Crystallographic Structural and Magnetic Properties of FePt Nanoparticles

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Abstract: $Fe_{60}Pt_{40}$ nanoparticles have been successfully prepared by sonochemistry method from the solution of Fe($C_2H_3O_2$)₂ and H_2PtCl_6 . The as-prepared sample then has been annealed at temperatures from 450 $^{\circ}$ C to 650 $^{\circ}$ C. Annealed samples have ordered L1_o phase with face-centered tetragonal (fct) structure and show hard magnetic properties. The nanoparticles annealed at 550° C show highest coervicity of 4.36 kOe measured at room temperature. The annealed-temperature dependence of the coercivity is consistent with the appearance of the order phase L_0 in the samples.

Keywords: FePt nanoparticles, coercivity, squareness, annealing temperature, sonochemistry.

1. Introduction*

FePt nanoparticles have attracted much attention due to their applicability in ultrahigh density magnetic storage media and microelectronic mechanical systems (MEMS) [1] mainly because of their hard magnetic properties and chemical stability in the ordered face-centered tetragonal (fct) $L1_0$ structure. This ordered fct $L1_0$ phase of FePt materials are normally obtained from the disordered facecentered cubic (fcc) materials via the order-disorder transition. In the ordered phase, FePt alloys show excellent hard magnetic properties with high room-temperature saturation magnetization, $\mu_0 M_s$, of 1.4 T, the Curie temperature T_c of 750 K and high crystalline anisotropy K_1 , of 7 MJ/m³, which is an order of magnitude higher than that of the currently used Co-Cr based alloys [2].

Properties of FePt nanoparticles vary due to the synthesizing method. In general, FePt nanostructured materials can be prepared by physical techniques such as mechanical deformation [3], arc-melting [4], vacuum evaporation (sputtering) [5, 6], laser ablation pulse [7], chemical methods [8-

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10], and physicochemical method such as electrodeposition [11, 12]. Among them, the vacuum evaporation is the most used method. In physicochemical methods, sonoelectrochemistry was used to synthesize FePt nanoparticles [13]. An alternative approach for synthesizing nanomaterials is sonochemistry (see [14] and references therein). It is a very useful synthetic method, in which the application of ultrasonic energy could increase the rate of electrolytic water cleavage. The effects of ultrasonic radiation on chemical reactions are due to the very high temperatures and pressures, which develop in and around the collapsing bubble [15]. In this paper, we successfully prepared FePt nanoparticles by the sonochemical method and we report structure and the hard magnetic properties of the prepared samples.

2. Experimental

The synthesis of FePt nanoparticles was conducted by sonochemical reaction from the solution of $Fe(C_2H_3O_2)$ and H₂PtCl₆. This solution was prepared with pure water in a 150 mL flask. The solution in flask was ultrasonicated with power of 375 W and frequency of 20 kHz emitted by a Sonic VCX 750 ultrasound emitter within 240 minutes. The FePt nanoparticles were collected from the solution by using a centrifuge (Hettich Universal 320) at 9000 rpm, 30 min. The nanoparticles were then dried at 75^oC and these as-prepared samples then were annealed at various temperatures from 450° C to 650° C under $(Ar+5\%H_2)$ atmosphere for 1 h.

The chemical composition of the samples was studied by energy dispersive spectroscopy (EDS OXFORD-ISIS 300). XRD patterns were taken by X-ray diffractormeter (Bruker D5005). The magnetic properties of the samples were studied by using a vibrating sample magnetometer (VSM) MicroSence EZ9.

3. Results and discussion

The energy dispersive spectrum shown in Figure 1 indicated that the estimated chemical composition of the sample is almost close to the nominal composition of $Fe₆₀Pt₄₀$. Figure 2 shows the X-ray diffraction patterns of as-prepared and annealed Fe₆₀Pt₄₀ nanoparticles. Evidence of chemical ordering for the annealed samples was signified by appearance of the (001), (110), (111), (200), (002) peaks. The as-prepared sample has the structure with partly nanocrystalline structure fcc with a broad peak at $2\theta = 40.8^{\circ}$. The annealed samples have almost same crystalline structure at all annealed temperatures. The ordered phase was assigned to $L1_0$ fct structure. The $L1_0$ fct phase is obtained by annealed over 450° C. However, the intensity of diffraction peaks vary and then decrease at annealed temperatures of above 550° C.

Figure 1. The energy dispersive spectrum of $Fe_{60}Pt_{40}$ sample prepared by sonochemistry.

Figure 2. X-ray diffraction patterns of the $Fe_{60}Pt_{40}$ nanoparticles: as-prepared and annealed at 450°C, 500°C, 550°C, 600°C, 650°C.

From the X-ray diffraction, we have calculated the lattice parameters *a* and *c* of $Fe₆₀Pt₄₀$ samples. The results show in Table 1. From Table 1 it can be seen that the *c* axis is compressed and the *a* axis is expanded with increasing annealing temperature T_{an} up to 550°C. This is one of evidences of the appearance of ordered phase in annealed FePt samples. The compress of *c* is strongest in the sample annealed at 550°C with the lowest c/a ratio of 0.97. With further increasing T_{an} , *c* and *a* have an opposite tendency.

The order parameter s, determined by arrangement of atoms over the entire crystal, can be estimated as the area ratio of the peaks (001) and (002) [16,17]. From this, the order parameter values

are deduced for annealed $Fe₆₀Pt₄₀$ nanoparticles as shown in Figure 3. The order parameter increases with increasing T_{an}, attains a maximum value of 0.54 at 550°C and decreases with further increasing T_{an}.

m \blacksquare and	c(A)	a(A)	c/a
450	3.697	3.761	0.980
500	3.674	3.777	0.972
550	3.670	3.780	0.970
600 650	3.697	3.720	0.990
	3.701	3.710	0.997

Table 1. The dependence of lattice parameters on annealing temperature T_{an} of Fe₆₀Pt₄₀ nanoparticles.

The as-prepared $Fe₆₀Pt₄₀$ exhibits soft magnetic properties as shown in Figure 4. Figure 5 shows the room-temperature hysteresis loops of $Fe_{60}Pt_{40}$ nanoparticles annealed at 450^oC to 650^oC. The annealed samples exhibit hard magnetic properties. When increasing the annealing temperature, the coercivity H_c increases and attains maximum value of 4.36 kOe at T_{an} = 550°C. The increase of H_c with increasing annealing temperature may be attributed to the forming of fct $L1_0$ phase as well as the chemical ordering in the nanoparticles. Further increase of the annealing temperature decreases the coercivity, which may be due to the formation of soft phase $Fe₃Pt$ at higher temperature. This dependence of coercivity on the annealing temperature is consistent with the behavior of changing of the order parameter s with the annealing temperature as discussed above.

The dependence of magnetic squareness $S = M_r/M_s$ on the annealing temperature is shown in Figure 6. In general, the annealing-temperature dependence of magnetic squareness is similar to that of H_c . The highest value for *S* of 0.87 is obtained for the sample annealed at 500°C. This value of *S* is higher than those obtained for L1₀ FePt and CoPt nanoparticles prepared by sonoelectrodeposition at room temperature [13,18].

Figure 3. The annealing-temperature dependence of the order parameter s for $Fe_{60}Pt_{40}$ nanoparticles.

Figure 4. Room-temperature hysteresis loop of as-prepared $Fe_{60}Pt_{40}$ nanoparticles.

Figure 5. Room-temperature hysteresis loops of $Fe_{60}Pt_{40}$ nanoparticles annealed at 450° C $\div 650^{\circ}$ C.

Figure 6. The annealing-temperature dependence of magnetic squareness $S = M_r/M_s$ of Fe₆₀Pt₄₀ nanoparticles.

4. Conclusion

The structure and hard magnetic properties of the $Fe₆₀Pt₄₀$ nanoparticles prepared by sonochemistry have been studied. The lattice parameters and the order parameter have been determined. The room-temperature coercivity started to increase abruptly from annealing temperature of 450 $^{\circ}$ C, has the highest value of 4.36 kOe at annealing temperature of 550 $^{\circ}$ C. The chemical order degree shows similar tendency, indicating the hard magnetic properties strongly depend on the order of the $L1_0$ phase of FePt nanoparticles.

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