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Sb–C Nanofibers with Long Cycle Life as an Anode Material for High-performance Sodium-ion Batteries

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Experimental Section

1. Material synthesis

Experimental procedures are schematically presented in Fig. 1. A typical experiment is as following: 0.75 g polyacrylonitrile (PAN, Mw =150,000, Aldrich) and 0.75 g SbCl₃ (99% purity, National Medicine Co., Ltd, Shanghai, China) were dissolved in 9 ml dimethylformamide (DMF) solvent at 60 °C with vigorous stirring for 12 hour. The obtained mixture was used as the working fluid for electrospinning. The precursor solution was loaded into a 10 mL syringe equipped with a size seven needle. The flow rate of solution was 10 μ L min⁻¹ controlled by a syringe pump (Model LSP01-1A, Shanghai, China). The needle was connected to a high voltage power CDF, Tianjin, China). A voltage of 10 kV was supply (Model DW-N503-1A applied between the needle and the aluminum foil. The aluminium foil was used to collect the nanofibers. The as-collected nanofibers and comparison composite were all first heat- treated in a tube furnace at 2 °C min⁻¹ up to 280 °C in Ar (92 vol. %) / H₂ (8 vol. %) for 6 h to stabilize the fiber structure by reaction of PAN. Subsequently, the Sb-C nanofibers were obtained by carbonthermal reaction at 700 °C for 6 h in Ar (92 vol. %) / H₂ (8 vol. %). For comparison, the preparation process of the Sb/C composite prepared by a conventional method is the same as the Sb-C nanofibers just without the electrospinning process.

2. Sample characterization

Morphological characterizations of the Sb-C nanofibers before and after sodium insertion were performed by scanning electron microscopy (SEM, ULTRA/PLUS, ZEISS) and by transmission electron microscopy (TEM, JEOL, JEM-2010-FEF). The crystalline structures of the as-prepared nanofibers were characterized by X-ray

diffraction (XRD, Shimadzu XRD-6000). The composition analysis of the Sb-C nanofibers was performed with TG measurement (Diamond TG/DTA6300).

3. Electrochemical measurements

The Sb-C anodes were prepared by mixing 70 wt% Sb-C composite, 20 wt% super P, and 10 wt% Polyacrylic acid (PAA, 25 wt%) to form a slurry, which was then coated onto a copper (Cu) foil and dried at 60 °C overnight under vacuum. The area of the electrode is ~ 1.13 cm² while the loading of the whole material is about 2.5 mg/cm². The charge-discharge performances of the electrode were examined by 2032 coin-type cells using the Sb-C anode as a working electrode and a Na sheet as counter and reference electrode, 1 M NaPF₆ dissolved in ethylene carbonate (EC)/diethyl carbonate (DEC) (1:1 by volume) with 5% fluoroethylene carbonate (FEC) as the electrolyte (about 0.1 mL), and a Celgard 2400 microporous membrane as separator. The Na sheets were home-made by rolling sodium lumps into thin plate, and then cut into circulated disks. All the cells were assembled in a glove box with water/oxygen content lower than 1 ppm and tested at room temperature. The galvanostatic charge-discharge test was conducted on a LAND cycler (Wuhan Kingnuo Electronic Co., China). The discharge/charge capacities were calculated based on the Sb mass in the composites by deducting the capacity contribution of the pyrolyzed corbon from PAN nanofiber and Super P. Cyclic voltammetric measurements were carried out with the coin cells at a scan rate of 0.1 mV s⁻¹ using a CHI 660 c electrochemical workstation (ChenHua Instruments Co., China).



Fig. S1 TG curve of the Sb-C nanofibers.



Fig. S2 XRD pattern of the combustion product of Sb-C nanofibers



Fig. S3 The initial two discharge/charge profiles of the pyrolyzed carbon from PAN nanofibers (a) and Supper P (b) between 0.01 V and 2.0 V vs. Na^+/Na at a current rate of 100 mA g⁻¹.



Fig. S4 (a) Cycling performance of the pyrolyzed carbon from PAN nanofibers at a cycling rate of 100 mA g⁻¹; (b) C rate capability of the carbon electrode at various current rates from 100 mA g⁻¹ to 800 mA g⁻¹.



Fig. S5 (a)Cycling performance of the Sb-C composites without electrospinning at a cycling rate of C/3 (200 mA g⁻¹); (b) TG curve of the Sb-C composites without electrospinning; (c,d) SEM images of the Sb-C composites without electrospinning.



Fig. S6 XRD patterns of the Sb-C nanofibers electrode after the first discharging to 0.01 V at 100 mA g⁻¹.