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Characterization of ZnO Nanobelt-Based Gas Sensor for H₂, NO₂, and Hydrocarbon Sensing

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Abstract—A conductometric H₂, NO₂, and hydrocarbon gas sensor based on single-crystalline zinc oxide (ZnO) nanobelts has been developed. The nanobelt sensitive layer was deposited using a radio frequency (RF) magnetron sputterer. The microcharacterization study reveals that the nanobelts have a single crystal hexagonal structure with average thickness and width of about 10 and 50 nm, respectively. The sensor was exposed to H₂, NO₂ and propene gases at operating temperatures between 150 °C and 450 °C. The study showed that optimum operating temperatures for the sensor are in the range of 300 °C–400 °C for H₂, 300 °C–350 °C for NO₂, and 350 °C–420 °C for propene sensing.

Index Terms—Conductometric, gas sensor, RF sputtering, ZnO nanobelts.

I. INTRODUCTION

DEVICES based on semiconductor metal oxide (SMO) thin films are the most promising among solid-state gas sensors, due to their small dimensions, low cost, online operation, and high compatibility with microelectronic processing [1]. They have been used extensively for gas sensing based on film conductivity changes caused by interaction with gas molecules [2]–[4]. Intense research and development have been conducted to design highly sensitive, selective and stable gas sensors since Seiyama first observed gas sensing effects in metal oxides [5]. Semiconductor metal-oxide-based gas sensors are used for environmental and emission monitoring, automotive, domestic, industrial, and medical applications. The gas sensing mechanism in these materials is governed by the reactions which occur at the sensor surface between the thin-film sensitive layer and the target gas molecules. It involves chemisorption of oxygen on the oxide surface followed by charge transfer during the reactions of oxygen with target gas molecules [6]. The adsorbed gas atoms inject electrons into or extract electrons from the semiconducting material, depending on whether they are reducing or oxidizing agents, respectively [7]. This mechanism results in a change

of the film conductivity, which corresponds to the gas concentration. Although semiconductor metal-oxide gas sensors are promising, low selectivity, high-power consumption, and lack of long term stability have prevented their use in more demanding applications [8]. In the literature, there are several approaches to reduce these limitations, such as use of catalysts and promoters, multisensor array systems, optimization of sensors' operating temperature, and use of nanotechnology.

The performance of solid-state gas sensors improves with a reduction in the size of the oxide particles [9], as the entire thickness of the sensitive layer can be affected by the redox reaction during the interaction process. As a result, the performance of a gas sensor is directly related to granularity, porosity, and ratio of exposed surface area to volume. Recent advances in the synthesis, structural characterization, and investigation of physical properties of nanostructured metal oxides provide the opportunity to greatly improve the response of these materials for gas sensing. Devices based on nanostructured oxides have already been fabricated for gas sensing, for example, SnO₂ nanobelts for CO and NO₂ sensing [1], In₂O₃ nanowires for NO₂ detection [10], WO₃ nanowires for NO₂ sensing [11], and TiO₂ nanotubes for H₂ sensing [12].

Among the semiconductor metal oxides, zinc oxide (ZnO) was one of the earliest discovered and is the most widely applied oxide gas sensing materials due to its high mobility of conduction electrons and good chemical and thermal stability under operating conditions [13]. ZnO gas sensors have been fabricated in various forms, including single crystals, sintered pellets, thick films, thin films, and heterojunctions which were studied to detect H₂ [3], NO₂ [14], NH₃ [15], CH₄ [16], O₂ [17], CO [14], and ethanol [18]. Catalytic elements, such as palladium (Pd) and platinum (Pt) are often used to modify surface reactions for improving sensing properties [16], [19]. Still, a common concern about the ZnO thin-film-based gas sensor is the lack of selectivity and higher operating temperature. In general, its optimum operating temperature is in the range of 400 °C–500 °C [20].

Semiconducting zinc oxide in the form of nanobelts, nanorods, and nanowires is of growing importance for the development of highly sensitive gas sensors. ZnO nanobelts, with a distinct structural morphology (wurtzite family), characterized by a rectangular cross section and a uniform structure, are very promising for the development of stable gas sensors [21]. Nanobelts form porous structures when deposited as thin films. Their large surface to volume ratio's and nanoscale dimensions allow quick diffusion of gases into and out of the belt, which increases the intensity of reactions resulting in high sensitivity and fast sensor response and recovery times. As a result, most

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literature reports on nanostructured ZnO gas sensors [1], [19], [22]–[26] indicate that significant gas sensing performance improvement has been achieved. Wang *et al.* [22] developed ZnO nanorods coated with Pd to detect H_2 down to 10 ppm at 25 °C. Rout *et al.* [19] have employed ZnO nanorods, nanowires, and nanotubes to detect H_2 and ethanol. They found high sensitivity at 200 °C and even reduced operating temperature to 150 °C after impregnating them with 1% Pt. Xiangfeng *et al.* [23] developed a ZnO nano-tetrapod-based sensor to detect ethanol and methane and achieved high sensitivity at 300 °C. Baratto *et al.* [24] developed nanostructured fibers of ZnO to detect NO_2 down to 0.4 ppm at 100 °C. Zhang *et al.* [25] developed ZnO nanorod and nanowire-based humidity sensors and found that they are highly sensitive towards humidity and have good long-term stability and fast response time.

Various ZnO nanostructure growth techniques have been reported in literature, such as wet chemical process [26], molecular beam epitaxy [27], solid vapor deposition [28], vapor-phase transport process [25], pulsed laser deposition [29], metal-organic vapor phase epitaxy [30], electrochemical deposition [19], thermal evaporation [23], [31], and sputtering [32]. In this paper, RF sputtered ZnO nanobelt-based sensor has been developed and the dynamic responses of the sensor to different concentration of H_2 , NO_2 , and propene between 150 °C and 450 °C have been investigated.

II. EXPERIMENTAL

The sensor was designed and fabricated to operate as a resistive element. The device comprises of a sensitive ZnO nanobelt layer deposited over platinum (Pt) sputtered interdigital electrodes on a $3 \times 3 \text{ mm}^2$ sapphire substrate. A platinum sputtered heater also fabricated onto the substrate which is separated from the sensing layer and the electrodes by the electrically insulating sapphire layer.

Zinc oxide layer was deposited on the substrate using RF sputtering technique without the presence of a metal catalyst [33]. Initially, the sputtering chamber was evacuated to a pressure lower than 10^{-5} torr. Deposition of ZnO was then carried out at a pressure of 40 mtorr and an RF power of 300 W for a period of 60 min. The ZnO target was prepared by conventional solid-state method from 99.9% pure ZnO powder. The sputtering process was performed under argon (Ar) atmosphere without external heating of the substrate.

Since no metal catalyst was used in the synthesis, it is likely that the growth mechanism of nanostructured ZnO is governed by a vapor-solid (VS) process. It is believed, at first, island-shape nanoparticles of ZnO were formed on the substrate similar to Stranski-Krstanov type growth model [34]. These nanoparticles acted as nucleation sites or seed for subsequent growth of ZnO nanobelts as the surface energy of these nucleation sites is higher than that of the flat surface.

In addition, when the sputtering power is low (for instance lower than 200 W), the deposited ZnO layer is transparent, indicating that a thin film of highly ordered crystallites has been formed. High sputtering power is needed for the formation of ZnO nanostructures which produces a supersaturation deposition condition [33]. In this work, the ZnO films deposited at an

Fig. 1. SEM image of ZnO nanobelts on alumina substrate in 5 μm scale [39].

Fig. 2. SEM image of ZnO nanobelts on alumina substrate in 1 μm scale [39].

RF power of 300 W appear white. This indicates randomly oriented nanostructures.

The sensor was mounted inside an enclosed environmental cell. Four mass flow controllers (MFC) were connected to form a single output that supplies gas to the cell. A constant flow rate of 0.2 liters per minute was delivered via the MFCs. A computerized gas calibration system was used to vary the concentration of H_2 , NO_2 , and propene in synthetic air. A high precision Keithley 2001 multimeter was used to measure the variation of sensor resistance. Custom LabVIEW-based software was used to autonomously control the experimental setup and take measurement of the sensor. The sensor responses were displayed in real-time and saved for offline processing and analysis. Gas exposure time was fixed for each pulse of analyte gas and the cell was purged with synthetic air for fixed periods of time between each pulse to allow the surface of the sensor to recover to the atmospheric conditions.

Heating for the device is provided by a micro heater fabricated on the back side of the sapphire substrate with a patterned platinum resistive element. A regulated DC power supply was connected to the heater to control the operating temperature of the sensor between the range of 150 °C and 450 °C in increments of approximately 30 °C. A thermocouple was used to obtain a real-time reading of the sensor surface temperature with

Fig. 3. (a) TEM bright field image of a ZnO nanobelt, the inset is a zoom of $\bar{1}100$ spot. (b) Associated SADP of ZnO layer [33].

1 °C accuracy. The sensor was exposed to H₂ gas pulse sequence of 0.06%, 0.12%, 0.25%, 0.50%, 1%, and 0.12% concentration in synthetic air, NO₂ gas pulse sequence of 0.51, 1.06, 2.12, 4.25, 8.5, and 1.06 ppm concentration in synthetic air and propene gas pulse sequence of 0.25%, 0.50%, 1%, and 0.25% concentration in synthetic air.

III. RESULTS

Structural Characterization: The scanning electron microscopy (SEM) (Figs. 1 and 2) images indicate that the as-grown ZnO layer on the substrate consists of a large quantity of wire-like nanostructures with typical lengths in the range of several micrometers. The wire-like nanostructures are not evenly distributed across the surface; instead there are islands with thousands of nanowires. The transmission electron microscopy (TEM) bright field image and the associated selected area diffraction pattern (SADP) of wire-like ZnO nanostructure are shown in Fig. 3(a) and (b), respectively. From the TEM trace analysis, it was found that the ZnO nanostructure grew along $[11\bar{2}0]$ and $[2\bar{1}10]$ directions on the (0001) plane [33]. The TEM image and the cross streaking of the $\bar{1}100$ spot in the inset of Fig. 3(a) indicate that the geometrical shape of the ZnO nanostructures can be categorized as nanobelts. The analysis of the TEM image also suggests that the ZnO nanobelts have a distinct structural morphology characterized by a rectangular cross section and a uniform structure. The analysis of SEM and TEM images show that the as-grown nanobelts have a single crystal hexagonal structure with average thickness and width of about 10 and 50 nm, respectively.

Electrical Characterization: The dynamic properties of the sensor such as sensitivity, stability, response, and recovery times were all found to be temperature dependent. The sensor requires an elevated operating temperature to enhance redox reactions to achieve the optimum conditions [35]. Dynamic responses of the sensor to different concentrations of H₂ at 385 °C and 420 °C and NO₂ at 300 °C and 325 °C are shown in Figs. 4 and 5, respectively. It was observed that with the increase of temperature, baseline resistance of the sensor was decreased and the response and recovery times of the sensor were reduced. For H₂ gas, the dynamic response was not linear with respect to gas concentrations. It is believed that at high concentrations of H₂, saturation may take place due to a lack of adsorbed oxygen ions

Fig. 4. Dynamic response of the conductometric sensor to different H₂ gas concentrations in synthetic air at 350 °C and 420 °C.

Fig. 5. Dynamic response of the conductometric sensor to different NO₂ gas concentrations in synthetic air at 300 °C and 325 °C.

to react with analyte molecules. It is well known that in an air environment, oxygen molecules adsorb onto the surface of the ZnO layer to form O₂⁻, O⁻, and O₂²⁻ ions by extracting electrons from the conduction band depending on the temperature [6], [36]. Takata *et al.* [37] found that the stable oxygen ions were O₂⁻ below 100 °C, O⁻ between 100 °C and 300 °C, and O₂²⁻ above 300 °C. The oxygen adsorptions on the surface of nanobelts can be explained by the following equations:

The positively charged ZnO surface and negatively charged adsorbed oxygen ions form a depletion region at the surface. Since ZnO nanobelts have single crystalline structure with average thickness less than 50 nm (less than Debye length), adsorbed oxygen can easily penetrate through the bulk of the belt. As a result, free carriers can travel through the bulk of the belt in a similar way to the channel of a field-effect transistor (FET) [1]. When the device is exposed to a target gas, two different extremes may occur: pinch-off and fully conductive states. A complete depletion of carriers inside the belt will produce a pinched-off channel. A complete removal of adsorbed oxygen from the belt will produce a highly conductive channel. When an n-type ZnO nanobelt surface is exposed to a reducing gas such as hydrogen or propene at elevated temperatures, analyte molecules dissociate and combine with the adsorbed oxygen, thereby reinjecting electrons. Above 300 °C, the reactions can be expressed by the following equations:

Due to increase concentration of electrons, the depletion region decreases to produce a conductive channel along the belt, which strongly increases belt conductivity.

In the case of strong oxidizing gas NO_2 , reactions take place directly with the oxide surface rather than with the oxygen chemisorbed at surface. During the interaction process, NO_2 molecules consume conduction electrons and subsequently increase the depletion region at the surface. Thus, the ZnO nanobelt layer conductivity is reduced after exposure to NO_2 . The sensor response to NO_2 can be explained by the following reactions:

The sensitivity of a semiconducting oxide gas sensor is defined as follows:

$$(a) \text{ for reducing gas: } S = [\text{R}_{\text{air}} - \text{R}_{\text{gas}}]/\text{R}_{\text{gas}};$$

$$(b) \text{ for oxidizing gas: } S = [\text{R}_{\text{gas}} - \text{R}_{\text{air}}]/\text{R}_{\text{air}};$$

where R_{air} is the resistance of the sensor in air, R_{gas} is the resistance of the sensor in presence of gas.

The sensitivity of the sensor towards H_2 and NO_2 as a function of operating temperature is shown in Fig. 6 and towards propene in Fig. 7. The sensitivity versus operating temperature curve shows a maximum which depends on the target gas. This could be explained by the temperature dependence of the adsorption and desorption process on the metal-oxide surface [38]. It was observed that the sensitivity towards NO_2 is highest at 220 °C–250 °C but other performance parameters such as response and recovery times are long, repeatability is poor, and the baseline resistance is not stable. Therefore, the tradeoff between different parameters is needed in choosing the operating temperature. Analysis of the experimental results suggest that the optimum operating temperatures for the sensor are in the range of 300 °C–400 °C for H_2 , 300 °C–350 °C for NO_2 , and 350 °C–420 °C for propene sensing. However, it was found that the sensor has the fastest response and recovery with greater repeatability and baseline stability at operational temperatures of

Fig. 6. Sensitivity versus operating temperature for 1% H_2 and 8.5 ppm NO_2 gas concentrations in synthetic air.

Fig. 7. Sensitivity versus operating temperature for 1% propene concentration in synthetic air.

Fig. 8. Dynamic response of the conductometric sensor to different H_2 gas concentrations in synthetic air at 385 °C [39].

385 °C, 350 °C, and 370 °C for H_2 , NO_2 , and propene gas, respectively. Figs. 8–10 show the dynamic responses of the sensor to different concentrations of H_2 at 385 °C, NO_2 at 350 °C, and propene at 370 °C, respectively. At these temperatures, the sensitivity of the sensor was calculated to be 14.3 for 1% H_2 , 0.81 for 8.5 ppm NO_2 , and 0.17 for 1% propene. Fast response time of 48, 180, 72 s, and recovery time of 336, 268, 252 s were observed for 1% H_2 , 1.06 ppm NO_2 , and 1% propene gas, respectively.

Reproducibility was observed as indicated when a second pulse of 0.12% H_2 , 1.06 ppm NO_2 , and 0.25% propene were introduced into the sensor chamber. It was found that the ZnO nanobelt-based sensor produce repeatable responses of the same magnitude with good baseline stability.

Fig. 9. Dynamic response of the conductometric sensor to different NO₂ gas concentrations in synthetic air at 350 °C [39].

Fig. 10. Dynamic response of the conductometric sensor to different propene gas concentrations in synthetic air at 370 °C.

IV. CONCLUSION

A conductometric gas sensor has been fabricated based on ZnO nanobelts synthesized by RF sputtering of a zinc oxide target under controlled conditions. Novel gas sensor based on these ZnO nanobelts has been investigated towards H₂, NO₂, and hydrocarbon at different operating temperatures between 150 °C and 450 °C. Study shows that the optimum operating temperatures for the sensor are in the range of 300 °C–400 °C for H₂, 300 °C–350 °C for NO₂, and 350 °C–420 °C for propene sensing. The fastest response and recovery with greater repeatability for H₂, NO₂, and propene gases occurred at 385 °C, 350 °C, and 370 °C, respectively. The results demonstrate that the developed sensors are promising for industrial applications.

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