Electronic Supplementary Material (ESI) for RSC Advances

Palladium nanoparticles on the inner wall of Tin oxide hollow nanosphere with enhanced hydrogen sensing property

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Experimental Details

Synthesis of SiO_2 nanosphere: SiO_2 nanosphere was first produced by a modified stöber method[1]. Briefly, 6.0 mL NH_3•H_2O (28.0 wt% aqueous solution) was added to 180 mL ethanol to form solution A. 2.0 mL tetraethyl orthosilicate (TEOS) was added to 10.0 mL ethanol to form solution B. Then 2.0 mL solution B was added to Solution A and after stirring for 10 min the residual solution B was added to A. The mixed solution was stirred for another 3 h and then the white SiO_2 nanosphere with the diameter of about 300 nm was collected by centrifugation and washing.

Preparation of Pd/SiO_2 nanostructure: Pd nanoparticles were loaded on the SiO_2 nanosphere with the assistance of $SnCl_2$ according to the reference[2]. In a typical process, 100 mg SiO_2 nanosphere was dispersed in 50mL distilled water and stirred for 10 min as part A. 0.1 g $SnCl_2$ was dissolved in 20 mL 0.02 M HCl solution as part B. Parts A and B were mixed together under stirring for 10 min. Then the suspension was centrifuged. After washing with distilled water five times, the precipitate was dispersed in 50 mL distilled water. Then a certain amount of $PdCl_2$ solution (0.0564 M) was added into it. Ten min later, 10 mL of 0.15 M sodium formate solution was added following stirring for 5 h. After centrifugation and washing with distilled water five times, the precipitate was dried at 60 ºC for 12 h.

Preparation of Pd/SnO_2 nanostructure: The Pd/SiO_2 composite was encapsulated by SnO_2 according to Lou's work[3, 4]. Typically, 1.8g (30 mmol) urea and 0.266g (1 mmol) K_2SnO_3•$3H_2O$ was dissolved in 34 mL (de-ionized) DI water first, then 18mL ethanol was added in the solution to form thin milky suspension under mind stir. 240 mg Pd/SiO_2 spheres was dispersed in another 4 mL DI water by ultra sonication. The two suspensions were mixed and transferred to a 100 mL teflon autoclave, which was

then heated in an electric oven at 170 °C for 36 h. After the autoclave cool down, the products were centrifuged and washed with DI water. The SiO_2 core can be etched in 2M NaOH in a 45 °C oven in 3–6 hours, and the hollow spheres were centrifuged and dried in an oven at 110 °C. In the control experiment, SnO_2 hollow sphere was obtained in the same procedure except that SiO_2 was instead of Pd/SiO_2.

Sensor fabrication and measurement: The gas sensing properties was measured in a home made sensor testing system according to the reported procedure of our group[5]. Before the sample deposition, all samples including the $Pd@SiO_2$ composite and SnO2 hollow sphere were annealed at 400 °C for 2 h. Then a certain amount of sample (commonly 5 μL suspension with a concentration of 100 mg/ml in water) was deposited on a commercial sensor electrode by spin coating. A constant flux of synthetic air of 200 mL/min was used as gas carrier. Hydrogen was used as the testing gas and was introduced into the testing chamber by mass flow controller with a concentration of 10 ppm, 20 ppm, 30 ppm, 40 ppm and 50 ppm, respectively. All the measurements were executed in a temperature-stabilized sealed chamber at 300 °C under controlled humidity. The electronic conductivity is measured by sourcemeter (KEITHLEY, Model 2400). The conductivity of the sensor film was measured by two probe method. In the sensor device, a 21 v voltage was applied and the current flow was measure. The resistance of the Pd/SnO_2 sensor films was obtained according to the formula below:

$R=U/I$

Supporting Figures

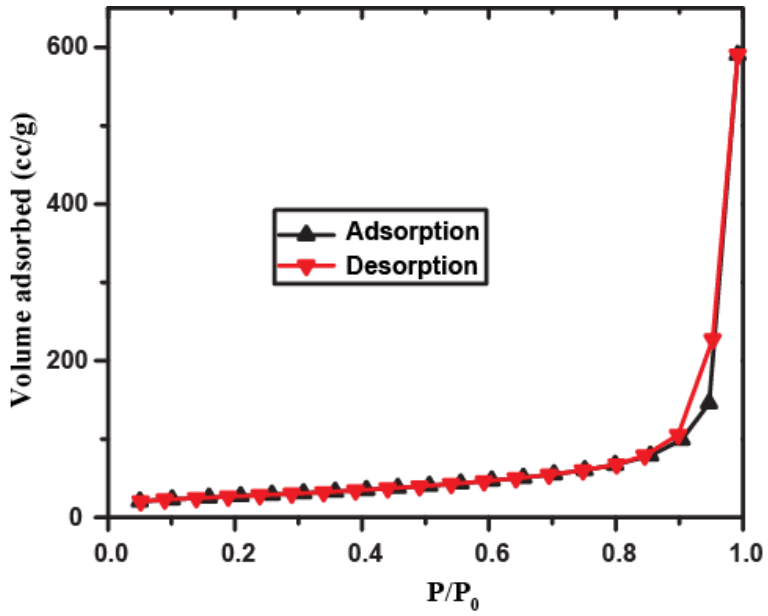

Figure S1 N_2 adsorption-desorption isomer of the Pd@SnO_2 composite. The calculated BET surface area is 96.4 m^2/g.

References:

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