

Supporting Information

A facile fabrication of hierarchical hollow microspheres assembled by titanate nanotubes

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Experiment.

Preparation of amorphous $\text{TiO}_2 \cdot n\text{H}_2\text{O}$ microspheres: All chemicals were used as received. Monodispersed amorphous $\text{TiO}_2 \cdot n\text{H}_2\text{O}$ spheres were prepared by controlled hydrolysis of titanium tetraisopropoxide (TTIP, $\text{Ti}(\text{OC}_3\text{H}_7)_4$, 97%, Aldrich) in ethanol. Typically, 35 mL of ethanol was mixed with 0.15 mL of 0.1 M aqueous potassium chloride, followed by the addition of 0.8 mL of TTIP at ambient temperature. The solution was mixed completely using a magnetic stirrer for about 10 min until a white precipitate appeared. The suspension was aged in a static condition for 24 h in a closed container at room temperature in air atmosphere. The $\text{TiO}_2 \cdot n\text{H}_2\text{O}$ precipitate (~ 0.1 g) at the bottom of the vessel was washed several times with ethanol and then was used as the precursor for the preparation of titanate hollow spheres.

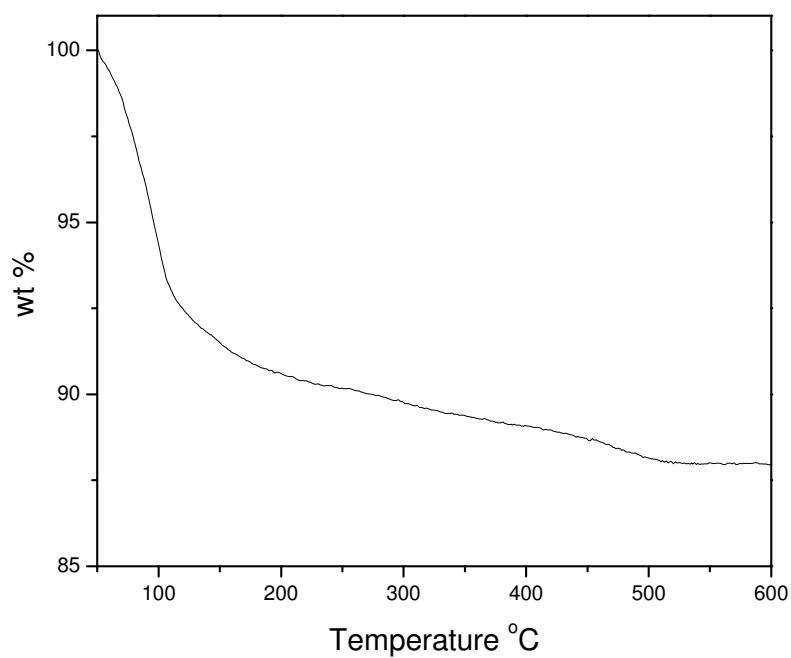


Figure S1 Thermogravimetry behavior of protonated titanate. The TG curve implies that the protonated titanate shows a weight loss of about 12% after heating up to 600 °C. The steep weight loss is found before 200 °C, which is usually attributed to the adsorbed water and interlayer water. The weight loss above 200 °C is thought to be the dehydration of structural water.

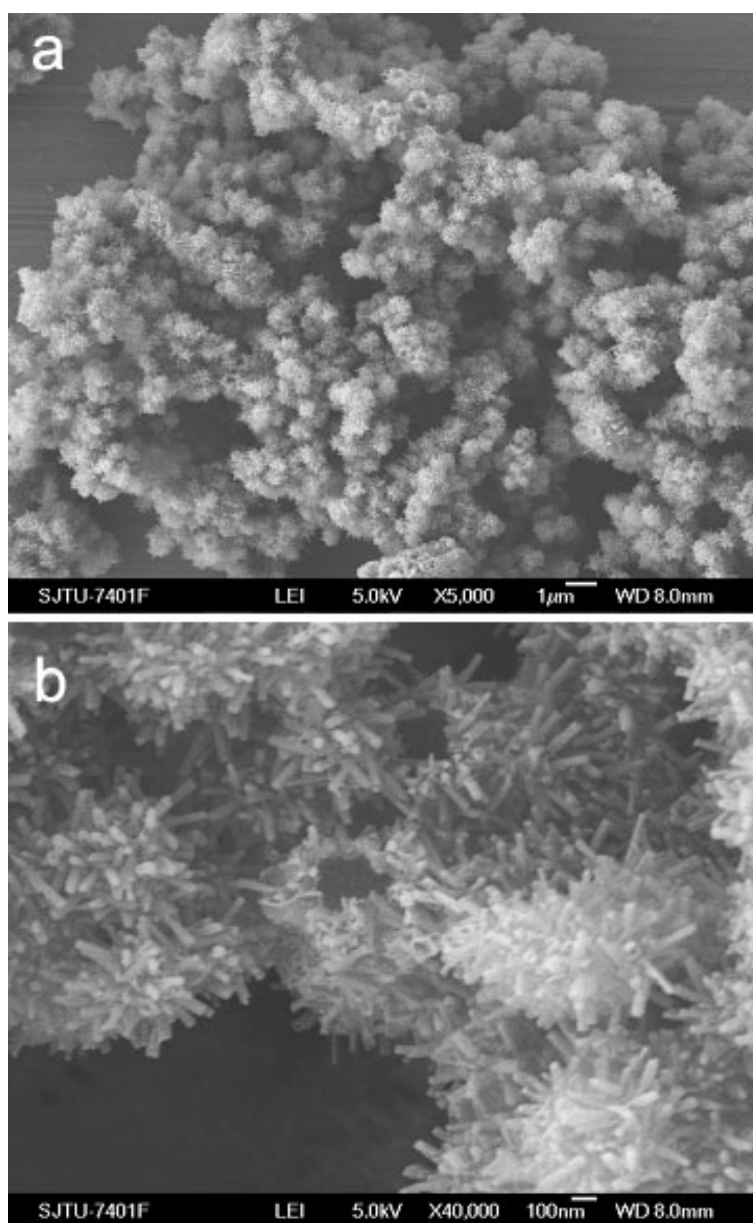


Figure S2 FESEM images of hierarchical protonated titanate hollow microspheres.

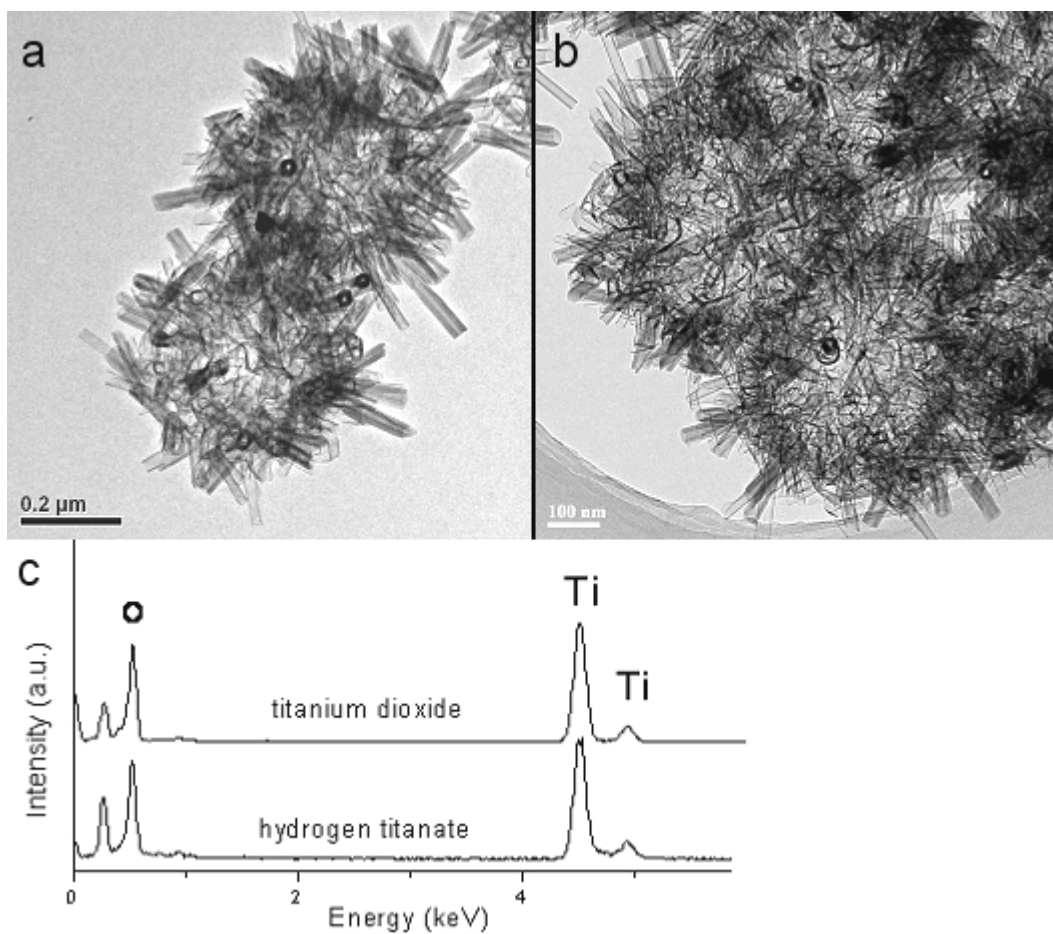


Figure S3 The typical TEM images of titanate hollow microspheres after acid washing (a) and following calcination (b), and their corresponding EDX spectrums (c).

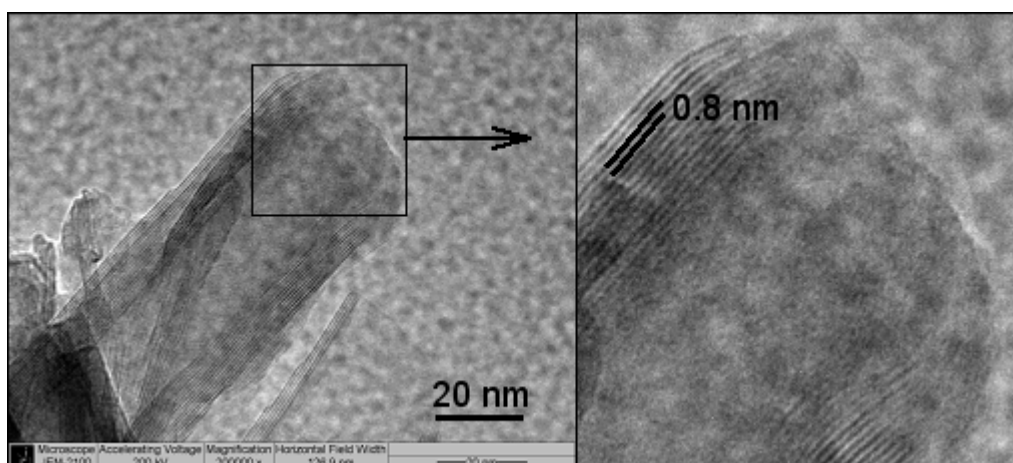


Figure S4 TEM image of one titania nanotube and its drawing of partial enlargement.

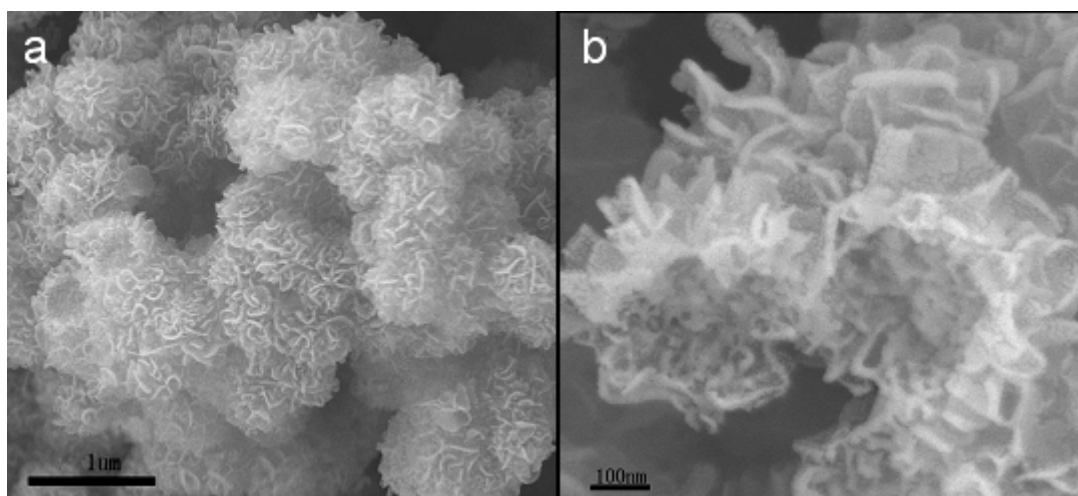


Figure S5 FESEM images of the product from the hydrothermal system without H₂O₂.

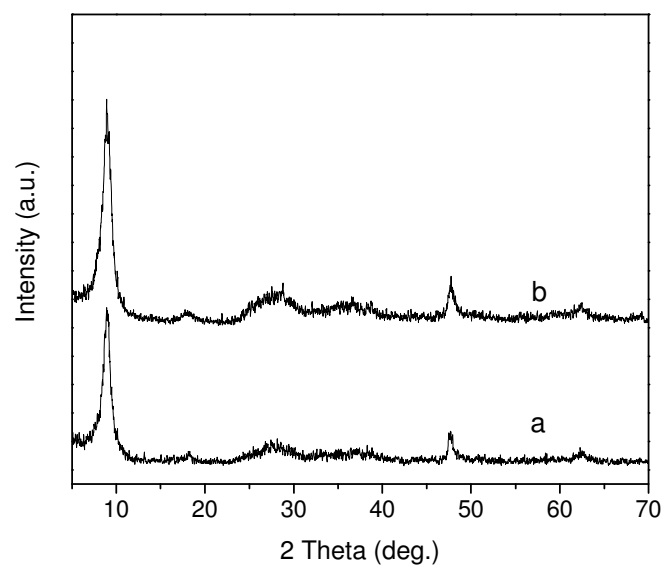


Figure S6 XRD patterns of the hollow spheres composed of titanate nanosheets (a) and the hollow spheres composed of titanate nanotubes (b).

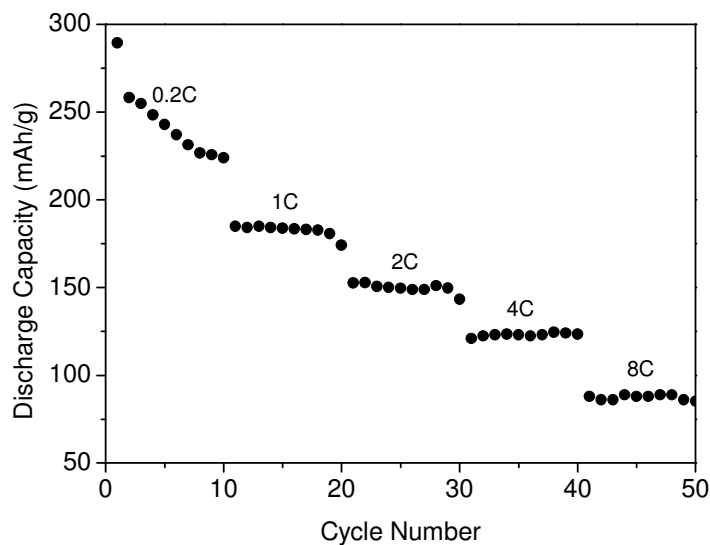


Figure S7 The rate performance of hierarchical anatase TiO₂ hollow microspheres at 0.2-8C rate. The electrochemical characterization was measured with coin cells in which a lithium metal foil was used as the counter electrode. The electrolyte employed was 1M solution of LiPF₆ in ethylene carbonate and dimethyl carbonate (1:1 in volume). The composite electrodes were made of the active materials powder (60 wt%), acetylene black (20 wt%) and polyvinylidene fluoride binder (20 wt%) homogeneously mixed in N-Methyl pyrrolidinone solvent and then coated uniformly on an aluminium foil. Finally, the electrode was dried under vacuum at 110 °C for 10 h. Cell assembly was carried out in an argon-filled glove box. The coin cells were cycled under different current densities between cutoff voltages of 3.0 and 1.0 V on a CT2001A cell test instrument (LAND Electronic Co.) at room temperature.

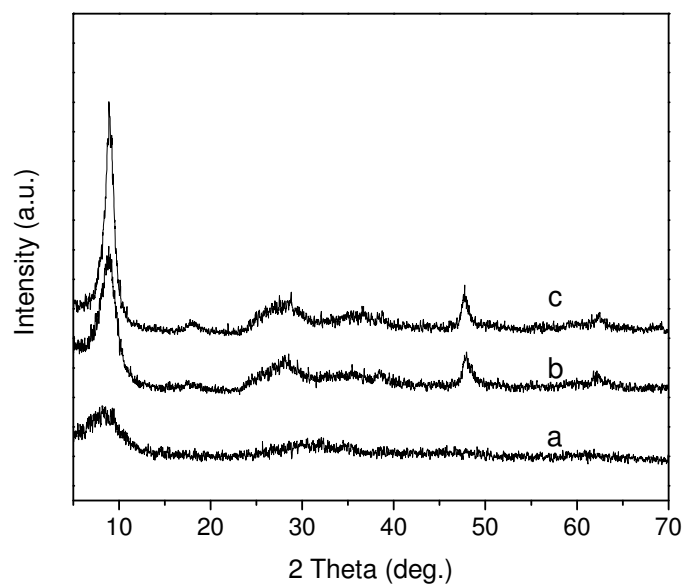


Figure S8 XRD patterns of the products obtained at 180°C for different reaction times:

(a) 1 h, (c) 2 h, and (d) 10 h.