



Size Control of FePt Nanoparticles

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ABSTRACT

Monodisperse FePt nanoparticles with diameters 2 to 16 nm have been made from thermal decomposition of iron pentacarbonyl in octyl ether in the presence of oleic acid and oleyl amine. The size of the nanoparticles can be controlled by regulating the amount of surfactant, solvent, heating rate and other reaction parameters. The as-synthesized particles have chemically disordered FCC structure and can be transformed into the chemically ordered FCT structure after proper thermal treatments. The FCT structured thin film FePt nanoparticle assemblies exhibit hard magnetic properties with coercivity as high as 2.7 T. These hard magnetic nanoparticles can be used in many technological applications.

INTRODUCTION

Chemically synthesized FePt nanoparticles¹ have unique magnetic and structural characteristics and can be applied in many advanced technological applications. For instance, the particles can be directly applied in biomedical applications such as drug delivery after treating with surfactants. The FePt nanoparticles with strong magnetic anisotropy can also be used as building blocks of magnetic recording media and soft and hard magnetic thin films or bulk magnets. Different applications require different particle sizes. However, producing FePt particles with the desired size and acceptable size distribution remains a challenge. The Ostwald ripening, which has been successfully applied to production of iron oxide nanoparticles of various sizes² does not work for FePt nanoparticles synthesis. In this poster, we report our recent work in synthesizing FePt nanoparticles with diameters of 2-16 nm.

EXPERIMENTAL

A typical synthesis route of the chemical synthesis of FePt nanoparticles is schematically shown in Fig. 1. After the synthesis, nanoparticles and their assemblies are characterized by transmission electron microscopy (TEM), X-ray diffraction (XRD), alternating gradient magnetometer (AGM) and superconducting quantum interference device (SQUID) magnetometer.

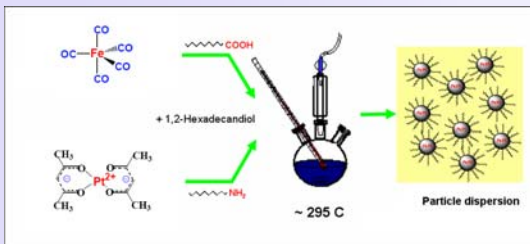


Fig. 1 Schematic showing the chemical synthesis of the FePt nanoparticles.

2 nm FePt nanoparticles are synthesized as follows: 0.5 mmol Pt(acac)₃ is charged to 125 ml flask containing a magnetic stir bar and 20 ml of octyl ether. Argon gas was flowed throughout the whole experiment. After purging with Argon for 30 minutes at room temperature, the flask was heated up to 200°C and 1.1 mmol of Fe(CO)₅, 0.5 mmol each of oleic acid and oleyl amine were added and the temperature is kept constant for 30 minutes. Then the flask is heated up to 295 °C and refluxed for 30 minutes before cooling to room temperature under the Argon blanket. A heating rate of 5°C per minute was maintained during the experiment. The resulting black product was precipitated by adding ethanol and the precipitate was redispersed in hexane and stored under refrigeration. Samples for magnetic characterization were prepared by depositing 2 to 3 layers of the dispersion onto 3x3 mm silicon substrate, which led to the formation of FePt nanoparticle assemblies. The samples were then annealed in forming gas (Ar + 7% H₂) atmosphere for 1hr at temperatures at 650°C.

If the iron precursor and surfactant were added at 100°C instead of 200°C, 4 nm particles are obtained. Likewise, for 8 nm particles 10 ml of octyl ether is used with 8 times of surfactant. For 16 nm Pt/Fe₃O₄ core-shell particles, first 10 nm Pt cores are synthesized by addition of Pt precursor at 290°C in a mixture of octyl ether and surfactants and then they are coated with iron⁴. After annealing, these particles are converted to FCT FePt nanoparticles (see Fig 2) which are confirmed from the XRD and magnetic measurements. The reaction conditions for all particles have been summarized in Table 1.

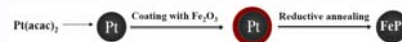


Fig 2 Schematic of 16 nm core-shell Pt/Fe₃O₄ nanoparticles

Particle Size (nm)	Pt addition temperature (°C)	Fe addition temperature (°C)	Surf addn temperature (°C)	HDD (mmol)	Surfactant (mmol)	Octyl Ether (ml)	Highest Coercivity (T)	Required Fe(CO) ₅ (mmol)
2	20	200	200	1.5	0.5	20	2.7	1.2
4	20	100	100	1.5	0.5	20	2.4	1.1
8	20	130	130	0	4	10	1.6	1.0
16	290	220	20	1.5	0.5	5	1.8	0.9

Table 1 Reaction parameters for various size of particles

RESULTS

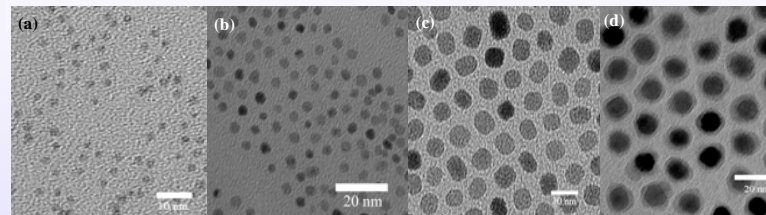


Fig 3 TEM image of as synthesized FePt nanoparticles of (a) 2 (b) 4 (c) 8 and (d) 16 nm

Fig. 3 shows the TEM image of the as-synthesized FePt particles of diameter 2, 4, 8 and 16 nm. In Fig. 5 one can find XRD patterns of the as-synthesized FePt particles of various diameters and annealed FePt nanoparticles. The as-synthesized particles exhibit a disordered FCC crystal structure. In case of 16 nm core-shell Pt/Fe₃O₄ particles, because the peaks come from the Pt atom, they are slight left compared to FCC FePt peaks. Annealing leads to formation of chemically ordered FCT structure, as indicated by the (111), (200) and (220) peaks shift and evolution of (110) and other peaks.

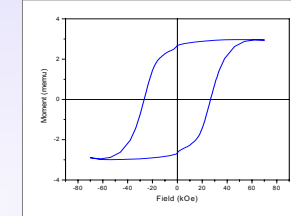


Fig. 4 Magnetic hysteresis loop of 2 nm nanoparticle assembly annealed at 650°C in FG for 1 hour.

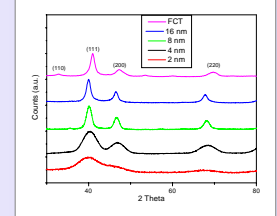


Fig. 5 X ray diffraction patterns of the as-synthesized particles of 2, 4, 8 and 16 nm.

The 2 nm particles showed the highest coercivity of 2.7 T (see Fig. 4, magnetization not normalized) while 4, 8 and 16 nm showed 2.4, 1.6 and 1.8 T, respectively. The high coercivity of annealed particles clearly proves beyond doubt that these particles are transformed into the FCT phase with high magnetocrystalline anisotropy after annealing.

The composition of FePt nanoparticle materials is tuned by varying the amount of Fe(CO)₅, however amount of Pt(acac)₃ (0.5 mmol) has been kept constant. It has already been seen¹ that not all the Fe(CO)₅ contributes to the FePt formation and almost double moles of Fe(CO)₅ yields high quality FCT phase and highest coercivity. However, the composition also depends on the size of the particles³. The amount of required Fe(CO)₅ decreases as the size of the particles increases. The details are given in the Table 1.

CONCLUSIONS

Monodisperse FePt particles of diameter 2 to 16 nm have been successfully synthesized by varying the synthesis conditions. Meanwhile, composition control has been realized which resulted in correct stoichiometry of the hard magnetic FePt FCT phase. After annealing, the nanoparticles assemblies give high coercivity. In the future we will use the hard magnetic particles as building blocks to produce advanced nanostructured thin film and bulk magnets.

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4. Teng X., Yang H., *J. Am. Chem. Soc.*, **125**, 2003, 14559.

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