Highly sensitive H₂S detection sensor at low temperature based on hierarchically structured NiO porous nanowall

arrays

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Fabrication of the thick film sensor

For the fabrication of the thick film NiO sensors, the as-obtained NiO nanosheets were mixed with a small amount of terpilenol to form NiO paste, which was then brush-coated onto the surface of an Al_2O_3 microtube with a pair of Au electrodes attached with Pt wires. After being dried at 80 °C, it was sintered at 300 °C for 2 h in air to form a thick sensing film.

The schematic drawing of gas sensing set-up was provided, as shown in Fig. S1. 1 ppm standard H₂S gas was purchased from Dalian special gas Co. Ltd. Before measurement, a 10 L glass chamber was evacuated by pump in advance, and then the corresponding volume of H₂S gas (1 ppm standard gas) was injected into the container by a syringe to obtain different concentration of H₂S, according to calculation formula (V_c= C×10 L/1 ppm, where C is the target concentration of H₂S for sensing measurement). Then clean air with 25 RH% was used as the dilute gas and passed into the container to balance the pressure of the inner and outer of container. The gas response can be measured when the sensor was put in/taken out of the gas container. In order to prevent the influence of the residual H₂S gas on the gas response of the sensor, the gas sensing measurement was followed the sequence from low concentration to high concentration. The sensing measurement of the sensors for other gases was similar with that of H₂S gas.



Fig. S1 The schematic drawing of gas sensing set-up.



Fig. S2 XRD patterns of the as-prepared precursor powder (a) and the calcined powder (b) after heat treatment at 500 °C for 2 h.



Fig. S3 SEM images of the arrays film sensor (a) and the thick film sensor (b).



Fig. S4 The relationship between the responses of two sensors and different working temperatures (a) and sensing transients of NiO arrays film sensor (b) and thick film sensor (c) to H_2S .



Fig. S5 The reproducibility and long-term stability of the NiO nanowall arrays film sensor to 10 $ppm H_2S$ gas measured at 92 °C.



Fig. S6 The response of hierarchically structured NiO nanowall arrays film sensor at different relative humidity atmospheres.



Fig. S7 XPS spectra of full survey (a) and the fine spectrum of Ni 2p (b) of NiO powder

Fig. S8 shows the Gas chromatogram and Mass spectrum of the gaseous product after the H_2S sensing measurement of the sensor. Fig. S8a shows the gas chromatogram of SO₂, the retention time (Rt) is 1.414 min, which was confirmed by

mass spectroscopy from the molecular ion m/z=64 (Fig. S8b). The fragment m/z=34 corresponds to the gas of H₂S. By this means, it is convinced the generation of sulfur dioxide when NiO nanowall arrays was exposed to H₂S.



Fig. S8 Gas chromatogram (a) and Mass spectrum (b) after NiO nanowall arrays sensor exposure to H_2S .