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Supplementary Information

Novel and Scalable solid-state synthesis of nanocrystalline FeF_3/C composite and its excellent electrochemical performance

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Experimental Section

Material preparation

Nanocrystalline FeF_3/C composite was synthesized by solid-state reaction. Poly-tetrafluoroethylene (PTFE, Sigma-Aldrich) and $Fe(C_2O_4)\cdot 2H_2O$ (iron oxalate, Alfa) were mixed at 1:1, 1.5:1, 2:1, and 2.5:1 w:w, then ball-milled overnight. (Mixtures will be identified using these ratios.) The mixtures were dried on a hot plate, then ground and molded into pellets. Various pellets were fired at different temperatures *T* (at ramping rate of 200 °C/h) for 1 h in Ar atmosphere in a covered alumina crucible. The 2:1 sample synthesized at 600 °C was used for electrochemical tests.

Material characterizations

X-ray diffraction (XRD) patterns of the samples were obtained using an Rigaku using Cu Kα radiation in the 2θ range from 20° to 70° and a rate of 3°/min. The obtained XRD patterns were analyzed using MDI jade 6 software program and refined using X'pert Highscore plus. The mix of precursors was subjected to thermal gravimetric analysis (TGA). The amount of carbon was measured using a Vario EL III (Analysensysteme, GMBH). Morphologies were observed using a transmission electron microscope (TEM, JEM-2100F, JEOL).

Electrochemical properties

Electrodes were fabricated by manually mixing the resulting material with PTFE binder in a

ratio of 95:5 (w:w). Electrodes were punched with diameter of 8 mm. Electrochemical properties were measured using Swagelok-type cell. Cells were assembled with the composite electrode, separator (Celgard 2400), electrolyte ($LiPF_6$ in EC/DMC, 1:1), and Li metal as a negative electrode. All cell assembly processes were performed in an Ar-filled glove box. All electrochemical tests were performed at room temperature.

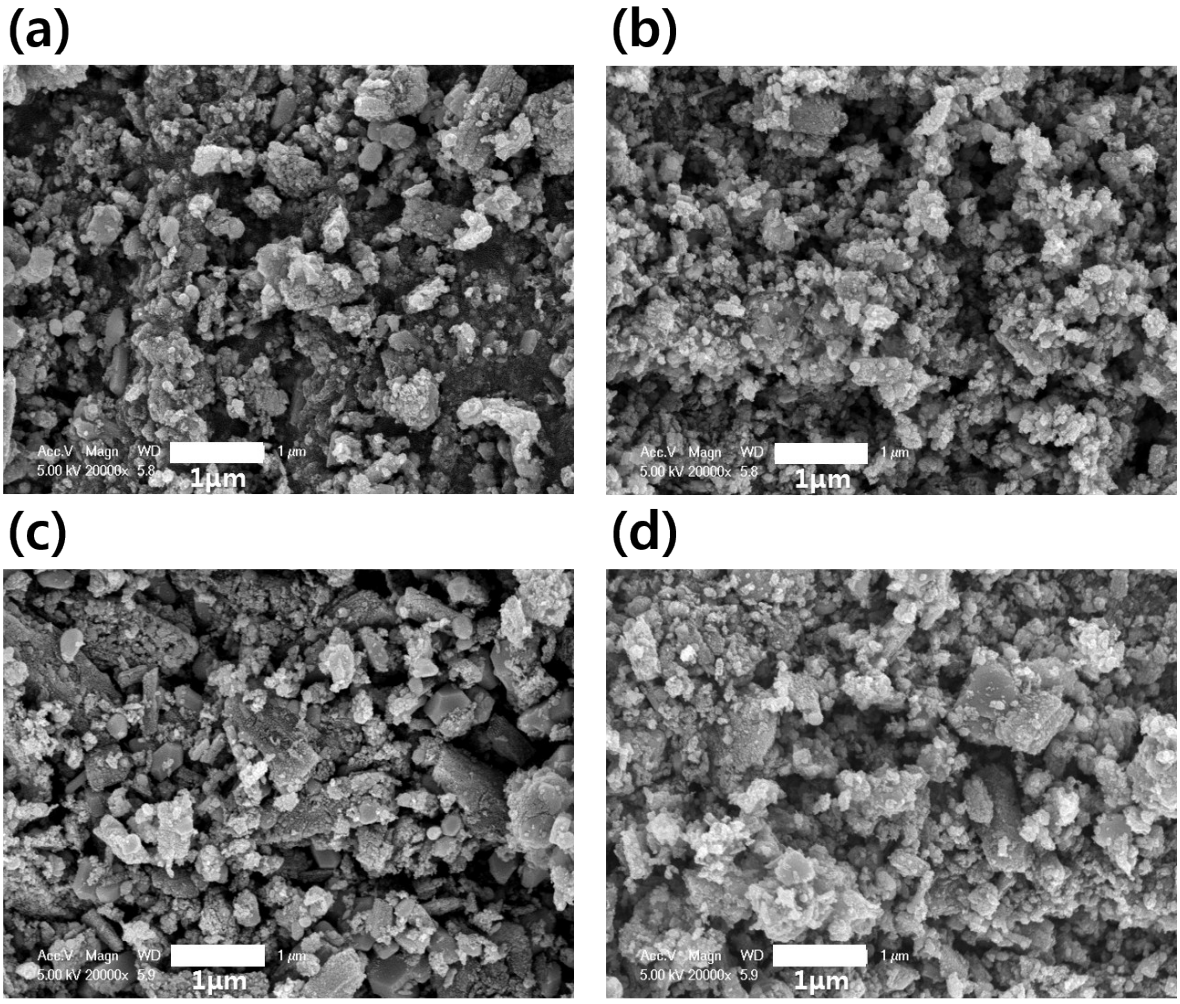

Figure S1 **SEM images of the samples obtained by the different PTFE ratios: 1:1 (a), 1.5:1 (b), 2:1 (c), 2.5:1 (d)**

The SEM images of the samples obtained by the different PTFE ratios were shown in Figure S1. As all samples were synthesized at 600°C for 1h under argon with considerable amount of PTFE and Fe source, the morphologies of all samples were similar having wide range of particle size.

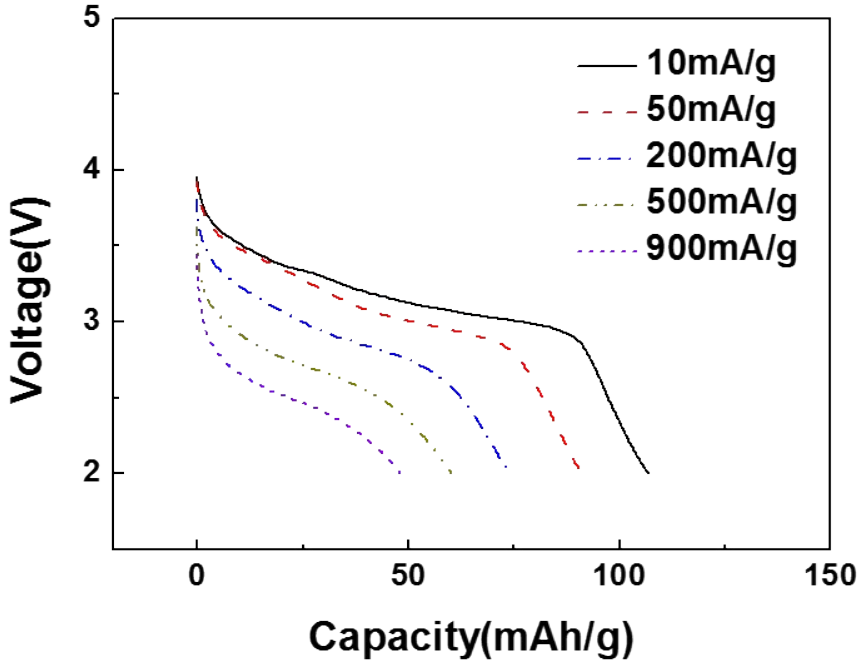

Figure S2 Rate capability of the FeF_3/C composite synthesized at 700 °C, electrode composition is 80(material): 15(Super-P): 5 (PTFE binder)

Even though the electrode was composed with 15% of Super-P for conductive agent, the sample synthesized at 700 °C shows poor electrochemical activity. The electrode shows 105mAh/g of discharge capacity at a current rate of 10mA/g and about 50mA/g of discharge capacity at a current rate of 500mA/g.

1. F. Badway, F. Cosandey, N. Pereira and G. G. Amatucci, *Journal of The Electrochemical Society*, 2003, **150**, A1318.