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Excellent gas sensing of hierarchical urchin-shaped Zn doped cadmium sulfide

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ABSTRACT

Urchin-like hierarchical Zn doped CdS powders were successfully synthesized via simple one-pot hydrothermal process. Their SEM and TEM images indicated that the hierarchical structure were assembled by single crystal nanorods with the hexagonal wurtzite phase. EDS element mapping verified that Zn ions were homogeneously distributed among the hierarchical microstructure. The performances of gas sensors based on pure and Zn doped CdS were measured and compared. The results indicated that Zn doping could enhance their responses to some volatile organic compounds and improve its selectivity to ethanol and toluene as well. The possible reasons for this enhancement were investigated. In addition, the sensor based on Zn doped CdS exhibited the ultrafast response and recovery to ethanol ($\tau_{response} < 1$, $\tau_{recovery} = 8s$), indicating that the Zn doped CdS could be a promising gas sensing candidate for online monitoring of ethanol.

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1. Introduction

Gas sensors are playing more and more vital roles on the air-quality monitoring and medical diagnosis. Chemiresistive gas sensors, based on semiconductor oxides, are widely investigated in the past few decades because of their superior gas sensing properties, low cost and ease for operation [1–7]. Gas sensing relies on the surface chemical reaction between target gas and chemical absorbed oxygen on the surface of sensing material. The microstructure of sensing materials embody their comprehensive properties related with surface activity, gas transportation etc. Therefore, their sensing properties can be further adjusted and optimized by controlling their microstructure [8,9]. Compared with low dimension nanomaterials such as one-dimensional(1D) and two-dimensional(2D) building blocks, three-dimensional(3D) hierarchical sensing materials may present more fascinating sensing properties owing to large surface area, favorable gas diffusion and well-defined morphology. Consequently, simple and efficient ways to synthesize the 3D hierarchical nanomaterials are highly expected to explore gas sensor with excellent performance. For the resistance-type gas sensor, semiconductor oxides, such as ZnO [10–12], α-Fe_2O_3 [13], In_2O_3 [14,15], WO_3 [16–19], SnO_2 [20,21], are dominantly be studied. However, research reports on the metal chalcogenides in the field of gas sensor have rarely been reported in the past years [22–24], in spite of their extraordinary optoelectronic properties in visible-light detector, sensors, waveguide photodetectors, lasers, field-effect transistors, solar cell and photocatalysis etc. Ruan et al. [25] reported that Zn1-xCdxS nanowire had response of 12 toward 50 ppm ethanol. Ma et al. [26] verified that CdS microparticle could response to 30 ppm NO with sensitivity of 65.8 at the operating temperature of 180 °C. Thus it can be seen that CdS is a potential good sensing material. Its sensing performance is worthy of further investigation. In order to improve the sensing performance, several effective methods such as doping, noble metal loading and construction of heterojunction have been adopted. Doping is a simple and effective method. Zn^{2+} ion was selected to be doped in the CdS because of the similar crystal structure of ZnS and CdS. In our work, unique urchin-like hierarchical CdS assembled by a large number of nanorods firstly prepared by one-step hydrothermal route. The gas sensors based on the as-synthesized CdS were fabricated and their gas sensing performances were investigated and compared. The results showed

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that Zn doped CdS exhibited high response sensitivity and fast response-recovery property to ethanol at 225 °C.

2. Experimental

2.1. Synthesis of urchin-like hierarchical CdS

All the reagents in the experiment were purchased from Beijing Chemical Reagent and used as received without further purification. 0.1M cadmium nitrate and thiourea solutions were prepared for the cadmium and sulfide precursors, respectively. In a typical experiment, 0.05 g ZnO was added in 40 ml mixture of deionized water and ethylenediamine (1:2 v/v) with vigorous stirring till it was solved completely. Then appropriate amounts of cadmium nitrate and thiourea solution (1:1 v/v) were mixed with the above solution. Being stirred for 20 min, the mixed solution was transferred into a 45 ml Teflon-lined stainless autoclave and maintained at 180 °C for 12 h. On cooling down, the precipitates were collected by centrifugation, repeatedly washed with distilled water and absolute ethanol for several times, and dried at 80 °C in air. Different amounts of cadmium nitrate (0.25 ml, 0.50 ml, 0.75 ml) were added in the hydrothermal procedure, and the corresponding products were labeled as S1, S2 and S3, respectively. They were classified as Zn doped CdS. If the above hydrothermal process was repeated with the fixed 0.25 ml cadmium nitrate and no addition of ZnO, the obtained sample was labeled as pure CdS.

2.2. Characterization

The phase structures of the as-synthesized products were analyzed by a Rigaku TTRIII X-ray diffractometer with Cu $K\alpha_1$ radiation ($\lambda = 1.5406A$). The surface morphology and microstructure of the prepared samples were observed by field emission scanning electron microscope (SEM, JEOL JSM-7500F) at an acceleration voltage of 15 kV and transmission electron microscopy (TEM, JEOL JEM-3010) at 200 kV. The energy dispersive X-ray spectroscopic (EDS) elemental mappings were investigated by the TEM attachment.

2.3. Fabrication and measurement of gas sensor

The gas sensor was fabricated as follows: the as-synthesized powder was first dispersed in deionized water in volume ratio of 5:1 to form homogeneous slurry. Subsequently, it was coated onto an alumina tube (4 mm in length, 1.2 mm in external diameter, and 0.8 mm in internal diameter, attached with a pair of gold electrodes) with a brush to form a thick sensing film. Being baked for 10 min under an infrared lamp, it was sintered at 300 °C for 2 h to improve its thermal stability. Then a Ni-Cr alloy coil was inserted into the alumina tube as a heater in order to control the operating temperature of the sensor. A RQ-2 series Intelligent Test Meter (made in China) was applied to investigate the sensing performance of the sensor. The measurement was carried out by a static process, that is to say, the sensor was alternately placed into the test chambers full of air and a kind of target gas with certain concentration and the corresponding resistance (R_a or R_g) was measured respectively. The resistance ratio of R_g/R_a in an oxidative target gas was defined as response value. On the contrary, in a reductive gas, the response was expressed as R_a/R_g.

3. Results and discussion

3.1. Structural and morphological characteristics

Fig. 1a shows the X-ray diffraction (XRD) patterns of pure and Zn doped CdS (S1) products. As seen in Fig. 1a, all the diffraction peaks can be indexed to those of hexagonal wurtzite CdS (JCPDS card No. 89-2944) (a = 4.14 Å, c = 6.715 Å). No extra diffraction peaks are observed. To further differentiate the corresponding peak positions for pure and Zn doped CdS, their enlarged diffraction patterns in the range of 20–35° (shown in Fig. 1b) are evaluated. Compared with pure CdS, the peak positions for Zn doped CdS shift slightly towards large diffraction angle, confirming that Zn^{2+} ions with smaller ion radii than Cd^{2+} are doped into the CdS lattice (R_{Zn}:74pm, R_{Cd}:95pm). The morphologies and microstructures of the as-prepared products were illustrated by FESEM photographs (shown in Fig. 2). It is observed that both Zn-doped and pure CdS exhibit urchin-like hierarchical structure, with the former having more regular and homogenous structure. The urchin-like hierarchical structures are assembled by a lot of nanorods with the diameter between 30 and 50 nm and length between 0.5 and 0.8 μm. Moreover, HRTEM and TEM elemental mapping were employed to further analyze the lattice structure and the elemental distribution of the as-prepared Zn doped CdS hierarchical structure. Fig. 3a, b, f display the TEM imagines of Zn doped CdS microstructure, showing that urchin-like hierarchical structure consists of numerous nanorods. The clear lattice fringes in a single nanorod

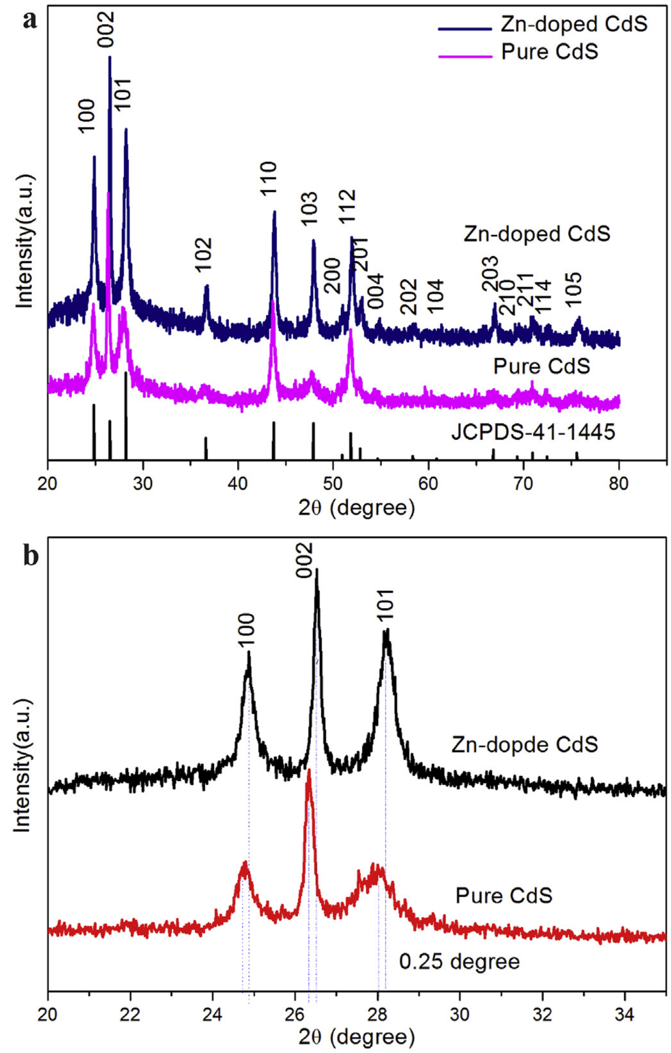

Fig. 1. a XRD patterns of pure and Zn doped CdS (S1)samples. b Enlarged XRD patterns between the range of 20 and 35 degree.

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