Gas Sensing with Hollow α-Fe_2O_3 Urchin-Like Spheres Prepared via Template-Free Hydrothermal Synthesis

Peng Sun, Zhe Zhu, Peilu Zhao, Xishuang Liang*, Yanfeng Sun, Fengmin Liu and Geyu Lu*

State Key Laboratory on Integrated Optoelectronics, College of Electronic Science and Engineering, Jilin University, Changchun 130012, People's Republic of China. Fax: +86 431 85167808; Tel: +86 431 85167808;
E-mail: lugy@jlu.edu.cn, liangxs@jlu.edu.cn

Experimental

Materials preparation

All the reagents in the experiment were analytical grade (Beijing Chemicals Co. Ltd.) and used as received without further purification. Hollow α-Fe_2O_3 urchin-like spheres were synthesized by annealing FeOOH precursor obtained via a hydrothermal reaction of ferric sulphate ($Fe_2(SO_4)_3$) in aqueous solution at suitable temperature. In a typical synthesis, $Fe_2(SO_4)_3$ was dissolved in deionized water to form a clear solution of a certain concentration, which was transferred into a Teflon-lined stainless steel autoclave, maintained at 140 °C for 12 h, and allowed to cool to room temperature naturally. The resulting precipitate was collected by centrifugation, washed several times with deionized water and ethanol, and finally dried in vacuum at 80 °C. Then the precursor was annealed at 600 °C for 2 h, hierarchical α-Fe_2O_3 hollow spheres were obtained. The concentration of $Fe_2(SO_4)_3$ was varied in the range from 0.005 to 0.1 M.

Materials characterization

The X-ray diffraction (XRD) pattern of the as-prepared product was performed on a Rigaku TTRⅢ diffractometer with Cu Kα radiation (λ=1.5418 Å) in the range of 20-80°. Filed-emission scanning electron microscopy (FESEM) observations were carried out with a JEOL JSM-7500F microscope, operated at an acceleration voltage of 15 kV. Transmission electron microscopy (TEM) observations were obtained on a JEOL JEM- 2100 microscope operated at 200 kV. The specific surface area was estimated using the Brunauer-Emmett-Teller (BET) equation based on the nitrogen adsorption isotherm obtained with a Micromeritics Gemini Ⅶapparatus (Surface Area and Porosity System).

Sensing properties measurements

A gas sensor was fabricated as follows: the as-prepared products were mixed with water, and then coated on 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) by a small brush to form a thick film. After drying under air at room temperature for 1h, the devices were sintered at 400 ℃ for 2 h. A Ni-Cr alloy coil was placed through the tube to control the operating temperature of the sensor. The measurement was processed by a static process in a test chamber (50 L in volume) under laboratory conditions (50 RH %, 22 ℃). A given amount of the tested gas was injected into the test chamber, and the sensor was put into the chamber for the measurement of the sensitive performance. The gas response S is defined as the ratio R_a/R_g, where R_a and R_g are the resistances measured in air and the tested gas atmosphere. The response and recovery time is defined as the time taken by the sensor to achieve 90% of the total resistance change in the case of adsorption and desorption, respectively.

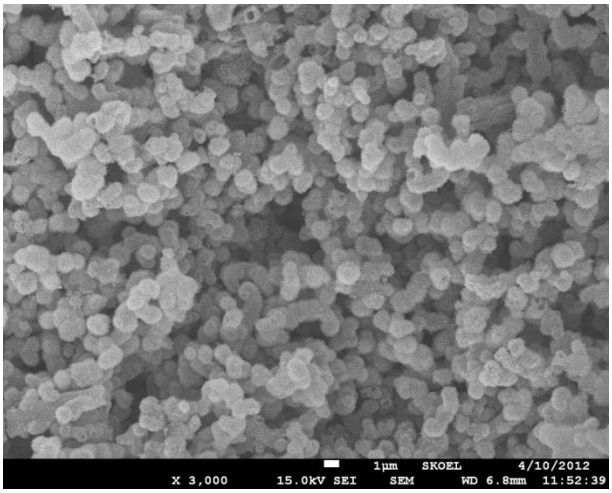

Fig. S1† A typical low-magnification FESEM image of the as-prepared α-Fe_2O_3 hollow spheres

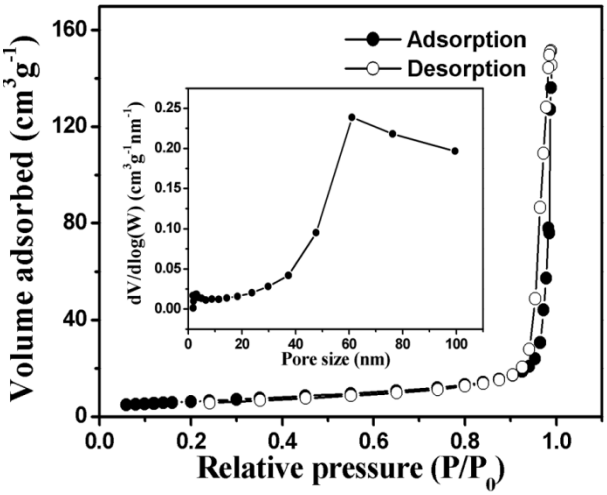

Fig. S2† N_2 adsorption-desorption isotherm of α-Fe_2O_3 hollow spheres

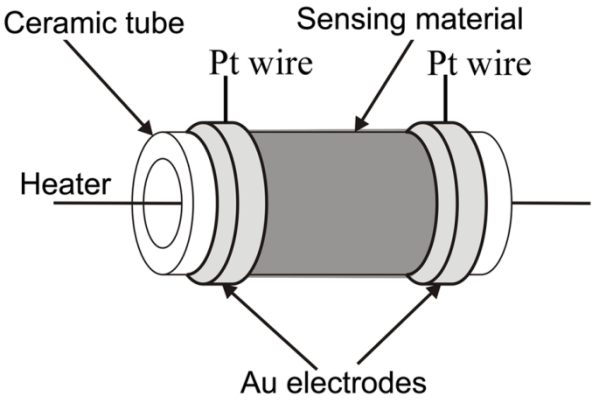

Fig. S3† The schematic structure of the gas sensor