Electronic Supplementary Material (ESI) for Journal of Materials Chemistry A. This journal is © The Royal Society of Chemistry 2014

Supporting information

A high efficiency H₂S gas sensor material: Paper like Fe₂O₃/Graphene nanosheets and structural alignment dependency of device efficiency

Zaixing Jiang^{a,b,†}, Jun Li^{a,†}, Hüsnü Aslan^{b,†}, Qiang Li^b, Yue Li^a, Menglin Chen^b, Yudong Huang^{a,*}, Jens Peter Froning^c, Michal Otyepka^c, Radek Zbořil^c, Flemming Besenbacher^b, Mingdong Dong^{b,*}

^aDepartment of Polymer Science and Technology, School of Chemical Engineering and Technology, Harbin Institute of Technology, 150001 Harbin, People's Republic of China ^bInterdisciplinary Nanoscience Center (iNANO), Aarhus University, DK-8000, Aarhus C, Denmark ^cRegional Centre of Advanced Technologies and Materials, Department of Physical Chemistry, Faculty of Science, Palacký University, 77146 Olomouc, Czech Republic

[†]These authors contributed equally to this work.

1. Experimental details

Graphite oxide was synthesized from expanded graphite (KP9935-300, Qingdao, P.R. China) by a modified Hummers method. All the materials used in this work were of analytical grade, which were commercially obtained and used as received. In a typical experiment to prepare the Fe₂O₃/graphene nanocomposite, about 2.0 mmol of Fe(NO₃)₃·9H₂O was added to 25.0 mL of GO ethanol solution. The reaction mixture was treated under ultrasonic (500 W) for 30 min to obtain homogenous solution. Then, the mixture was moved to a 80.0 mL stainless steel vessel. And the vessel was charged with CO₂ up to 5.0 MPa at 0°C. After that, the vessel was sealed and moved to an oven at 120°C and maintained at this temperature for 2h. Subsequently, the autoclave was put into a salt-bath furnace of 350°C and maintained for 1h. After it was cooled to ambient temperature, the vessel was slowly depressurized, and the black powder product was collected carefully. The as-prepared black powder of 200.0 mg was resolved in water with the aid of

ulrasonic, which was used to prepare VAFe/GN. The thicknesses of paper prepared ranged form 3µm to 10µm.

XPS analysis was carried out on a FEI Sirion 200 spectrometer (Royal Dutch Philips Electronics Ltd., Netherlands), using monochromatic Al $K\alpha$ radiation at 12.5 kev and 300 W. A pass energy of 300 eV was used for the survey spectra. Take-off-angles relative to sample surface of 45° was employed. The SEM (Quanta 200FEG, FEI, US) was employed to characterize the vertical aligned graphene nanosheets. The morphology and microstructure of the Fe₂O₃/graphene nanosheet was examined by TEM on a H-600 transmission electron microscope (Hitachi, Japan).

The CL measurement for the obtained VAFe/GN in response to H_2S was similar to that reported in our previous work. The air from the pump was flowed through the quartz tube. At 190°C, H_2S was injected into the tube and oxidized on the surface of the sample by the oxygen in the flowing air. The subsequent CL intensity was directly measured with an ultraweak CL analyzer (Biophysics Institute of the Chinese Academy of Science, P.R. China). The detection limit has been determined through adjusting the concentration of H_2S . The carrier-gas flow rate was about 200.0 mL/min.

2. Supporting Figures



Figure S1. TEM image of aggregated Fe₂O₃ nanoparticles synthesized in the absence of graphene.



Figure S2. Effect of $Fe(NO_3)_3 \cdot 9H_2O$ on the distribution of Fe_2O_3 nanoparticles on graphene surface. The amount of $Fe(NO_3)_3 \cdot 9H_2O$ used are (a) 0.5 mmol, (b) 1.0 mmol, (c) 2.0 mmol, (d) 4.0 mmol. As shown, the amount of Fe_2O_3 on graphene increased with the amount of $Fe(NO_3)_3 \cdot 9H_2O$ as expected. In the cases where $Fe(NO_3)_3 \cdot 9H_2O$ amounts used as 0.5 mmol and 1.0 mmol, the graphene surface was not fully covered by Fe_2O_3 nanoparticles (see Figure S2 a and b). The Fe_2O_3 nanoparticles were excessive attached on graphene surface when the amount of $Fe(NO_3)_3 \cdot 9H_2O$ was 4.0mmol. The graphene surface was uniformly and densely covered by Fe_2O_3 nanoparticles when $Fe(NO_3)_3 \cdot 9H_2O$ used was 2.0 mmol. The scale bar in Fig S2 is 200nm.



Figure S3. VAFe/GN films with different thickness. (a) $\sim 2\mu m$ thick, (b) $\sim 4\mu m$ thick.



Figure S4 Response curves of H_2S on VAFe/GN paper at 130°C. The detection limit of the as-prepared VAFe/GN is about 9ppm H_2S at 130°C. The air flow rate is 200 ml/min.



Figure S5. Cross section SEM image of HAFe/GN.



Figure S6. Top SEM image of VAFe/GN film.