Supporting Information for

SnO₂ Nanosheet Hollow Spheres with Improved Lithium Storage Capabilities

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Experimental section

Materials Synthesis

In a typical synthesis, 12.5 mg of sulfonated polystyrene hollow spheres (sPSHSs) was dispersed into 40 mL of a 10 mM mercaptoacetic acid (97 wt%, Alfa Aesar) solution by sonication for 10 minutes, followed by addition of 0.3 mL of HCl solution (37 wt%, Sigma-Aldrich). After stirring for 5 minutes, 100 mg of Tin (II) chloride dihydrate (SnCl₂·2H₂O, 99%, Sigma-Aldrich) and 0.5 g of urea were added. After being stirred for another 5 minutes, the reaction solution was transferred to a 60 mL Teflon-lined stainless steel autoclave and kept in an electric oven at 120 °C for 6 h. The autoclave was then taken out of the oven and left to cool down to room temperature. The dark yellow precipitate was collected by centrifugation, washed thoroughly with ethanol, and dried at 80 °C for 12 h. The products were further calcined at 400 °C in air for 2 h with a heating rate of 1 °C min⁻¹ to obtain highly crystalline SnO₂.

Materials Characterizations

The morphology and structure of samples were examined using field-emission scanning electron microscopy (FESEM; JEOL, JSM-6700F, 5 kV) and transmission electron microscopy (TEM; JEOL, JEM-2100F, 200 kV). Crystallographic information of the samples was collected from powder X-ray diffraction (XRD; Bruker, D8 Advance X-ray diffractometer, Cu K α radiation, $\lambda = 1.5406$ Å). The surface area and pore size distribution of the sample were measured using a Quantachrome Instrument (Autosorb AS-6B). Thermogravimetric analysis (TGA) was carried out under a flow of air with a temperature ramp of 20 °C min⁻¹.

Electrochemical Measurements

The electrochemical measurements were carried out using two-electrode Swagelok cells (X2 Labwares, Singapore) with pure lithium foil as both the counter and the reference electrodes at room temperature. The working electrode consists of the active material, conductive agent (carbon black, Super-P-Li) and polymer binder (poly(vinylidene difluoride), PVDF, Aldrich) in a 70:20:10 weight ratio. The electrolyte used was 1.0 M

LiPF6 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 (0.005–2.5 V, 0.5 mV s⁻¹) was performed using an electrochemical workstation (CHI 660C). The charge/discharge tests were performed using a NEWARE battery tester at a current rate of 160 mA g⁻¹ (~0.2C) with a voltage window of 0.01-1.2 V.



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



Figure S2. XRD patterns of samples: (I) $sPS@SnO_x$ (*x*=1, 2) NSHSs (II) SnO_2 NSHSs after calcination in air. The peaks marked in red and black in pattern I are due to SnO_2 and SnO, respectively.



Figure S3. TGA curve of sPS@SnO_x NSHSs.