

Supplementary Information

Magnetization of Amorphous FeOOH Chrysanthemum-like Nanosheets Under Ambient Conditions

Yan Si,^a Siyu Shi,^b Jingyun Jing,^c Yun Bai,^d and Qian Wang^{*a}

^a Institute of Chemistry, Chinese Academy of Sciences, Beijing 100190, China

^b Aerospace Science & Industry Defense Technology Research and Test Center, Beijing 100854, China

^c Beijing Key Laboratory of Metro Fire and Passenger Transportation Safety, China Academy of Safety Science and Technology, Beijing 100012, China

^d Institute of Analysis and Testing Beijing Academy of Science and Technology (Beijing Center for Physical and Chemical Analysis), Beijing 100094, China

Experimental section

Materials

Cumyl hydroperoxide (CHPO) was purchased from ACROS. Iron sulfate heptahydrate ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$) was purchased from China Pharmaceutical Group Co. Chloroauric acid (HAuCl_4) was purchased from Bellingway Technology. Sodium borohydride (NaBH_4) was purchased from Guanghua Chemical Factory and n-decane was purchased from Xilong Scientific Co., Ltd. All chemicals were analytical grade and used as received.

Preparation of the iron oxide nanosheet with Chrysanthemum-like structure

10.0 mL of n-decane dissolved 10.0 μL of CHPO was mixed with 50.0 mL of water and stirred at 300 rpm to form a dynamic soap-free emulsion under continuous ultrasound. The frequency of the ultrasound was fixed at 40 KHz and the power was 150 W. 183.0 mg of $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ dissolved in 5.0 mL of water was added to the emulsion slowly by a peristaltic pump in 1 hour. The chrysanthemum-like nanosheet was obtained after the dispersion was centrifuged and washed with ethanol.

Preparation of the magnetized nanosheets

20 mg of prepared nanosheets were dispersed in 50 mL water before 10 μ L of 1 g/L of HAuCl_4 solution was added. The solution was stirred at 200 rpm for 1 hour to absorb Au^{3+} onto the nanosheets. After the removal of excess HAuCl_4 by centrifugation, the nanosheets were re-dispersed in 50 mL of water and followed by the addition of a 15 mg NaBH_4 dissolved in 1 mL of water. The solution changed to black from yellowish as soon as the NaBH_4 solution was added. The magnetic nanosheet was obtained after being centrifuged and then washed with ethanol and water.

Characterization

Transmission electron microscope (JEOL 1011 at 100 kV), scanning electron microscope (FEI QUANTA FEG 250 at 15 kV) equipped with an energy-dispersive X-ray (EDX) analyzer were used to observe morphology of the sample. TEM samples were prepared by spreading dilute dispersions in ethanol onto carbon-coated copper grids. The samples for SEM observation were prepared by vacuum sputtering with Pt after ambient drying. Fourier transform infrared (FT-IR) spectra were performed after scanning samples 32 times with a Bruker EQUINOX 55 spectrometer. X-ray diffraction (XRD) patterns were recorded on Rigaku DMAX-2500 power X-ray diffractometer using $\text{CuK}\alpha$ radiation (60 kV, 200mA). The sample was scanned in the range of $2\theta = 10\text{--}80^\circ$ (wide-angle-XRD) at a rate of $8^\circ/\text{min}$. Hysteresis loop analysis and magnetization-temperature (M-T) curves were measured by a VSM magnetic property measurement system (VSM-PPMS9, Quantum Design, Inc). X-ray photoelectron spectroscopy (XPS) data were taken on an AXIS Ultra instrument from Kratos Analytical. All XPS spectra were corrected using C1s line at 284.6 eV. Morphology of the emulsion droplets and the arrangement of nanosheets in a magnetic field were observed under OLYMPUS FV1000-IX81 microscope. High-resolution transmission electron microscope (HRTEM) images were obtained using a TEM (JEM-F200, JEOL, Japan) operated at 200 kV. The EDAX detector linked to the TEM was used to confirm the elemental mappings of the sample.

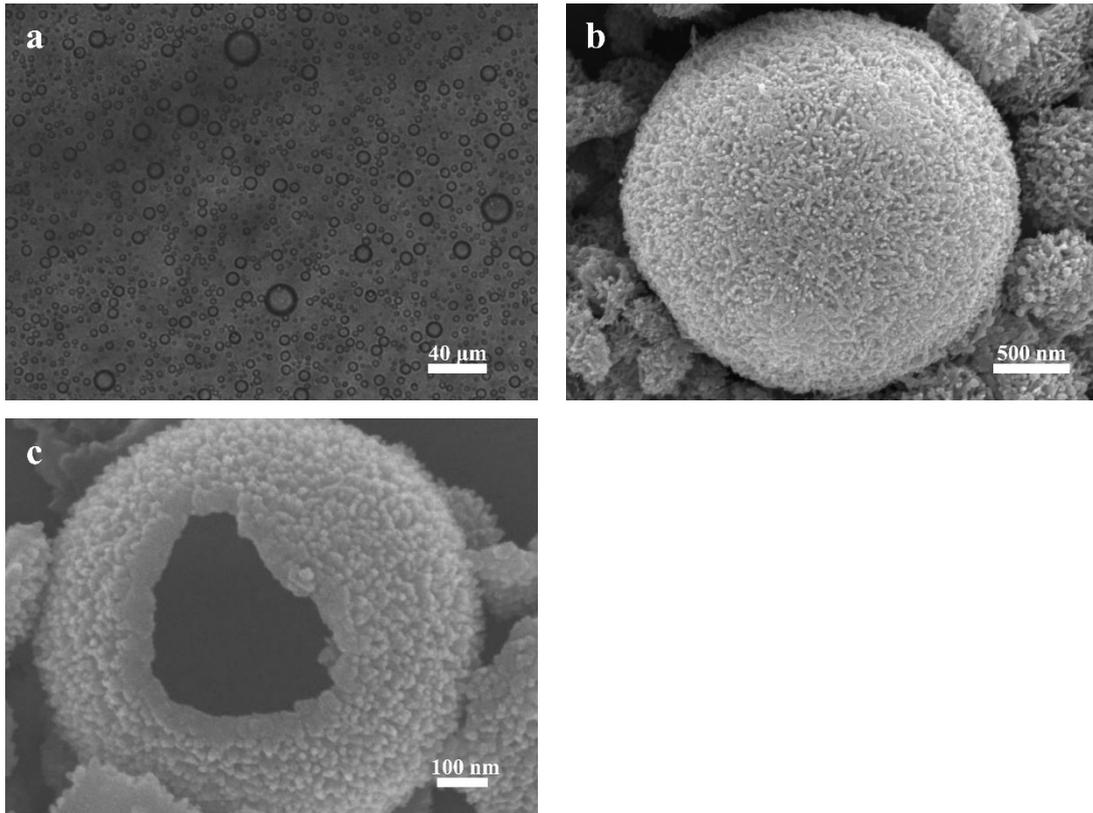


Figure S1. a) Optical microscopy image of emulsion droplets formed by continuous ultrasound, SEM image of b) intact and c) broken hollow sphere found in the chrysanthemum-like nanosheets.

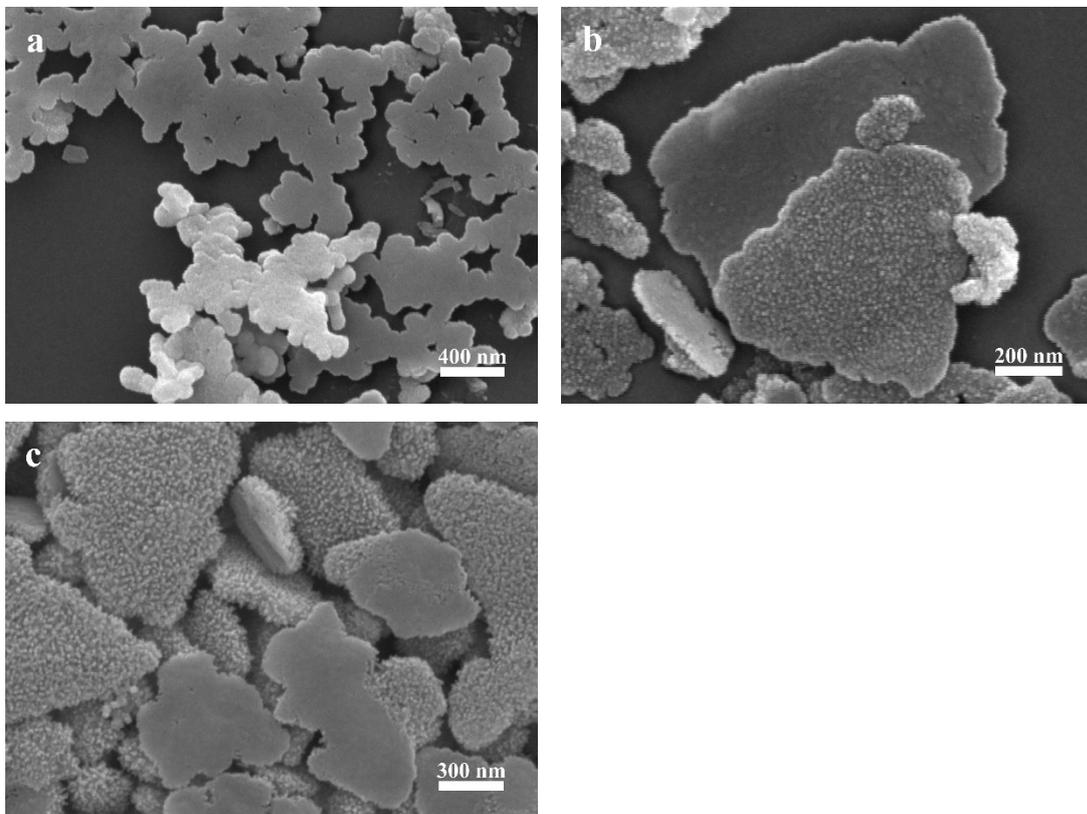


Figure S2. SEM images of nanosheets obtained with a) 0%, b) 25%, and c) 50% of maximum ultrasonic power (150 W).

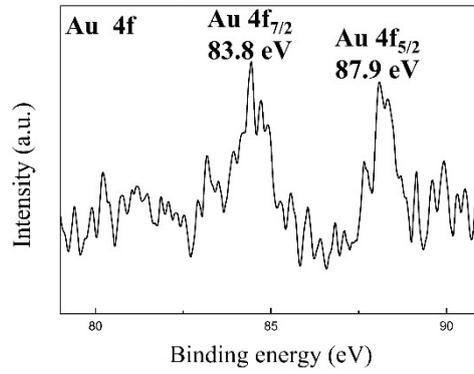


Figure S3. XPS spectrum of Au 4f portion of magnetized nanosheets.