Supplementary information for

Facile Synthesis of Urchin-like Glass/Nickel Core/Shell Composite Hollow Spheres

S1 Experimental details

All agents were of analytical grade and used without further purification. A typical experiment was as following. 10g of the hollow glass spheres (HGS) were washed with diluted HCl and deionized water for 2 times respectively, then redispersed in 3-aminopropyltriethoxy silane (KH550, a silane coupling agent) ethanol solution (30g/L), shaken at 50℃ for coupling, separated again after 40min and dried in vacuum drying chamber at 120℃ for 1.5h. The glass spheres gained from the coupling step were dispersed in ~300ml of a solution containing 0.6g/L of $PdCl_2$. The activated hollow glass spheres were separated, rinsed twice with deionized water and dried at 50℃.

In a typical hydrothermal procedure, an aqueous solution was prepared by dissolving $NiSO_4 \cdot 6H_2O$ (0.1M) in 23 ml of distilled water containing different amounts of NaOH（0～1.0M）. After adding 7ml $N_2H_2 \cdot H_2O$ (hydrate hydrazine 50%) and 0.3g of the pretreated hollow glass spheres, the mixture was stirred vigorously for 5min. Then the suspension was transferred into a Teflon-lined stainless-steel autoclave with a capacity of 50ml for hydrothermal treatment at 120℃ for 12h. After the reaction was completed, the resulting products were collected, rinsed thrice with distilled water, then vacuum-dried at 60℃.

XRD analysis was carried out on a Regaku D/max2200PC diffractometer with Cu-Kα radiation (λ=1.5406 Å). XPS data were obtained with a ESCALab220i-XL electron spectrometer from VG Scientific using 300W Mg-Kα radiation. The binding energies were referenced to the C1s line at 284.8 eV from adventitious carbon. The scanning electron microscopy (SEM) images and energy dispersive X-ray (EDX) were obtained using a Hitachi S-4300 microscope and EMAX Horiba, respectively. The particle density of the composite hollow spheres was measured by Archimedes method with water as the immersion fluid. High-resolution transmission electron microscopy (HRTEM) images were performed with a Philips TECNAI-20 transmission electron microscope at an acceleration voltage of 200 kV. Magnetic measurements were carried out at room temperature using a vibrating sample magnetometer (VSM, Lakeshore 7307, USA) with a maximum magnetic field of 1T.

Figure S1. XPS survey spectrum of Ni 2p of the composite spheres.

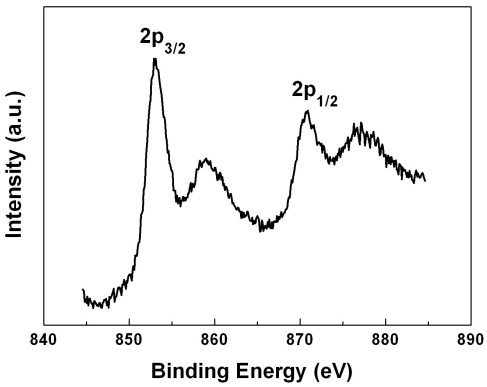
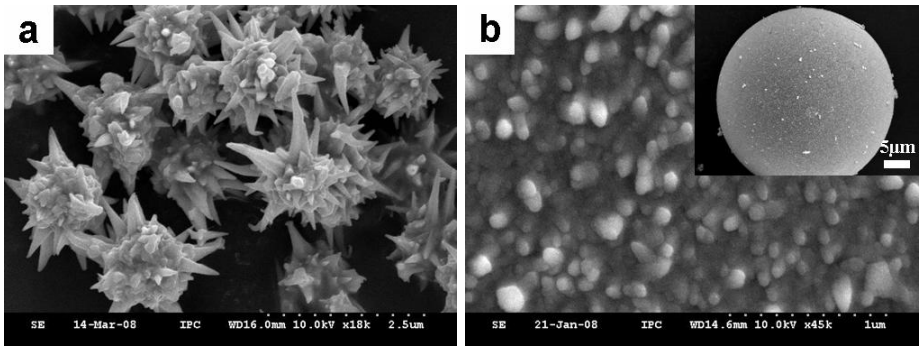


Figure S2. SEM images of: a) isolated nickel particles obtained at 120℃ using pristine HGS, b) core/shell composite hollow spheres obtained at 150℃.



Figures S3. SEM images of the products obtained with different NaOH concentrations: a 0.05M, b 0.1M, c 0.4M, d 1.0M.

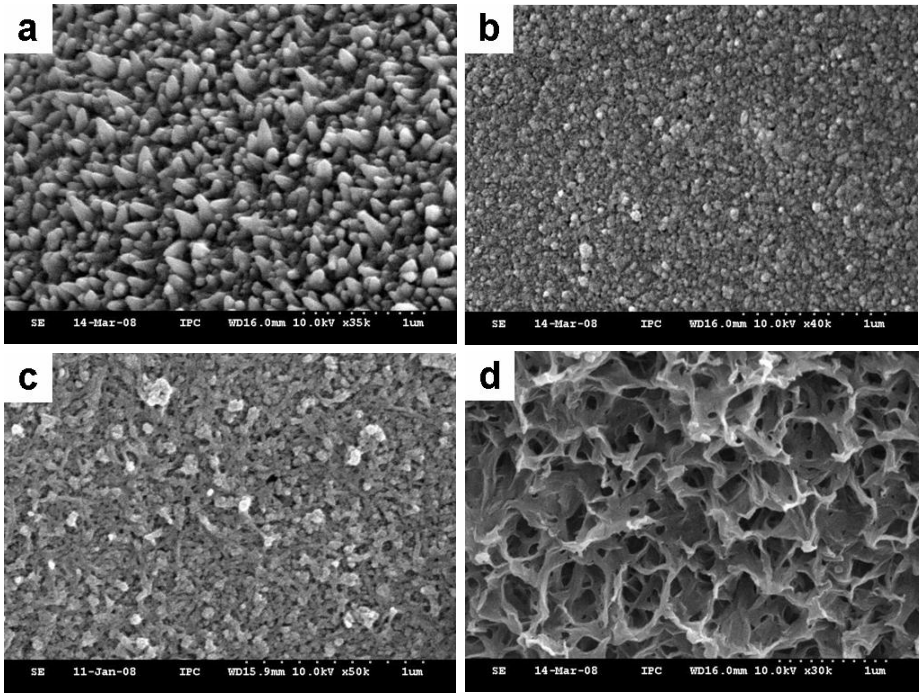


Figure S4. SEM observations of the nickel shells at different reaction stages: a 1h; b 3h; c 6h; d 12h. reaction temperature 120℃, without sodium hydrate.