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Facile synthesis and enhanced ethanol sensing properties of the brush-like ZnO–TiO_2 heterojunctions nanofibers

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ABSTRACT

Three-dimensional brush-like ZnO–TiO_2 hierarchical heterojunctions nanofibers have been successfully obtained by the combination of the electrospinning and hydrothermal process. The FESEM images showed that the brush-like ZnO–TiO_2 hierarchical heterojunctions nanofibers are composed of uniform ZnO nanorods layer of approximately 100–300 nm in diameter grown on the side surface of TiO_2 core nanofibers. The gas sensing studies revealed that the ZnO–TiO_2 sensors exhibited enhanced sensing performance to ethanol compared with the pristine TiO_2 nanofibers and pristine ZnO nanorods, which might be attributed to the unique hierarchical structure and great degree of electron depletion of the interface based on the synergistic effect among the two components of TiO_2 and ZnO.

1. Introduction

Three-dimensional hierarchical nanostructures with different morphologies and elements are of great interest because their unique structures enable physical properties that make them attractive materials for applications such as photonic devices, catalysis, chemical sensors, drug delivery, and energy conversion and storage systems [1–3]. In recent years, electrospinning has been widely investigated as a simple and robust technique for the production of nano- and microfibers of various materials such as polymers, metal oxides, and even metals. One-dimensional nanofibers appear to have several amazing characteristics such as very large surface area to volume ratio, flexibility in surface functionalities, and superior mechanical performance compared with any other known form of the material [4–6]. Therefore, surface-functionalized nanofibers have been of great significance because the resulting products may possess improved physical and chemical properties, which should find applications in a variety of fields such as photocatalysts, gas sensors and supercapacitors [7–9]. However, it is important to select suitable materials and appropriate methods to introduce the desired functionality onto nanofiber surfaces to meet specific needs.

Inorganic semiconductor nanostructures are among some of the most attractive nanomaterials for gas sensors applications [10]. Among various semiconductor oxides, TiO_2 and ZnO are two kinds of great technologically important materials due to their excellent electronic, chemical and optical properties [11–13]. However, pristine TiO_2 is with a high resistance, which may bring about the problem of accuracy of detection for gas sensors [14]. Therefore, incorporating two of these materials into an integrated structure could improve this problem. As a versatile wide band gap semiconductor (E_g = 3.37 eV), ZnO exhibits high mobility of conduction electrons and other excellent functional properties for widely potential applications in UV absorbers, gas sensors, and photocatalysis [15–17]. Besides the capabilities of ZnO for the above potential applications, it is undoubted that ZnO is one of the most widely researched and used gas sensing materials [18]. Recently, ZnO nanowire arrays and nanorods were grown on flat substrates or inorganic nanofibers by a simple seeding method [19]. However, to the best of our knowledge, the reports of synthesis of ZnO nanorods surface-functionalized inorganic nanofibers by an electrospinning and hydrothermal route and achieve unusual gas sensing characteristics are still sparsely.

In this work, we report a facile and effective strategy to prepare brush-like ZnO–TiO_2 hierarchical heterojunctions nanofibers composed of highly dispersed ZnO nanorods well-grown on electrospun TiO_2 nanofibers. Excellent ethanol sensing properties such as high response and quick response–recovery based on our sensor have been observed at 320 °C. Especially, in comparison with TiO_2 nanofibers and ZnO nanorods, the brush-like ZnO–TiO_2 hierarchical heterojunctions sensor shows high response, low detection limit, rapid response and better selectivity to ethanol. The results demonstrate a promising approach to fabricate high-performance ethanol sensors with high response and better selectivity.

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Fig. 1. Strategy for growing ZnO nanorods onto TiO_2 nanofibers surface using hydrothermal method.

2. Experimental

2.1. Materials

All the reagents were of analytical grade and used without further purification. Tetrabutyl titanate and glacial acetic were purchased from Sinopharm Chemical Reagent Co. (Shanghai, China).

2.2. Synthesis process

2.2.1. Preparation of TiO_2 nanofibers

TiO_2 nanofibers were synthesized followed the reported literature [19] with some modification. 2 g of tetrabutyl titanate was mixed with 2 g of glacial acetic and 7.5 g of ethanol. Then the homogeneous solution was added to poly-(vinlypyrrolidone) (PVP) solution of about 11.5 wt%, followed by vigorous stirring at room temperature for 4 h. Composite PVP/tetrabutyl titanate nanofiber film was fabricated by electrospinning from a syringe at an applied electric voltage of 20 kV. TiO_2 nanofibers were obtained by calcining the composite nanofibers at 500 °C for 2 h in air.

2.2.2. Preparation of ZnO nanorods

30 mL of aqueous solution containing 0.05 g $Zn(NO_3)_2{\cdot}6H_2O$ and 5 mL of hexamethylenetetramine (HMT, $C_6H_{12}N_4$, 0.05 mol L^{-1}) was transferred into a stainless Teflon-lined 50 mL autoclave. Then the autoclave was sealed and kept at 100 °C for 3 h in an oven. The obtained products were collected and washed with absolute ethanol and distilled water several times and then dried in an oven at 60 °C.

2.2.3. Fabrication of ZnO/TiO_2 heterojunctions

For the synthesis of the ZnO/TiO_2 hierarchical structures, 30 mL of aqueous solution containing 0.05 g $Zn(Ac)_2{\cdot}2H_2O$ and 0.05 mol L^{-1} hexamethylenetetramine (HMT, $C_6H_{12}N_4$) was transferred into a stainless Teflon-lined 50 mL autoclave. After TiO_2 fibers were added into the reaction system, the autoclave was sealed and kept at 100 °C for 3 h in an oven. The obtained products were collected and washed with absolute ethanol and distilled water several times and then dried in an oven at 60 °C, the obtained products were collected for further analyses. The experimental procedure is shown in Fig. 1.

2.3. Characterization

The crystal structures of the products were determined by a Rigaku D/Max 2550 X-ray diffractometer with Cu-Kα radiation (λ = 0.15418 nm) in the range of 20–80° (2θ) at room temperature. Field emission scanning electron microscope (FESEM: SHIMADZU Japan, SSX-550) images of the products were taken. Some of the product was placed on a conductive adhesive for transmission electron microscope for analysis (TEM: Japan RILI H-8100).

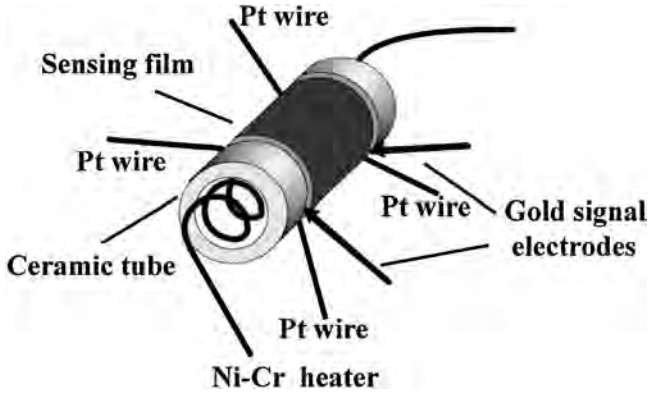

Fig. 2. Schematic structure of the gas sensor.

2.4. Fabrication and measurement of gas sensor

The products were mixed with deionized water at a weight ratio of 4:1 to form a paste. The sensors were made by coating ceramic tube with the paste to form a thin 10 μm sensing film. A pair of gold electrodes was installed at each end of the ceramic tube before it was coated with the paste; each electrode was connected with two Pt wires. A Ni–Cr heating wire was inserted into the tube to form an indirect-heated gas sensor. The structure of the sensor is shown in Fig. 2. The details of the sensor fabrication are similar to those reported in the literature [20].

The electrical properties of the sensor were measured by using a CGS-8 Intelligent Gas Sensing Analysis System (Beijing Elite Tech Co., Ltd.). The sensor response was defined as the ratio ($S = R_a/R_g$) of the resistance of the sensor in dry air (R_a) to that is target gases (R_g). The operating temperature of the sensor was varied between 260 and 400 °C. The response and recovery time were defined as the time taken by the sensor to achieve 90% of the total resistance change in the case of adsorption and desorption, respectively.

3. Results and discussion

3.1. Structural and morphological characteristics

XRD patterns in Fig. 3 reveal the overall crystal structure and phase purity of the three samples: ZnO nanorods (Fig. 3a), TiO_2 nanofibers (Fig. 3b) and brush-like ZnO–TiO_2 heterojunctions nanofibers (Fig. 3c), respectively. All the diffraction peaks for the two products match well with those of standard XRD patterns of anatase TiO_2 (JCPDS No. 21-1272) and hexagonal wurtzite-type ZnO (JCPDS No. 36-1451). No other impurity peaks are detected, which indicates the absence of other impurities.

The morphology and size of the as-prepared TiO_2, ZnO and ZnO–TiO_2 products were observed by using a field emission scanning electron microscope (FESEM). Fig. 4a shows the FESEM images

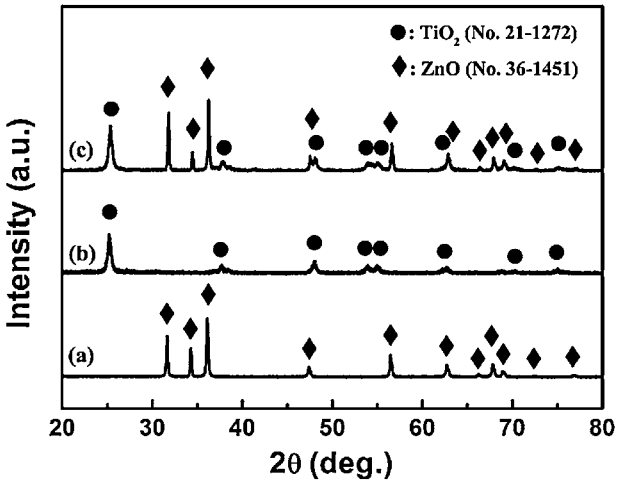

Fig. 3. XRD patterns of (a) ZnO nanorods, (b) TiO_2 nanofibers, and (c) ZnO–TiO_2 heterojunctions.

of the pristine TiO_2 nanofibers. The nanofibers have a uniform diameter distribution of about 100 nm and possess a relatively smooth surface. For comparison, the FESEM image of pristine ZnO nanorods is shown in Fig. 4b. The diameters of the ZnO nanorods ranged from 100 to 300 nm, and the average length was about 2 μm. For ZnO–TiO_2 heterojunctions, a typical image of such structure is shown in Fig. 4c and d. After applying the hydrothermal growth of ZnO nanorods, the initially smooth TiO_2 nanofibers branched out, formed a hierarchical structure resembling the combs and brushes. The ZnO branches stand perpendicular to the side surfaces of the TiO_2 nanofibers as multiple rows in a parallel manner. The ZnO nanorods are not of uniform diameter, which is characteristic of solution-grown ZnO nanorods. It is noted that some branches were broken off from the trunk by sonication during the FESEM sample preparation process (Fig. 4c). The high-magnification image (Fig. 4d) revealed that high density of the secondary ZnO nanorods were dispersed on the primary TiO_2 backbones nanofibers. It is

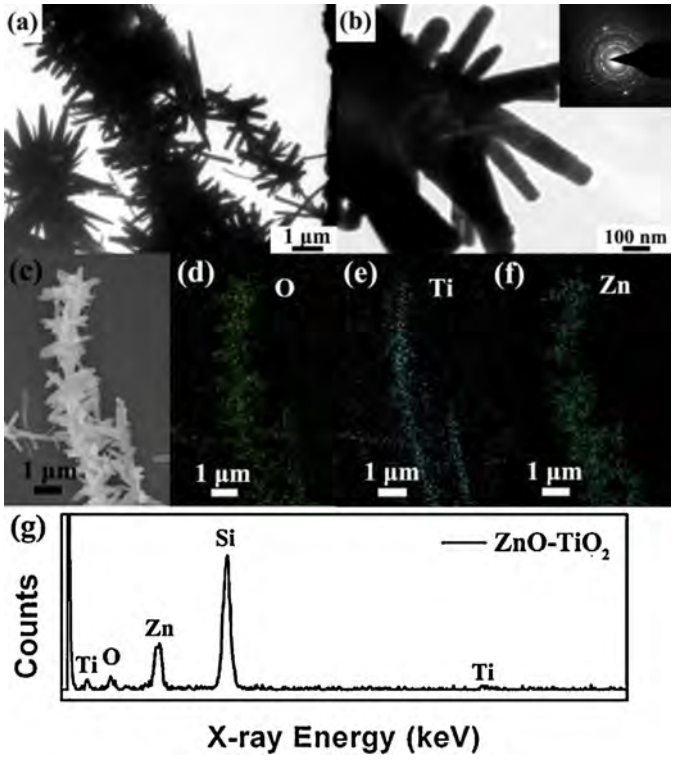

Fig. 5. (a) and (b) TEM image pattern of the typical ZnO–TiO_2 heterojunctions. The inset of (b) is a selected-area electron diffraction (SAED) pattern. (c) The SSEM image of ZnO–TiO_2 heterojunctions. (d)–(f) EDX elemental maps and (g) spots pattern of O, Ti, and Zn, respectively.

obvious that the hydrothermal treatment successfully achieved brush-like ZnO–TiO_2 heterojunctions nanofibers which integrated the wurtzite phase ZnO nanorods with the anatase TiO_2 nanofibers.

The brush-like structure of the as-prepared ZnO–TiO_2 heterojunctions was further confirmed by transmission electron microscopy (TEM). As shown in Fig. 5a, the TiO_2 nanofibers can

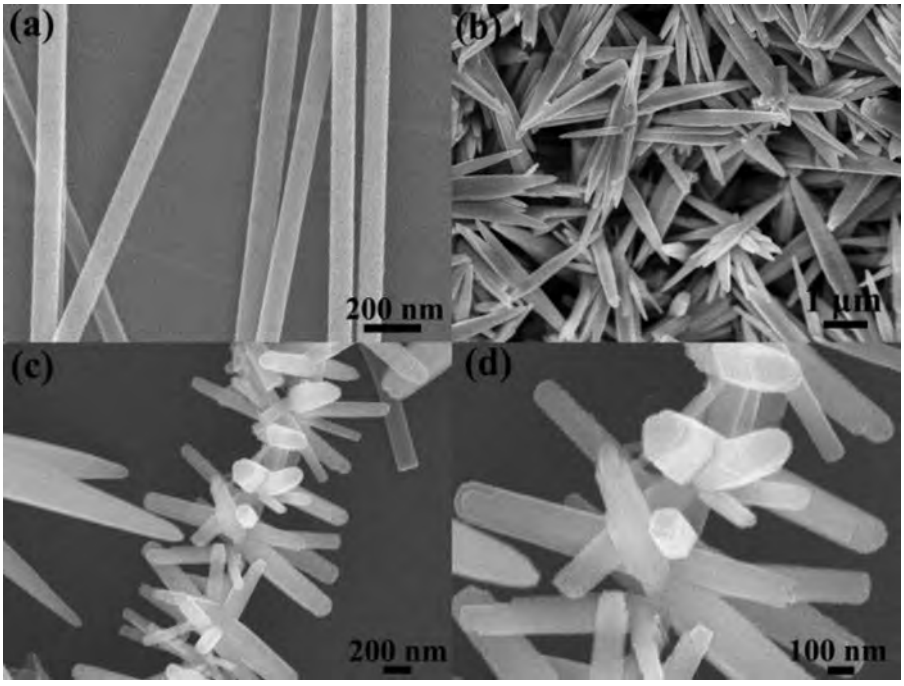

Fig. 4. The FESEM images of the as-prepared (a) pristine TiO_2 nanofibers, (b) pristine ZnO nanorods and (c) and (d) ZnO–TiO_2 heterojunctions.

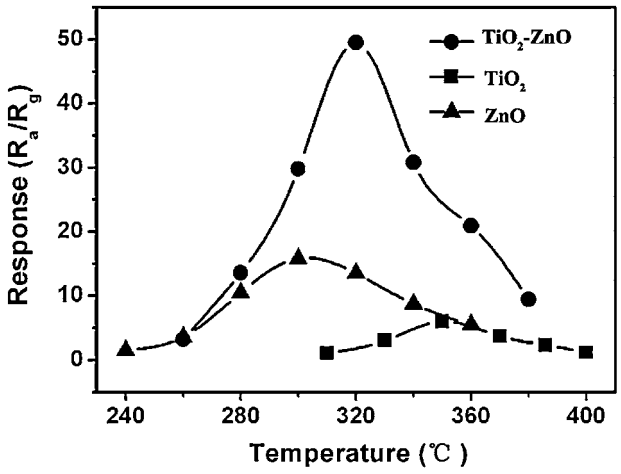

Fig. 6. Responses of pristine ZnO nanorods, TiO_2 nanofibers and ZnO–TiO_2 heterojunctions versus operating temperature to 500 ppm of ethanol, respectively.

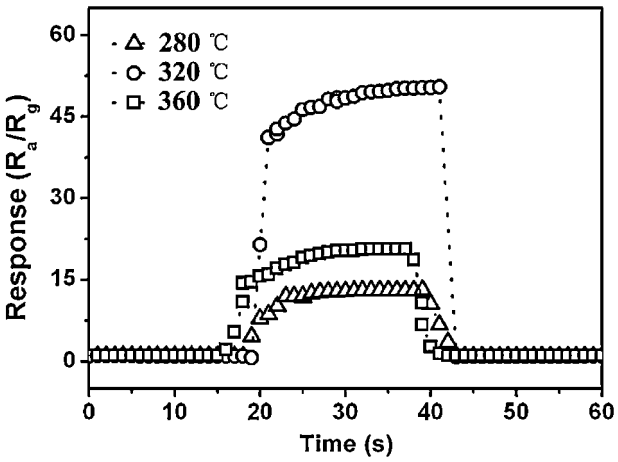

Fig. 7. Response transients of ZnO–TiO_2 heterojunctions to 500 ppm of ethanol at different operating temperature, respectively.

be densely coated by ZnO nanorods after the process. More details can be observed in Fig. 5b, which demonstrates that the final size of the ZnO nanorods is in accordance with Fig. 4b and the exterior of each brush-like structure is very loose. This network is believed to be favorable for gas sensors, which can facilitate the inward and outward gas diffusion. The corresponding SAED pattern (inset of Fig. 5b) confirms that the brush-like ZnO–TiO_2 heterojunctions nanofibers are polycrystalline structures in nature. The scanning SEM (SSEM) image of a single heterojunctions after the hydrothermal treatment, combined with EDX elemental mapping and spot scanning (Fig. 5c–f), clearly reveals the TiO_2 nanofibers can be densely coated by ZnO nanorods after the hydrothermal process. The elemental compositions of the as-prepared products were studied by EDX and the results are displayed in Fig. 5g. The data confirm that the samples are composed of Ti, Zn, and O. This indicates the high purity of the product.

3.2. Ethanol sensing properties

The gas response is usually dependent on the sensor operating temperature and addition. Fig. 6 shows the response as a function of sensor operating temperature for the three sensors to 500 ppm ethanol. It can be observed that sensors based on pristine ZnO and TiO_2 show relatively low response at the operating temperature in the range from 260 to 400 °C, with the maximum response of 15.7 and 6.0 at 300 °C and 350 °C, respectively. In contrast, ZnO–TiO_2 sensor exhibits a rapid increase of response to reach the maximum value of 50.6 at the operating temperature of 320 °C. Most importantly, the operating temperature of ZnO–TiO_2 sensor has a significant decrease, compared to the pristine TiO_2 nanofibers.

Response and recovery time are very important factors to a gas sensor, to judge whether a gas sensor is good or not also depend on that. A sensor with fast response and recovery usually made it an excellent real-time detector. The response transients of ZnO–TiO_2 nanostructures at different operating temperature to ethanol for one single period are shown in Fig. 7. It can be observed that the response and recovery time of the ZnO–TiO_2 sensors are obviously different at different given operating temperature. At the optimal operating temperature of 320 °C, the corresponding response and response/recovery time are about 50.6 and 5–10 s to 500 ppm of ethanol, respectively. However, the responses are about 13.9 and 21.5 at the temperature of 280 °C and 360 °C, and the corresponding response time is found to decrease with increasing the operating temperature. It can be explained from the kinetics and mechanics

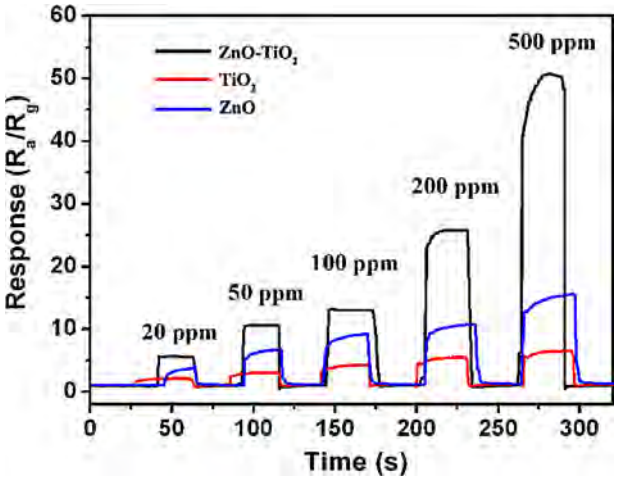

Fig. 8. Dynamic ethanol sensing transients of the pristine ZnO nanorods, TiO_2 nanofibers and ZnO–TiO_2 heterojunctions.

of the gas adsorption and desorption on the surface of ZnO or other similar semiconducting metal oxide [21].

Fig. 8 presents the dynamic response–recovery curves of pristine ZnO, TiO_2 and ZnO–TiO_2 sensors to different ethanol concentrations at a working temperature of 300 °C, 350 °C and 320 °C, respectively.

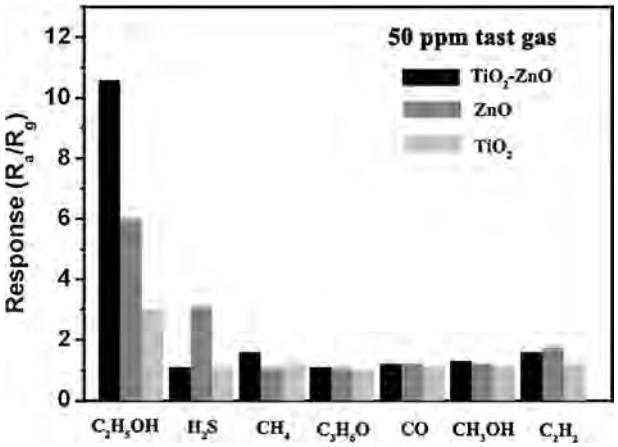

Fig. 9. Comparison in sensor response to different gas with a same concentration for ZnO–TiO_2 (black), ZnO (dark gray) and TiO_2 (light gray) of 50 ppm ethanol.

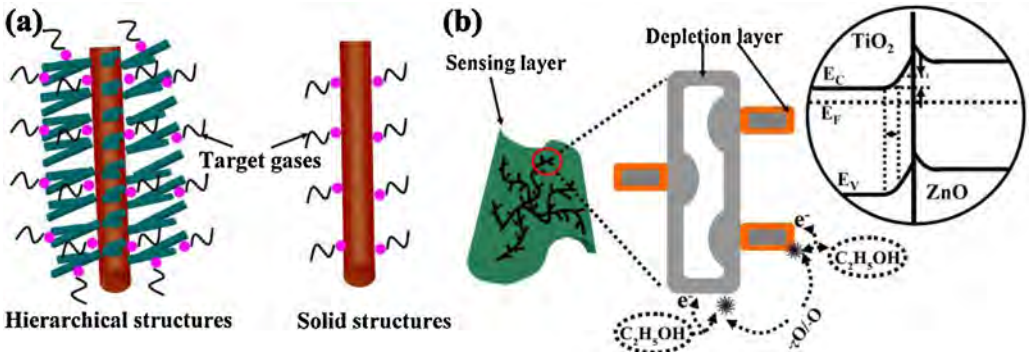

Fig. 10. (a) Key parameters to determine the gas response in hierarchical structures. (b) A schematic diagram of catalytic reactions and ideal band structure at the ZnO–TiO_2 heterojunctions.

The gas responses of ZnO–TiO_2 sensors to 20, 50, 100, 200 and 500 ppm of ethanol were 4.1, 10.6, 13.2, 26.1 and 50.6, respectively. These results indicated that the ZnO–TiO_2 sensor displayed about 2–3- and 3–8-fold enhancement in sensitivity compared to pristine ones, which in turn directly verifies the promotion effect of the unique nanostructure and synergistic effect among the two components of TiO_2 and ZnO.

The good selectivity is also a very important factor to a gas sensor when it is detecting a target gas in the presence of multi-gas molecules, especially those with similar physicochemical properties. For systematic analysis, the sensitivity values to various gases (C_2H_5OH, H_2S, CH_4, C_3H_6O, CO, CH_3OH and C_2H_2) at 50 ppm were measured and the results are shown in Fig. 9. These results imply that the hierarchical ZnO–TiO_2 heterostructure sensor exhibits obvious response for C_2H_5OH and lesser effects for H_2S, CH_4, C_3H_6O, CO, CH_3OH and C_2H_2. The ZnO decorated of TiO_2 results in high selectivity to ethanol, which is proved to be one of the direct adsorption at the TiO_2 surface site [22,23]. The enhanced gas sensing response mechanism for ethanol in semiconductor is discussed by Bahadur et al. [22].

The gas sensing property of the ZnO–TiO_2 samples for ethanol is much better than that of pristine ones. This may be attributed to two factors. One is that hierarchical structures exhibit well-aligned structures and high surface area [21]. In our work, it can be observed (in Fig. 4c and d) that the inner surfaces of the structure of the brush structures are very rough and have comb structures with well-aligned structures. And the specific surface areas of the hierarchical ZnO–TiO_2, pure TiO_2 nanofibers and ZnO rods were 16.8, 8.4 and 3.5 $m^2\,g^{-1}$, respectively. When sample units are exposed to the target gas, the gas is easy to spread inward by well-aligned structures, the effective contacts surface area will increase and the amount of target gas absorbed onto the surface of materials also will increase, compare to the single structures as shown in Fig. 10a. Thus, high response was obtained. Gas diffusion length and gas diffusion speed are important factors that affect the response and recovery time of semiconducting oxide gas sensors. Outer structure with nanorods nanostructures is generally assembled in highly periodic and porous manners. Thus, the gas diffusion toward the entire sensing surface is not hampered, it is a well-established that the fast response and recovery time. The other is the incorporation of the interface between ZnO nanorods and TiO_2 nanofibers [24]. When the ZnO–TiO_2 heterojunctions are surrounded by air, by the assistance of ZnO, oxygen molecules can be more easily adsorbed on the surface of TiO_2 (Fig. 10b) [25]. Because the work function of ZnO (5.2–5.3 eV) [26] is larger than that of TiO_2 (4.2 eV) [27], the electrons in TiO_2 nanofibers will transfer to ZnO nanorods, thus this process increases both the quantity of adsorbed oxygen and the molecule-ion conversion rate resulting in the greater and faster degree of electron depletion from TiO_2 nanofibers and ZnO nanorods [28]. Compared with TiO_2 nanofibers, the conduction channel of ZnO–TiO_2 is much narrower. Moreover, the attachment of ZnO nanorods onto TiO_2 nanofibers induces more active sites for the adsorption of oxygen molecules. Thus, these ZnO regions on the surface of TiO_2 nanofibers become high-performance gas sensing elements. When the reductive ethanol is introduced, the ethanol molecule is oxidized by the oxygen species on the surface, so the electrons are released much easily from the surface reaction back into the conduction band. Simultaneously, the resistance decreases. These regions close to the ZnO–TiO_2 interface make the sensors more active in gas detection compared with that of the surface of pristine TiO_2 nanofibers and ZnO nanorods, thus the gas sensing performance is enhanced.

4. Conclusions

In summary, we have successfully synthesized three-dimensional ZnO/TiO_2 heterojunctions with novel hierarchical architectures by combining electrospinning and hydrothermal process. The effects of ZnO loading on the ethanol sensing properties of TiO_2 products have been investigated. The results reveal that the gas responses of the ZnO–TiO_2 sensor upon exposure to 20–500 ppm of ethanol were found to be 2–3- and 3–8-fold higher than that of pristine ones. The response and recovery times are about 5–10 s to 500 ppm of ethanol at 320 °C. The gas-sensing mechanism to ethanol is briefly introduced. The results demonstrate that our sensor is a potential candidate as high-performance gas sensors.

Acknowledgements

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