Carbon-Coated FeP Nanoparticles Anchored on Carbon Nanotube Networks as Anode for Long-Life Sodium-Ion Storage

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Experimental

Materials and Methods

Synthesis of CNT@Fe\textsubscript{3}O\textsubscript{4}-C. The nanoparticle was produced by a one-pot hydrothermal method and a subsequent carbonization process. In a typical synthesis, 50 mg of CNT, 1g of Fe(NO\textsubscript{3})\textsubscript{3}.9H\textsubscript{2}O and 1g of glucose were dissolved into 60 ml of 30\% ethanol/H\textsubscript{2}O solutions and sonicated for 1 h. Then the solution was transferred into a Teflon-lined stainless-steel autoclave and heated at 160 °C for 12h. After that, the resulting product were collected by centrifugation, washed with ethanol and water for three times, and dried at 80 °C. Finally, the black precipitate was annealed at 400°C for 2h under Ar atmosphere.

Synthesis of CNT@FeP-C. The CNT@FeP-C was fabricated by the phosphidation of CNT@Fe\textsubscript{3}O\textsubscript{4}-C. In a typical procedure, CNT@Fe\textsubscript{3}O\textsubscript{4}-C and sodium hypophosphite (NaH\textsubscript{2}PO\textsubscript{2}) were placed at two separate positions in one closed crucible. Subsequently,
the products were heated to 300 °C for 3h with a heating rate of 2 °C min⁻¹ in a flowing Ar atmosphere.

**Characterization**

The X-ray diffraction (XRD) patterns were obtained by Rigaku D/max-2200/PC with filtered Cu Kα radiation. Raman spectra were recorded in a plus laser Raman spectrometer (Bruker Optics Senterra R200-L) with an excitation laser beam wavelength of 514.5 nm. The morphologies of the as-prepared materials were analyzed by a scanning electron microscopy (SEM, Sirion 200, FEI) and transmission electron microscopy (TEM, JEM-2100F, JEOL). XPS characterization was performed using a SAGE Spectrometer (Specs, Berlin, Germany) using non-monochromatized MgKa with 100 W radiation. Electronic conductivity measurement was carried out at room temperature using the four-point technique (DP-SB100A/20, Beijing Ya’ou De Peng technology co. Ltd., China). Sample pellet was made by cold pressing powder under 15 MPa.

**Electrochemical Measurements**

The 2016-type coin cells were fabricated in a argon-filled glove box. The working electrode was prepared by mixing 80% active material, 10% super P and 10% CMC, and then casted onto a Cu foil and dried at 90 °C for 12 h. The testing cell was made of the working electrode, sodium metal as the counter and reference electrode, a glass fiber separator and the electrolyte using 1M NaClO₄ in the mixture of EC/PC (1:1). Galvanostatic charge-discharge tests were carried out on a Land Battery Measurement System at various currents with a potential
window of 0.01-3 V. CV tests were performed at a scan rate from 0.1 to 5 mV/s using CHI605D electrochemical workstation. Electrochemical impedance spectroscopy was recorded by AUTOLAB.

Supplementary Figures

Figure S1. (a) XRD pattern and (b) Raman spectrum of the CNT@FeP-C.

Figure S2. TGA profiles of FeP and CNT@FeP-C.
Figure S3. The SEM image of pure CNT.

Figure S4. Nitrogen adsorption/desorption isotherms of CNT@FeP-C.
Figure S5. Cycling performance of CNT and FeP at the current rate of 1A g⁻¹.
Figure S6. The real axis $Z'$ with the reciprocal square root of the lower angular frequencies $\omega^{-1/2}$. 