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Enhanced gas sensing by assembling Pd nanoparticles onto the surface of SnO_2 nanowires

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ABSTRACT

SnO_2 nanowires with an average 0.6 μm in length and about 25 nm in diameter were prepared by a hydrothermal method. The sensors were fabricated using SnO_2 nanowires assembled with Pd nanocrystals. The sensing properties of the sensors such as selectivity, response–recovery time and stability were tested at 290 °C. After assembling Pd nanocrystals onto the surface of SnO_2 nanowires, the gas sensing properties of the sensors toward H_2S were improved. The sensors based on Pd nanoparticle@SnO_2 nanowires exhibit high stability owing to stable single crystal structure. The mechanism of promoting sensing properties with Pd nanoparticles is discussed.

1. Introduction

Tin oxide (SnO_2) with a wide band gap (Eg = 3.6 eV, at 300 K) is one of the most important and extensively used metal oxide semiconductor materials for gas sensors [1–4]. SnO_2 nanoparticles have been employed in gas sensors for decades owing to its good gas sensing properties, however, the cross selectivity and low stability of SnO_2 nanoparticles obstruct their broad application [5,6].

In recent years, many efforts have been made to improve the gas sensing properties by utilizing one-dimensional SnO_2 nanomaterials for its single crystalline nanostructure, its large surface-to-volume ratio and the congruence of the facets with their lateral dimensions [7–12]. For example, Kolmakov et al. have succeeded in synthesizing SnO_2 nanowires to detect CO and O_2 [13]. However, one-dimensional single crystal nanomaterials did not exhibit satisfactory gas sensitivity. How to make a further improvement of the gas sensitivity and selectivity with good stability is the main tendency of gas sensor development. The researchers try to enhance the gas response by doping and compounding various elements to the nanomaterials [14–18]. For instance Kumar et al. [19] successfully synthesized Cu-doped SnO_2 nanowires and nanobelts under argon atmosphere at ambient pressure, and seen its higher gas response than pure SnO_2 nanowires. However, these techniques often require high temperature and induce impurities in the final products when catalysts and templates are introduced to a reaction system. The active sites of doping nanowires are not high enough too. Therefore, taking some appropriate process to modify SnO_2 nanowires may be an effective way to enhance the gas sensing properties. Self-assemblies process is a simple and effective method for synthesizing in solution system under low temperature and ambient pressure. Recently, it was reported that ZnO nanowires with Pd nanoparticles self-assembled onto their surfaces showed excellent gas sensing properties [20]. The gas sensing properties of the SnO_2 nanowires are not as good as SnO_2 nanoparticles. It is thereby a challenge for us to enhance the gas response of SnO_2 nanowires. So self-assemblies and directed assemblies maybe a possible method for improving the response of SnO_2 nanowires.

In this report, a simple and efficient process was proposed for synthesizing and assembling SnO_2 nanowires and Pd nanoparticles with their improved gas sensing properties. Compared with pure SnO_2 nanowires, Pd nanoparticle@SnO_2 nanowires exhibit not only high gas performance due to the effect of Pd nanoparticles but also high stability attributed to the stable structure of their single crystal. The mechanism of promoting sensing properties of Pd nanoparticles is also discussed. The results show that self-assembly process is a promising method for improving the performance of the SnO_2 nanowires based gas sensors.

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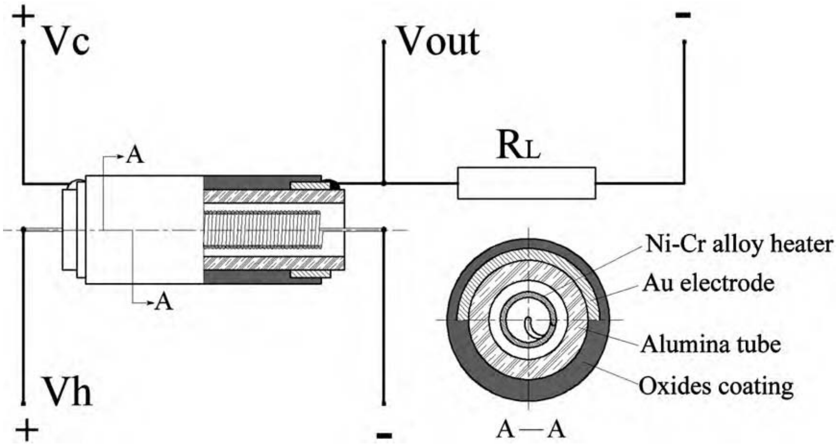

Fig. 1. Working principle of the gas sensing measurement system and the structure of the sensor (V_h, heating voltage; V_c, circuit voltage; V_{out}, signal voltage and R_L, load resistor).

2. Experimental

All of the commercial reagents were analytical grade, purchased from Shanghai Chemical Industrial Co. Ltd. (Shanghai, China) and used in the experiments without further purification.

2.1. Synthesis of SnO_2 nanowires

SnO_2 nanowires were synthesized with the method mentioned in the literature [12]. 1.50 mmol $Na_2SnO_3{\cdot}4H_2O$ was added into a solution of 18 mol/l NaOH (20 ml). The solution had been stirred about 5 min, absolute ethanol (20 ml) was added, and then a white and translucent suspension was obtained. The mixture was transferred into a stainless steel autoclave (50 ml) which was sealed, and then heated in an electric oven at 280 °C for 24 h. After cooling down naturally, white precipitation was collected by centrifugation and washed with deionized water and ethanol for at least 4 times. The products were dried at 80 °C for 24 h for further characterizations.

2.2. Synthesis of Pd nanoparticles

PVP-stabilized monodisperse Pd nanoparticles were synthesized using a modified polyol process described in the literature [21]. Briefly, Na_2PdCl_4 (0.1384 g) was reduced by ethylene glycol (EG) (5 ml) at 110 °C in the presence of poly(vinyl pyrrolidone) (PVP) (K-30, 0.0800 g) for 9 h. After being centrifuged and dried at 60 °C under vacuum for 4 h to remove EG, Pd nanoparticles protected with PVP were obtained.

2.3. Self-assembly of Pd nanoparticles on the surfaces of SnO_2 nanowires

The prepared Pd nanoparticles (0.06 mg, 0.1 mg, 0.2 mg and 0.4 mg) were dispersed in distilled water (2 ml) and mixed with SnO_2 nanowires (20.0 mg). The mixtures were ground in an agate mortar for 1 h and then dried at 100 °C for 2 h. The grey solids of SnO_2 nanowires with Pd nanoparticles on their surfaces (0.3 wt%, 0.5 wt%, 1 wt% and 2 wt%) were finally prepared by calcining at 600 °C for 2 h to remove PVP.

2.4. Characterization

The phase structure and purity of the synthesized product were examined by X-ray diffraction (XRD) with a Rigaku D/max-2550 V diffractometer employing Cu Kα radiation ($\lambda = 1.54056$ Å) at a scanning rate of 0.02°/s ranging from 10° to 80°. Their microscopic nanostructures were observed by a transmission electron microscopy (TEM, JEOL JEM-200CX working at 160 kV). The composition elements of the sample were analyzed by the energy-dispersive spectroscopy (EDS).

2.5. Gas sensor fabrication and gas sensing properties test

The SnO_2 nanowires decorated with Pd nanoparticles were slightly ground with an adhesive terpineol in a mortar to form a slurry suspension. Then the slurry suspension was coated onto the surface of ceramic tubes with a pair of Au electrodes to obtain thick films. The coated ceramic tubes were then dried at 100 °C for 2 h and subsequently calcined at 600 °C for 2 h. In general side-heating type devices were aged at 290 °C for several days to improve the stability before testing.

The gas sensing properties of the sensors fabricated were measured using a static test system of HW-30A (Hanwei Electronics Co. Ltd., Henan Province, China). For reducing gases which decrease the resistance of the sensors, the gas response (*S*) is calculated according to $S = R_a/R_g = (V_c - V_a)V_g/(V_c - V_g)V_a$, where R_a is the sensor resistance in air (its relative humidity (RH) is about 25%), R_g is that in the gas tested, V_a is the output voltage of a sensor in air, and V_g is that in the gas tested. The response time is defined as the time required for the variation in conductance to reach 90% of the equilibrium value after a test gas was injected and the recovery time as the time necessary for the sensor to return to 10% above the original conductance in air after releasing the test gas. Fig. 1 shows the working principle of the gas sensing measurement system and structure of the sensor.

3. Results and discussion

3.1. Structure and morphology

The TEM image as shown in Fig. S1(a) (see supporting material) displays a sphere-like morphology of SnO_2 nanowires with a uniform diameter of 25 nm and a length up to 0.8 μm as well as a large aspect ratio in the range of 20–35. The corresponding electron diffraction pattern (ED, Fig. S1(b) inset (see supporting material)) indicates the single crystal structure of SnO_2 nanowires. Fig. S2 (see supporting material) depicts the typical XRD patterns of SnO_2 nanowires. The sharp diffraction peaks caused by its high crystallinity can be indexed well to the tetragonal rutile structures of SnO_2, and this was in agreement with the reported data (JCPDS, 41-1445).

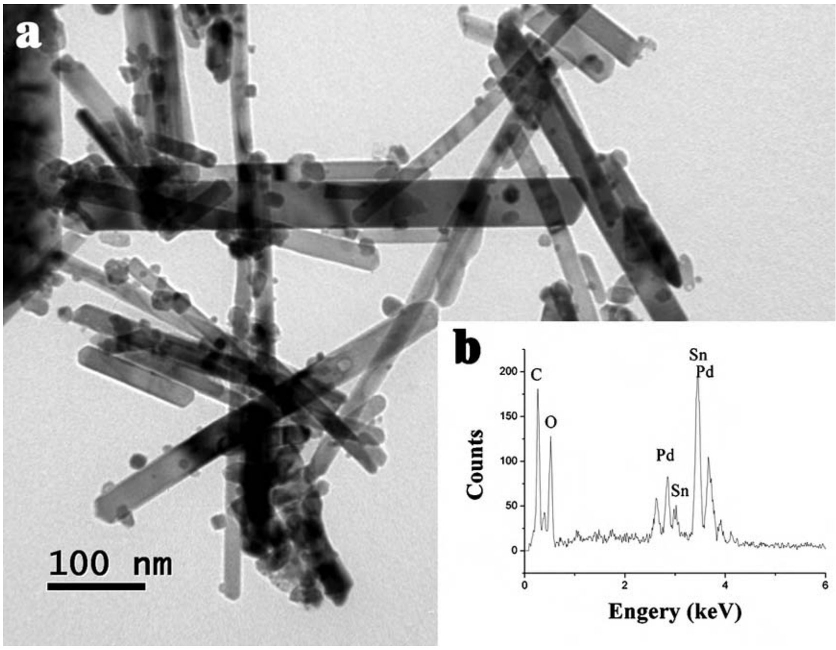

Fig. 2. (a) TEM image and (b) EDS (inset) of Pd nanoparticle@SnO_2 nanowires.

Fig. S3 (see supporting material) shows TEM images of Pd nanoparticles synthesized by reducing Na_2PdCl_4 with EG in the presence of poly PVP. The well-dispersed black dots with diameter of about 7 nm are Pd nanoparticles. Fig. S4 (see supporting material) shows the Pd nanoparticles XRD pattern, confirming that polycrystal diffraction (JCPDS No. 05-0681) of pure phase of Pd nanoparticles.

Fig. 2(a) displays TEM image of Pd nanoparticle@SnO_2 nanowires. The nano-architectures consist of nanowires and black dots, showing Pd nanoparticles well assembled onto the surfaces of nanowires. PVP has played a significant role in anchoring Pd nanoparticles onto the surfaces of SnO_2 nanowires through coordinative bonding during self-assemblies [22]. After calcination at 600 °C for 2 h, PVP was removed. Pd nanoparticles landed onto the surfaces of SnO_2 nanowires to form nano-architectures shown in Fig. 2(a). The energy-dispersive spectroscopy (EDS) analysis (Fig. 2(b), inset) confirms that the composition elements of the sample are Sn, Pd and O. The peaks corresponding to C come from carbon adhesive plaster. So we successfully obtained SnO_2 nanowires assembled with Pd nanoparticles.

3.2. *Gas sensing properties of the sensor*

For comparison, the pure SnO_2 nanowires and Pd assembled SnO_2 nanowires were used to fabricate gas sensors and the gas sensing properties of them both were tested. The gas sensing properties of the sensors for gases are influenced by the working temperature, so we should firstly investigate the optimum working temperature for the fabricated sensors. From Fig. 3 we can find that the Pd nanoparticle@SnO_2 nanowire sensors show the highest response to H_2S at 290 °C. Then, the optimum working temperature is settled at 290 °C for the subsequent measurements.

The effect of Pd nanoparticles loading on the surfaces of SnO_2 nanowires on the gas responses of the sensors was investigated at 290 °C. The gas sensing properties of the SnO_2 nanowire sensors loaded 0.0 wt%, 0.3 wt%, 0.5 wt%, 1.0 wt% and 2.0 wt% Pd nanoparticles were tested respectively. Fig. 4 shows that the response toward 50 ppm H_2S increases after decorating the Pd nanoparticle onto the surfaces of SnO_2 nanowires. The response of the sensor adding 0.5 wt% Pd nanoparticles reaches 19.8. However, adding more Pd nanoparticles further onto the surfaces of SnO_2 nanowires lead to reduce responses of the sensors. The optimum amount of Pd nanoparticles to be loaded on the surfaces of SnO_2 nanowires is 0.5 wt%.

Fig. 5(a) shows the response of pure SnO_2 nanowires and Pd nanoparticle@SnO_2 nanowire sensors in various gas environments, such as 50 ppm H_2S, H_2, formaldehyde, acetone, ethanol, methanol and ammonia at 290 °C. It is found that the responses of Pd nanoparticle@SnO_2 nanowires toward 50 ppm various gases were indeed enhanced at the working temperature. Especially, Pd nanoparticle@SnO_2 nanowire sensors exhibit H_2S gas much higher selectivity against the gases tested. For instance, the response value of Pd nanoparticle@SnO_2 nanowire sensors is 19.87 which is 7 times higher than that of pure SnO_2 nanowire sensors. The relative standard deviation (RSD) of the response ($n = 5$) was 2.66% for 50 ppm H_2S gas. In addition, the potential interference from

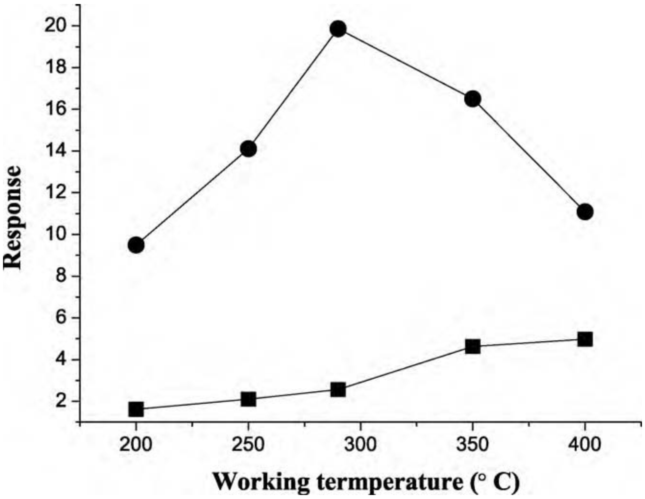

Fig. 3. Sensors response vs. temperature for Pd nanoparticles@SnO_2 nanowires and pure SnO_2 nanowires. (●) Pd nanoparticles@SnO_2 nanowires and (■) pure SnO_2 nanowires (at 50 ppm H_2S and 25% RH).

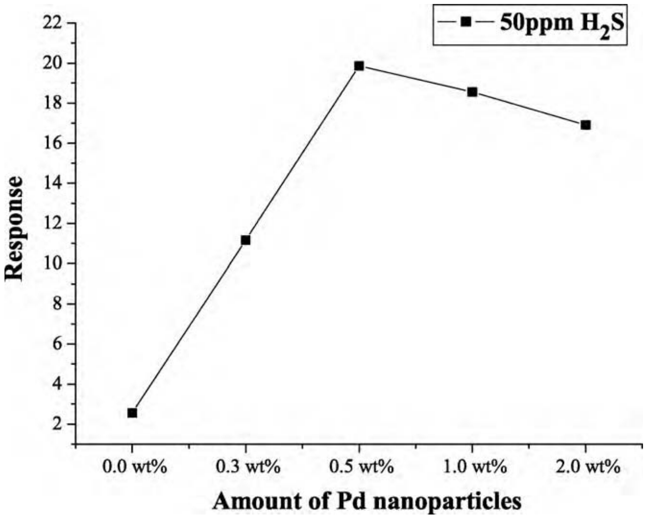

Fig. 4. Relationship between amount of Pd nanoparticles on the surfaces of SnO_2 nanowires and response to 50 ppm H_2S. (■) Pd nanoparticles@SnO_2 nanowires (at 290 °C and 25% RH).

ethanol, methanol, acetone, ammonia, carbon monoxide and so on was tested at considerably higher concentrations, which were much higher than the real world air samples. The concentrations of the most common gases in the real world [23,24] and the compound gas response value are listed in Table S1. From the result, we can find that the response of the sensors was hardly influenced by other gases in this experimental, and the Pd nanoparticle@SnO_2 nanowire sensors show good selectivity to H_2S gas.

Fig. 6(b) shows the gas response of the sensors made from Pd nanoparticle@SnO_2 nanowires and pure SnO_2 nanowires with H_2S concentration ranging from 5 ppm to 100 ppm, respectively. The gas responses of sensors increase in accompany with the increase of H_2S concentration and the gas responses of sensors fabricated with Pd nanoparticle@SnO_2 nanowires are higher than that of the sensors from pure SnO_2 nanowires. The gas response of the Pd nanoparticle@SnO_2 nanowire sensors can reach 5.55 for H_2S with concentration as low as 5 ppm, while the pure nanowires have nearly no response.

The long-term stability of Pd nanoparticle@SnO_2 nanowire sensors toward 50 ppm H_2S gas was investigated, while the sensors were continuously running in the aging system (TS-64) for 56 days. From Fig. 5(b), it is observed that the response values decrease nearly 10% for 56 days, however it tend to stabilize approximately starting from the 21st day. The average of long-term response is 18.71, and the RSD of the long-term response ($n = 8$) was 4.57% for 50 ppm H_2S gas.

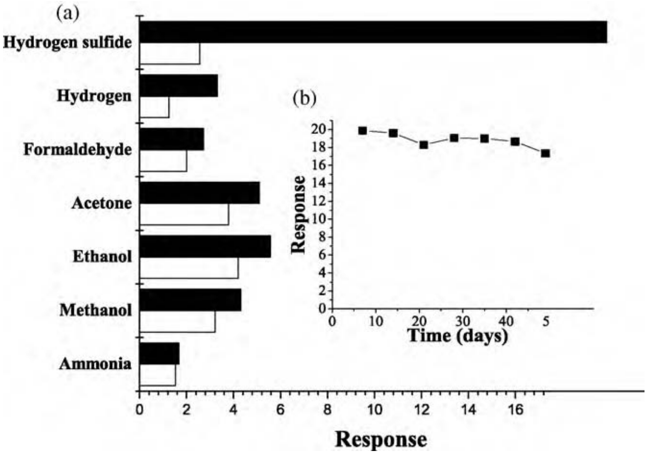

Fig. 5. (a) Selectivity of Pd nanoparticles@SnO_2 nanowires and pure SnO_2 nanowires (all concentration is 50 ppm at 290 °C) and (b) long-term stability of the Pd nanoparticle@SnO_2 nanowire sensors (at 50 ppm H_2S, 290 °C and 25% RH). (Solid) Pd nanoparticles@SnO_2 nanowires; (open) pure SnO_2 nanowires; and (■) Pd nanoparticles@SnO_2 nanowires.

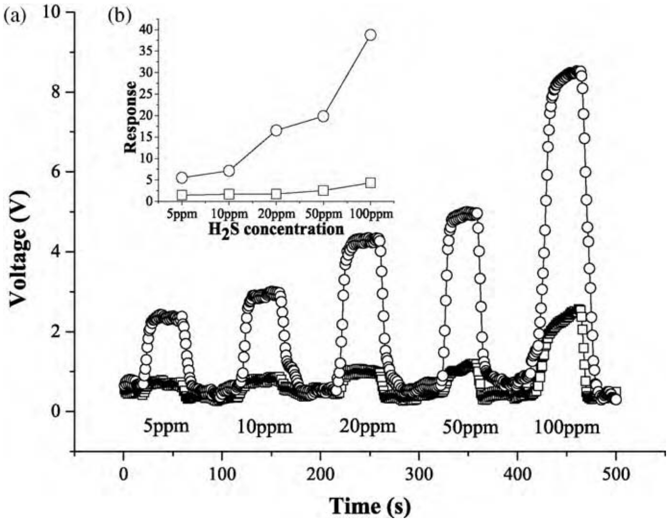

Fig. 6. (a) Typical response curve of pure SnO_2 nanowires and Pd nanoparticles@SnO_2 nanowire sensors exposed to H_2S (at H_2S concentration ranging from 5 ppm to 100 ppm, 290 °C and 25% RH) and (b) response vs. H_2S concentration (at 50 ppm H_2S, 290 °C and 25% RH). (○) Pd nanoparticles@SnO_2 nanowires and (□) pure SnO_2 nanowires.

Fig. 6(a) shows the response variation of Pd nanoparticle@SnO_2 nanowires and pure SnO_2 nanowire sensors with H_2S concentration ranging from 5 ppm to 100 ppm. The response of sensors increased with the variation of H_2S concentration and the response and recovery times of the Pd nanoparticle@SnO_2 nanowire sensors are 7 s and 10 s for 50 ppm H_2S gas, respectively. Compared to pure SnO_2 nanowires, there is no apparent enhancement of the response and recovery times.

In addition, the short-term stability of Pd nanoparticle@SnO_2 nanowire sensors was also investigated for 12 times under the same experimental conditions in one day. Fig. 7 displays that the sensors shows well short-term stability of response and recovery for 50 ppm H_2S gas which meant the good repeatability of the measurements. Thus the Pd nanoparticle@SnO_2 nanowire sensors

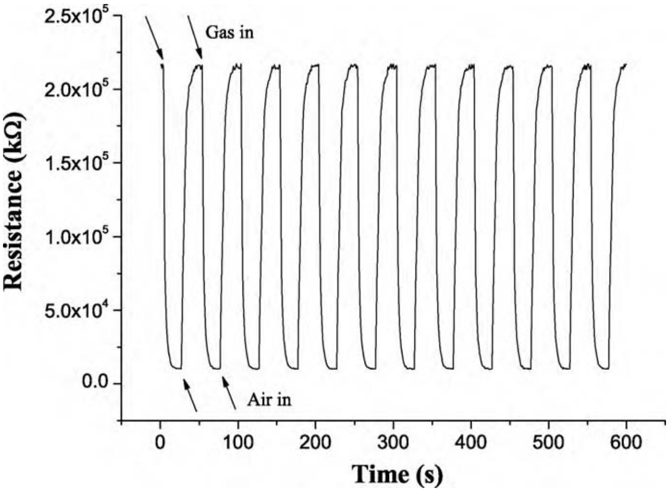

Fig. 7. Short-term stability and repeatability of Pd nanoparticle@SnO_2 nanowire sensors (at 50 ppm H_2S, 290 °C and 25% RH).

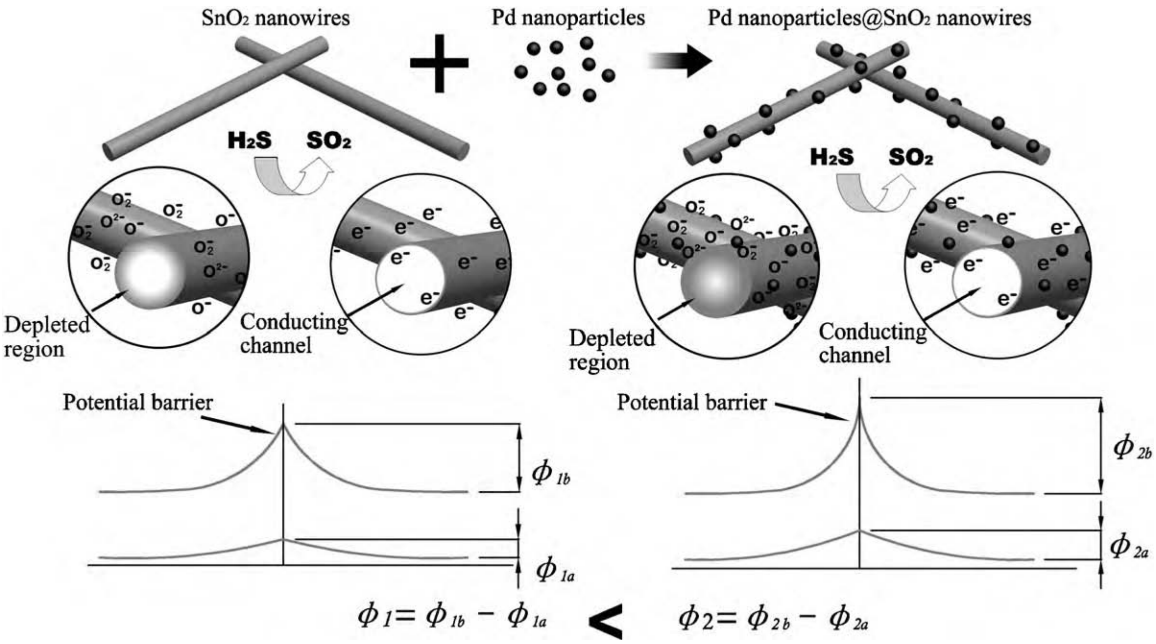

Fig. 8. Schematic drawing of H_2S-sensing mechanism on pure SnO_2 nanowires and Pd nanoparticles@SnO_2 nanowires.

exhibit satisfactorily long-term stability and short-term repeatability in their response and recovery for detecting H_2S gas.

From the reported literature [25–33], we find that the H_2S sensors' response of modified SnO_2 nanomaterials depend on its test condition, its nanostructure and sensing mechanism. For example, Fang et al. [29] had synthesized CeO_2–SnO_2 thin films for enhancing the response about 4 times to 20 ppm H_2S at room temperature, though the response value was about 4.5. Most CuO-doped SnO_2 sensors showed a high response and slow recovery regardless of the sensors' morphology [30–32], because the detection mechanism of H_2S could be understood in the framework of the specific chemical interaction between H_2S and CuO and its consequent effect on the p–n junction structure. For example, Hwang et al. [30] reported that CuO-doped SnO_2 nanowires showed the response value of 1280 and slow recovery time about 360 s toward 80 ppm H_2S at 300 °C. Jin et al. [33] reported that the response increased when the film was doped with Cu or Ag. They found that the response value of SnO_2 porous film doped with 16 nm Ag film was extremely high as 5900 toward 1 ppm H_2S at 200 °C, however its recovery time was more than 2000 s. Apparently, our experimental results not only show a considerable gas response (response value is 19.87) but also a short recovery (recovery time is 10 s) toward 50 ppm H_2S at 290 °C.

Apparently, our experimental results show a considerable increase of gas response by assembling Pd nanoparticles on the SnO_2 nanowires. The promoting mechanism of Pd nanoparticles in gas response can be explained by depletion layer modulation model [34,35]. When the sensors are exposed to air atmosphere, oxygen will be physically and chemically adsorbed on the surface of SnO_2 nanowires [36]. The adsorbed oxygen act as electron acceptors and then form O_2^-, O and O^{2-}, generating depleted layers, which results in increase of the width and height of the potential barrier at the contacts among the nanowires and consequently leads to resistance increase of the nanowire sensors (Eq. (1)).

$$(1/2)O_2(\text{gas}) \rightarrow (1/2)O_2(\text{phys}) \rightarrow (1/2)O_2^-(\text{chem}) \rightarrow O^-(\text{chem}) \rightarrow O^{2-}(\text{chem}) \qquad (1)$$

$$2H_2S(\text{ads}) + 3O_2^- \rightarrow 2SO_2(\text{gas}) + 2H_2O + 3e^- \qquad (2)$$

$$H_2S(\text{ads}) + 3O^- \rightarrow SO_2(\text{gas}) + H_2O + 3e^- \qquad (3)$$

When Pd nanoparticles are loaded, more oxygen can be absorbed and easy dissociated on the surface of SnO_2 nanowires [37]. This process increases the population of oxygen, resulting in increase the length of depleted layer together with the width and height of the potential barrier. After the sensors are exposed upon a reducing gas such as H_2S, the adsorbed O^- on the surface of the sensors reacted with the reduced gas (Eqs. (2) and (3)). The trapped electrons were released back to the conduction band. The depleted layer was fading away in accompany with the reaction until system reached equilibrium. At the contacts of SnO_2 nanowires, the width and height of the barrier potential decreased, thus lead the decrease of sensor resistance (as shown in Fig. 8).

In comparison to the pure SnO_2 nanowires, there are more trapped electrons released back to the conduction band, more shift of depleted layer length and barrier potential width and height at the contacts. Therefore, the sensors from Pd nanoparticle@SnO_2 nanowires show the better H_2S-sensing properties than those from pure SnO_2 nanowires. The responses of H_2S sensors decreased after assembling more Pd nanoparticles onto the surfaces of SnO_2 nanowires, because there were not enough exposed surfaces of SnO_2 nanowires to receive dissociated oxygen adsorbs. And less exposed surfaces of SnO_2 nanowires also affected the functionalities of the sensors [38]. Thus it is important to control density of Pd nanoparticles onto the surfaces of SnO_2 nanowires at an appropriate proportion. With this self-assembly method, we conveniently design and fabricate other better chemical sensors from semiconductor materials and monodispersive nanoparticles or quantum dots [39].

4. Conclusions

Sensors with effectively enhanced H_2S-sensing properties were achieved by assembling appropriate monodisperse Pd nanoparticles onto the surfaces of SnO_2 nanowires. The sensors can detect H_2S down to 5 ppm (response value is 5.55) in a temperature of 290 °C. The response and recovery times are about 7 s and 10 s, respectively. The considerable increase of gas response is proposed to the benefit of depletion layer modulation after assembling Pd nanoparticles. The assembly process could be applied extensively to various materials including the design of highly sensitive gas sensors.

Acknowledgments

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.talanta.2010.04.053.

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