Electronic Supplementary Information

Self-organized sheaf-like Fe_3O_4/C hierarchical microrods with superior lithium storage properties

Fei-Xiang Ma,[ab] *Hao Bin Wu,*[a] *Cheng-Yan Xu,*[b] *Liang Zhen,*[b] *and Xiong Wen (David) Lou*[*a]

[a] School of Chemical and Biomedical Engineering, Nanyang Technological University, 62 Nanyang Drive, Singapore, 637459. Email: xwlou@ntu.edu.sg

[b] School of Materials Science and Engineering, Harbin Institute of Technology, Harbin 150001, China.

Experimental

Materials Synthesis: All chemicals were purchased from Sigma-Aldrich and used without any purification. In a typical synthesis of sheaf-like Fe_3O_4/C hierarchical microrods with nanowires as building blocks, 0.303 g of $Fe(NO_3)_3$ $6H_2O$ and 0.2 g of glucose were completely dissolved into 30 ml of ethylene glycol (EG). Then the mixture was transferred into a 40 ml Teflon-lined stainless steel autoclave and heated at 220 ^oC for 12 h in an electric oven, followed by cooling naturally to room temperature. The resultant Fe-glycolate precursor was washed with ethanol for several times through centrifugation and dried in an oven at 70 ^oC overnight. Finally, the as-prepared precursor was calcined at 350 ^oC for 3 h with a heating rate of 1 ^oC min^{-1} in N_2 gas flow to obtain Fe_3O_4/C microrods.

Materials Characterization: X-ray diffraction (XRD) patterns were collected using a Bruker D8 Advanced X-Ray Diffractometer. The morphology and structure of the products were characterized by field-emission scanning electron microscope (FESEM, JEOL JSM-6700F), and transmission electron microscope (TEM, JEOL JEM-2010) with an accelerating voltage of 200 kV. Thermogravimetric analysis (TGA) was performed on SDT Q600 (TA Instruments). Nitrogen adsorption-desorption was performed on Autosorb 6B at -196 ℃. Raman spectrum was taken under ambient conditions by using a micro-Raman spectrometer (Reinshaw Raman Scope RM3000).

Electrochemical Measurements: Electrochemical measurements were carried out using two-electrode Swagelok-type cells with pure lithium foil as the counter and reference electrode at room temperature. The working electrode was prepared by pasting a mixture of active materials, super P and sodium carboxymethyl cellulose (CMC) with a weight ratio of 70:20:10 onto a copper plate. The electrodes were dried in vacuum at 70 ℃ for 12 h before assembling battery in an Ar-filled glovebox. The typical loading density of the active material was approximately 1.0 mg cm^{-2}. The capacity of the electrode was calculated based on the total weight of Fe_3O_4/C microrods. The electrolyte was composed of 1 M $LiPF_6$ in a 50:50 w/w mixture of ethylene carbonate (EC)/dimethyl carbonate (DMC) mixture solution. Cyclic voltammetry (CV) measurement was carried out on a CHI660E electrochemical workstation. Charge-discharge measurement was performed on a Neware battery tester.

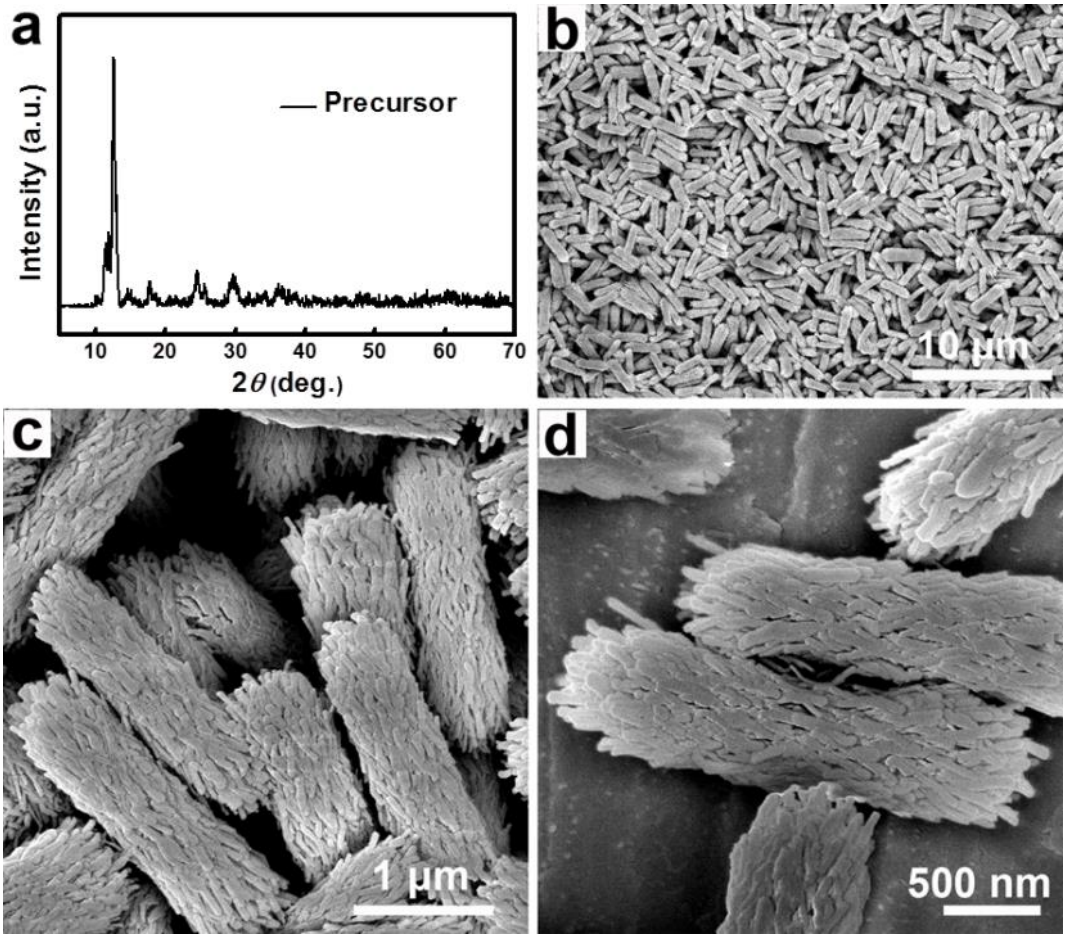

Figure S1. (a) XRD pattern and (b-d) FESEM images of the as-prepared Fe-glycolate precursor microrods.

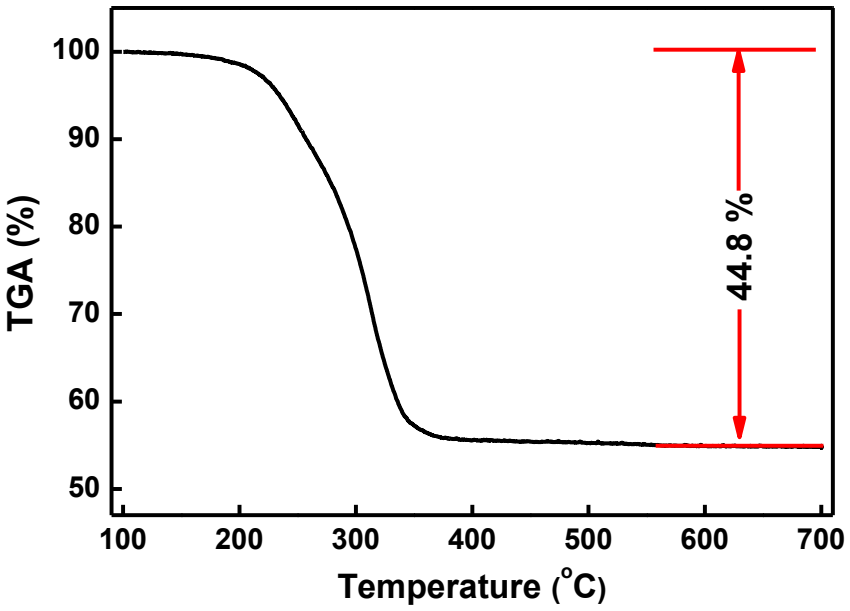

Figure S2. TGA curve of the as-prepared Fe-glycolate precursor from 100 to 700 ^{o}C under N_2 gas flow with a temperature ramp of 10 ºC min^{-1}.

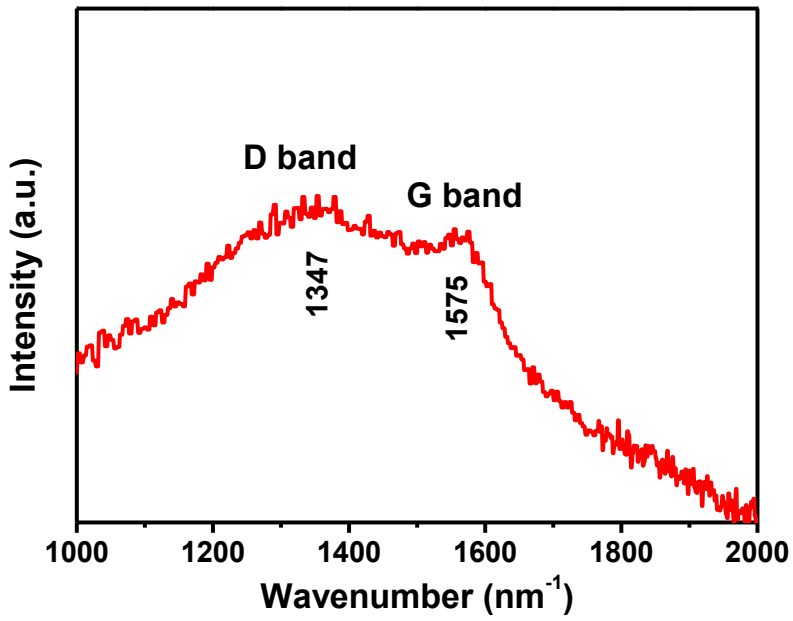

Figure S3. Raman spectrum of the as-prepared Fe_3O_4/C microrods. The high intensity of D band relative to G band indicates the amorphous nature of the carbon material.

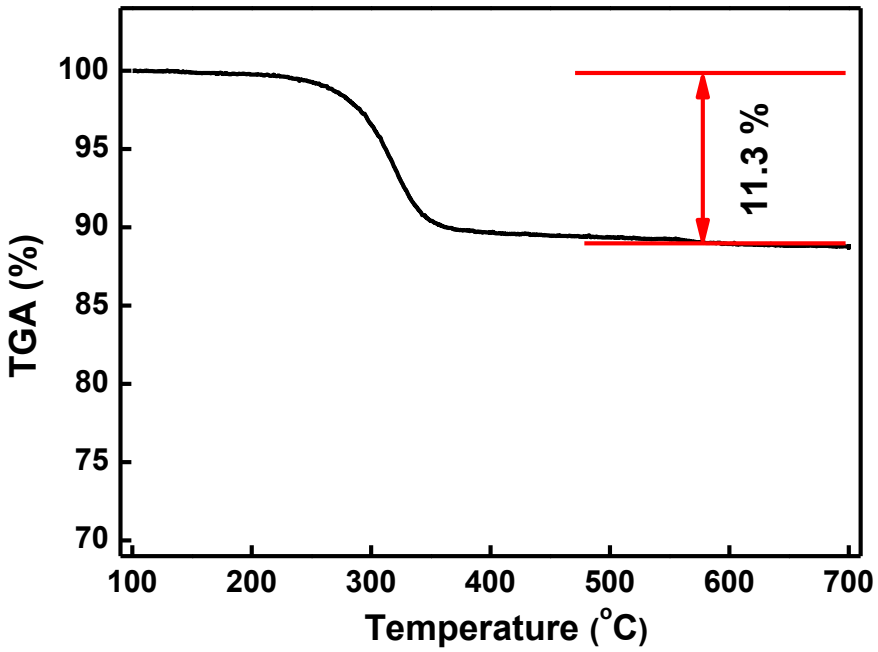

Figure S4. TGA curve of the as-prepared Fe_3O_4/C microrods from 100 to 700 ^oC under air gas flow with a temperature ramp of 10 ^oC min^{-1}.

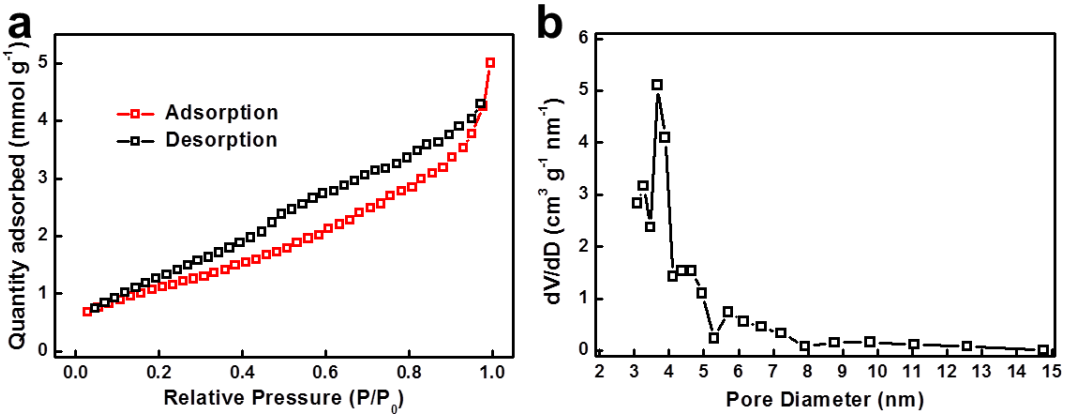

Figure S5. (a) N_2 adsorption-desorption isotherm plots and (b) corresponding BJH pore size distribution of the Fe_3O_4/C microrods.

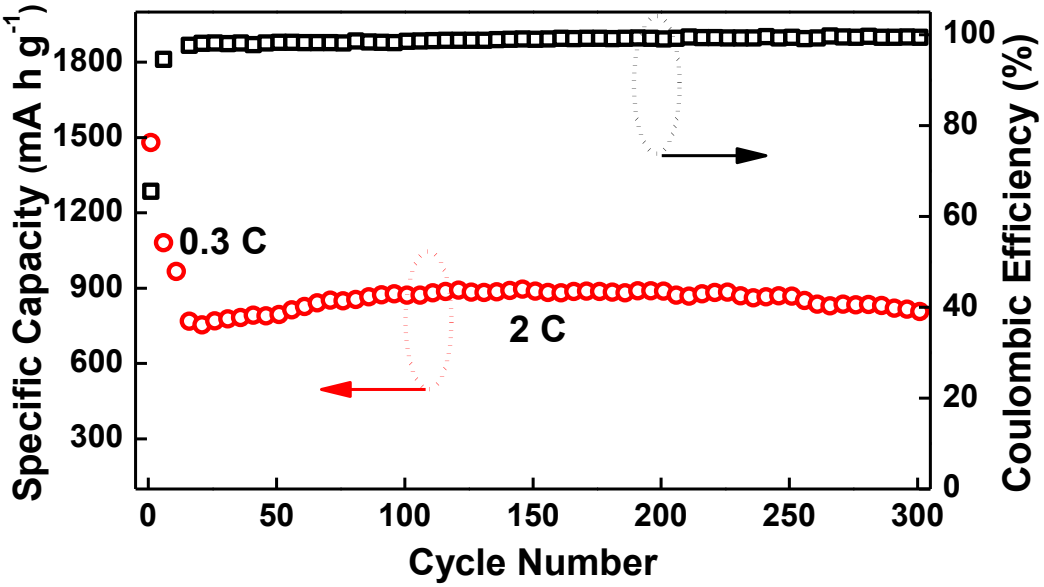

Figure S6. Cycling performance of the Fe_3O_4/C microrods electrode at a current density of 2 C (1 C=1000 mA g^{-1}). The electrode was firstly activated at 0.3 C for the initial 10 cycles.