Supplementary Information

Facile one-step synthesis of 3D macroscopic SnO_2/graphene aerogel and its application as a superior anode material for Li-ion batteries

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Experimental Details

Chemical reagents and materials

Graphite powder (325 mesh, with purity >99.99%) was obtained from Alfa Aesar. All other chemicals (purchased from Beijing Chemical Co.,Ltd.) used in this experiment were analytical grade and were used without further purification.

Preparation of SGA

GO was synthesized from natural graphite powder by the modified Hummer's method as originally presented by Kovtyukhova et al.[1] The resulting solid graphite oxide was dispersed in water and subjected to dialysis for 7 days to completely remove metal ions and acids. In order to obtain GO nanosheets dispersed in water, the solution after dialysis was sonicated for 1 h with a frequency of 40 kHz (KH-500, Kunshan, Hechuang Ultrasonic Cleaner Inc.). Subsequently, the suspension of GO nanosheets was treated by high-speed centrifugation (4000rpm, 30min) to remove any undispersed solid, and afterwards, a brown homogeneous supernatant was collected.

In a typical synthesis of the SGA, we prepared a mixture of GO, $SnCl_2$ and HCl in a 20 mL cylindrical sampler vial with concentrations of 2 mg/ml, 0.004 and 0.08 M, respectively. The resulting mixture was reacted at 90℃ for 1 h without stirring. Finally, the 3D black monolith was taken out, washed with distilled water, and freeze-dried into an aerogel for further characterizations and electrochemical measurements.

2.3. Characterizations

The structures and compositions of the as-prepared products were characterized by X-ray powder diffraction (XRD) using a Rigaku Dmax 2200 X-ray diffractometer with Cu Kα radiation (λ= 1.5416 Å). The morphology of the as-prepared sample was investigated by JEOL JSM-7001F field-emission scanning electron microscope (FESEM). Transmission electron microscopy (TEM) and high-resolution TEM (HRTEM) investigations were carried out by a JEOL JEM-2100F microscope. The as-prepared samples were dispersed in ethanol and dropped onto a copper grid for the drying process in air. Raman spectrometer was recorded on a LabRAM HR800(HORIBA Jobin Yvon) confocal Raman spectrometer, with an excitation laser wavelength of 488.5 nm. All samples were deposited on silicon wafers in powder form without using any solvent. The XPS data were taken on an AXIS Ultra instrument from Kratos Analytical. The IR spectra were carried out through a Nicolet iN10 MX (Thermo Scientific) in the infrared domain 600-4000 cm^{-1}

2.4. Electrochemical measurements

The electrochemical properties of the SGA as anode materials in lithium ion cells were evaluated by galvanostatic charge/discharge technique. The test electrodes were prepared by mixing 80 wt % active material with 10 wt % carbon black and 10 wt % polyvinylidene fluoride (PVDF) dissolved in N-methyl-2-pyrrolidone (NMP) to form a slurry, which was then coated onto a copper foil (current collector), dried at 80 °C for 10 h and finally pressed under pressure of 10 MPa. Afterwards, CR2016 type coin cells were assembled in an highly-pure argon-filled glovebox using the test electrodes, the metallic lithium counter/reference electrode, a polypropylene separator (Celgard 2400), an electrolyte of 1 mol/L $LiPF_6$ in ethylene carbonate and diethyl carbonate (EC/DMC, 1:1 vol) (Tianjin Jinniu Power Sources Material Co., Ltd. China). Charge-discharge measurements were carried out galvanostatically at a current density of 100 $mA \cdot g^{-1}$ in the voltage range of 0.005 V ～ 1.5 V using a battery test system (LAND CT2001A model, Wuhan Jinnuo Electronics. Ltd., China).

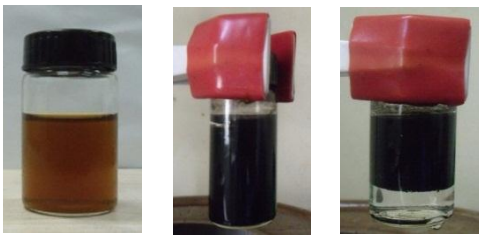

Fig. S1 Photographs of the time-dependent formation process of the SGH

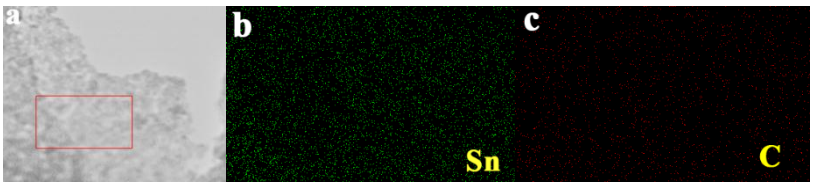

Fig. S2 TEM image obtained from the SGA (a) with corresponding EDS maps for Sn (b) and C (c).

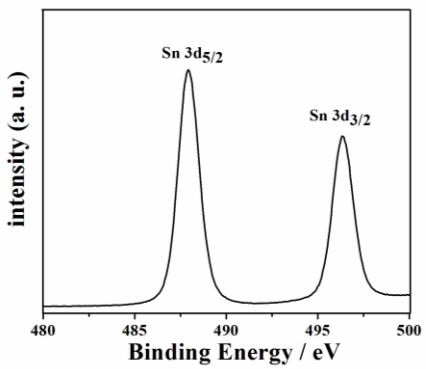

Fig. S3 Sn 3d XPS spectrum of the SGA

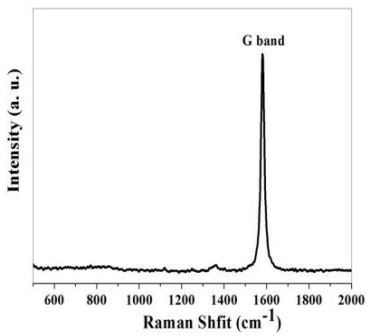

Fig. S4 The Raman spectrum of graphite

Table S1. Summary of reversible specific capacities of SnO_2/graphene composites reported previously and in our work.

Current density ($mA\ g^{-1}$)	Initial capacity ($mA\ h\ g^{-1}$)	Cycle number (n)	Residual capacity ($mA\ h\ g^{-1}$)	Ref.
50	810	30	670	10a
50	1080	30	694	10b
156	786	50	558	10c
55	765	100	520	10e
200	541	35	377	10f
67	978	30	840	10h
100	819	50	626	10i
100	690	20	433	10k
10	unknown	110	625	10r
100	902	60	602	Our work

Reference

1 N. I. Kovtyukhova, P. J. Ollivier, B. R. Martin, T. E. Mallouk, S. A. Chizhik, E. V. Buzaneva and A. D. Gorchinskiy, *Chem. Mater.*, 1999, **11**, 771.