Supporting Information

Facilely constructing 3D porous NiCo₂S₄ nanonetworks for high performance supercapacitors

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Experimental

1. Synthesis of the porous NiCo₂S₄ nanonetworks

Nickel foams were used as substrate and cleaned by 1.0 M HCl with ultrasound. The NiCo₂O₄ nanosheets/NF was prepared by hydrothermal treatment of fresh nickel foam in thehomogeneous solution containing 2 mmol Ni(NO₃)₂·6H₂O, 4 mmol Co(NO₃)₂·6H₂O, 24 mmol urea and 100 ml deionized water at 100°C for 6 h. After that, the obtained product was rinsed several times with deionized water, and then annealed at 250°C for 2 hin air, resulting in the NiCo₂O₄ nanosheets/NF. To obtain the porous NiCo₂S₄ nanonetworks/NF, the NiCo₂O₄ nanosheets/NF was immersed into 0.2 M Na₂S solutionand heated at 100°C for 24 h under hydrothermal condition. The loading mass ofNiCo₂S₄ on nickel foam is ~2 mg cm⁻².

2. Characterization

Scanning electron microscopy (SEM) images were taken with a field-emission scanning electron microscope (JEOL 7500F) operating at 5 kV. Transmission electron

microscopy (TEM) images were collected on a Tecnai G2 20 s-twin with an accelerating voltage of 200 kV. Inductively coupled plasma mass spectrometry (ICP-MS) data were collected on an Aglient 7500a ICP-MS spectrometer. X-ray diffraction (XRD) pattern of the product was recorded on a Rigaku D/MAX-3B using Cu K α radiation at a scanning speed of 10°(2 θ)/min in the range of 30-80°. The X-ray photoelectron spectroscopy (XPS) experiments were performed with an ESCA LAB 250 spectrometer using a focused monochromatic Al K α (hv = 1486.6 eV) X-ray beam with a diameter of 200 µm.

3. Electrochemical Measurements

Electrochemical experiments were performed on a CHI 660D electrochemical analyzer (CH Instruments, Chenhua Co., Shanghai, P.R. China). A conventional three-electrode cell was used, including $NiCo_2S_4$ networks/NF as working electrode, an Ag/AgCl (saturated KCl) electrode as reference electrode, and a platinum wire as counter electrode in 2 M KOH solution.

Figures



Fig. S1 SEM images of the (a) bare NF, (b) $NiCo_2O_4$ nanosheets, and (c) 3D porous $NiCo_2S_4$ nanonetworks at low-magnification.



Fig. S2 SEM images of the $NiCo_2S_4$ -based nanonetworks obtained with at different sulfuration time: (a) 6 h, (b) 12 h, (c) 24 h, and (d) 36 h.

Sample	Cell system	Electrolyte	Current density (A g ⁻¹)	Specific capacitance (F g ⁻¹)	Ref.
NiCo ₂ S ₄ nanotubes	Three- Electrode System	1.0 M KOH	0.93	202.3	1
urchin-like NiCo ₂ S ₄		6.0 M KOH	1	1149	2
$NiCo_2S_4$ nanotubes		6.0 M KOH	1	933	4
3D porous NiCo ₂ S ₄ nanonetworks		2.0 M KOH	1	1501.2	This work

Table S1. Comparison of the specific capacitance of reported $NiCo_2S_4$ based materials and our sample for supercapacitors.

1. J. Xiao, L. Wan, S. Yang, F. Xiao and S. Wang, Nano Lett., 2014, 14, 831-838.

 H. Chen, J. Jiang, L. Zhang, H. Wan, T. Qi and D. Xia, *Nanoscale*, 2013, 5, 8879-8883.

3. H. Wan, J. Jiang, J. Yu, K. Xu, L. Miao, L. Zhang, H. Chen and Y. Ruan, *Crystengcomm*, 2013, **15**, 7649-7651.