Robust 3D Nanowebs Assembled from Interconnected and Sandwich-like

C@Fe₃O₄@C Coaxial Nanocables for Enhanced Li-ion Storage

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Supplementary experimental details

Materials characterization: Scanning electron microscope (SEM) images were obtained using Hitachi S-8010 equipment operated at 15 kV. Transmission electron microscope (TEM) images were obtained using Tecnai G2 F20 S-TWIN. N₂ adsorption/desorption isotherms were measured using Micromeritics TriStar II apparatus with liquid nitrogen at 77 K. X-ray diffraction (XRD) patterns were collected using PANalytical X'Pert PRO MRD diffractometer with Ni-filtered Cu *Ka* radiation. Thermogravimetric (TG) analysis was carried out using a SEIKO TG/DTA 7300 thermal analyzer. Raman spectra were recorded with JobinYvon LabRAM HR800 Raman spectrometer with an excitation wavelength of 532 nm.

Electrochemical testing: The working electrode slurry was prepared by dispersing the active material (NCW@Fe₃O₄@NCS, NCW, bulk Fe₃O₄, or NCW@Fe₃O₄), Super P-Li, and poly(vinylidene fluoride) (PVDF) binder in N-methylpyrrolidone with a weight ratio of 7:2:1. The slurry was spread onto copper foil disks and dried in a vacuum oven at 120 °C overnight. Lithium foil as the counter electrode, and 1.0 M LiPF₆ in ethyl carbonate/dimethyl carbonate (1:1 v/v ratio) as the electrolyte, and Celgard 2500 as the separator were used to assemble a CR2032 coin cell.

Galvanostatic discharge and charge tests were performed with a cycle tester from LAND Electronic Co., and the cut-off potential window was set between 3.0 V and 0.01 V.



Fig. S1 SEM images of PPy at low magnification.



Fig. S2 SEM image of PPy prepared with a higher concentration of starting materials. The detailed concentrations are as follows. 7.3 g of cetrimonium bromide (CTAB) was dissolved in 120 mL of HCl solution (1 mol L⁻¹). 13.7 g of ammonium persulfate was then added into the above solution. At last, 8.3 mL of pyrrole monomer was dropwise added. The diameter of the PPy nanofibers increases to 80-100 nm, much larger than that obtained at low concentration of starting materials.



Fig. S3 TG curves of PPy precursor and CTAB under Ar flow. Obviously, the weight loss of PPy precursor between 200 and 300 °C can be attributed to the decomposition of CTAB-based soft template.



Fig. S4 N_2 adsorption/desorption isotherms of NCW.



Fig. S5 SEM image of two layers of polyelectrolytes-covered NCW.



Fig. S6 High-resolution TEM image of two layers of polyelectrolytes-covered NCW.



Fig. S7 SEM image of NCW/FeOOH prepared in the absence of two-layered polyelectrolytes.



Fig. S8 SEM images of 1D (a) carbon nanotubes (CNTs), (b) CNTs@Fe₃O₄@NCS, (c) electrospun carbon nanofibers (CNFs), and (d) CNFs@Fe₃O₄@NCS. CNTs@Fe₃O₄@NCS and CNFs@Fe₃O₄@NCS were prepared through the similar polyelectrolyte-assisted approach except that CNTs and CNFs were used as the carbon substrates, respectively. The uniform distribution of Fe₃O₄ on carbon substrates is clearly observed, demonstrating the wide applicability of this polyelectrolyte-assisted strategy.



Fig. S9 TG curve of NCW@Fe₃O₄@NCS under air flow.

Considering the weight loss of carbon and the oxidation reaction of Fe_3O_4 to Fe_2O_3 , the content of Fe_3O_4 in NCW@Fe_3O_4@NCS hybrid is calculated according to the following equation.

(1-X)×100%-X×3.4%=30%

where X is the weight percentage of Fe₃O₄.

X is calculated to be 68%.



Fig. S10 SEM images of bulk Fe₃O₄.



Fig. S11 SEM and TEM images of core-shell NCW@Fe₃O₄.



Fig. S12 The initial three discharge/charge curves of bulk Fe₃O₄.



Fig. S13 The cycling performance of bulk Fe₃O₄.



Fig. S14 The initial three discharge/charge curves of core-shell NCW@Fe₃O₄.



Fig. S15 N₂ adsorption/desorption isothermal curves of NCW@Fe₃O₄@NCS.



Fig. S16 SEM images of bulk Fe₃O₄ electrode before cycling.



Fig. S17 SEM images of bulk Fe₃O₄ electrode after 100 cycling.