

## Soft-Templating Synthesis of Granular Magnetite Nanowires and Their Magnetic Properties†

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A facile synthesis of granular  $\text{Fe}_3\text{O}_4$  nanowires in aqueous mediums is reported. The results showed that the proteins can be used as a soft template for synthesis of  $\text{Fe}_3\text{O}_4$  nanowires. The as-prepared  $\text{Fe}_3\text{O}_4$  nanowires are made of many magnetite nanoparticles and have excellent magnetic properties ( $M_s = 81 \text{ emu/g}$ ,  $H_c = 1450 \text{ Oe}$ ) at room temperature.

**Key Words:** Magnetite, Nanowires, Proteins, Soft-templating.

### INTRODUCTION

Nanostructured magnetite ( $\text{Fe}_3\text{O}_4$ ) is among the most important magnetic materials and has aroused extensive attention because of their potential applications in magnetic separation<sup>1,2</sup>, contrast agents in magnetic resonance imaging (MRI)<sup>3,4</sup> and other aspects<sup>5</sup>. To date, various methods such as magnetic-field induction<sup>6</sup>, oxide precursor reduction<sup>7,8</sup> and hard template methods<sup>9</sup> were used, respectively. However, use of hard templates or precursors usually suffers from disadvantages related to impurity and tedious synthetic procedures, which may hinder them being used in large-scale practical applications. Therefore, it remains a challenge to develop simple and effectual approaches to synthesize some real 1D magnetite nanostructures.

In the present work, a simple one-pot soft-template synthesis of well-crystallized granular magnetite nanowires are reported based on ferric chloride hexahydrate ( $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ ), citric acid and proteins raw materials.

### EXPERIMENTAL

In a typical preparation of  $\text{Fe}_3\text{O}_4$  nanowires, 1.42 g  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$  and 3 g citric acid was added to 40 mL deionized water continuous stirring for 20 min. Then, 1 g proteins were added into the above solution to form colloidal solution. Finally, the colloidal solution was transferred quickly into a 60 mL Teflon-lined autoclave and maintained at 180 °C for 12 h. After being cooled to ambient temperature naturally, the solid powder in the autoclave was collected by centrifugation,

washed several times with deionized water and ethanol and finally vacuum-dried at 80 °C for 8 h.

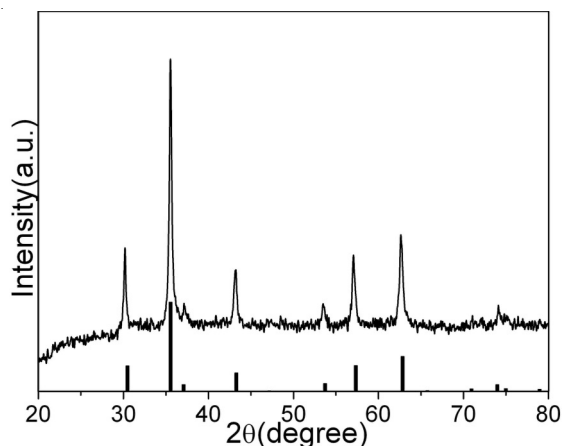
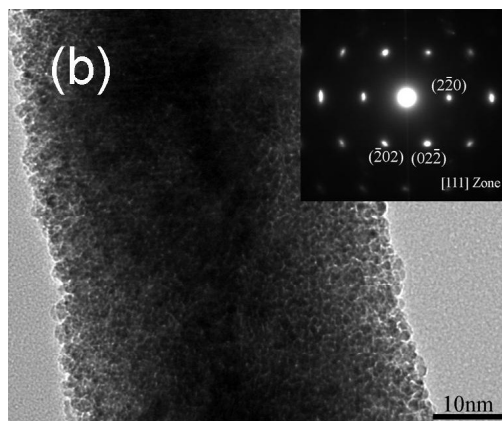
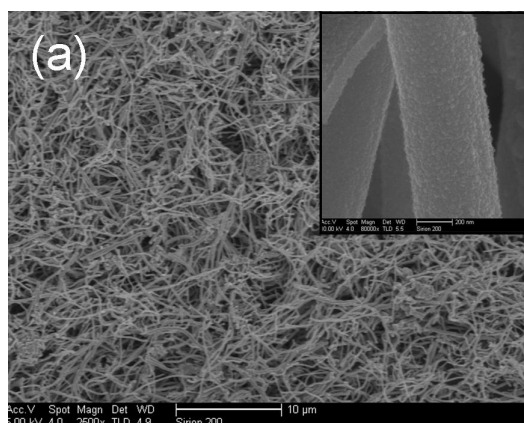
The phase of the as-prepared samples was investigated by powder X-ray diffraction (XRD, Philips X'pert PRO with  $\text{CuK}\alpha$  (0.15419 nm) radiation). The morphology and microstructure of the as-synthesized samples were analyzed with scanning electron microscopy (FESEM, Sirion 200) and transmission electron microscopy (TEM, Hitachi-800, JEOL 2010, 200 kV). Magnetization measurements were carried out on a superconducting quantum interference device (SQUID) at room temperature.

### RESULTS AND DISCUSSION

The nanowires were shown by powder XRD (Fig. 1) to consist of pure  $\text{Fe}_3\text{O}_4$  phase (magnetite), which has a face-centered structure with lattice parameters of  $a = b = c = 0.8395 \text{ nm}$ . The XRD peaks are labeled by using the three- and four-index systems, as for hexagonal nanostructure. The four-index system facilitates the discussion below.

Scanning electron microscopy (FESEM) and transmission electron microscopy (TEM) images provide further insight into the microstructure and morphology of the product. Fig. 2A displays a representative SEM image of an as-prepared sample at low magnification. It shows that the sample is made up of more than 95 % wirelike structures. Fig. 2B shows a high-magnification bright-field TEM image of a single  $\text{Fe}_3\text{O}_4$  nanowire. The inset in Fig. 2B shows a selected-area electron diffraction pattern (SAED) that was obtained by aligning the electron beam perpendicular to the circular facets of an

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Fig. 1. XRD patterns of Fe<sub>3</sub>O<sub>4</sub> solid nanowiresFig. 2. (A) Low-magnification SEM image with a single nanowire of the obtained Fe<sub>3</sub>O<sub>4</sub> nanowires. (B) High-magnification TEM image of a single nanowire and SAED pattern indicating the single-crystal nature of the nanowires

individual nanowire. The hexagonal symmetry of these diffracted spots suggests that each Fe<sub>3</sub>O<sub>4</sub> nanowire was a single crystal. The SAED patterns on different nanowires or different positions of a given single nanowire were essentially identical, thus indicating that the nanowires are single-crystalline.

Fig 3 presents the corresponding magnetization as a function of magnetic field, or the *M* versus *H* curve. The saturation magnetization (*M<sub>s</sub>*) and the coercivity (*H<sub>c</sub>*) of the magnetite microspheres is about 81 emu/g and 145 Oe, which

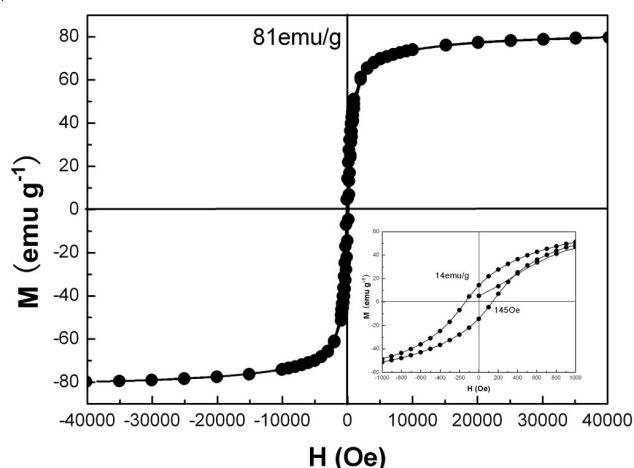


Fig. 3. Magnetization hysteresis loop of the porous magnetite microspheres. The down-right inset is an expanded low-field curve

are close to the values of bulk Fe<sub>3</sub>O<sub>4</sub> (85-100 emu/g and 115-150 Oe, respectively<sup>10</sup>), as shown in the down-right inset of Fig. 3, corresponding to an expanded low-field hysteresis curve.

## Conclusion

In summary, we have successfully prepared magnetite nanowires *via* a facile one-step sol-hydrothermal route. This method uses less expensive raw materials instead of a hazardous Fe(CO)<sub>5</sub> or cost iron acetylacetonate precursor. The as-prepared Fe<sub>3</sub>O<sub>4</sub> nanowires are made of many magnetite nanoparticles and have excellent magnetic properties at room temperature.

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