Supplementary information

Fabrication of porous NiS/Ni nanostructured electrode via a dry thermal sulfuration method and its application in lithium ion battery

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Experimental details

Fabrication procedure: The chemicals were analytical grade and purchased from Shanghai Chemical Reagents. In a typical procedure, 1 mmol thiourea were milled and transferred into a 50 ml teflonlined autoclave, Ni foams (disc with diameter of 14 mm, 100 PPI pore size, 380 g m^{-2} surface density, 1.5 mm thick, purchased from Changsha Lyrun New Material) were subsequently placed in it. The autoclave was at last sealed in atmosphere and placed in an oven, heated at 160 ^{o}C for 4h. The mass of Ni foam disc electrode before and after sulfuration was weighted by a microbalance. According to the reaction (S + Ni → NiS), the weight of the active NiS are derived from $m_{NiS} = \Delta m \times 90.8/32.1$, where Δm is the weight difference of Ni foam before and after sulfuration.

Structure characterization: The morphology and structure of the resulting products were characterized by field-emission scanning electron microscopy (FE-SEM JSM 7500F, JEOL) and X-Ray powder diffraction (Rigaku Ultima IV with 3kV high-frequency X-Ray generator, X-Ray tube Cu 9407F701, Scintillation counter probe HD031019, Graphite Momochromator HD05648, Cu Kα radiation λ=1.5406 Å).

Electrochemical characterization: For fabricating of Li-ion battery, the as-prepared NiS/Ni foams (disc electrode with diameter of 14 mm) were dried (120 ^{o}C, 24 h, vacuum). Coin-type cells (2025) of Li/1 M $LiPF_6$ in ethylene carbonate, dimethyl

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carbonate and diethyl carbonate (EC/DMC/DEC, 1:1:1 v/v/v)/ NiS/Ni disc electrode were assembled in an argon-filled dry box (MIKROUNA, Super 1220/750, H_2O<1.0 ppm, O_2 < 1.0 ppm). A Celgard 2400 microporous polypropylene was used as the separator membrane. The cells were tested in the voltage range between 0.1 and 3 V with a multichannel battery test system (LAND CT2001A). The Cyclic voltammetry (CV) measurement of the electrodes was carried out on a CHI660C electrochemical workstation at a scan rate of 0.5 mV s^{-1} between 0.02 and 3 V.

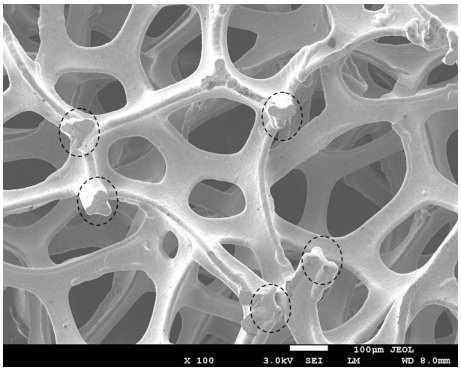

Fig. S1. A cross section SEM image of Ni foam.

As shown in Fig. S1, the Ni foam exhibits three-dimensional porous structure. Ni foam was cut off and placed vertically on sample table for SEM characterization. Due to the three-dimensional structure of Ni foam with a thickness of 1.5 mm, the cross section image of Ni foam is similar to surface image. However, the edges caused by cutting can be clearly seen.

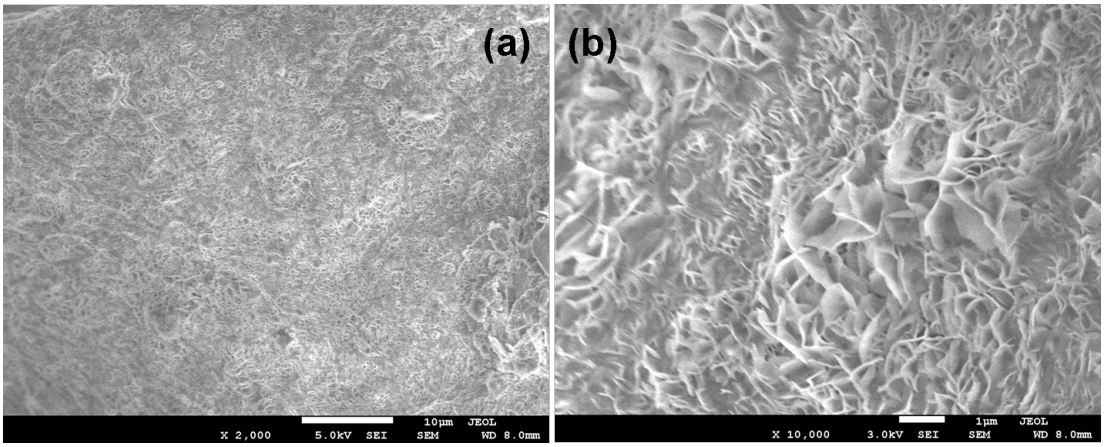

Fig. S2. SEM image of NiS/Ni obtained at 6h with low (a) and high (b) magnification.

Fig. S2 is SEM images of NiS/Ni obtained at 6h. As shown in a low magnification SEM image in Fig. S2(a), hole-like morphology can be generally observed. Fig. S2(b) is a high magnification SEM image of the NiS/Ni electrode, which shows a large number of interconnected nanowalls. Prolonging the reaction time to 6h can obtain clear NiS nanowall-like morphology, indicating the formation of porous architecture of NiS gradually evolved from nanoholes to nanowalls.