Phase Transformation and Magnetic Hardening in Isolated FePt Nanoparticles

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Abstract—Isolated monodisperse $L1_0$ FePt nanoparticles coated by carbon were obtained by adding enough surfactants that decomposed into carbon after the chemical synthesis and postannealing of the $A1$ FePt nanoparticles. The effect of isolation between FePt nanoparticles on the phase transition temperature and magnetic properties has been studied systematically by thermal, magnetic, and structural characterizations and analyses. It was found that the $A1$ to $L1_0$ phase transition temperature is dependent sensitively on the amount of isolation medium. The transition temperature shift reaches 150–200 °C from nonisolated particle assemblies to completely isolated particles, which may be attributed to the high activation energy of the phase transformation for the isolated particles.

Index Terms—Isolated $L1_0$ FePt nanoparticles, magnetic hardening, magnetocrystalline anisotropy, phase transformation.

I. INTRODUCTION

IRON–PLATINUM nanoparticles are a promising candidate for the future magnetic recording media with ultrahigh density beyond 1 Tbit/in² due to the high magnetocrystalline anisotropy ($K_u \sim 6 \times 10^6$ J/m³) [1], [2]. A key aspect of this technological development is to reduce the ferromagnetic particle size to 10 nm or less. The theoretical and experimental critical size room temperature ferromagnetic of FePt nanoparticles is about 3 nm [1], [3], while most other small magnetic particles are superparamagnetic at room temperature. Chemically synthesized FePt nanoparticles attract much attention because of their well-defined morphology since Sun *et al.* made the 2-D periodical nanoparticle arrays [4]–[10]. Unfortunately, as-synthesized nanoparticles take a disordered face-centered cubic (fcc) structure ($A1$ phase) that has vanishing magnetocrystalline anisotropy, and hence, they are superparamagnetic at room temperature [11]. Postannealing is therefore necessary to convert the $A1$ phase to the ordered $L1_0$ phase with face-centered tetragonal structure possessing high magnetocrystalline anisotropy. The heat treatments, however, always lead to undesired agglomeration or coalescence of the particles and also the concomitant loss

of the periodical assembly of chemically synthesized nanoparticles, which is disadvantageous for the application of magnetic recording media. Recently, the monodisperse $L1_0$ nanoparticles were obtained by several special methods, such as salt-matrix annealing [12], [13] and SiO₂ coating [14]. The principle of these methods is to prevent the sintering of the nanoparticles by adding separating media during high-temperature annealing. However, no attention has been paid on the effect of isolation on the $A1$ to $L1_0$ phase transition temperature and the magnetic hardening of isolated particles so far. For example, the coercivity of isolated particles does not increase significantly when the annealing temperature is lower than 700 °C [12]–[14], while the ordering temperature in thin films can be as low as 357 °C [15]. The significant increase of phase transformation temperature of the isolated particles was not understood yet and only some primary results were reported in our recent work [16]. In this paper, the isolation effect on the magnetic hardening and phase transformation of the FePt nanoparticles has been studied systematically by thermal, structural, and magnetic analyses of samples prepared with controlled amount of isolation medium. A simply annealing method by only adding enough surfactants was also introduced to develop the monodisperse $L1_0$ FePt nanoparticles in this study.

II. EXPERIMENTAL METHODS

The 8 nm disordered $A1$ FePt nanoparticles were synthesized by high-temperature decomposition of Fe(CO)₅ and reduction of Pt(acac)₂ in solution [2]. The detailed synthesis process is as follows: platinum acetylacetonate (197 mg, 0.5 mmol) and dioctylether (20 mL) were mixed and heated to 120 °C. Oleic acid (0.16 mL, 0.5 mmol), oleyl amine (0.17 mL, 0.5 mmol), and iron pentacarbonyl (1.0 mmol) were added and the mixture was heated to reflux at 298 °C for 1 h before cooling to room temperature under the argon blanket. The black product was precipitated by adding ethanol and was separated by centrifugation. The particles were redispersed in hexane. This process was repeated three times to remove the surfactants. Each 10 mL dispersed FePt nanoparticles were then dispersed in different amount of surfactants to control the degree of isolation status. In this study, we always added equal amount of oleic acid (OAc; 0.03–5 mL) and oleyl amine (OAm; 0.03–5 mL). The heat treatments were carried out at different temperatures under forming gas (93% Ar + 7% H₂). The composition of nanoparticles (around Fe₅₂Pt₄₈) was checked by energy-dispersive X-ray spectra (EDS) analysis. The morphology and structure were determined by transmission electron microscopy (TEM), selected-area electron diffraction (SAED), and powder X-ray diffraction

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Fig. 1. XRD patterns of (a) as-synthesized nanoparticles and 600 °C $\times$ 1 h annealed samples with different amount of surfactant addition: (b) without surfactant; (c) 0.12 mL OAc plus 0.12 mL OAm; (d) 5 mL OAc plus 5 mL OAm.

(XRD) with Cu $K\alpha$ radiation. Samples for TEM analysis were prepared by depositing a drop of the nanoparticles solution onto an amorphous carbon-coated copper grid from their hexane dispersions after ultrasonic. Small-angle X-ray scattering (SAXS) was also performed on Rigaku Ultima IV to determine the particle size distribution. The magnetic properties were characterized using a superconducting quantum interference device magnetometer with field strength up to 7 T. The differential scanning calorimetry (DSC) used in this study is a NETZSCH instrument (DSC 404 Pegasus®). The samples were placed on a Pt sample pan and were then heated to temperatures up to 800 °C at controlled scan rate of 5–50 K/min.

III. RESULTS AND DISCUSSION

A. Development of Isolated $L1_0$ FePt Nanoparticles

Our previous study showed that carbon-coated FePt particles were formed by annealing the salt-surfactant-particle mixtures, as a consequence of decomposition of the surfactants into carbon [17]. The carbon coating makes the FePt particles highly thermally stable. The role of surfactants in this study is similar to that reported in our previous study. Fig. 1 shows the XRD patterns of the as-synthesized FePt nanoparticles and the 600 °C annealed samples with different amount of surfactant additions. It was determined by Scherrer formula that the grain (particle) size of the as-synthesized particles was around 7.5 nm. After the annealing, particles without surfactant separation had serious sintering and significant grain growth (to around 23 nm). However, it was interesting to find that the grain growth was effectively suspended by addition

Fig. 2. Dependence of grain size on the annealing temperature with different amount of surfactant addition. The symbol star represents the grain size of the two-step annealed nanoparticles.

of the surfactants. For example, the grain size of the annealed sample is only around 8.3 nm by addition of 0.12 mL OAc and 0.12 mL OAm to 10 mL as-synthesized FePt nanoparticle solution. If the surfactant amount is further increased to 5 mL OAc and 5 mL OAm, the grain size decreases to 8.1 nm.

Fig. 2 compares the grain size change upon annealing of the nanoparticles with different amount of surfactant additions. It shows that the grain size increases linearly from 8 to 50 nm by increasing the annealing temperature (T_a) from 400 to 800 °C if no surfactants are added. The grain growth was significantly suppressed after adding a small amount of surfactants (for example, 0.12 mL OAc and 0.12 mL OAm, as shown in Fig. 2). By adding a large amount of surfactants, the grain size remained almost unchanged after annealing at temperatures below 600 °C. The size was only about 17 nm even after being annealed at 800 °C for 1 h, which is much smaller than that of annealed particle without surfactant addition (50 nm). These results indicate the importance of the surfactant addition in controlling particle sintering and grain growth during annealing.

Fig. 3 shows the TEM images of the as-synthesized nanoparticles and the particles annealed at different temperatures with addition of 5 mL OAc and 5 mL OAm to a 10 mL as-synthesized FePt nanoparticle solution. It was found that the particle size of the as-synthesized particles is around 8 nm [Fig. 3(a)]. Interestingly, the particle size did not increase after annealing at 600 °C [Fig. 3(b)], which agrees with the XRD analysis. The stability of the nanoparticles is attributed to the formation of carbon shell since the surfactants OAc and OAm mostly decompose at about 400–500 °C [18], [19]. However, the sintering of nanoparticles was still observed in the 800 °C annealed samples, as shown in Fig. 3(c), while the high-temperature annealing is necessary to develop the $L1_0$ structure from the disordered $A1$ structure of the as-synthesized particle. As one can see from Fig. 3(d)

Fig. 3. TEM images of (a) as-synthesized nanoparticles, the samples after annealing at (b) 600 °C for 1 h and (c) 800 °C for 1 h. SAED patterns of the (d) 600 °C and (e) 800 °C annealed particles.

and (e), the (0 0 1) and (1 1 0) rings are very weak, which means the ordering of the FePt nanoparticles is low even after the annealing at 600 and 800 °C for 1 h.

As mentioned earlier, sintering of the nanoparticles was observed in high-temperature annealed samples even with surfactants addition. This may be caused by the loss of surfactants during fast heating to high temperature, and thus, less carbon shells were left to prevent sintering at high temperatures. To obtain homogenous particles with fully ordered $L1_0$ structure, we used a two-step treatment: first, the mixture of as-synthesized particles with 5 mL OAc and 5 mL OAm addition was annealed at 500–600 °C for 1 h to form the carbon shell; the dry powders were then annealed at high temperature for long time, for example, 800 °C for 2 h. Fig. 4(a) shows the TEM image of the FePt nanoparticles after the two-step annealing treatment. It can be seen that homogenous nanoparticles with particle size about 8 nm were obtained. The particle size agrees with that determined by Scherrer formula using the XRD pattern (as shown by symbol star in Fig. 2). The high-resolution TEM image [Fig. 4(b)] shows that each particle is single crystalline. The (0 0 1) and (1 1 0) rings in the SAED pattern [Fig. 4(c)] are stronger than those of one-step annealed particles, which confirms that the particles are transferred to $L1_0$ structure with higher chemical ordering than the particles annealed by one-step method. EDS analysis shows the composition of the $L1_0$ nanoparticles is around $\text{Fe}_{52}\text{Pt}_{48}$, which is the same as the as-synthesized nanoparticles.

Fig. 5 shows the SAXS patterns of the as-synthesized and two-step annealed nanoparticles. The solid lines represent the experimental results, while the dash lines are the simulated data obtained from the average diameter and dispersion of the particles. The size distributions of the particles were obtained by analyzing the scattering profile, as shown in the inset of Fig. 5. The average diameters were estimated to be 8.5 nm for the

Fig. 4. (a) TEM images of two-step annealed (600 °C $\times$ 1 h plus 800 °C $\times$ 2 h) particles; (b) high-resolution TEM image of an individual particle; (c) selected-area electronic diffraction pattern of the annealed particles; (d) EDS spectra of the annealed particles.

Fig. 5. Small-angle X-ray scattering patterns of the as-synthesized and two-step annealed nanoparticles. The solid and dash lines represent experimental and fitted data. The inset gives the size distribution of the as-synthesized and annealed nanoparticles.

as-synthesized particles and 9.0 nm for the two-step annealed particles, which approximately agree with the XRD and TEM analyses. The deviation only increased from 9.8% to 12.8% for the annealed particles.

Fig. 6. Dependence of S on the annealing temperature with different amount of surfactant addition.

B. Phase Transformation and Magnetic Hardening in Isolated FePt Nanoparticles

As one can see in Fig. 1, the surfactant addition significantly reduced the particle sintering and grain growth during the annealing. However, it was also observed that the intensity of the (0 0 1) and (1 1 0) superlattice peaks of the $L1_0$ phase decreases with increasing amount of surfactant additions by annealing at the same conditions, indicating that the chemical ordering degree of the isolated particles is lower than the sintered particles. To evaluate the effect of surfactant addition on the atomic-scale chemical ordering of the $L1_0$ phase in a quantitative way, the ordering parameter S was determined by $S \cong 0.85(I_{001}/I_{002})^{1/2}$ [13], [20], where I_{001} and I_{002} are the integrated intensity of (0 0 1) and (0 0 2) diffraction peaks from XRD patterns, respectively. S is zero for the disordered phase while it is one for a fully ordered $L1_0$ phase. Fig. 6 shows the dependence of chemical ordering parameter on annealing temperature with different amount of surfactant additions. It was found that the phase transformation from A1 to $L1_0$ phase starts from 400 °C to 450 °C for the particles without surfactant addition. The chemical ordering parameter S increases from 0.65 ± 0.05 to 0.98 ± 0.02 with increasing annealing temperature from 450 °C to 650 °C. With the addition of 0.12 mL OAc and 0.12 mL OAm, the phase transformation starts from 500 °C, while S is only 0.20 ± 0.05 . Further increase of the surfactant addition to 5 mL OAc and 5 mL OAm suppresses the starting temperature of phase transformation to 550 °C with $S = 0.36 \pm 0.08$. S is only about 0.8 even after annealing at 700 °C–800 °C, which is still far from fully ordered. By comparing the S – T_a curves as show in Fig. 6, one can see that the shift of phase transformation temperature reaches about 150 °C–200 °C due to the surfactant addition if we choose samples with the similar S for comparison (for example, $S = 0.6$ – 0.7). Fig. 6 also shows the chemical ordering parameter of the two-step annealed particles for comparison (symbol star). It was found that the highly ordered particles ($S = 0.93 \pm 0.04$) were obtained by the two-step annealed pro-

Fig. 7. (a) DSC traces for the FePt nanoparticles without surfactant, with addition of 0.03 mL OAc plus 0.03 mL OAm and 5 mL OAc plus 5 mL OAm. The sample with 5 mL OAc plus 5 mL OAm surfactants addition was annealed at 500 °C for 1 h before the DSC measurement to remove the surfactants. (b) The DSC traces with different scanning rate of the particles with addition of 5 mL OAc and 5 mL OAm after annealing at 500 °C for 1 h. The inset gives the Kissinger plots of the samples with and without surfactant additions.

cess. It should be mentioned that the chemical ordering of the two-step annealed particles is higher than the one-step annealed particles since longer annealing time always results in higher ordered structure, which agrees with the SAED analysis.

To understand the phase transformation in the isolated FePt nanoparticles, DSC traces were measured for the FePt particles with different amount of surfactant additions and different scanning rate (Fig. 7). As shown in Fig. 7(a), there are two sharp exothermic peaks for the particles without addition of extra surfactant. The strong peak around 417 °C is attributed to the transformation from A1 to $L1_0$ phase of most particles that have sintered together while the peak around 610 °C is due to some isolated particles surrounded with surfactants. It should be noted here that a small amount of surfactant still exists in the as-synthesized FePt nanoparticles even when the particles were washed three times. However, only one sharp peak around 630 °C was observed for the particles with the addition of 0.03 mL OAc plus 0.03 mL OAm and the samples with addition of 5 mL OAc plus 5 mL OAm after annealing at 500 °C for 1 h. This observation may be related to the fact that separation media suppressed the phase transformation of the FePt nanoparticles. The difference in phase transition temperature of the samples

with and without addition of surfactant is around 200 °C, which is consistent with the measurements in chemical ordering and magnetic hardening. In addition to the sharp peaks, it was also found that there is a broad peak from 340 to 480 °C for both the samples without surfactant addition and with addition of 0.03 mL OAc plus 0.03 mL OAm. This may be attributed to the decomposition of the surfactants, as also reported in [17] and [18]. There is also indirect evidence that the broad peak was not observed in the DSC curve of the 500 °C preannealed samples with addition of 5 mL OAc and 5 mL OAm since the surfactants almost decomposed during preannealing.

As discussed earlier, the particle separation suppressed the phase transformation from $A1$ to $L1_0$ structure. Thus, understanding the thermal dynamics of the phase transformation is important in controlling the $L1_0$ structure formation in the isolated FePt nanoparticles. Fig. 7(b) shows the DSC traces with different scanning rate of the particles with addition of 5 mL OAc and 5 mL OAm. Using the Kissinger plot [21]–[23], which is based on the analysis of temperature shift of the DSC peak with heating rate, the activation energy E_a of the phase transformation can be obtained by using the linear relationship $\ln(T_p^2/\beta) = E_a/k_B T_p$, where T_p is the temperature of the transformation peak, β is the heating rate, and k_B is the Boltzmann's constant. The corresponding Kissinger plots of the FePt nanoparticles without and with surfactant additions are shown in the inset of Fig. 7(b). The obtained activation energy of the particles without surfactant addition is 2.07 ± 0.09 eV, which is quite similar to that of the FePt thin films [24], [25]. However, the activation energy of the particles with surfactant additions significantly increased to 2.87 ± 0.15 eV, which is of about 38% enhancement. The increased activation energy of the isolated FePt nanoparticles indicates a more difficult phase transformation from $A1$ to $L1_0$ structure, which may be the major reason for the higher phase transformation temperature of the isolated FePt nanoparticles compared to that of the sintered particles.

The magnetic hardening of FePt particles is closely related to the phase transformation from the disordered $A1$ structure to the ordered $L1_0$ structure. Fig. 8 shows the room temperature demagnetization curves of the annealed samples without surfactants and with addition of 5 mL OAc and 5 mL OAm. It was noted that the demagnetization curves of the annealed samples without surfactant are smooth while those with addition of surfactants have kinks near zero magnetic field, which is similar to the salt-matrix annealed monodispersed $L1_0$ FePt nanoparticles [12], [13]. This may be attributed to the composition inhomogeneity in the isolated FePt particles. In other words, the annealing led to a homogenous composition distribution in the samples without surfactant due to atomic diffusion, while the formed carbon shell prevents the diffusion between particles in the samples with surfactant addition, and thus, a considerable composition deviation from $\text{Fe}_{50}\text{Pt}_{50}$ in some particles.

As shown in Fig. 8, one can see that the coercivity of FePt particles without surfactants was around 3.4 kOe after annealing at 450 °C and increased fast with increasing annealing temperature up to 550 °C. However, the coercivity of the FePt particles with the addition of 5 mL OAc and 5 mL OAm was only 0.7 kOe even after being annealed at 650 °C. The high coercivity was

Fig. 8. Room temperature demagnetization curves of the annealed samples (a) without surfactants and (b) with addition of 5 mL OAc and 5 mL OAm.

Fig. 9. Dependence of H_c on the annealing temperature with different amount of surfactant additions.

only obtained by annealing at temperatures higher than 700 °C. Fig. 9 summarizes the dependence of coercivity on the annealing temperature with different amount of surfactant additions. The onset temperature of magnetic hardening was significantly shifted to high temperature when large amount of surfactants were added. The shift of temperature reaches 200 °C–250 °C from the nonisolated particle assemblies to completely isolated particles if we choose samples with coercivity of 10 kOe for comparison. As can be seen, the dependence of H_c on annealing temperature is quite similar to that for S (as shown in Fig. 6). This means that both the magnetic hardening and the $A1$ to $L1_0$ phase transformation of FePt nanoparticles depend sensitively on the isolation conditions.

The development of the high magnetocrystalline anisotropy is very important for applications of the FePt nanoparticles since the disordered FePt nanoparticles are superparamagnetic at room temperature. As one can see in Fig. 9, the high coercivity means that the high magnetocrystalline anisotropy was

Fig. 10. Temperature-dependent H_s of the particles annealed at different temperatures with addition of 5 mL OAc and 5 mL OAm.

obtained. However, it was found that the magnetic hardening temperature and chemical ordering temperature are different if one compares Figs. 6 and 9. For example, the chemical ordering parameter of the FePt nanoparticles with 5mL OAc and OAm addition increases to 0.36 ± 0.08 after annealing at 550 °C. However, the coercivity is still close to zero until the annealing temperature increases to 700 °C. The study of low-temperature magnetic properties can help explain the difference between the $S \sim T_a$ and $H_c \sim T_a$ curves. Fig. 10 shows the temperature dependence of H_c on the measurement temperatures. It shows that the as-synthesized particles are superparamagnetic at room temperature and H_c is only about 1.3 kOe even at 5 K. Annealing at 500 °C has only slightly increased H_c to 1.9 kOe at 5 K, which means that the phase transformation did not really start. However, the H_c at 5 K significantly increased to 13.1 kOe while the room temperature H_c is only 1.4 kOe by annealing at 600 °C, which means that the $L1_0$ structure was partially obtained. This is consistent with the chemical ordering measurement in Fig. 6. Further increase of annealing temperature led to high H_s and stable temperature dependence of H_c due to the development of highly ordered $L1_0$ structure. The anisotropy constant K_u of the FePt nanoparticles can be approximately determined by fitting the temperature dependence of coercivity using Sharrock formula [3], [26], [27]. It was found that K_u of the as-synthesized nanoparticles is only $0.42 \pm 0.17 \times 10^6$ J/m³. K_u increases to $2.53 \pm 0.47 \times 10^6$ J/m³ after annealing at 600 °C, which means that the phase transformation already started at 600 °C even the room-temperature coercivity was low. The two-step annealing process can further increase K_u to 7.1×10^6 J/m³, which is of the order of the anisotropy constant of bulk FePt alloy.

IV. CONCLUSION

In summary, isolated FePt nanoparticles coated by carbon have been produced by adding extra amount of surfactants and by annealing the particle-surfactant mixtures. It is found that magnetic hardening and the $A1$ to $L1_0$ phase transition in the FePt nanoparticles depend sensitively on the isolation condition.

The shift of magnetic hardening and phase transformation temperature reaches 150 °C–200 °C upon addition of the isolation medium into the FePt nanoparticles. The high phase transformation temperature of the isolated nanoparticles is attributed to the high activation energy.

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