High Coercivity in FePt Nanoparticle Assemblies

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Abstract

Ultra-fine FePt nanoparticles have been synthesized via a novel chemical solution synthesis route. Without using a reducing agent, stoichiometric FePt nanoparticles were produced by the decomposition of iron acetylacetonate and platinum acetylacetonate in octyl ether in the presence of oleic acid and oleyl amine. The particle size was determined by transmission electronic microscopy (TEM) observation to be around 2 nm. The particles were then deposited on a substrate to form thin-film-like assemblies and subsequently heat treated. Upon annealing, the as-synthesized nanoparticles transformed from the FCC phase to the magnetically anisotropic FCT phase leading to magnetic hardening in the assemblies. Coercivity up to 2.7 T was obtained in the samples with the Fe:Pt molar ratio of 1.2:1 after being annealed at 650°C for 1 hour in forming gas (Ar + 7% H2). The high coercivity indicates a highly completed phase transition from the FCC structure to the FCT structure.

Introduction

Chemically synthesized FePt nanoparticles[1,2] are promising candidates for developing ultrahigh density magnetic recording media and high energy product permanent magnets. These nanoparticles are chemically stable and possess high magnetocrystalline anisotropy in the $L1_0$ phase. The magnetic properties of the FePt nanoparticles are very sensitive to the particle composition. In the methods proposed earlier,[1] a volatile and toxic compound $Fe(CO)_5$ has been used as a precursor. It is difficult to carry over the initial molar ratio of metal precursors to the final product with this precursor. In this poster, we report a simple and compositionally controlled route to synthesize FePt nanoparticles of 2 nm range by decomposition of $Fe(acac)_3$ and $Pt(acac)_2$ under controlled conditions.

Experimental

A 1.5 mmol Pt(acac)2, 1.5 mmol Fe(acac)3, 0.75 mmol of oleic acid and 0.75 mmol of oleyl amine was added to 125 ml flask containing a magnetic stir bar and 30 ml of octyl ether. Argon gas was passed throughout the whole experiment while heating, refluxing and cooling to room temperature. After purging with Argon for 30 minutes at room temperature, the flask was heated up to 200° C and held for 30 minutes. Then the flask was heated up to 295 ° C and refluxed for 30 minutes before cooling to room temperature under an Argon blanket. The heating rate of 5° C per minute was maintained during the experiment. The schematic representation of the chemical synthesis is shown in Fig. 1.

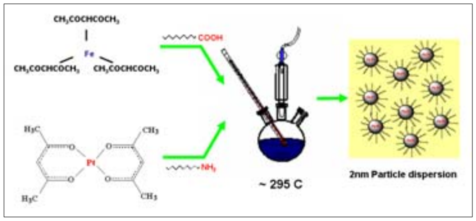

Fig. 1. Schematic showing the chemical synthesis

The resulting black product (5mL) was precipitated by adding ethanol (40mL) and separated by centrifugation. After discarding the light brown supernatant, the precipitate was redispersed in hexane (15mL) and ethanol (5mL) was added to precipitate out irregular and large size particles by centrifugation. The black precipitate was discarded and ethanol (25mL) was then added to the supernatant to precipitate the fine and monodisperse particles. These particles were 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, evaporating the solvent at room temperature and further drying in vacuum, which led to the formation of FePt nanoparticle assemblies. The samples were then annealed in argon or forming gas (Ar + 7% H_2) atmosphere for 1 hour at temperatures at 650°C. Magnetic hysteresis loops of the samples were measured by AGM and SQUID magnetometers. TEM and X-ray analysis were used to identify the particle sizes and crystal structure.

Results and Discussion

Figure 2 shows the TEM image of the as-synthesized FePt particles. The particle size is approximately 2 nm with quite uniform size distribution. Figure 3 shows XRD pattern of as synthesized FePt nanoparticles which possess a disordered FCC crystal structure. The nano range of particles size can be easily confirmed by the broad peak. Also shown is the standard XRD pattern for FCT FePt particles.

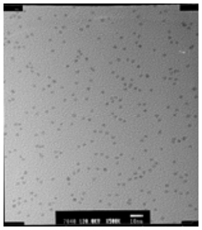

Fig. 2. TEM image of nanoparticles

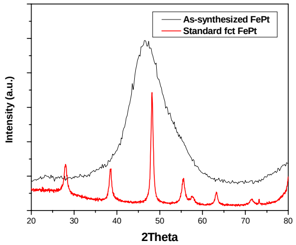

Fig. 3. XRD patterns

A graph of coercivity of nanoparticles annealed in forming gas at 600° C for 1 hour with different molar ratios of Iron precursor is shown in Fig. 5. The highest coercivity achieved is 1.3 T at 1.2:1 Fe:Pt ratio. However, coercivity depends on the other reaction parameters too. For instance, if we put the surfactant at 200 C instead at room temperature, these particles showed a coercivity of 1.9 T or if we reflux at 295 C for 48 hrs, the particles show 2.7 T from the magnetic hysteresis measurements using SQUID (Fig. 4). The average particle composition of the FePt nanoparticles synthesized using a 1:1 mole ratio of the precursors was determined to be $Fe_{46}Pt_{54}$ from the ICP analysis. It clearly shows that atomic composition of Fe:Pt must be almost equal for the highest magnetic properties of FePt nanoparticles which have already been proved earlier[1]. The high coercivity of annealed particles clearly proves beyond doubt that these particles are transformed into the FCT phase with high magnetic crystalline anisotropy after annealing.

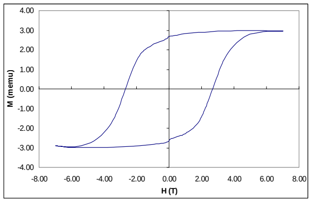

Fig. 4. Magnetic hysteresis loop

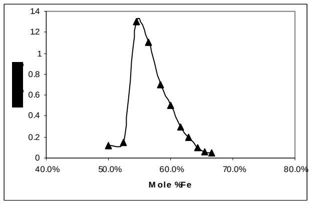

Fig. 5. Coercivity trend

Conclusion

In conclusion, Monodispersed FePt nanoparticles with particle size of 2 nm range have been synthesized by a novel chemical solution method by decomposition of ferric acetyl acetonate, Fe(*acac*)3 , and platinum acetyl acetonate, Pt(*acac*)2 in octyl ether. The coercivity up to 2.7 T at room temperature can be obtained after annealing the samples at 650° C for 1 hour in forming gas. This novel route of FePt nanoparticle synthesis opens more possibilities in the nanoparticles size control and magnetic property improvement.

References

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