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Room temperature non-balanced electric bridge ethanol gas sensor based on a single ZnO microwire*

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In this paper, ultra-long and large-scaled ZnO microwire arrays are grown by the chemical vapor deposition method, and a single ZnO microwire-based non-balanced electric bridge ethanol gas sensor is fabricated. The experimental results show that the gas sensor has good repeatability, high response rate, short response, and recovery time at room temperature (25 °C). The response rate of the gas sensor exposed to 90-ppm ethanol is about 93%, with a response time and recovery time are 0.3 s and 0.7 s respectively. As a contrast, the traditional resistive gas sensor of a single ZnO microwire shows very small gas response rate. Therefore, ethanol gas sensor based on non-balanced electric bridge can obviously enhance gas sensing characteristics, which provides a feasible method of developing the high performance ZnO-based gas sensor.

Keywords: ZnO microwire, gas sensor, room temperature, ethanol

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1. Introduction

In recent years, volatile organic compounds (VOC) gas sensors have attracted much attention due to the increasing demand for health care and environmental protection.[1–3] In all VOC sensors, ethanol (C_2H_5OH) gas sensor plays an important role in the chemical industry, food, and biomedical science.[4] Therefore, it is very important to develop an ethanol gas sensor with high sensitivity, fast response, and low cost. Among the various gas sensing materials, ZnO material exhibits the characteristics required for an excellent gas sensor such as large band-gap energy of 3.37 eV, large exciton binding energy of 60 meV, high mobility of conduction electrons (200 $cm^2/(V\cdot s)$), good chemical and thermal stability.[5–7] The different forms of ZnO gas sensors have been prepared, such as powders, films, heterojunctions and micro/nanostructures. Among these forms, ZnO micro/nanostructures, especially micro/nanowires are particularly important for gas sensing due to their high length-to-diameter ratios and high surface-to-volume ratios.[8–10] Therefore, ZnO-micro/nanowire gas sensors are expected to be able to detect lower concentration gas and exhibit better sensing performance than bulk and thin film gas sensors.[11]

Generally, traditional ZnO gas sensors need operate at high temperatures of 100 °C–400 °C to achieve good sensing performance. Apart from high power consumption, the device operating at high temperatures also causes the flammable and explosive gases to be ignited.[12] Furthermore, the high operating temperature adversely affects sensor's reliability and durability.[13] Therefore, ZnO-based gas sensors operating at room temperature without heating devices are more practicable and cost-effective, according to the current developing trend. Moreover, to the best of our knowledge, the research on ZnO-based non-balance electric bridge gas sensor has not been reported. In this paper, the large-scaled ZnO microwires (MWs) arrays are grown by the chemical vapor deposition method. The diameter and length of MWs are about 20 μm–50 μm and 1 cm–2.5 cm, respectively. The non-balanced electric bridge ethanol gas sensor based on a single ZnO microwire is fabricated. And the gas sensing properties are also investigated at room temperature. The experimental results indicate that the non-balanced electric bridge gas sensor has ultrahigh sensitivity, wonderful selectivity, rapid response, and recovery time ethanol compared with traditional resistive gas sensor of single ZnO microwire.

2. Experimental procedure

2.1. Synthesis of ZnO microwires

The ZnO microwires were synthesized by the chemical vapor deposition (CVD) method. The well-mixed ZnO powders and graphite powders with a mass ratio of 20 : 3 were placed in a quartz boat and the boat was located in the center of the tube furnace. The cleaned Si substrate was placed horizontally on the downstream side of the source 15 cm far. High purity Ar carrier gas was passed into the furnace at a rate of 200 sccm during the entire synthesis. In the reaction pro-

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cess, oxygen gas was introduced into the reacting chamber at a flow rate of 30 sccm. The temperature was kept at 1000 °C for 30 min, and then the furnace was cooled down to room temperature. It could be found that a large-scaled ZnO MW arrays visible to the naked eye were observed on the Si substrate.

2.2. Characterizations

The surface morphologies of the samples were characterized by digital camera and scanning electron microscopy (Hitachi TM3030). The elemental analysis of single ZnO microwire was performed by energy dispersive x-ray spectrometer (EDS) equipped in a scanning electron microscope. Photoluminescence (PL) measurements were performed by using a micro-PL system at room temperature. For PL measurements, He–Cd laser with 325 nm in wavelength was used as an excitation source.

2.3. Fabrication and measurement of gas sensors

In this work, the ethanol gas sensor is based on the principle of non-balance electric bridge by using a single ZnO microwire as one bridge arm R_4 and other three fixed-value resistors R_1 (10 kΩ), R_2 (10 kΩ), and R_3 (50 kΩ) to form the four bridge arms. The schematic diagram is shown in Fig. 1. The sheet glass and the conductive silver paste were used as the substrates and electrodes of single ZnO microwire, respectively. The sheet glass was washed with acetone, ethanol, and deionized water under sonication in sequence. The selected single ZnO microwire was placed on the sheet glass. And the two ends of the microwire were fixed by the conductive silver paste, then connected a single ZnO microwire into the non-balanced electric bridge as R_4. The as-prepared devices were located in the chamber of our homemade gas sensing system. The Keithley 4200 semiconductor characterization system was used to monitor the sensing property by using the two-point (A and B) measurement as indicated in Fig. 1. Experimental tests were conducted under an applied voltage of DC 5 V at room temperature (25 °C) in dark room.

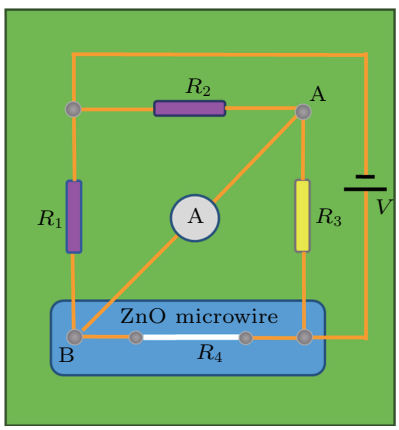


Fig. 1. Schematic diagram of non-balanced electric bridge ethanol gas sensor based on single ZnO microwire.

Gas-sensing measurements were performed in ambient air at 26% relative humidity. The sensing performance was characterized by the change in current when the sensor was exposed to the ethanol gas, and the sensor response rate was defined as[14–16]

$$S = \left| \frac{(I_g - I_a)}{I_a} \right| \times 100\%, \tag{1}$$

where I_a and I_g are the current value of the sensor in air and in gaseous ethanol, respectively. The response and recovery time of the gas sensor are also crucial parameters.

3. Results and discussion

3.1. Morphology and structure

Figure 2(a) shows the photograph of as-grown ZnO microwires. As shown in Fig. 2(a), a large number of white and transparent microwires are prepared on the entire Si substrate, which have the length ranging from 1 cm–2.5 cm. Furthermore, it can be seen that the ZnO microwires have a large size and high density, which makes it convenient to prepare the gas sensor. Figure 2(b) shows the scanning electron microscope (SEM) image of the individual ZnO microwire. The single ZnO microwire has a perfect smooth surface and a diameter of 30 μm. To determine the composition of ZnO microwires, we perform the EDX analysis, and the result indicates that the sample is composed of O, Zn elements without any impurities. And the molar percentage of O and Zn in microwire are about 48.5% and 51.5%, respectively. That is, the atomic ratio of O/Zn is close to 1 : 1.

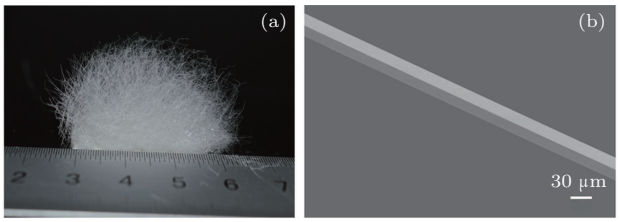


Fig. 2. (a) Photograph of ZnO microwires arrays, and (b) SEM image of single ZnO microwire.

3.2. Optical properties

Room temperature micro-PL measurements of a single ZnO microwire are carried out and shown in Fig. 3. The PL spectrum exhibits a strong near-band-edge (NBE) emission centered at about 377 nm (3.29 eV) and a broad green emission centered around 510 nm (2.43 eV). The NBE is normally ascribed to the recombination of free excitons.[17–19] The green band emission accounts for the recombination of the excited donor electrons with the deep level donor or acceptor like defects such as zinc interstitials (V_{Zn}) and oxygen vacancies (V_O).[20,21]

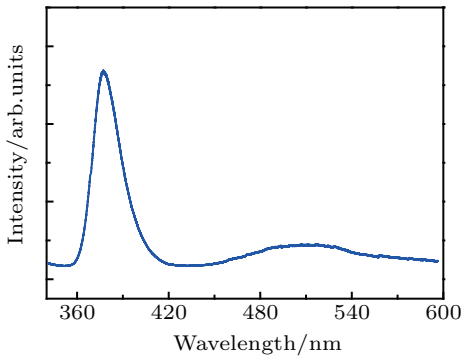


Fig. 3. Room-temperature micro-PL spectrum of single ZnO microwire.

3.3. Gas sensing properties

The large size microwire is chosen for easy manipulation under an optical microscope or naked eye. The single ZnO microwire with a length as long as 2.0 cm and diameter of about 30 μm for gas detector is selected under an optical microscope to prepare the sensor.

Figure 4 showed the *I*–*V* characteristics of a single ZnO microwire at room temperature, with the distances between the both electrodes being 1.5 cm. The *I*–*V* curves are measured with a bias voltage in a range from −10 V to +10 V. It can be seen that the *I*–*V* curve is nearly linear, indicating a fairly good Ohmic contact between silver electrode and ZnO microwire. It ensures that all upcoming behaviors of electrical devices represent the gas-sensing properties but not the contact between the materials and the electrodes. In addition, from the slope of a straight line, the ZnO resistance value and the resistivity can be calculated to be about 87 kΩ and 10 Ω·cm, respectively.

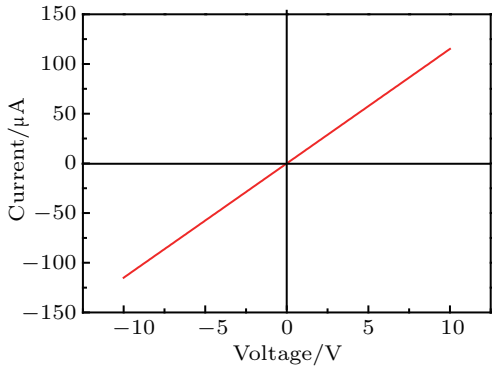


Fig. 4. Room temperature *I*–*V* characteristics of a single ZnO microwire.

The time-evolving dynamic response–recovery curves of the non-balanced electric bridge gas sensor based on a single ZnO microwire for ethanol gas concentration sequence of 10, 30, 50, 70, 90 ppm at room temperature are shown in Fig. 5(a). It is clearly seen that the rectangle-shaped sensing behavior and sensor presents sensitive and reversible response to ethanol with different concentrations. The current reveals a drastic decrease with ethanol gas concentration increasing and recovers to its initial value after exposing gas to air. Figure 5(b) shows the corresponding response rate *versus* ethanol concentration in a range from 10 ppm to 90 ppm. From the curves, it is found that the response rate increases with tested gas concentration increasing, showing its nearly linear correlation with ethanol concentration. When the ethanol concentration is 90 ppm, the response rate of the device can reach 93%.

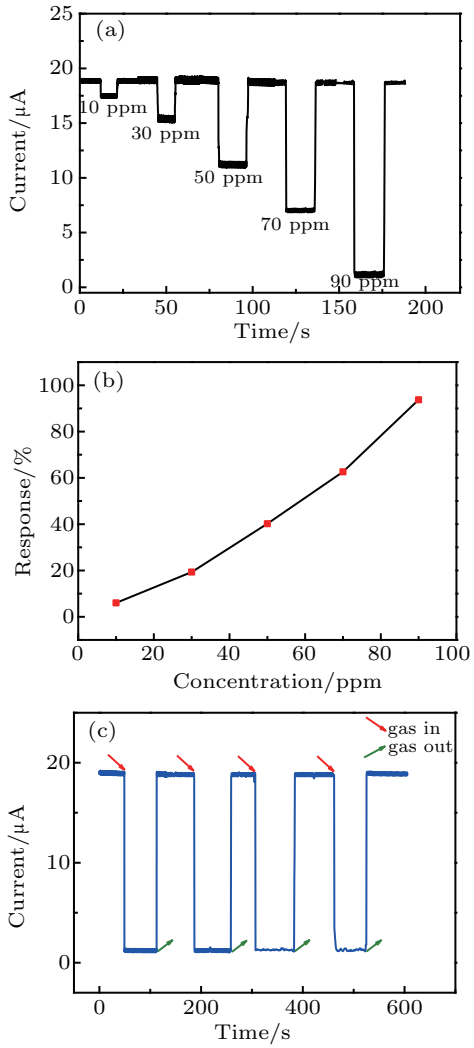


Fig. 5. (a) Dynamic response–recovery curves of the non-balanced electric bridge gas sensor based on a single ZnO microwire for different ethanol concentrations at room temperature, (b) response of the sensor to ethanol concentration in a range of 10 ppm–90 ppm, and (c) reproducibility and stability of the sensor exposed to ethanol gas of 90 ppm at room temperature.

Reproducibility and stability are also very important characteristics of gas sensors, and are required by gas sensors for long life service-term and the ability to respond successfully to testing gases without a visible decrease in sensor response rate.[22,23] Figure 5(c) shows the response rate–recovery curves of gas sensor exposed to four representative reversible cycles of continuous tests for 90 ppm of ethanol gas at room temperature. It can be observed from Fig. 5(c) that when sensors are exposed to gaseous ethanol, the current decreases rapidly and then returns to its initial value when the sensor is re-exposed to air. The gas sensor shows good response and recovery characteristics among the four cycles, which indicates that the gas sensor has a perfect replicability

and good stability. Both response and recovery are very fast, taking about 0.3 s and 0.7 s, respectively.

In order to study the difference in gas sensitivity between non-balanced electric bridge structure gas sensor and the conventional resistive gas sensor (the electrodes at both ends of the ZnO microwire are directly connected to the power source), at the same test temperature of 25 °C, applied voltage of DC 5 V and ethanol gas concentration of 90 ppm, we test the response curves of the two sensors, and the results are shown in Figs. 6(a) and 6(b). In Figs. 6(a) and 6(b), the conventional resistive sensor has a responsivity of 0.5%, the response and recovery time of 6.5 s and 2.1 s, respectively, and the gas sensor of the non-balanced electric bridge structure has a response rate of 93% and the response and recovery time of 0.3 s and 0.7 s, respectively. It can be clearly seen that device using non-balanced electric bridge structure can significantly increase its sensor response rate, and reduce its response and recovery time.

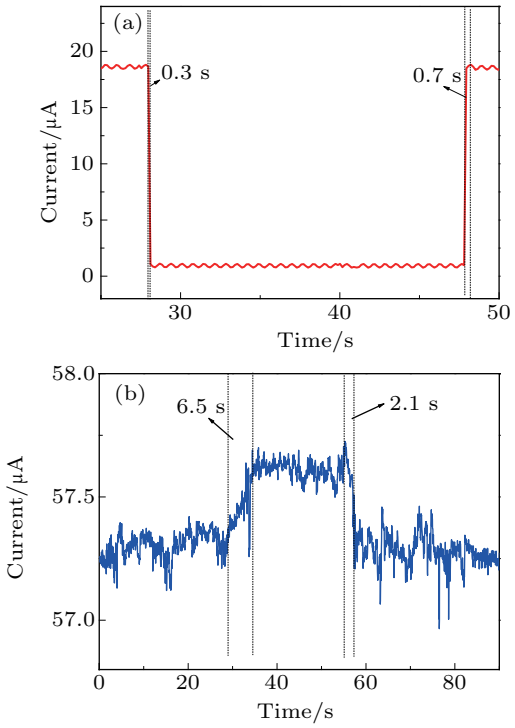


Fig. 6. Response and recovery curves of the two kinds of gas sensors at 90-ppm ethanol: (a) non-balanced electric bridge structure, and (b) conventional resistive structure.

In order to prove that the current changes of the device in the ethanol gas are from the ZnO microwire, we make four non-balance electric bridge devices by replacing the bridge arm R_4 with the fixed resistances of 10 kΩ, 20 kΩ, 50 kΩ, and 100 kΩ, respectively. Figure 7 shows the responses of the five sensors to ethanol gas of 90 ppm at room temperature with applying biase voltage of 5 V. It can be seen that the device is immersed into ethanol gas for 20 s and then taken out and placed in the air for 60 s. It is found that when R_4 value is any of the four fixed resistance values, the device does not respond to the ethanol gas. In addition, it is also found that with resistance R_4 decreasing, the current value between points A and B in Fig. 1 decreases gradually.

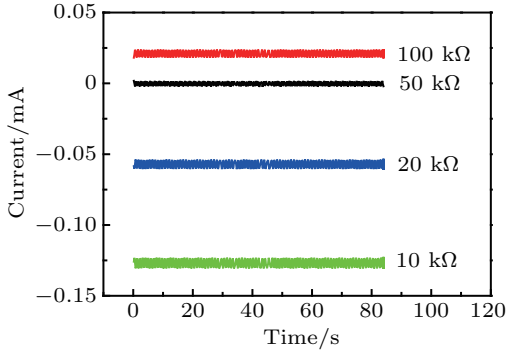


Fig. 7. Response curve of fixed value resistance acting as bridge arm R_4 at ethanol gas concentration of 90 ppm.

3.4. Gas sensing mechanism

The ZnO gas sensing mechanism, which has been widely accepted, is the depletion theory, including the model of charge depletion layers induced by the adsorption/desorption of oxygen molecules on the surface of sensing material.[24–26] When the gas sensing material is exposed to air, oxygen molecules are adsorbed on the ZnO microwire surface. The adsorbed oxygen molecules can extract electrons from the sensing ZnO material due to their large electronegativity and form oxygen species such as molecules (O_2^-) and (O^-, O^{2-}) by capturing electrons from the ZnO conduction band at temperatures of 25 °C–500 °C, as described in the following reaction equations:[27]

$$O_{2(gas)} \rightarrow O_{2(ads)}, \tag{2}$$

$$O_{2(ads)} + e^- \rightarrow O^-_{2(ads)}$$
(O_2^- dominates at 25 °C–150 °C), (3)

$$O^-_{2(ads)} + e^- \rightarrow 2O^-_{(ads)}$$
(O^- dominates at 150 °C–350 °C), (4)

$$O^-_{(ads)} + e^- \rightarrow O^{2-}_{(ads)}$$
(O^{2-} dominates above 350 °C). (5)

Of these oxygen species, O_2^- is dominant at room temperature.[28]

Thus, the electron depletion layers are formed in the surface regions of ZnO microwire as shown in Fig. 8. Here, the concentration of electrons, as majority carriers, is very low and eventually increases the resistance of the gas sensor. Secondly, when ethanol is added into the atmosphere, the O_2^- ions on the surface of ZnO microwire react with the ethanol gas molecules at room temperature, releasing the trapped electrons back to the conduction band of ZnO. This process leads the resistance of the sensor to increase, which can be described with the following reaction equation:

$$C_2H_5OH + 3O^-_{2(ads)} \rightarrow 2CO_2 + 3H_2O + 3e^-. \tag{6}$$

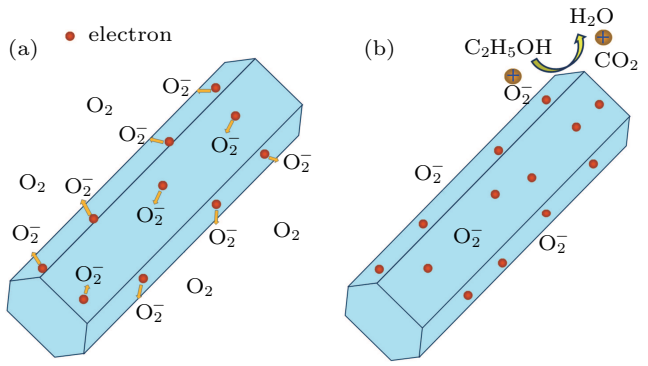


Fig. 8. Schematic illustration forsensing mechanism of ZnO microwire sensor to ethanol: (a) exposed to air, and (b) exposed to ethanol.

According to the analysis of the possible gas sensing mechanism, when the ZnO microwires are placed in the ethanol gas, the resistance of the microwire sensor decreases, that is, the resistance R_4 decreases.

The expression of the output voltage V_g between the two points A and B is given. The output voltage V_g between the two points A and B can be formulated as follows:[29]

$$\begin{aligned} V_g &= \left(\frac{R_2}{R_2+R_3}-\frac{R_1}{R_1+R_4}\right)V \\ &= \frac{R_2(R_1+R_4)-R_1(R_2+R_3)}{(R_1+R_4)(R_2+R_3)}V, \end{aligned} \tag{7}$$

where V is the bias voltage.

The equivalent resistance R between points A and B is expressed by the following relation:

$$\begin{aligned} R &= \frac{R_1R_4}{R_1+R_4}+\frac{R_2R_3}{R_2+R_3} \\ &= \frac{R_1R_4(R_2+R_3)+R_2R_3(R_1+R_4)}{(R_1+R_4)(R_2+R_3)}. \end{aligned} \tag{8}$$

Therefore, the current I_g can be obtained as

$$\begin{aligned} I_g &= \frac{V_g}{R} = \frac{R_2(R_1+R_4)-R_1(R_2+R_3)}{R_1R_4(R_2+R_3)+R_2R_3(R_1+R_4)}V \\ &= \frac{R_2R_4-R_1R_3}{R_1R_4(R_2+R_3)+R_2R_3(R_1+R_4)}V. \end{aligned} \tag{9}$$

In this experiment, $V = 5$ V, $R_1 = R_2 = 10$ kΩ, and $R_3 = 50$ kΩ. For easy calculation, the unit of R_4 is kΩ and the unit of I_g is mA. The above equation (9) can be simplified into

$$I_g = \frac{R_4-50}{22R_4+100}. \tag{10}$$

According to the above analysis, the ZnO microwires serving as resistance R_4 is strongly dependent on ethanol concentration. When the ZnO microwires are placed in the ethanol gas, the resistance value R_4 decreases, and the current also decreases as indicated by formula (10). This result is consistent with the decrease in current value in Fig. 5(a). Furthermore, we can see from the relevant literature that the non-balanced electric bridge structure can amplify a small amount of resistance change,[30] so the gas sensor can significantly enhance the response rate, response time, and recovery time of the device.

4. Conclusions

Ultra-long and large-scaled ZnO microwire arrays are grown by the chemical vapor deposition method without using metal catalyst. The surface of the ZnO microwire is very smooth, with neither particles nor clusters attached to it. The length of the microwires is about 1 cm–2.5 cm. The ethanol gas sensor is based on the principle of non-balanced electric bridge, by using a single ZnO microwire as one bridge arm R_4 and other three fixed-value resistors R_1 (10 kΩ), R_2 (10 kΩ), and R_3 (50 kΩ) to form a four-arm bridge. The experimental results show that the gas sensor has good repeatability, high sensitivity, short response, and recovery time at room temperatures. When the ethanol concentration is 90 ppm, the response rate of ethanol gas sensor can reach 93%, the response time is about 0.3 s, and the recovery time is about 0.7 s. The traditional resistive gas sensor of a single ZnO microwire has very small gas response rate at room temperatures. The conventional resistive sensor has a response rate of 0.5%, the response time and recovery time are 6.5 s and 2.1 s, respectively. It can be seen that the ethanol gas sensor manufactured by using the non-balanced electric bridge structure can significantly enhance the gas sensing property.

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