

Solvothermal Synthesis of Lithium Iron Phosphate Nanoplates

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Supporting Information

S1. Experimental Details

Synthesis of LiFePO_4 Nanoplates: In order to insure the reactants to be well dissolved, all the ethylene glycol used in the experiment was pre-heated to 50 °C. In a typical synthesis of LiFePO_4 Nanoplates, 6 mmol $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ and 6 mmol H_3PO_4 were dissolved in 20 ml ethylene glycol at first. At the same time, 16.2 mmol $\text{LiOH} \cdot \text{H}_2\text{O}$ was dissolved in 15 ml ethylene glycol to form a clear solution (the molar ratio of $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$, H_3PO_4 and $\text{LiOH} \cdot \text{H}_2\text{O}$ was 1:1:2.7). Then, the LiOH -ethylene glycol solution was slowly added into the FeSO_4 - H_3PO_4 -ethylene glycol solution with constantly stirring for five minutes. At last the mixture was performed into a 40 ml autoclave and heated at 180 °C for 10 h. The LiFePO_4 nanoplates were formed in the bottom. The precipitate was washed by water and ethanol before dried at 60 °C for 6 h.

Synthesis of LiFePO_4/C Nanoplates: To prepare LiFePO_4/C nanoplates, the as-obtained LiFePO_4 nanoplates were mixed with 11 wt. % sucrose as carbon source (ratio of LiFePO_4 : C is about 1:0.05, w/w). The mixture was first calinated in Ar atmosphere at 200 °C for 0.5 h, and then heated to 550 °C for 2.5 h. The heating rate is 3 °C min⁻¹.

Electrochemical measurements: A composite electrode used for electrochemical measurements was prepared by mixing active material, acetylene black and poly (vinylidene fluoride) (PVDF) with weight ratio 75:15:10. Lithium metal was used as the counter electrode. The electrolyte was a 1 M LiPF_6 in a 1:1 mixture of ethylene carbonate (EC)/diethyl carbonate (DEC) and the separator was Celgard 2500. The CR2016 cell was assembled in the glove box filled with highly pure argon gas. The charge/discharge experiment was performed between 2.0 and 4.3 V at different current density.

S2. Characterization of LiFePO₄/C nanoplates

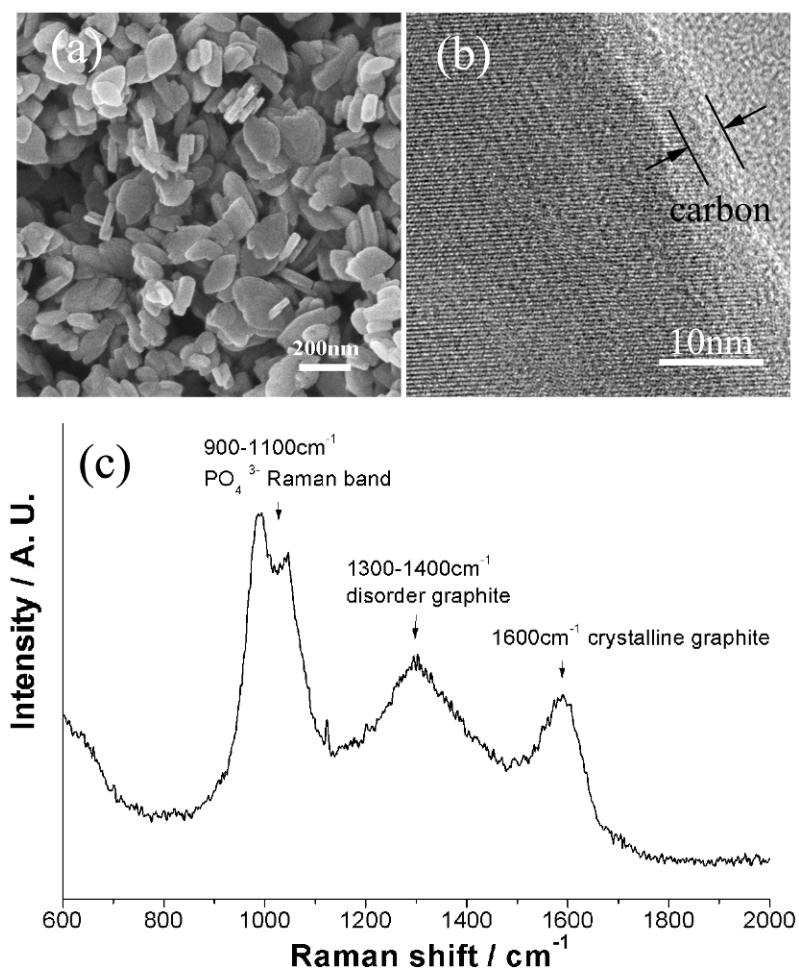


Fig. S2. (a) SEM image of LiFePO₄/C nanoplates; (b) HRTEM image of LiFePO₄/C nanoplates;
(c) Raman spectra of LiFePO₄/C nanoplates

Fig. S2a shows the SEM image of LiFePO₄/C nanoplates. It can be found that the LiFePO₄/C nanoplates maintain the size and crystal orientation of LiFePO₄ nanoplates after calcined with sucrose at 550 °C for 2.5 h. Fig. S2b shows that there is a poorly crystallized thin layer on the surface of LiFePO₄/C nanoplates, labeled by black arrow. This layer may be the carbon layer, which generated by carbonization of the sucrose. In order to confirm the carbon coating on LiFePO₄, Raman spectrum of the LiFePO₄/C nanoplates is shown in fig. S2c. The most intense peak at about 1000 cm⁻¹ corresponds to the PO₄ stretching modes of LiFePO₄. The bands at 1350 and 1580 cm⁻¹ are assigned to carbon. These results proved the existence of carbon coating on LiFePO₄.

S3. First charge-discharge curve of micron-sized LiFePO_4 and no plate-like LiFePO_4

In order to show the special charging and discharging plateau of LiFePO_4 nanoplates, we synthesized a micron-sized LiFePO_4 and a no plate-like LiFePO_4 for comparisons.

Synthesis of micron-sized LiFePO_4 : The micron-sized LiFePO_4 plates were synthesized by hydrothermal method, using 5 mmol $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$, 5 mmol H_3PO_4 , 13.5 mmol $\text{LiOH} \cdot \text{H}_2\text{O}$ and 0.15 g ascorbic acid as reactants ($\text{Li:Fe:PO}_4 = 2.7:1:1$, $\text{PH}=8$). The reactants was dissolved in 35 ml water and transferred to 40 ml Teflon vessel to react at 180°C for 10 h. The plate is about $\sim 10\ \mu\text{m}$ in length, $\sim 5\ \mu\text{m}$ in width and $\sim 30\ \mu\text{m}$ in thick.

Synthesis of no plate-like LiFePO_4 : The preparation of no plate-like (fusiform-like) LiFePO_4 sample followed the same procedures of LiFePO_4 nanoplates except that the reactant ratio of $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$, H_3PO_4 , $\text{LiOH} \cdot \text{H}_2\text{O}$ was 1:1:3.

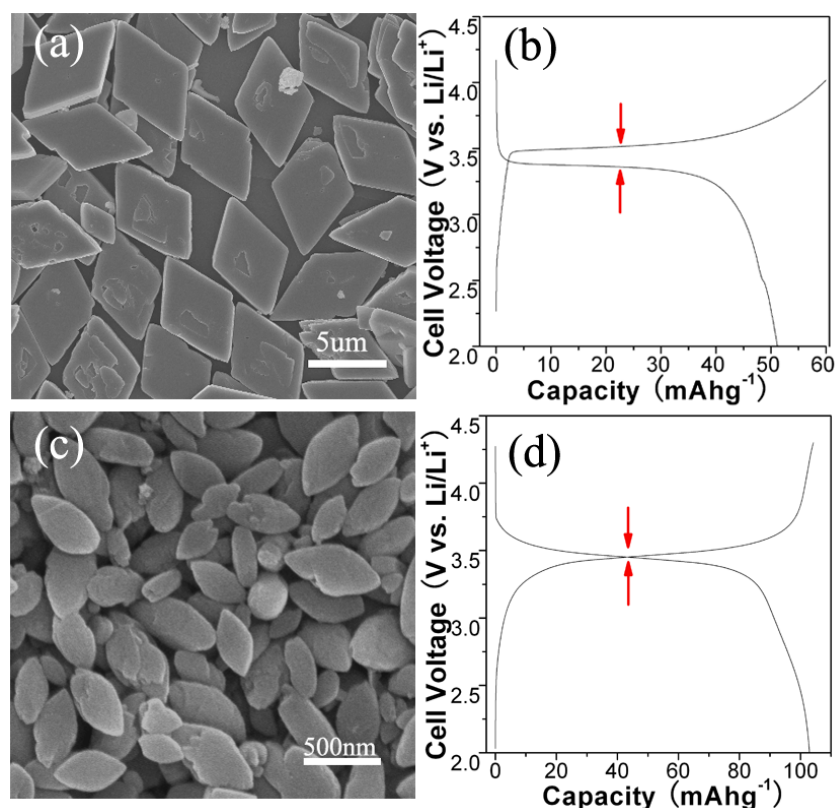


Fig. S3. a) SEM image of micron-sized LiFePO_4 . b) First charge-discharge curve of micron-sized LiFePO_4 at 0.1C. c) SEM image of no plate-like (fusiform-like) LiFePO_4 . d) First charge-discharge curve of no plate-like (fusiform-like) LiFePO_4 at 0.1C