Supporting Information for Hierarchical MoS₂/MoO₃ nanotubes with excellent electrochemical performance: MoS₂ bubbles on MoO₃ nanotubes

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Experimental section

All the reagents are of analytical purity grade without further purification. In order to obtain MoO₃ nanorods, MoO₃ powder (10 mmol) was dissolved into aqueous H_2O_2 solution (30%, 10 mL) under stirring. The molybdenum concentration was controlled to be 0.9 mmol L⁻¹ by adding HNO₃ solution (2 mmol L⁻¹). Then, the as-prepared solution was kept for 4 days until its color is transparent deep yellow. Subsequently, 48 mL of precursor solution was poured into a Teflon-lined stainless steel autoclave (80 mL), which was heated to 170 °C for 24 h. Finally, the precipitate could be collected after cooling down to room temperature by centrifuging and rinsing with deionized water. The as-obtained powder was dried at 60 °C in vacuum for overnight. To prepare hybrid MoS₂/MoO₃ hierarchical nanotube, 0.1 g of MoO₃ nanorods was dispersed in 80 mL of N-methyl-2-pyrrolidone (NMP) which was transferred into a three-necked flask (250 mL), which was dropwise added with 5 mL of N₂H₄•H₂O under stirring. Then the flask was heated to 80 °C in an oil bath under reflux for 24 h. After being naturally cooled, the precipitates were collected and washed with deionized water. To dry the product, the precipitate was washed with ethanol for several times and heated at 60 °C for overnight. MoS₂ bubble-like hollow nanoparticles can be obtained by the same procedure except adding MoO₃ nanorods.

The morphologies of samples were characterized by scanning electron microscopy (SEM, JSM-6701F) operated at an accelerating voltage of 5 kV and transmission electron microscopy (FEI Tecnai TF20). XRD patterns of products can be recorded by Rigaku RINT-2000 instrument using Cu K α radiation (40 kV). ESCALAB 250Xi X-ray photoelectron spectrometer (XPS) was used to investigate the oxidation states of elements in as-prepared samples.

The electrochemical properties of samples were measured on a CHI660 (Chenhua, Shanghai, China) with three electrode system. In order to prepare the electrode, 8.5 mg of active material, 1.0 mg of carbon black and 0.5 mg of poly(tetrafluoroethylene) were mixed to form a homogeneous slurry. Then the slurry was coated on the nickel foam with coating area of 1 cm², which was dried at 60 °C for 6 h to obtain the working electrode. The mass of active materials on the 1 cm² nickel foam was measured to be 8.5 mg. In addition, a saturated calomel electrode (SCE) and platinum sheet

were used as the reference and counter electrodes, respectively. During the electrochemical process, 1 M KCl aqueous solution was used as electrolyte. In addition, the electrochemical impedance spectroscopy (EIS) profile was performed in the frequency in a range of $10 \text{ mHz}-10^5 \text{ Hz}$.

Additional Figures



Fig. S1 (A) TEM and (B) HRTEM image of hierarchical MoS₂/MoO₃ nanotube.



Fig. S2 (A) scanning TEM (STEM) micrograph and EDX mapping images of (B) Mo (C) S and (D) O.



Fig. S3 (A) XRD pattern of samples; (B) SEM image (C) TEM image and (D) high-resolution TEM (HRTEM) image of

MoO₃ nanorods.



Fig. S4 SEM images of product obtained at (A) 6 h (B) 12 h (C) 18 h during hydrothermal reaction.



Fig. S5 (A and B) SEM images of product without adding MoO_3 nanorods.



Fig. S6 CV curves of (A) MoS₂ bubble-like hollow nanospheres (B) MoO₃ nanorods and (C) samples at 10 mV s⁻¹ (D) specific capacitances of electrodes at different scan rates.



Fig. S7 SEM images of samples after (A) 1000 and (B) 3000 charge-discharge cycles at 1.17 A g⁻¹.