Crystalline Carbon Hollow Spheres, Crystalline Carbon-SnO₂ Hollow Spheres, and Crystalline SnO₂ Hollow Spheres: Synthesis and Performance In Reversible Li Ion Storage

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Supporting Information 1

The following FE-SEM images (at same magnification) compare two types of crystalline hollow carbon prepared at 1000 and 900 °C. A smoother surface could be observed for the carbon prepared at 1000 °C compared to the sample at 900 °C.

Supporting Information 2

The following XRD patterns compare two types of crystalline hollow carbon prepared at 1000 and 900°C. The carbon prepared at a higher temperature (1000°C) has a more graphitic crystalline structure.

Supporting Information 3

The following thermogravimetric analysis (TGA) curves of crystalline carbon hollow spheres prepared at 1000°C and 900°C show that the silica template had been completely removed by aqueous HF solution because there were no residues left at temperatures above 800 °C. Weight losses were observed between 600-800°C and 530-730°C for carbons prepared at 1000°C and 900°C, respectively. A higher decomposition temperature indicates higher thermal stability, and thus a higher graphitic extent. The conclusion from the TGA measurements is in agreement with the XRD patterns in Supporting Information 2.

Supporting Information 4

The following two FE-SEM images compare the carbon-supported SnO₂ nanoparticles prepared by microwave-assisted heating method (300 W at 85°C for 3 min) and by conventional heating method (water bath at 85°C for 3 h). A detectable agglomeration of particles could be observed for the sample prepared by conventional heating. The microwave-assisted method was found to produce a more uniform dispersion of nanoparticles on the carbon surface with less agglomeration.

Supporting Information 5

The following FE-SEM image shows that carbon spheres were intact at temperatures as high as 500°C. However there was a detectable particle size growth for the SnO₂ nanoparticles (6-8 nm), compared to the SnO₂ nanoparticles (1-3 nm) heat treated at 350°C (Figures 3).

Supporting Information 6

The following XRD patterns show the SnO₂-encapsulated/decorated crystalline carbon hollow spheres after heating at 350 °C and 650 °C respectively. Broad SnO₂ (101) and (211) diffractions were observed for the 350 °C sample, indicating very small particle size. After heating at 650 °C for 3 h, these peaks became shaper and a few other weaker peaks corresponding to the (200), (220), (310), (112), and (301) diffractions also appeared, indicating substantial growth in particle size and increased crystallinity. All the diffraction peaks can be indexed to the rutilelike SnO₂ with tetragonal lattice constants $a_0 = 4.74 \text{ \AA}$ and $c_0 = 3.19 \text{ \AA}$ (JCPDS 41-1445).

It should be noticed that (110) reflection ($\sim 26.5^\circ$) of SnO₂ was shadowed by the comparatively strong (002) reflection ($\sim 25.7^\circ$) of crystalline carbon hollow spheres. The (110) reflection peak could be clearly seen after the decomposition of carbon at 650 °C.

Supporting Information 7

The following HRTEM image shows the crystalline carbon hollow spheres after 200 cycle repetitive charge and discharge at the 1C rate. Similar to Figure 2C, packed graphene sheets could be still observed.

Supporting Information 8

The following FE-SEM image shows the SnO₂-carbon composite with a low loading of SnO₂ nanoparticles (~25.6 wt%, as determined by ICP).

Supporting Information 9

The following Figures show the charge and discharge curves of crystalline carbon hollow spheres and crystalline carbon hollow spheres with 25.6 wt% of SnO_2 . (a1) the first cycle charge and discharge curves of crystalline hollow carbon; (b1) the first cycle charge and discharge curves of 25.6 wt% SnO_2 -carbon composite; (a2) the 100th cycle charge and discharge curves of crystalline hollow carbon; (b2) the 60th cycle charge and discharge curves of 25.6 wt% SnO_2 -carbon composite. Measurement conditions: 0.2 C, 5 mV- 2 V. The first cycle columbic efficiencies were measure to be ~72.5%, and 62.4% for the crystalline carbon hollow spheres and 25.6 wt% of SnO_2 -carbon composite respectively.

Supporting Information 10

The following Figures show the first cycle cyclic voltammograms of (top) crystalline hollow carbon and (bottom) 25.6 wt% SnO_2 -carbon composite (cyclic voltammetry was performed on an EG&G model 273 potentiostat/galvanostat, using the active anode as the working electrode and lithium as both the counter and reference electrodes. The scan range of 0-1.5 V (vs. Li) was swept at the rate of 0.1 mV/s). The cathodic peaks at ~0.6-0.8 V could be attributed to the solid electrolyte interface (SEI) formation on the active materials and to the reduction reaction of SnO_2 to Sn.