Crystallographic Structural and Magnetic Properties of FePt Nanoparticles

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Abstract: Fe₆₀Pt₄₀ nanoparticles have been successfully prepared by sonochemistry method from the solution of Fe(C₂H₃O₂)₂ and H₂PtCl₆. The as-prepared sample then has been annealed at temperatures from 450°C to 650°C. Annealed samples have ordered L₁₀ phase with face-centered tetragonal (fct) structure and show hard magnetic properties. The nanoparticles annealed at 550°C show highest coercivity of 4.36 kOe measured at room temperature. The annealed-temperature dependence of the coercivity is consistent with the appearance of the order phase L₁₀ 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) L₁₀ structure. This ordered fct L₁₀ phase of FePt materials are normally obtained from the disordered face-centered 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, μ₀Mₛ, of 1.4 T, the Curie temperature Tₑ of 750 K and high crystalline anisotropy K₁, 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-
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$_2$H$_3$O$_2$)$_2$ and H$_2$PtCl$_6$. 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°C 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 diffractometer (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 Fe$_{60}$Pt$_{40}$. Figure 2 shows the X-ray diffraction patterns of as-prepared and annealed Fe$_{60}$Pt$_{40}$ 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 2θ = 40.8°. 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 Fe$_{60}$Pt$_{40}$ 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 Fe$_{60}$Pt$_{40}$ 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 $T_{an}$.

Table 1. The dependence of lattice parameters on annealing temperature $T_{an}$ of Fe$_{60}$Pt$_{40}$ nanoparticles.

<table>
<thead>
<tr>
<th>$T_{an}$ (°C)</th>
<th>c (Å)</th>
<th>$a$ (Å)</th>
<th>$c/a$</th>
</tr>
</thead>
<tbody>
<tr>
<td>450</td>
<td>3.697</td>
<td>3.761</td>
<td>0.980</td>
</tr>
<tr>
<td>500</td>
<td>3.674</td>
<td>3.777</td>
<td>0.972</td>
</tr>
<tr>
<td><strong>550</strong></td>
<td><strong>3.670</strong></td>
<td><strong>3.780</strong></td>
<td><strong>0.970</strong></td>
</tr>
<tr>
<td>600</td>
<td>3.697</td>
<td>3.720</td>
<td>0.990</td>
</tr>
<tr>
<td>650</td>
<td>3.701</td>
<td>3.710</td>
<td>0.997</td>
</tr>
</tbody>
</table>

The as-prepared Fe$_{60}$Pt$_{40}$ 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°C to 650°C. 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 Fe$_3$Pt 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 L1$_0$ FePt and CoPt nanoparticles prepared by sonoelectrodeposition at room temperature [13,18].

![Figure 3](image-url)  

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$^\circ$C÷650$^\circ$C.
4. Conclusion

The structure and hard magnetic properties of the Fe\textsubscript{60}Pt\textsubscript{40} 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\degree C, has the highest value of 4.36 kOe at annealing temperature of 550\degree C. The chemical order degree shows similar tendency, indicating the hard magnetic properties strongly depend on the order of the L1\textsubscript{0} phase of FePt nanoparticles.

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References