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Synthesis and Characterization of Fe-C Core-shell Nanoparticles

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Magnetic nanoparticles are suitable for applications such as biosensing [1] and contrast agents for magnetic resonance imaging [2] etc. Iron nanoparticles have the advantage of high saturation magnetization for these applications. However, oxidation at ambient condition makes this advantage rarely available. Encapsulating iron nanoparticles by graphitic carbon is one of the solutions to this problem.

In this work, Fe-C core-shell nanoparticles are successfully synthesized. The approach is similar to Kong's method for carbon nanotubes [3]. For the transmission electron microscopy (TEM) study, the nanoparticles are transferred to a holey carbon TEM grids by applying a drop of nanoparticle suspension to the grids which are then air-dried. A Philips CM300 TEM operated at 150KeV was used for the TEM study.

The nanoparticles are typically 5~10 nm in diameter (Fig. 1). Each nanoparticle is composed of an Fe core and a graphitic shell (Fig. 2). High-resolution TEM images and Fast Fourier Transforms (FFT) of selected areas reveal that the core of the nanoparticles can either be α-Fe (BCC, lattice parameter 2.87Å) or γ-Fe (FCC, lattice parameter 3.56 Å) (Fig. 3 and Fig. 4). The magnifications in both images were calibrated by the (0002) spacing of graphite (3.35 Å). Neither iron oxide nor iron carbide is observed in the core of the particles. Note that in Fig. 3 and Fig. 4, the graphitic shells surrounding both particles are not clearly seen because of the background, damage by the electron beam, and by the imaging defocus conditions.

A single magnetic domain would be more energetically stable for a magnetic particle below a certain critical size (e.g. ≈10 nm for iron) and thermal agitation would fluctuate the magnetization direction of the particle, giving rise to the superparamagnetic (SPM) state [4]. Therefore, the nanoparticles synthesized in this work are expected to be SPM. Zero Field Cooling (ZFC) and Field Cooling (FC) and magnetization versus field measurements were performed on the as-grown material by a Superconducting Quantum Interference Device (SQUID). In ZFC and FC, the sample is cooled from room temperature to 2 K at zero magnetic field. Once the temperature reaches 2 K, a 1000 Oe magnetic field is applied and kept for the remainder of the ZFC and FC measurement. Then the temperature is increased to 320K and the magnetization versus temperature is measured. After the temperature reaches 320K, the sample is cooled back to 2 K and the magnetization versus temperature is measured again (Fig. 5 (a)). The peak value of the ZFC curve is generally regarded as the blocking temperature (the temperature at which the particles undergo transition from ferromagnetic to SPM states). The blocking temperature of the nanoparticles is about 3.5K, suggesting their magnetocrystalline anisotropy energy is very small. Magnetization versus magnetic field is measured at 300K and no hysteresis is observed (Fig. 5 (b)), which is another piece of evidence of the SPM state. It is noteworthy that the saturation magnetization (Ms) of the nanoparticles is only about 9% of that of bulk α-Fe. This is significantly lower than the Ms observed by Sun et al for carbon-encapsulated Fe nanoparticles (37.6% of bulk α-Fe)[5], this may be due to a

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higher percentage of γ-Fe nanoparticles and/or smaller particle size (more significant finite size effect on the Ms), which are observed by high-resolution TEM. Therefore, the TEM study suggests that the microstructure and size of such nanoparticles have significant impact on the magnetic properties [6].

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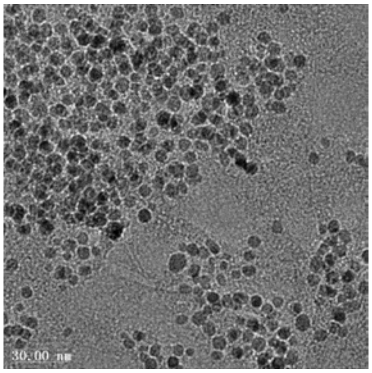

Fig.1. Low-magnification image of the nanoparticles.

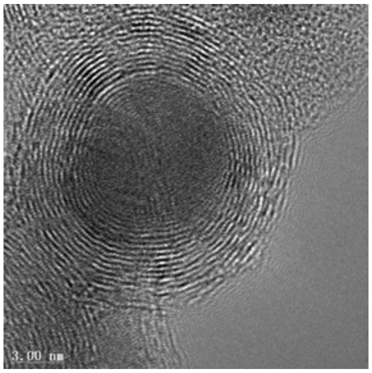

Fig.2. TEM image showing the Fe-C core-shell structure.

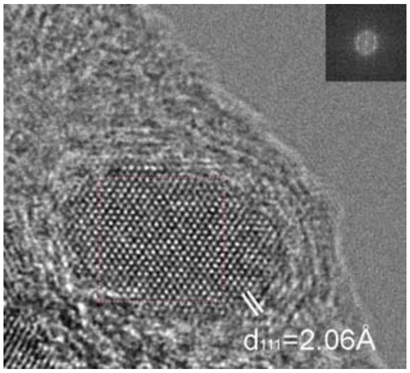

Fig.3. [110] zone axis TEM image and FFT of γ-Fe.

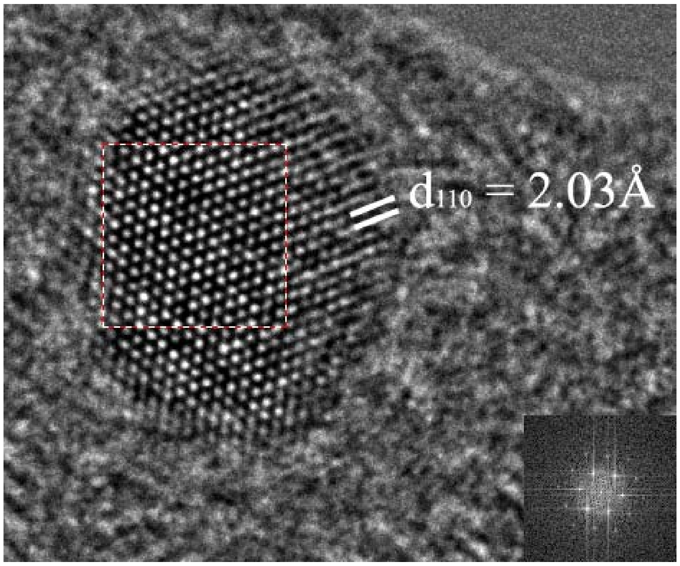

Fig.4. [111] zone axis TEM image and FFT of α-Fe.

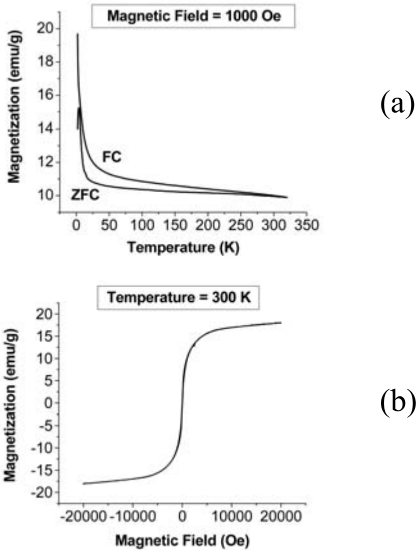

Fig.5 Temperature dependence of magnetization (a) and M vs. H curve (b)

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