Preparation and H_2S gas-sensitive properties of hierarchical flower-like Ag/ZnO composites

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2.1 Preparation of hierarchical flower-like Ag/ZnO composites

0.16 g of zinc acetylacetonate was ultrasonically dispersed in 30 mL anhydrous ethanol, and then 1 mL of ammonia solution (2.5 $mol \cdot L^{-1}$) was added dropwise to the mixture, followed by magnetic stirring for 1 h. The resulting solution was then transferred to a 50 mL stainless steel reaction vessel lined with polytetrafluoroethylene and heated at 180 °C for 8 h. After natural cooling to room temperature, the white precipitate was obtained after washing three times with deionized water and ethanol by centrifugation, followed by drying at 70 °C for 8 h and calcination at 400 °C in air for 1 h, resulting in flower-shaped ZnO material.

2.2. Characterization

The synthesized samples were characterized for phase and crystal structure using X-ray powder diffraction (XRD, Bruker, D8-advance) at an operating voltage of 15 kV. The morphology of the samples was observed using field emission scanning electron microscopy (FESEM, Hitachi, S-4300). The fine structure of the synthesized materials was observed using transmission electron microscopy (TEM, Hitachi, H-7650) at an

operating voltage of 200 kV. Nitrogen adsorption-desorption analysis was carried out using a fully automated gas adsorption analyzer (BET, Quanta chrome, Autosorb-iQ) at 77 K to calculate the specific surface area and pore size distribution. Surface qualitative and elemental valence analysis of the samples was performed using X-ray photoelectron spectroscopy (XPS, Thermos, Escalab 250Xi), with the binding energy of the C 1s peak at 284.8 eV used for energy calibration.

2.3. Fabrication and measurement of gas sensors

An Al_2O_3 tube (4 mm long, 1.2 mm outer diameter, 0.8 mm inner diameter) with a pair of Au electrodes and four Pt wires at both ends was coated with a hierarchical floral Ag/ZnO composite, and then a Ni-Cr alloy wire was inserted into each hollow cylinder to control the operating temperature, and the two ends of the heating wire and four platinum wires were welded to the six-column base to form a gas sensor.

The gas sensor test system (JF02F) was used to test the gas sensing performance of the prepared hierarchical structure flower-like Ag/ZnO composite sensor, and the test process is as follows: firstly, the gas sensor reaches a stable initial resistance Ra in the air, and the sensor is inserted into a container (10 L) prepared with a certain concentration of target gas, and when the resistance is stable, the resistance value is recorded as Rg. The sensor's response to the gas is expressed by S, which is the ratio of the resistance Ra in the air to the resistance Rg in the target gas, i.e., S = Ra/Rg. Response-recovery time is defined as the time it takes for a sensor to reach a 90% change in resistance (Ra-Rg) during response and recovery. That is, the time required for the resistance of the gas sensor to change from Ra to Ra-90% after the gas sensor is exposed to the measured gas is called the response time and is denoted as t_{res}. The time it takes for the gas sensor to change from Rg to Rg+90% after leaving the measured gas is called the recovery time and is denoted as t_{rec}. The selectivity coefficient is K=S target gas/S other gases. In the humidity resistance test, the response of the hierarchical structure floral Ag/ZnO composite sensor to relative humidity was tested with LiCl, CH_3COOK, K_2CO_3, $CuCl_2$, NaCl and KNO_3 saturated salt solutions. At room temperature (25±1°C), the air humidity above the saturated solution was 11.3 RH%, 22.5 RH%, 43.2 RH%, 67.0% RH%, 75.3 RH%, and 93.6 RH%, respectively. Before

testing, store the saturated salt solution in a 5 L glass container for at least 24 h.

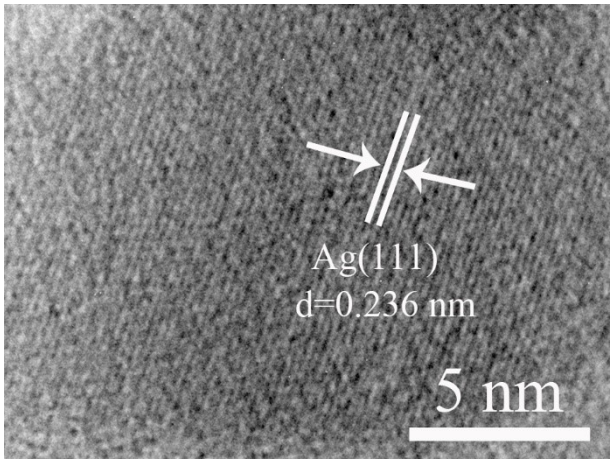

Fig S1 TEM image of Ag/ZnO material

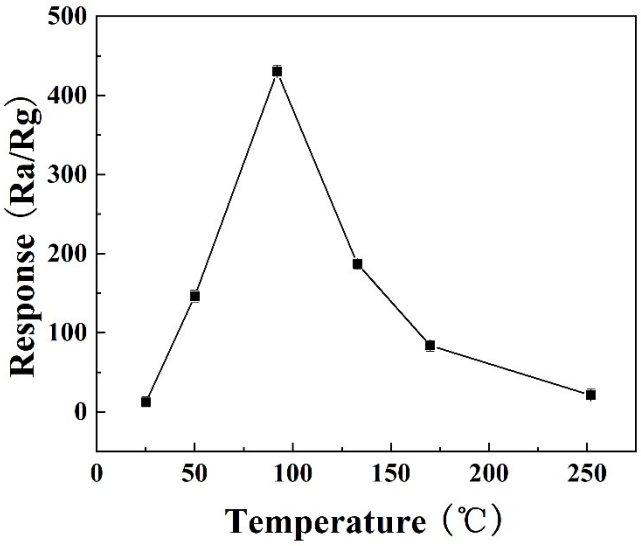

Fig S2. Responses of sensors to 100 ppm of H_2S gas at different working temperatures

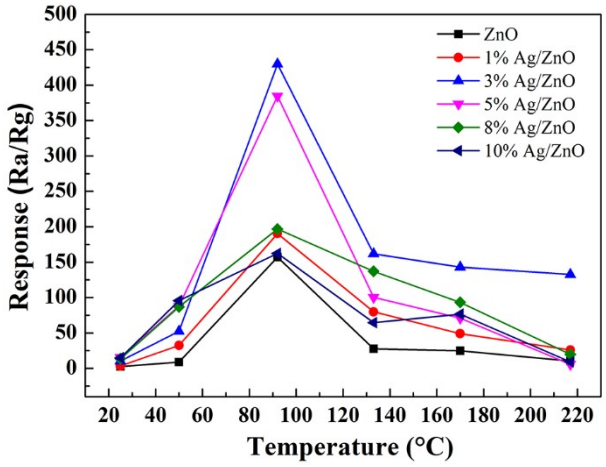

Fig S3. Responses of Ag/ZnO and ZnO sensors loaded with different Ag molar ratios

(1 at%、3 at%、5 at%、8 at% and 10 at%) to 100 ppm H_2S at different working temperatures