

Nonequilibrium Synthesis of Silica-Supported Magnetite Tubes and Mechanical Control of Their Magnetic Properties

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Movie: growth.avi

The movie is recorded using a stationary camera showing tube growth within a small portion of the reaction vessel. The video clip shows 10 s of the actual experiment. Notice the gradual change of the tube color as the movie proceeds. Width of the glass rod: 5 mm. Rod velocity: 2 mm/s. Flow rate: 10 mL/h. The data in Figure 4 are obtained from this experiment. The movie is digitally compressed. The image quality of the raw data is higher.

Figure 1S shows representative scanning electron microscopy (SEM) data of the precipitation tubes. (a,b) SEM images revealing crack formation on the inner surface of the tube wall. We suggest that crack formation is responsible for the observed fragility of the silica-magnetite tubes. (c) SEM image showing distinct leaf-like patterns that were not observed in the similarly prepared copper hydroxide-silica tubes.¹ Energy dispersive spectroscopy (EDS) data (d) reveals iron as the main constituent element of the leaf-like patterns. This shows that the leaf-like microstructures are not an artifact from carbon coating during sample preparation. The EDS data are also in agreement with the previously reported results that the inner surface of the tube wall is rich in metal oxide/hydroxide.^{2,3}

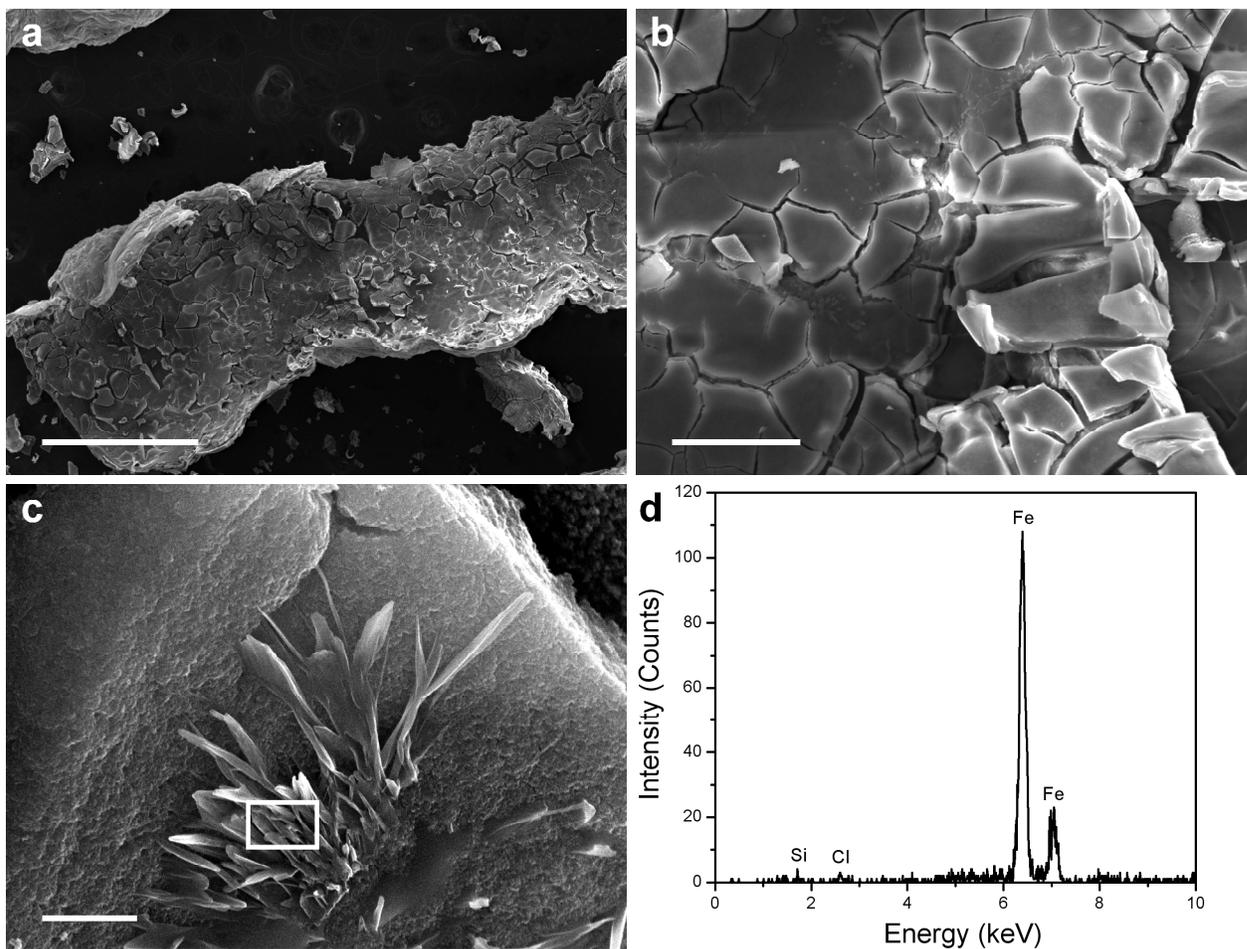


Figure 1S. (a,b,c) Scanning electron micrographs of the tubular reaction products. (a) SEM image of the inner surface of the tube wall. (b) A higher magnification image revealing formation of polygonal segments separated by micro-sized cracks (rod velocity: 3 mm/s, flow rate: 13 mL/h). (c) SEM image revealing distinct leaf-like patterns (rod velocity: 3 mm/s, flow rate: 25 mL/h). (d) EDS spectrum obtained from the material within the white rectangle in (c). Scale bars: 1 mm (a), 100 μm (b), 5 μm (c).

Figure 2S shows the variation of the magnetic properties (remanent magnetization M_r , coercivity H_c , and saturation magnetization M_s) along the length of several silica-magnetite tubes. During the synthesis of these samples, the velocity of the glass rod and the flow rate of the injected solution were kept constant at 3 mm/s and 25 mL/h, respectively. For the measurements, each tube was cut into five segments with essentially constant lengths of 1.3 cm and sorted into groups of equal distance d from the glass capillary. The data in Figure 2S suggest no systematic variation of the magnetic properties.

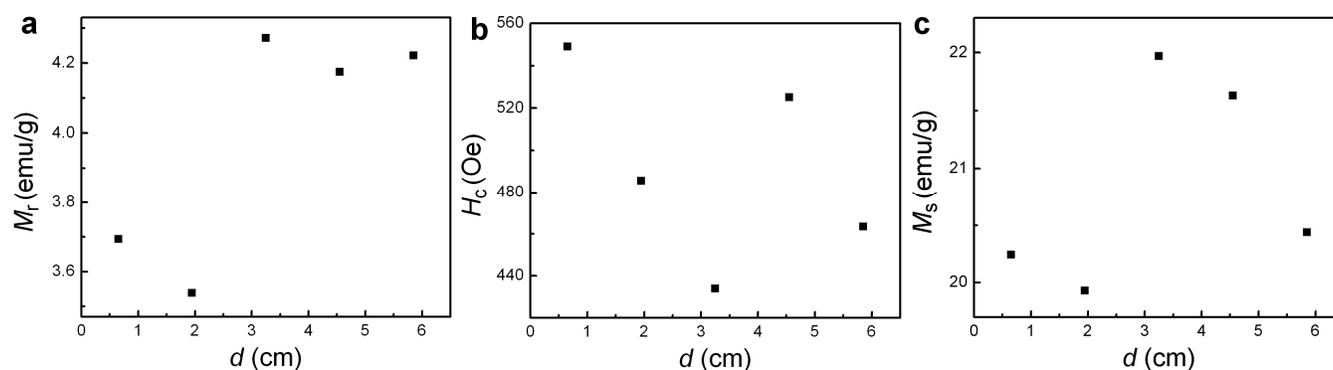


Figure 2S. Variation of the magnetic properties (M_r , H_c , M_s) along the length of the formed tubes. Rod velocity = 3 mm/s. Flow rate = 25 mL/h.

References

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- (3) Balköse, D.; Özkan, F.; Köktürk, U.; Ulutan, S.; Ülkü, S.; Nişli, G. *J. Sol-Gel Sci. Technol.* **2002**, *23*, 253–263.