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Electronic Supporting Information

Direct synthesis of FeS/N-doped carbon composite for high-performance sodium-ion batteries

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Experimental Section

Synthesis of the FeS/NC composite

First, 0.5 g $Fe(NO_3)_3 \cdot 9H_2O$ and 10 g urea were dissolved in 30 mL distilled water and then evaporated the solvent in an oven at 80°C for 24 h. Subsequently, the obtained mixture was annealed at 700°C with a heating rate of 3°C min^{-1} for 1 h under Ar atmosphere to prepare Fe-precursor. Finally, the FeS/NC composite was achieved by mixing the Fe-precursor with sulfur powder (mass ratio 1: 3) and annealed at 650ºC with a heating rate of 2ºC min^{-1} for 3 h in a mixture of Ar and 5% H_2. As control groups, pure FeS powder was prepared without the addition of urea under the same conditions and the pure NC material was obtained by dissolving Fe-precursor in 2 M HCl solution for 48 h.

Characterization

The structure of samples were checked by using X-ray diffraction (XRD) technique (Bruker D8 Advance (Germany) with Cu Kα radiation, $\lambda = 1.5418$ Å). Raman spectroscopic measurement was performed using a Renishaw RM1000 microspectroscopic system. XPS was carried out on a Thermo K-Alpha XPS spectrometer (Thermo Fisher Scientific) equipped with a monochromatic Al Kα X-ray source (hν =1468.6 eV). The morphology of the samples were investigated by scanning electron microscopy (SEM, FEI Quanta 200 FEG) and high-resolution transmission electron microscopy (HRTEM, Tecnai G2 F20 S-TWIN, Japan) operating at 200 KV. Thermogravimetric (TG) analysis was conducted on a STA-449C (Netzsch, Germany) in a temperature range of 30 to 700ºC with a heating rate of 10ºC min^{-1}.

Electrochemical Tests

The electrochemical performance of FeS/NC composite was studied by using 2032-type coin cell. The working electrode was prepared by casting the slurry composed of FeS/NC composite, carbon black, and a binder of polyvinylidene fluoride (PVDF) with a mass ratio of 80:10:10 on Cu foil current collector. The mass loading of the active material was about 0.6 mg cm^{-2}. The metal sodium foil, glass-fiber, and 1.0 M $NaCF_3SO_3$ in diglyme with 5% FEC were utilized as the counter/reference electrode, separator, and electrolyte, respectively. Then, the cells were assembled in glovebox filled with Ar. Galvanostatic charge and discharge measurements were carried out using a LAND CT2001A battery testing system (Wuhan, China) with cut-off voltage of 0.01−3.0 V. Cyclic voltammetry (CV) measurements and Electrochemical impedance spectroscopy (EIS) measurements were carried out using a CHI660A electrochemical workstation.

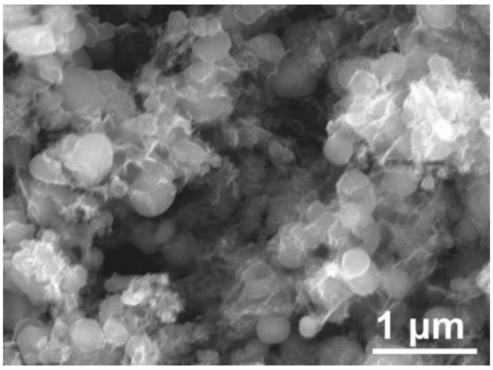

Figure S1. SEM image of the sample annealed at 700ºC for 1 h.

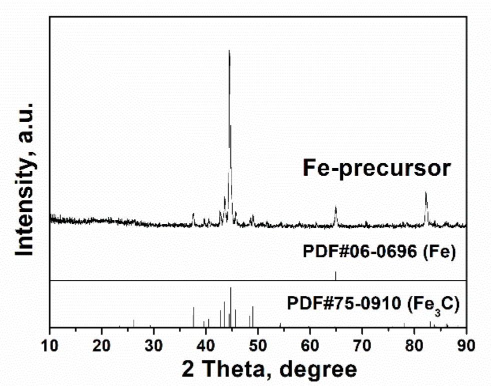

Figure S2. XRD pattern of the precursor.

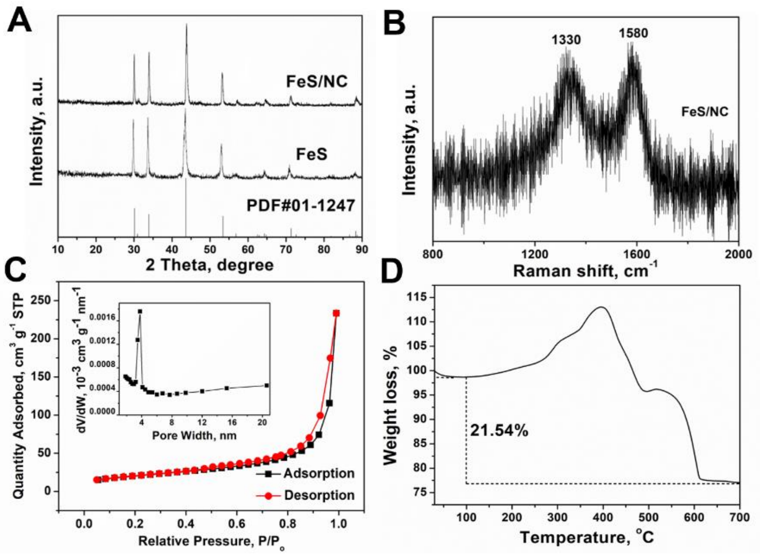

Figure S3. XRD pattern of the FeS/NC composite and pure FeS (A), Raman spectra of FeS/NC composite (B), Nitrogen adsorption-desorption isotherm and pore size distribution (insert) of the FeS/NC composite (C), and TG curve of the FeS/NC composite (D).

The weight decrease (1.35%) below 100°C was assigned to absorbed water. The weight increase in the temperature range from 100 to 400°C was attributed to the partial transformation of iron sulfides into $FeSO_4$. The next two steps of mass loss in the temperature range from 400 to 700°C, corresponding to weight loss of carbon and sulfur. The content of NC in FeS/NC composite is calculated based on the transformation from FeS to Fe_2O_3.

$$\mathrm{FeS/NC} \rightarrow \mathrm{Fe_2O_3} + \mathrm{gas}$$

Atomic weight: Fe (56), S (32), O (16)

$$x+\frac{[(56+32)-(56*2+16*2)/2]\ (1-x)}{(56+32)}=21.54\%$$

The total weight percentage of FeS/NC composite is defined as 100%, while the NC weight percentage is x. The weight loss percentage of transformation between FeS and Fe_2O_3 is

$$\frac{[(56+32)-(56*2+16*2)/2]\ (1-x)}{(56+32)}=\frac{1-x}{11}$$

Therefore, the NC content of x was calculated to be 13.69%.

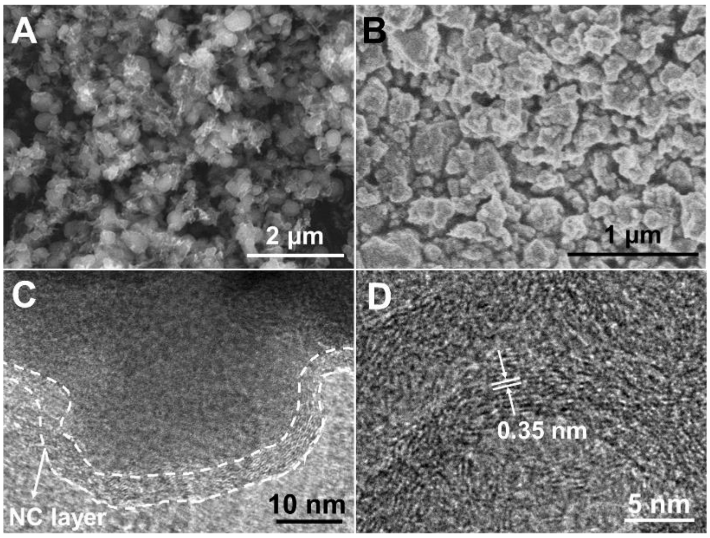

Figure S4. SEM images of the FeS/NC (A) and pure FeS (B), HRTEM images of the FeS/NC composite (C) and Nitrogen-doped carbon nanosheets (D).

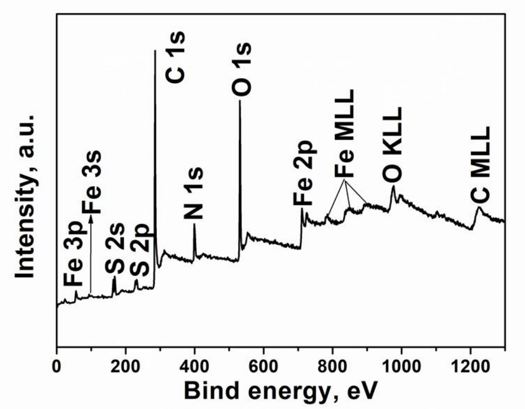

Figure S5. Survey XPS spectra of the FeS/NC composite.

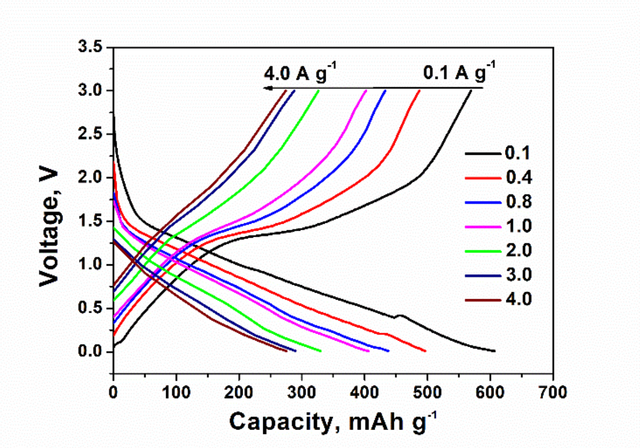

Figure S6. Charge and discharge profiles of the FeS/NC composite at various current densities.

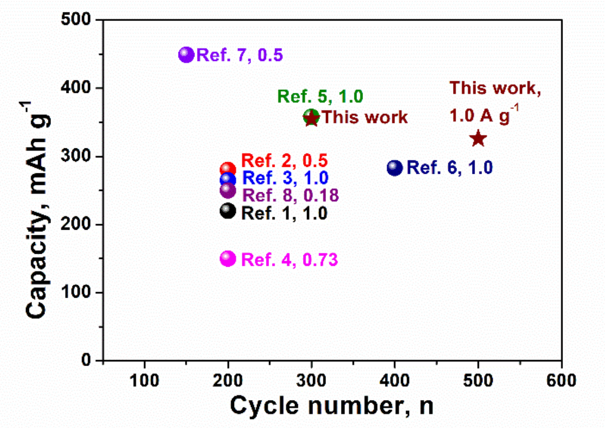

Figure S7. Comparison of specific capacity and cycle life between FeS/NC composite and some reported high-performance iron sulfide-based electrodes.[1, 2 3-8]

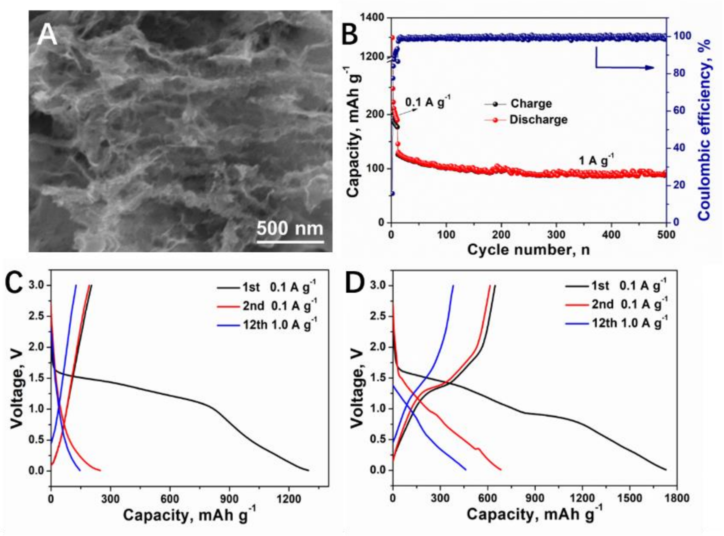

Figure S8. SEM image of pure NC (A), cycling performance of pure NC at 1.0 A g^{-1} (B), charge and discharge curves of the pure NC (C) and FeS/NC composite (D) at 0.1 and 1.0 A g^{-1}.

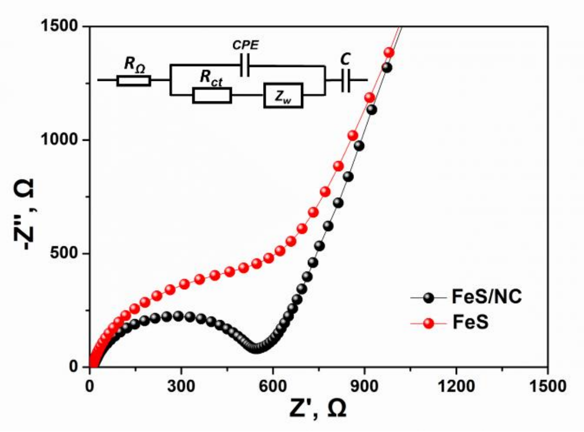

Figure S9. Electrochemical impedance spectroscopy for FeS/NC and pure FeS and equivalent circuit (insert).

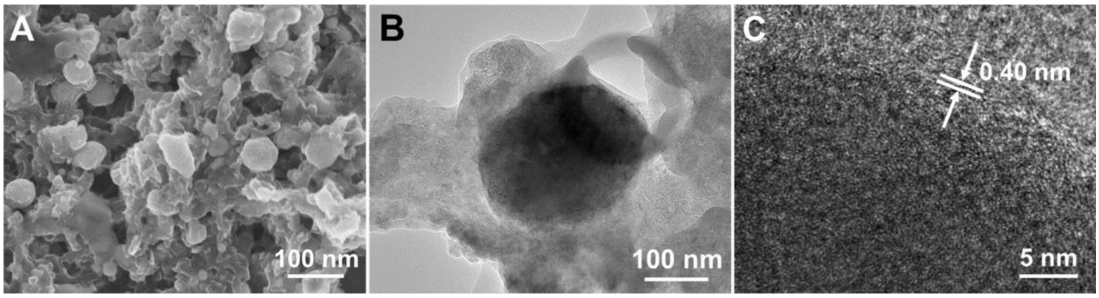

Figure S10. SEM image (A), TEM image (B), and HRTEM image (C) of FeS/NC composite after 30 cycles at 500 mA g^{-1}.

References

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