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Effect of Heat Treatment Conditions on the Properties of FePt Nanoparticles Produced by Sonochemistry

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Abstract: Magnetic properties of magnetic nanoparticles FePt prepared by sonochemistry were investigated. Upon annealing at temperature from 450ºC to 650ºC samples have ordered $L1_0$ structure and show hard magnetic properties. Influence of heat treatment conditions (annealing temperature, cooling rate) on the magnetic properties of the annealed samples has been studied.

Keywords: FePt, nanoparticle, sonochemistry, magnetic properties.

1. Introduction

FePt nanoparticles attract much attention as one of the most promising candidates for ultrahigh-density magnetic recording media because of their superior magnetic properties such as high magnetocrystalline anisotropy energy, high saturation magnetization and high chemical stability [1,2]. Meanwhile, FePt nanoparticles are also expected to be a high-performance nanomagnet for magnetic medicine, such as magnetic hyperthermia [3], immunomagnetic cell separation [4], and excellent contrast agents for magnetic resonance imaging [5].

Usually, FePt nanoparticles fabricated have a disordered face-centered cubic (fcc) structure, and thus a post thermal annealing is necessary to transform them into the desired ordered face-centered tetragonal (fct) $L1_o$ phase. Recently, magnetic properties of FePt nanoparticles prepared by sonoelectrodeposition have been reported by Nam *et al.* [6]. In this study, we report the hard magnetic properties of FePt nanoparticles synthesized by sonochemistry, which was developed to make nanoparticles [7]. Influence of heat treatment conditions (annealing temperature, cooling rate) on the magnetic properties of the annealed samples has been discussed.

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2. Experimental

The synthesis of FePt nanoparticles was conducted by sonochemical reaction. The mixture of H_2PtCl_6 and iron (II) acetate $[Fe(C_2H_3O_2)_2]$ with distilled water were prepared in a 150 ml flask in (Ar + 5% H_2) atmosphere. A little TSC (trisodium citrate) was added to the solution. The solution in flask was ultrasonicated with power of 375 W, frequency of 20 kHz emitted by a Sonic VCX 750 ultrasound emitter within 240 minutes. The FePt nanoparticles were washed and separated from the solution by using a centrifuge with alcohol at 9000 rpm for 30 minutes and then dried at 70°C-75°C. As-prepared samples then were annealed at various temperatures from 450°C to 650°C under continuous flow of (Ar + 5% H_2) gas for 1 h. After annealing, some samples were cooled gradually with furnace (normally cooled), the other part of samples were immediately pulled out of the furnace (fast cooled).

The structure of the as-prepared and the annealed FePt samples at various temperatures were studied by X-ray diffractormeter (Bruker D5005). The particle morphology was obtained from a transmission electron microscope (TEM JEM1010-JEOL). The chemical composition of the FePt nanoparticles was studied by using an energy dispersion spectroscopy (EDS OXFORD-ISIS 300) and revealed that the chemical composition of our sample is $Fe_{60}Pt_{40}$. Magnetic properties of samples were studied by using a Vibrating Sample Magnetometer (VSM) MicroSence EZ9.

3. Results and discussion

Figure 1 is the TEM image of typical as-prepared sample. Particles size of the as-prepared $Fe_{60}Pt_{40}$ sample was 3-5 nm.

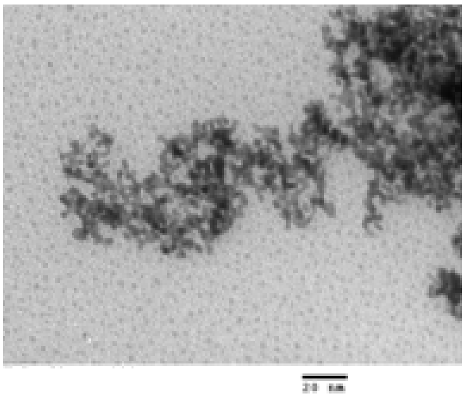

Figure 1. TEM image of the as-prepared $Fe_{60}Pt_{40}$ nanoparticles.

Figure 2 shows the X-ray diffraction (XRD) pattern of the as-prepared $Fe_{60}Pt_{40}$ nanoparticles. The XRD results showed the reflections of pure Pt structure, which is similar to other FePt nanoparticles produced by sonoelectrodeposition [6]. The reflections from Fe are very weak due to the fact that their atomic weight is much less than that of Pt which is similar to the XRD result of FePt foils prepared by cold deformation [8]. The Pt peaks in the as-prepared samples are broad due to the small size of the particles. The particles were not disordered FePt but they can be formed by many small domains of pure Fe and Pt. Figure 3 shows XRD patterns of $Fe_{60}Pt_{40}$ nanoparticles annealed at 550°C for 1 h for normally-cooled and fast-cooled samples. Upon annealing, the formation of the ordered $L1_o$ fct phase happened by the diffusion process between Fe and Pt domains.

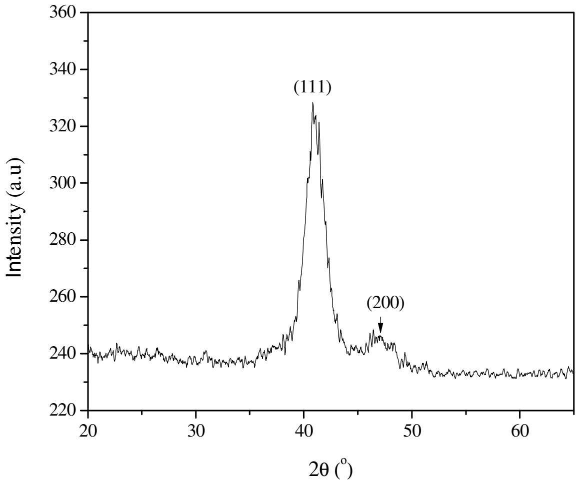

Figure 2. XRD pattern of as-prepared $Fe_{60}Pt_{40}$ sample.

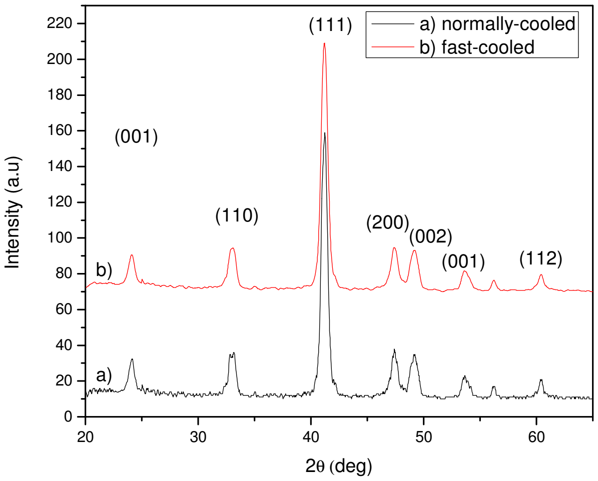

Figure 3. XRD patterns of $Fe_{60}Pt_{40}$ samples annealed at 550°C for 1 h:
a) normally-cooled, b) fast-cooled.

Magnetic measurements revealed low saturation magnetization (M_s) and coercivity (H_c) in all as-prepared samples (data not shown). The saturation magnetization of the unannealed particles was about few emu/g and the coercivity was just above 100 Oe. The low value of M_s of the as-prepared nanoparticles may be explained by the oxidation or hydroxidation of Fe atoms in nanoparticles which can result in the weak magnetic iron oxides and iron hydroxides. This is in agreement with the suggestion of separated Fe and Pt domains in as-prepared nanoparticles. It is known that FePt with high saturation magnetization is a chemically stable material. Therefore it is difficult to be oxidized to form weak ferromagnetic materials. After annealing the hard magnetic FePt phase was formed. Figure 4 presents the room-temperature magnetic curves of normally-cooled $Fe_{60}Pt_{40}$ nanoparticles annealed at different temperatures. The curves show a typical hard magnetic hysteresis loops with high H_c. Upon annealing, the saturation magnetization and the coercivity of the nanoparticles were improved significantly.

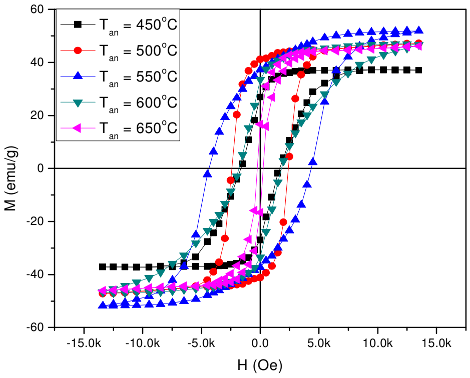

Figure 4. Room-temperature magnetic curves of normally-cooled $Fe_{60}Pt_{40}$ nanoparticles annealed at 450°C÷650°C for 1 h.

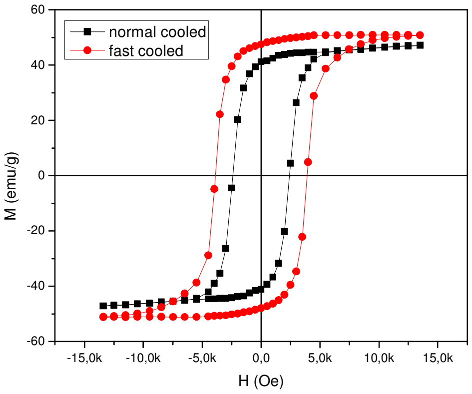

Figure 5. Room-temperature magnetic curves of normally-cooled and fast-cooled $Fe_{60}Pt_{40}$ nanoparticles annealed at 500°C.

Figure 5 shows room-temperature magnetic curves of normally-cooled and fast-cooled $Fe_{60}Pt_{40}$ nanoparticles annealed at 500°C as example. The magnetic curves of normally-cooled and fast-cooled $Fe_{60}Pt_{40}$ nanoparticles annealed at other temperatures have similar behavior. It is clearly seen that hard magnetic properties of fast-cooled samples are better that those of normally-cooled ones. We suggest that it is because chemically ordered fct $L1_o$ phase is maintained better due to fast cooling in fast-cooled samples compared to that in normally-cooled ones. The annealing-temperature dependence of coercivity of the normally-cooled and fast-cooled $Fe_{60}Pt_{40}$ nanoparticles is shown in Figure 6. The annealing temperature 550°C gives highest coercivity for both normally-cooled and fast-cooled samples. The coercivity is 4.36 kOe and 4.95 kOe for normally-cooled and fast-cooled samples annealed at 550°C, respectively.

The annealing-temperature dependence of magnetic squareness $S = M_r/M_s$ of the normally-cooled and fast-cooled $Fe_{60}Pt_{40}$ nanoparticles is shown in Figure 7. In general, magnetic squareness of the fast-cooled samples is higher than that of the normally-cooled ones. The highest value for S is obtained for both normally-cooled and fast-cooled samples annealed at 500°C.

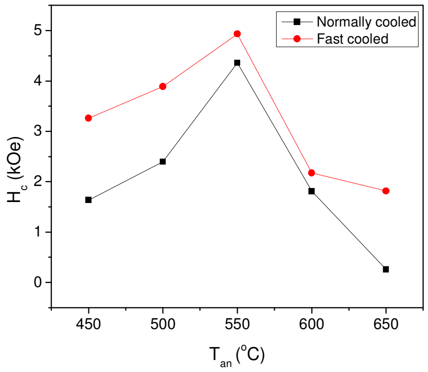

Figure 6. The annealing-temperature dependence of coercivity of the normally-cooled and fast-cooled $Fe_{60}Pt_{40}$ nanoparticles.

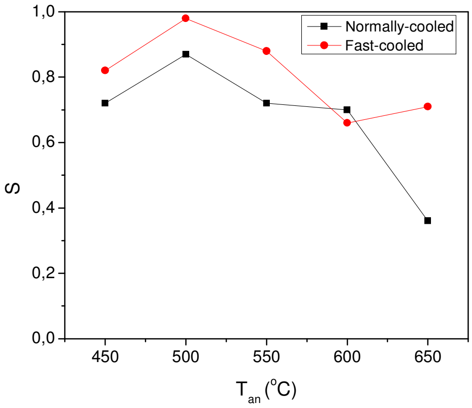

Figure 7. The annealing-temperature dependence of magnetic squareness $S = M_r/M_s$ of the normally-cooled and fast-cooled $Fe_{60}Pt_{40}$ nanoparticles.

4. Conclusion

The structure and magnetic properties of the $Fe_{60}Pt_{40}$ nanoparticles prepared by sonochemistry have been studied. The coercivity started to increase abruptly from annealing temperature $T_{an} = 450^{o}C$, gained the highest value at $T_{an} = 550^{o}C$. The room-temperature coercivity is 4.36 kOe and 4.95 kOe for normally-cooled and fast-cooled samples annealed at $550^{o}C$, respectively. Sonochemistry is a promising method to make FePt magnetic nanoparticles.

Acknowledgements

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References

- [1] S. Sun, C.B. Murray, D. Weller, L. Folks, and A. Moser, Science 287 (2000) 1989.
- [2] S. Sun, Adv. Mater. 18 (2006) 393.
- [3] S. Maenosono and S. Saita, IEEE Trans. Magn. 42 (2006) 1638.
- [4] H. Gu, P.-L. Ho, K.W.T. Tsang, L. Wang, and B. Xu, J. Am. Chem. Soc. 125 (2003) 15702.
- [5] S. Maenosono, T. Suzuki, and S. Saita, J. Mag. Mag. Mat. 320 (2008) L79.
- [6] Nguyen Hoang Nam, Nguyen Thi Thanh Van, Nguyen Dang Phu, Tran Thi Hong, Nguyen Hoang Hai, Nguyen Hoang Luong, J. Nanomaterials 2012 (2012) 801240.
- [7] A. Gedanken, “Novel methods (sonochemistry, microwave heating, and sonoelectrochemistry) for the preparation of nanosized iorganic compounds,” in Inorganic Materials: Recent Advances, D. Bahadur, S. Vitta, and O. Prakash, Eds., p.302, NarosaPublishing, Delhi, India, 2002.
- [8] N.H. Hai, N.M. Dempsey, D. Givord, J. Magn. Magn. Mater. 262 (2003) 353.