

## TiO<sub>2</sub> Hollow Spheres with Large Amount of Exposed (001) Facets for Fast Reversible Lithium Storage

Shujiang Ding,<sup>a</sup> Jun Song Chen,<sup>a</sup> Zhiyu Wang,<sup>a</sup> Yan Ling Cheah,<sup>b</sup> Srinivasan Madhavi,<sup>b</sup> Xiao Hu<sup>b</sup> and Xiong Wen Lou<sup>\*a</sup>

<sup>a</sup> School of Chemical and Biomedical Engineering, Nanyang Technological University, 70 Nanyang Drive, Singapore 637457.

E-mail: xwlou@ntu.edu.sg

<sup>b</sup> School of Materials Science and Engineering, Nanyang Technological University, 50 Nanyang Avenue, Singapore 639798.

### Experimental Section

**Materials Synthesis.** In a typical synthesis, 0.4 g of sulfonated polystyrene hollow spheres, was dispersed into 21 mL of isopropyl alcohol (IPA) by ultrasonication for 5 minutes. 0.3 mL of diethylenetriamine (DETA; 99 %, Sigma-Aldrich) was added to IPA solution. After the solution was gently stirred for 5 minutes, 0.3 mL of titanium(IV) isopropoxide (TIP; 97 %, Sigma-Aldrich) was added. The reaction solution was then transferred to a 60 mL Teflon-lined stainless steel autoclave and kept in an electric oven at 200 °C for 24 h. The autoclave was then taken out from the oven and left to cool down to room temperature. The pale yellow precipitate was collected by centrifugation, washed thoroughly with ethanol, and dried at 80 °C overnight. All of the products were calcined at 400 °C in air for 2 h with a heating rate of 1 °C min<sup>-1</sup> to obtain a highly crystalline anatase phase.

**Materials Characterizations.** The product morphology was examined using field-emission scanning electron microscopy (FESEM; JEOL, JSM-6700F, 5 kV) and transmission electron microscopy (TEM; JEOL, JEM-2100F, 200 kV). The elemental compositions of the samples were analyzed with energy-dispersive X-ray spectroscopy (EDX) attached to the FESEM instrument. Crystallographic information for the samples was collected using powder X-ray diffraction (XRD; Bruker, D8 Advance X-ray diffractometer, Cu K $\alpha$  radiation,  $\lambda = 1.5406 \text{ \AA}$ ). The nitrogen adsorption was performed using a Quantachrome Instrument (Autosorb AS-6B). Thermogravimetric analysis (TGA) was carried out under a flow of air with a temperature ramp of 5 °C min<sup>-1</sup>.

**Electrochemical Measurements.** The electrochemical measurements were carried out using two-electrode Swagelok cells (X2 Labwares, Singapore) with pure lithium metal as both the counter and the reference electrodes at room temperature. The working electrode consisted of active material (e.g., TiO<sub>2</sub> NSHSs), a conductive agent (carbon black, Super-P-Li), and a polymer binder (poly(vinylidene difluoride), PVDF, Aldrich) in a 70:20:10 weight ratio. The electrolyte used was 1.0 M LiPF<sub>6</sub> in a 50:50 (w/w) mixture of ethylene carbonate and diethyl carbonate. Cell assembly was carried out in an Ar-filled glovebox with concentrations of moisture and oxygen below 1.0 ppm. Cyclic voltammetry (1–3 V, 0.2 mV s<sup>-1</sup>) was performed using an electrochemical workstation (CHI 660C). The charge/discharge tests were performed using a NEWARE battery tester at different current rates (1C = 170 mA g<sup>-1</sup>) with a voltage window of 1–3 V.

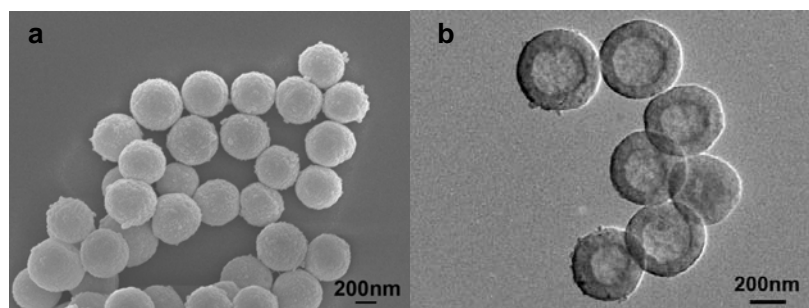


Figure S1. (a, b) SEM and TEM images of as-prepared PHS hollow spheres.

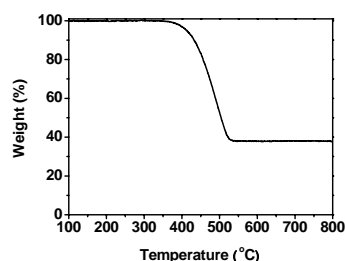


Figure S2. TGA curve of as-prepared carbon@TiO<sub>2</sub>-NSs hollow spheres.