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Enhancement of hydrogen monitoring properties based on Pd–SnO_2 composite nanofibers

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ABSTRACT

A nano-gas sensor based on Pd–SnO_2 composite nanofibers is fabricated by electrospinning technique and calcination procedure. The morphology, structure and composition of the as-prepared nanofibers are characterized by scanning electron microscopy (SEM), transmission electron microscopy (TEM), X-ray diffraction (XRD), X-ray photoelectron spectroscopy (XPS), respectively. The nano-gas sensor shows excellent hydrogen sensing properties such as high sensitivity and extremely fast response–recovery behavior (~9 s) at a lower operation temperature (280 °C). The detection limit of approximately 4.5 ppm H_2 is demonstrated. The function of adding Pd into the SnO_2 nanofibers and the sensing mechanism have also been discussed in this work.

1. Introduction

Hydrogen, as a renewable, abundant, efficient energy source, has been extensively investigated in the application of new energy sources [1–3]. However, hydrogen is colorless, explosive, and extremely flammable in the air. Consequently, a variety of robust and safety sensors are being developed for the reliable and continuous monitoring of H_2, particularly for combustion gas detection and fuel leak detection in spacecraft, autos and aircraft, fire detectors, and industrial process emissions [4–7]. Recently, the synthesis of hydrogen sensors based on wide bandgap semiconductors such as ZnO, SnO_2, TiO_2 and GaN has gained special focus owing to their sensitivity to surface charge and wide temperature stability [8–12]. Among these materials, SnO_2 is the most popular and commercial one for the development of resistive gas sensors. Till now, various SnO_2 structures such as sintered powders [13], thin and thick films [14,15], nanoparticles [16], nanowires [17], and nanosheets [18] are fabricated and applied in gas sensors. However, most of those gas sensors suffer from relatively low sensitivity or long response/recovery time [13–18].

Palladium (Pd) is a widely used catalyst for improving the selectivity and sensitivity of SnO_2 sensors to H_2 [19–24]. On the other hand, taking the advantages of large surface-to-volume ratio and unique electric properties based on one-dimensional nanostructures, many efficient gas sensors have been developed based on one-dimensional nanostructures [25–29]. Among the different methods for the fabrication of one-dimensional nanostructures, electrospinning has received considerable attention because of its simplicity and versatility [30,31]. Hitherto, several gas sensors have been successfully obtained for the detection of methanol [32], ethanol [33], humidity [34] and toluene [35] via electrospinning. But few H_2 sensors based on electrospun nanofibers have been reported previously [36].

In this paper, Pd-doped SnO_2 nanofiber has been fabricated via electrospinning technique and calcination process. The as-prepared nanofibers exhibit high sensitivity and ultra-fast response–recovery property. These results demonstrate a promising approach in development and realization of a low cost and high-performance hydrogen sensor.

2. Experiment details

2.1. Chemicals

Ethanol (>95%), N,N-dimethyl formamide (>95%) and $SnCl_2 \cdot 2H_2O$ were purchased from Tianjin Chemical Co. (China). $PdCl_2$ was purchased from Beijing Chemical Co. (China). Poly(vinyl pyrrolidone) (PVP, Mw = 1,300,000) was purchased from Aldrich. All chemicals were used as received without any further purification.

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2.2. Preparation of Pd–SnO_2 nanofibers

In a typical procedure, 0.4 g of $SnCl_2{\cdot}2H_2O$ was dissolved in 4.4 g of DMF and 4.4 g of ethanol under vigorous stirring for 10 min. Subsequently, 0.8 g of PVP and 12 mg of $PdCl_2$ were added into the above solution under vigorous stirring for 30 min. The mixture was electrospun (voltage: 12 kV; distance between anode and cathode: 20 cm) and followed by sintering at 600 °C for 5 h. The concentration of Pd in the final product was 3 wt%. The pure SnO_2 nanofibers were also obtained with no addition of $PdCl_2$ by the same procedure.

2.3. Fabrication and measurement of H_2 gas sensor based on our products

The as-prepared nanofibers were mixed with deionized water in a weight ratio of 100:25 to form a paste. The paste was coated onto a ceramic tube on which a pair of gold electrodes was previously printed, and then a Ni–Cr heating wire was inserted in the tube to form a side-heated gas sensor. Gas sensing properties were measured using a static test system. Saturated target vapor was injected into a glass test chamber (20 L in volume) by a syringe through a rubber plug. After fully mixed with air (relative humidity was about 25%), the sensor was put into the test chamber. When the response reached a constant value, the sensor was taken out to recover in air. The electrical properties of the sensor were measured by the CGS-1 intelligent test system (Beijing Elite Tech Co. Ltd., China). The sensor response was measured between 200 and 340 °C. The sensor response is defined as the ratio *Ra*/*Rg*, where *Ra* is the resistance in dry air, and *Rg* is the resistance in test gas. The time taken by the sensor to achieve 90% of the total resistance change was defined as the response time in the case of adsorption or the recovery time in the case of desorption.

2.4. Characterization

Nanofibers were characterized by means of scanning electron microscope (SEM, SSX-550, Shimadzu) transmission electron micrographs (TEM, Hitachi S-570), X-ray diffraction (XRD, Scintag XDS 2000 diffractometer with a Cu Kα radiation). Analysis of the X-ray photoelectron spectra (XPS) was performed on an ESCLAB MKII using Al as the exciting source.

3. Results and discussion

3.1. Morphological and structural characteristics of our products

Fig. 1a and b shows the typical SEM images of pure SnO_2 nanofibers and Pd–SnO_2 nanofibers, respectively, indicating diameters of the as-prepared nanofibers for all the samples are ranging from 80 to 120 nm. Further morphology characterization of our products was examined by TEM and shown in Fig. 1c and d, which indicated that the fibers have a rough surface due to the loss of PVP from the nanofibers. Those TEM images also show that both of the pure SnO_2 nanofibers and Pd–SnO_2 nanofibers are indeed composed of many interconnected grains of around 30 nm in size.

Fig. 2a shows XRD patterns of the pure SnO_2 nanofibers and Pd–SnO_2 nanofibers. All the strong diffraction peaks of both samples can be perfectly indexed as the tetragonal rutile structure for SnO_2 (JCPDS 41-1445). But there is no indication of the presence of any dopants-related diffraction peaks for the Pd-doped sample, attributing to the high dispersion or the poor crystallinity of dopants-related nanoparticles. To further prove the existence of Pd component in Pd–SnO_2 nanofibers, XPS has been used to investigate the composition and the chemical state of the elements existed in the as-prepared Pd–SnO_2 nanofibers, and the spectra are illustrated in Fig. 2b. The Pd 3d spectrum exhibited a doublet feature at 342.7 eV (Pd $3d_{3/2}$) and 336.8 eV (Pd $3d_{5/2}$). These peaks correspond to the Pd^{2+} oxidation state [37] and Pd therefore exists as PdO in the Pd–SnO_2 nanofibers. All of these results give the insight that the nanofibers are composed of PdO and SnO_2.

3.2. Sensing properties

The optimal operating temperature of the sensors for detecting H_2 is an important issue, and it was obtained by performing the gas sensing experiments at different temperatures. Before exposing to the target gas, each sensor was stabilized for 2 h at the working temperature. Fig. 3a presents responses to 100 ppm of H_2 for the pure SnO_2 nanofibers and Pd–SnO_2 nanofibers samples. For the pure SnO_2 nanofibers sensor, the response increases with a raise of operating temperature and reaches its maximum (about 4) at 320 °C, then decreases rapidly with increasing temperature. On the contrary, the Pd–SnO_2 nanofibers show higher response value (about 8.2) with respect to pure SnO_2 at lower temperature (280 °C). It is known that PdO can be reduced by H_2 and form PdHx at a relatively low temperature [38]. In our case, when hydrogen is passed over the PdO/SnO_2 nanofibers, Pd was formed from PdO via PdHx at low temperature. Then the Pd metal induces the surface and lattice oxygen reductions of Pd–SnO_2 nanofibers at much lower temperatures, in contrast to the pure tin oxide.

The concentration dependence of pure SnO_2 nanofibers and Pd–SnO_2 nanofibers was investigated in the range of 20–15,000 ppm H_2 and the plots of the gas response against the gas concentration are shown in Fig. 3b. As for the Pd–SnO_2 nanofibers, the results clearly indicate that even the lowest concentration of H_2, i.e. 20 ppm, the response is more than 3. The response value increases rapidly with increasing H_2 concentration at first (below 5000 ppm). Above 5000 ppm, the response slowly increases with increasing H_2 concentration. Although the similar trend was also observed for pure SnO_2 nanofibers, the responses were much weaker, which indicate a much lower sensitivity compared with the Pd–SnO_2 nanofibers. In addition, a low detection limit of the Pd–SnO_2 nanofibers is found to be around 4.5 ppm according to Fig. 3b, while the detection limit of pure SnO_2 nanofibers is about 25 ppm which is more than 4 times of that of the Pd–SnO_2 nanofibers. The detection limit is defined as the concentration at which response is ~1.1, corresponding to 10% change of resistance.

There are several reasons which should be considered to explain the improvement of the nanofibers' sensing performance upon PdO doping. To describe the sensitization mechanism more clearly, we present a simple model in Fig. 4. As shown in the schema, the whole process can be divided into three steps. Step 1 is the dissociation of molecular oxygen on the sensor surface (Fig. 4a). In this step, the PdO nanoparticles play a role as catalyst for activating the dissociation of molecular oxygen, the atomic products then diffuse to the metal oxide support. This step greatly increases both the quantity of oxygen that can repopulate vacancies on the SnO_2 surface and the rate at which this repopulation occurs, resulting in a greater and faster degree of electron withdrawal from the SnO_2 than for the pure SnO_2 nanofibers (Pd is a far better oxygen dissociation catalyst than SnO_2) [39,40]. This step is well-established in the catalysis literature and known as the "spill-over" effect. When hydrogen is introduced, PdO is reduced to metallic palladium, returning electrons to SnO_2, which is defined as Step 2 (Fig. 4b). In Step 3 (Fig. 4c), hydrogen molecules adsorbed on palladium simultaneously spillover the surface of SnO_2, activating the reaction between hydrogen and the adsorbed oxygen [38]. In this step, the dissociation of H_2 at the Pd surface forms atomic H that leads to additional reaction schemes [41]. Reviewing the basic reaction scheme between

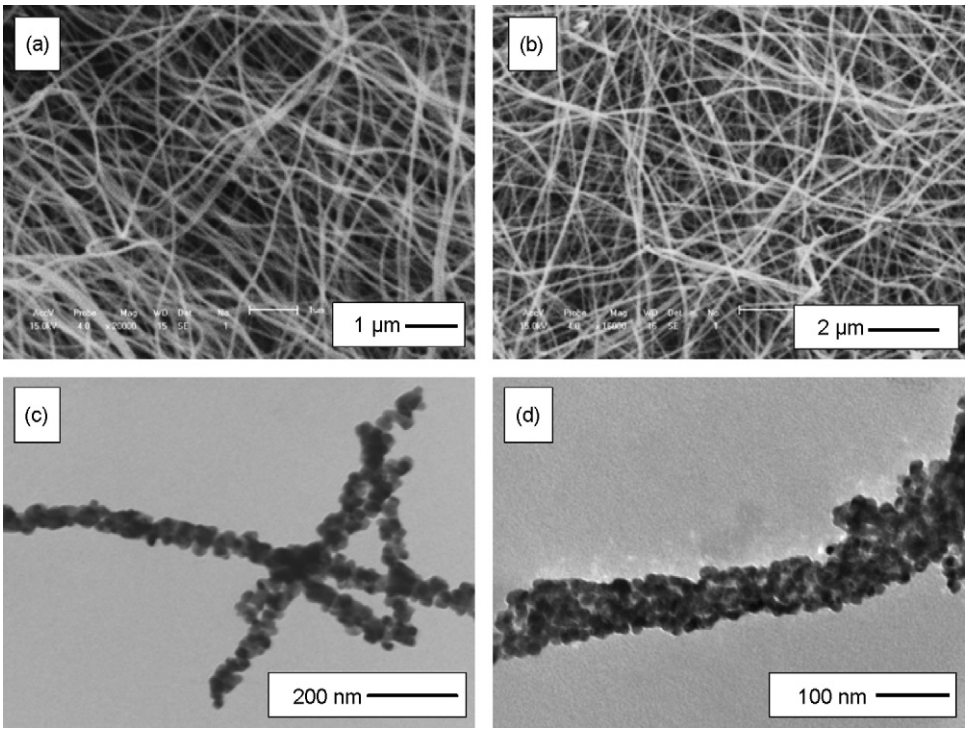

Fig. 1. SEM images of (a) pure SnO_2 nanofibers and (b) Pd–SnO_2 nanofibers. TEM images of (c) pure SnO_2 nanofibers and (d) Pd–SnO_2 nanofibers.

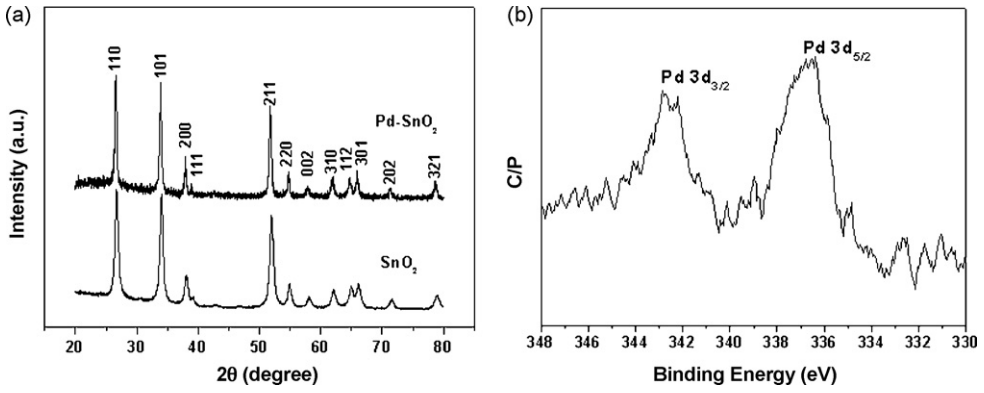

Fig. 2. (a) XRD patterns of the products and (b) XPS spectra of Pd 3p for Pd–SnO_2 nanofibers.

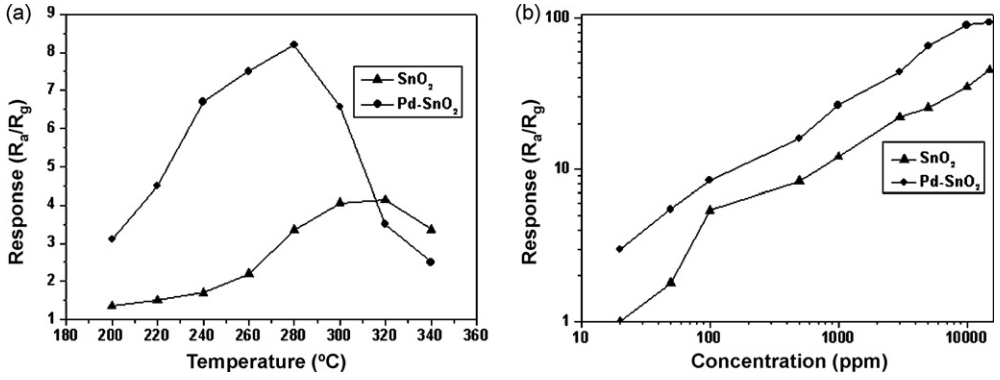

Fig. 3. (a) Responses of pure SnO_2 and Pd–SnO_2 nanofibers to 100 ppm H_2 as a function of operating temperatures. (b) Dependence of response on H_2 concentration for pure SnO_2 and Pd–SnO_2 nanofibers.

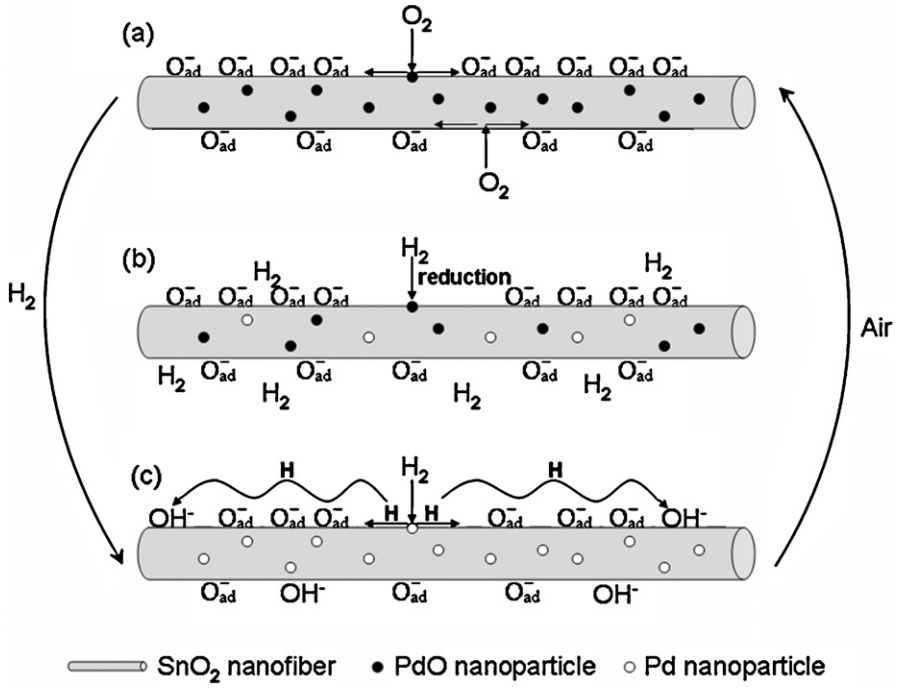

Fig. 4. Schematic model of the three steps for the sensitization mechanism of the as-prepared nanofiber sensor: (a) molecular oxygen dissociation on PdO nanoparticles followed by spill-over of the atomic species onto the SnO_2 surface; (b) reduction of PdO nanoparticles by H_2; (c) spill-over of H_2 on the SnO_2 surface and reactive with the oxygen species.

adsorbed oxygen species on the SnO_2 surface and molecular H_2 is a single step process presented below

$$H_2(g) + O_{ad}^{-}(SnO_2) \rightarrow H_2O(g) + (SnO_2) + e^{-} \quad (1)$$

With the dissociation of H_2 at the Pd surface, additional reaction schemes are supplied

$$H_2(g) \rightarrow H + H(\text{dissociation}) \quad (2)$$

$$H + SnO_2 \rightarrow H_{ad}(SnO_2) \quad (3)$$

$$H_{ad}(SnO_2) + O_{ad}^{-}(SnO_2) \rightarrow OH_{ad}^{-}(SnO_2) \quad (4)$$

$$OH_{ad}^{-}(SnO_2) + H_{ad}(SnO_2) \rightarrow H_2O(g) + (SnO_2) + e^{-} \quad (5)$$

In these reaction paths, $H_2O(g)$ is liberated as the final reaction product, resulting in accumulation of electrons at the surface which is responsible for the conductance increase, hence the better sensitivity to H_2 of the Pd–SnO_2 nanofiber sensor than pure SnO_2 nanofiber.

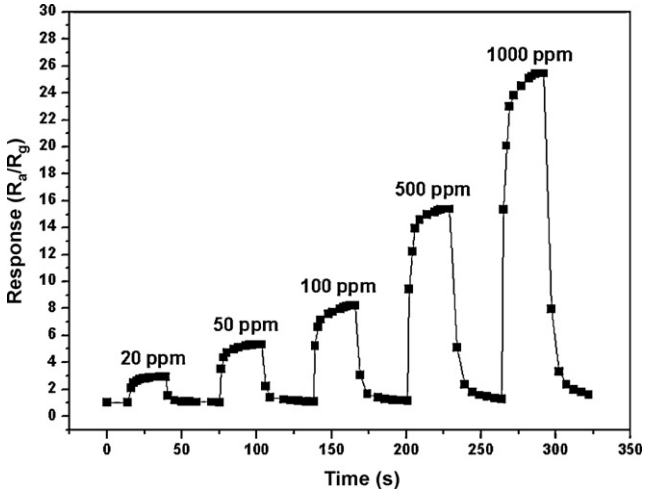

Fig. 5. Response and recovery characteristic curves of the sensor based on Pd–SnO_2 nanofibers to H_2 in the range of 20–1000 ppm at 280 °C.

It is well known that response and recovery characteristics are important for evaluating the performances of hydrogen sensors. To investigate the response–recovery behaviors of the Pd–SnO_2 nanofibers sensor, the sensor was sequentially exposed to 20, 50, 100, 500 and 1000 ppm H_2 at 280 °C. The sensor exhibits high response and fast response and recovery to H_2, as shown in Fig. 5. The response and recovery times are very short and change slightly with changing H_2 concentration. When exposed to H_2 with different concentrations, the response and recovery times are about 9 s. The extremely fast response and recovery behavior can be attributed to the one-dimensional nanostructure of Pd–SnO_2 nanofibers. It can facilitate fast mass transfer of the hydrogen molecules to and from the interaction region as well as improve the rate for change carriers to transverse the barriers along the fibers [34].

4. Conclusions

Pd–SnO_2 composite nanofibers have been fabricated via electrospinning and calcination. The as-prepared samples were evaluated for their gas sensing properties towards H_2. A maximum response value of 8.2 and extremely fast response–recovery behavior are obtained for 100 ppm H_2 detection at 280 °C, which exhibited better responses and lower operating temperature than that of the pure SnO_2 nanofibers. These results demonstrate a promising approach in development and realization of a low cost and high-performance hydrogen sensor.

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