2S is mainly contributed by change in grain boundary resistance and that to NO by both intragrain and grain boundary resistance. Isolated wire sensors, having no grain boundaries provide highly selective response to NO gas. At low temperatures, the response and recovery time of commercial powders are smaller than the nanowires sensors; however, at higher temperature, the nanowires gas sensors have much faster



response times than commercial ZnO powders. This bodes well for the use of ZnO nanowires as improved gas sensors. From the data of response and recovery time, we find that less time is needed as sensing temperature increase. In addition, nanowires with smaller diameters should have larger surface areas, which should leads to shorter response and recovery time. However, while there is a slight trend towards faster responses with smaller diameters, it is imperfect.

VII. CONCLUSIONS

Due to increased surface area, single nanowires and films made using nanowires to clearly differentiate the intragrain and grain boundary contributions as well as to develop sensors with better sensitivity/ selectivity. The effect of microstructure on response of n-typesemiconductor ZnO to H₂S and NO gases hasbeen investigated. It is seen that the response toH₂S arises due to changes in grain boundary resistance while that to NO arises due to changes inboth intragrain and grain boundary resistances. In addition, nanowires with smaller diameters should have larger surface areas, which should leads to shorter response and recovery timeOxygen vacancies in the lattice were alsoseen to help in improvement of sensor response

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