

Supplementary Information

Facile Synthesis of Carbon-decorated Single-crystalline Fe₃O₄ Nanowires and their Application as High Performance Anode in Lithium Batteries

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Experimental Section

Synthesis of Fe₃O₄ nanowires: In a typical synthesis, 8 mmol of FeSO₄·7H₂O (Fisher Scientific) and 4 mmol of Na₂S₂O₃·5H₂O (Fisher Scientific) were dissolved in 10 mL of de-ionized water, followed by the addition of 5 mL of polyethylene glycol (PEG-400, Alfa Aesar) under magnetic stirring. The mixture was stirred for few minutes, followed by the addition of 4 g of NaOH dissolved in 5 mL of de-ionized water. On adding NaOH, the Fe²⁺ ions form Fe(OH)₂, which oxidize to Fe(OH)₃ by reaction with the dissolved oxygen in the solvent. However, the weak reducing agent Na₂S₂O₃ present in the reaction mixture limits the oxidation of Fe(OH)₂ to a Fe³⁺/Fe²⁺ ratio of 2:1, resulting in the formation of phase pure Fe₃O₄ instead of the thermodynamically favorable hematite (Fe₂O₃) phase during the MW-HT process. The reaction precursors were then transferred and sealed in quartz vessels, which were fitted with a pressure and temperature probe. The rotor containing the closed quartz vessels was then placed on a turntable for uniform heating in an Anton Paar microwave synthesis system (Synthos-3000). The desired exposure time and temperature were programmed with the Synthos-3000 software. The automatic temperature and pressure control system allowed continuous monitoring and control of the internal temperature (± 1 °C) and pressure (up to 80 bar). The system was operated at a frequency of 2.45 GHz and a power of 600 W, the sample temperature was ramped to 150 °C, and kept at 150 °C for 15 minutes under the hydrothermal condition. After the MW-HT process, the reactor was cooled to room temperature by an in-built cooling fan capability in the Synthos-

3000 system. The black Fe₃O₄ precipitate formed after the reaction was repeatedly washed with ethanol and de-ionized water and dried at 70 °C under vacuum.

Synthesis of carbon-decorated Fe₃O₄ nanowires: The Fe₃O₄ nanowires (0.5 g) as synthesized above were dispersed in 20 mL of de-ionized water and sonicated for few minutes, followed by the addition of 0.5 g of D-glucose (Fisher Scientific). The reaction mixtures were then transferred to a quartz vessel and subjected to microwave-hydrothermal treatment at 180 °C for 15 minutes. The resulting material was isolated by centrifugation and further carbonized under Ar flow at 400 °C for 3 h.

Structural Characterization: TEM and SEM analysis were carried out, respectively, with a JOEL JEM-2010F TEM and Hitachi S-5500 Ultrahigh Resolution FE SEM. XRD patterns were recorded with a Philips PW-1830 X-ray diffractometer using filtered Cu K_α radiation. Raman spectroscopic analysis was performed with a Renishaw In-Via System utilizing a 514.5 nm incident radiation and a 50X aperture (N.A = 0.75), resulting in an ~2 μm diameter sampling cross section.

Electrochemical Characterization: Electrochemical performances were evaluated with CR2032 coin cells with an Arbin battery cycler. The test electrodes were prepared by mixing 70 wt. % active material with 15 wt. % conductive carbon (super P) as a conductive agent and 15 wt. % polyvinylidene fluoride (PVDF) dissolved in N-methyl-2-pyrrolidone (NMP) as a binder to form a slurry, which was then coated onto a copper foil and pressed and dried under vacuum at 100 °C for at least 4 h. The coin cells were finally assembled in an argon filled glove box with the Fe₃O₄ or carbon-decorated Fe₃O₄ test electrode, metallic lithium as the counter and reference electrode, 1 M LiPF₆ in 1:1 diethyl carbonate/ethylene carbonate electrolyte, and Celgard

polypropylene separator. Charge-discharge measurements were carried out galvanostatically at various C-rates over a voltage range of 0.01 to 3 V (vs Li/Li⁺).