Supplementary information

$(NH_4)_3FeF_6$ Mesocrystal Grown by Electric Field-Assisted in-situ Dissolution and Reaction of Anodic Iron Oxides

1. Experimental section

1.1. Preparation of ammonium iron fluoride mesocrystals

Fe metal foil (purity 99.9%) was cut into 1.5 cm x 10 cm pieces, and the Fe pieces were successively sonicated in anhydrous ethanol (98%) and deionized (DI) water for 20 min, respectively, followed by rinsing with DI water and then drying naturally. A glass beaker is used for the electrochemical reaction, and two pretreated Fe foils were used as anode and cathode. The solution used was ethylene glycol (98%) with addition of proper amount of water and ammonium fluoride (98%). Electrochemical reactions were carried out in gavanostatic mode at high current density for different times. Then the solution was centrifuged at 10000 r min^{-1} for 5 min and the obtained product denoted as $(NH_4)_3FeF_6$ was obtained which was then washed with anhydrous ethanol and dried in air at 60 °C in an oven.

1.2. Transformation to α-Fe_2O_3

The obtained ammonium iron fluoride mesocrystals were converted to α-Fe_2O_3 with nano-micro hierarchical and porous structure by annealing in air at 700 °C for 2 h in a muffle furnace.

1.3. Characterization

Field emission scanning electron microscope (FESEM, Merlin) and transmission

electron microscope (TEM, FEI Talos F200) were employed to characterize the morphology and microstructure of samples. The crystal phase of samples were analyzed by X-ray diffraction (XRD) measurements carried out on a BRUKER AXS D8 ADVANCE diffractometer with values of 2θ ranging from 5 ° to 90 ° by using Cu Ka radiation (1.5406 Å) operated at 40 kV and 40 mA with a step size of 0.013 °.

2. Supplementary figures

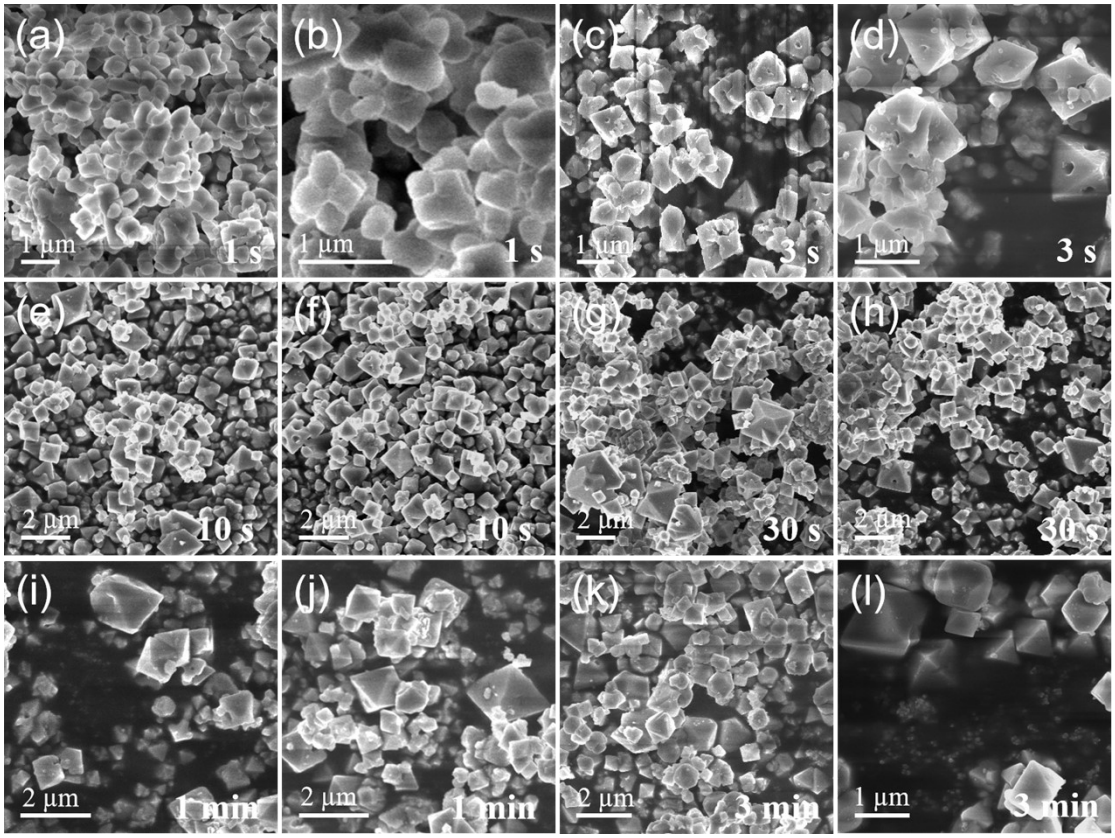

Figure S1 FESEM images of $(NH_4)_3FeF_6$ grown at different reaction times: (a,b) 1 s, (c,d) 3 s, (e,f) 10 s, (g,h) 30 s, (i,j) 3 min, and (k,l) 10 min.

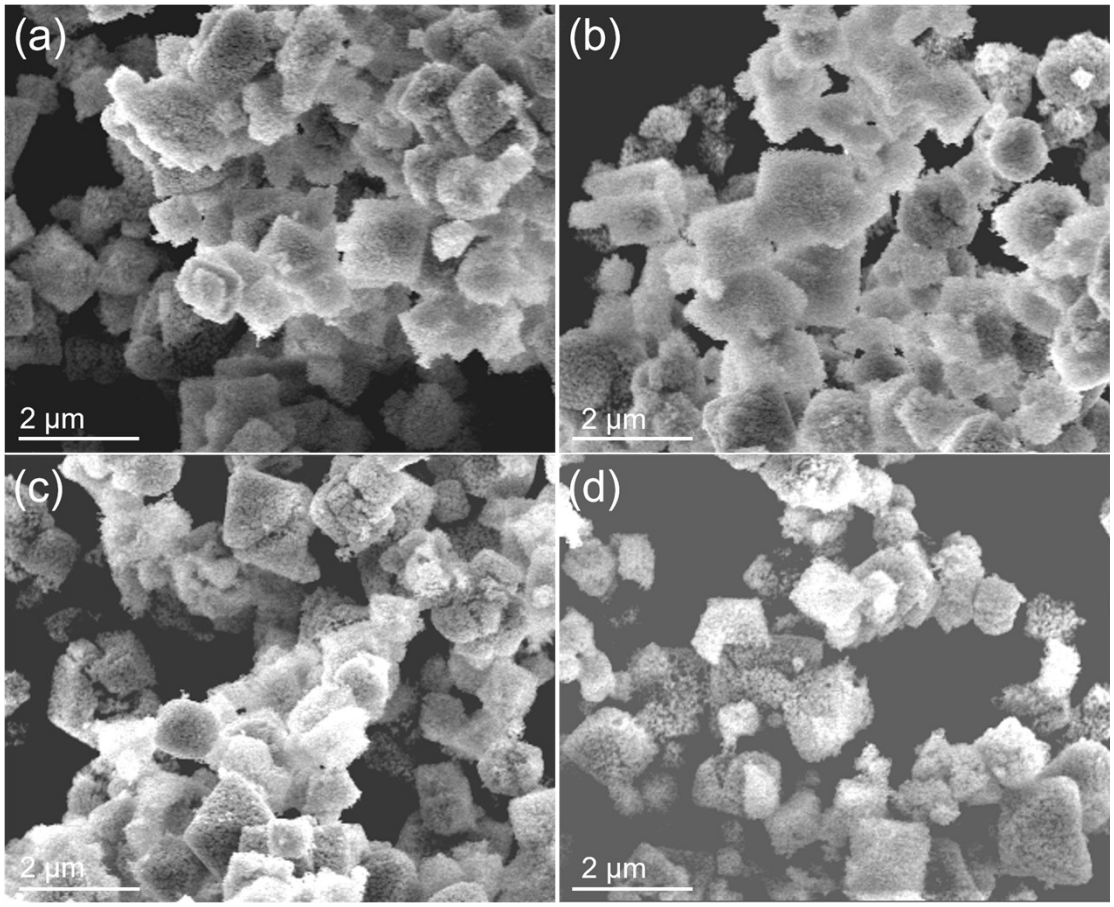

Figure S2 FESEM images of nano-micro hierarchical and porous α-Fe_2O_3 obtained from thermal annealing of as-prepared $(NH_4)_3FeF_6$ at 700 °C for 2 h.

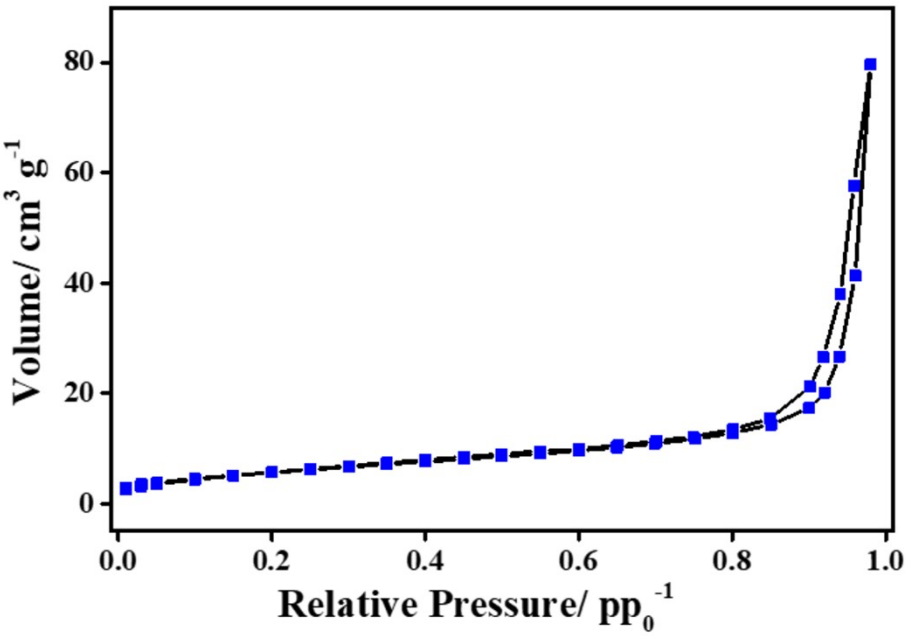

Figure S3 N_2 adsorption/desorption isotherms of α-Fe_2O_3.