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Magnetic properties of dilute FePt:C nanocluster films

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Nanocluster-assembled dilute $(\mathrm{FePt})_x\mathrm{C}_{100-x}$ films with $30 \geqslant x \geqslant 5$ (x denotes volume fraction) were produced using a gas-aggregation technique. FePt clusters with an average size of about 4.0 nm (with standard deviation $\sigma/d=0.09$) were embedded in high volume fraction of carbon matrix, which is used to isolate the FePt clusters. Postdeposition annealing was used to realize the high-anisotropy $L1_0$ phase. Single-crystal features of well-isolated clusters were observed by transmission electron microscope (TEM) in annealed dilute films. The coercivity of the films annealed at 700 °C for 5 min was a few hundred Oersteds at room temperature, while a coercivity about 4 kOe was observed at low temperature 10 K for $x=5$, indicating partial ordering of the clusters. The coercivity strongly depends on annealing temperature and annealing time, and increases with decreasing FePt volume fraction. For a dilute FePt cluster film with $x=5$ annealed at 700 °C for 60 min, a room-temperature coercivity of about 30 kOe and low-temperature (10 K) coercivity of about 40 kOe were obtained, which imply an anisotropy field of 83 kOe, assuming the clusters are noninteractive. The results are discussed by comparing with simulations for Stoner–Wohlfarth particles. © *2005 American Institute of Physics*. [DOI: 10.1063/1.1855457]

I. INTRODUCTION

Recently interest in films based on magnetic nanoclusters has grown enormously with increasing attention devoted to the extension of the magnetic recording areal density to 1 Tb/in^2, which depends likely on the development of high-anisotropy films with uniform size clusters or grains below 10 nm that are exchange decoupled or weakly coupled.[1–3] The equiatomic FePt nanoclusters with $L1_0$ phase are a promising candidate for such media because $L1_0$ ordered FePt phase has an anisotropy constant K_u of $\sim 7 \times 10^7$ erg/cm^3,[4] which helps to meet the requirement for both high signal-to-noise ratio and thermal stability of the media

While many efforts have been made to fabricate oriented $L1_0$ FePt nanoparticle or nanograin films with some exchange coupling,[5,6] understanding the magnetic properties of a collection of well-isolated clusters is of similar interest for exploring FePt clusters as a potential media for extremely high density recording. We have prepared FePt nanoclusters with small average cluster size ($d\sim 4$ nm) using a gas-aggregation technique.[7] The FePt clusters are nearly monodispersed with a narrow Gaussian size distribution (standard deviation $\sigma/d\sim 0.09$). In this paper dilute FePt:C nanocluster films have been prepared, in which the FePt volume fraction ranged from 5% to 30%. Carbon is used as matrix for isolating the FePt clusters to decrease the exchange interaction, and to reduce the cluster growth during high-temperature annealing.

II. EXPERIMENT

Dilute FePt:C nanocluster films were prepared with a multilayer method in which FePt cluster layers (produced by the gas-aggregation technique) and C layers (produced by a normal sputtering gun) were alternately deposited onto a Si substrate. The initial nominal dimensions of the multilayer are given by $[\mathrm{FePt}(x\ \mathrm{nm})/\mathrm{C}(y\ \mathrm{nm})]_{\times 12}$, where $x \approx 0.5$ nm and $1.2\ \mathrm{nm} \leqslant y \leqslant 9.5$ nm, corresponding to FePt volume fraction changing from 5% to 30%. FePt cluster content $x \approx 0.5$ nm in each layer implies only a partial occupancy of the cluster layer, thus allowing individual cluster being well isolated by C matrix.

In order to obtain $L1_0$ ordered FePt:C cluster films, postdeposition annealing was carried out using a rapid-thermal-annealing oven. Crystal structure and nanostructure of the cluster films were examined with a Rigaku x-ray diffractometer (XRD) and a JEOL 2010 transmission electron microscope (TEM). For TEM observations, clusters were directly deposited onto carbon-coated films supported by Cu grids. Magnetic properties were measured using a superconducting quantum interference device magnetometer operated between 5 and 300 K in magnetic fields up to 70 kOe.

III. RESULTS AND DISCUSSION

Figure 1 shows zero-field-cooled/field-cooled (ZFC/ZF) magnetization curves for as-deposited FePt:C cluster films with FePt volume fraction of 5% measured in a temperature range 5–300 K. The film is superparamagnetic at room temperature but show a peak in ZFC curves at low temperature. This peak marks a blocking temperature $T_b=36.5$ K under an external field of 200 Oe. For noninteractive single domain particles, $T_b=K_uV_p/25k_B$,[8] where K_u is anisotropy constant, V_p is the particle volume, and k_B the Boltzman constant. Taking the average cluster diameter as 4 nm, the K_u can be estimated as 3.76×10^6 erg/cm^3. With increase of volume fraction of FePt clusters, T_b increases and the peak width broadens somewhat, suggesting that the effective average

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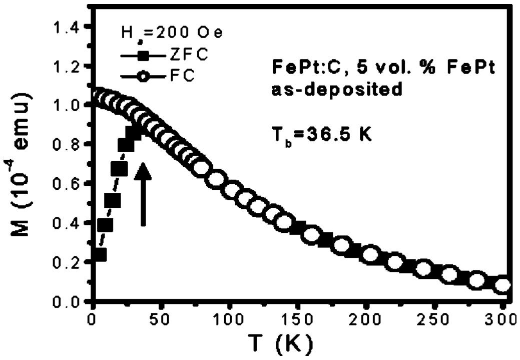

FIG. 1. Zero-field-cooled (ZFC) and field-cooled (FC) magnetization curves measured at $H=200$ Oe for as-deposited FePt:C cluster film with 5 vol % FePt.

cluster size increases with the increase of cluster volume fraction due to the coupling of clusters.

Figure 2 shows the temperature dependence of coercivity for FePt:C cluster films with 5 and 30 vol % FePt annealed at 700 °C for 5 min. At room temperature, a few hundred Oersteds of coercivity was observed for both films; at low temperature (10 K), the coercivity reaches 4 kOe for 5 vol % FePt film and 5.6 kOe for 30 vol % FePt, respectively. Increase of FePt cluster volume fraction increases the coercivity. These results suggest the partial ordering of $L1_0$ phase at short-time (5 min) annealing.

XRD measurement showed that as-deposited clusters were disordered fcc structure. High anisotropy $L1_0$ ordered clusters were obtained via postdeposition annealing. The fct structure peaks are present indicating the $L1_0$ ordered phase by thermal annealing. Figure 3 shows TEM images of FePt:C cluster films with 5 vol % FePt (a) and 20 vol % FePt (b) annealed at 700 °C for 10 min. Most clusters are well isolated with single crystal $L1_0$ structure. The arrows shown in Fig. 3(a) indicate the clusters with lattice fringe observable, suggesting a single-crystal structure. With an increase of FePt volume fraction, some large clusters were observed, probably due to the sintering of some clusters in contact with each other during deposition [Fig. 3(b)].

In-plane coercivity of dilute FePt:C films was measured at room temperature. Figure 4(a) shows the effect of FePt volume fraction on coercivity for the films annealed at 700 °C for different times. The coercivity increases with increase of annealing time, indicating the increase of $L1_0$ ordering degree. The coercivity tends to decrease with increase of FePt volume fraction, suggesting some exchange coupling among the clusters in FePt-rich films. This is in agreement with TEM observations shown in Fig. 3(b), i.e., some larger clusters due to a sintering effect may cause the decrease of coercivity. Figure 4(b) shows the effect of annealing temperature T_A on coercivity for the films with 5 and 30 vol % FePt, annealed at different temperatures for 10 min. Coercivity increased very rapidly from less than 2 kOe to higher than 17 kOe as T_A increased from 675 °C to 700 °C, indi-

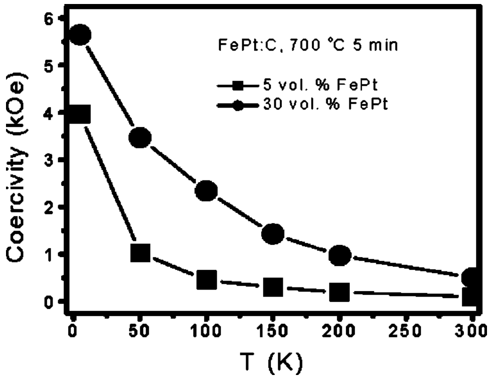

FIG. 2. Temperature dependence of coercivity for FePt:C cluster films with 5 vol % and 30 vol % FePt, annealed at 700 °C for 5 min.

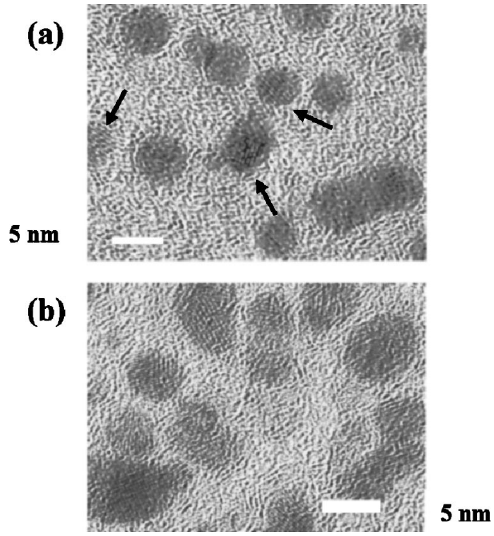

FIG. 3. High resolution TEM images of FePt:C cluster films with 5 vol % FePt (a) and 20 vol % FePt (b), annealed at 700 °C for 10 min.

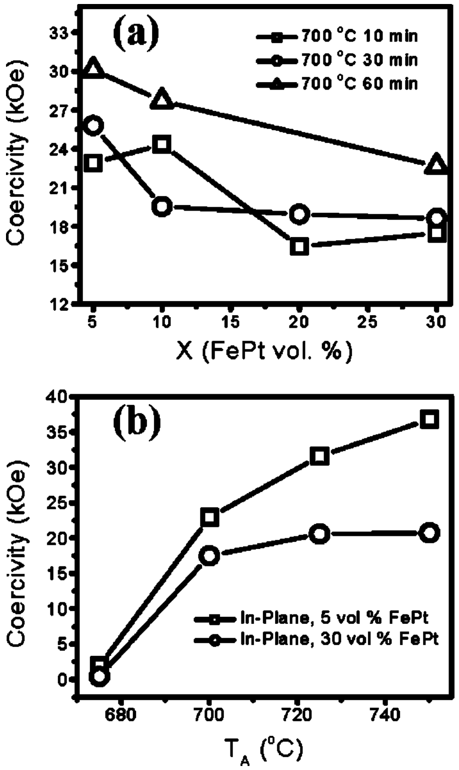

FIG. 4. In-plane coercivity of FePt:C cluster films: (a) effect of FePt volume fraction on coercivity for films annealed at 700 °C for different time, (b) effect of annealing temperature on coercivity for films with 5 vol % and 30 vol % FePt, annealing time: 10 min.

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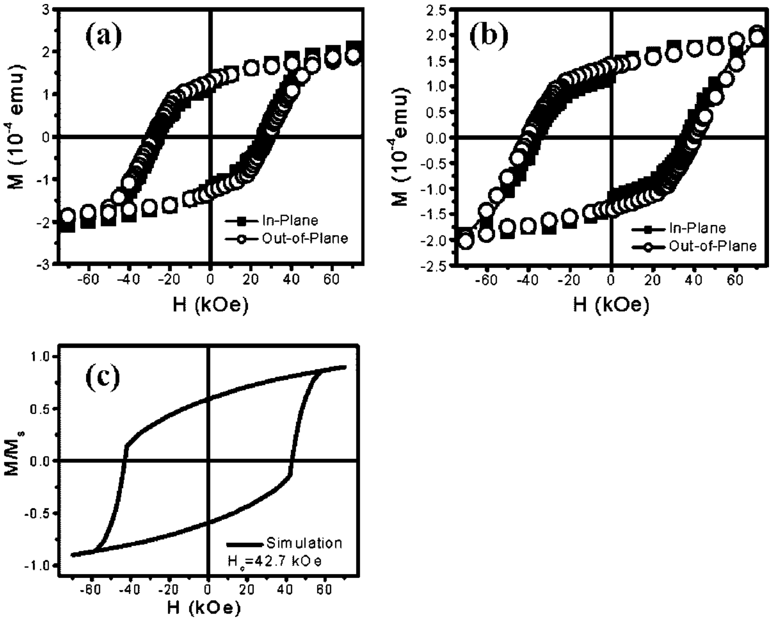

FIG. 5. In-plane and perpendicular hysteresis loops of FePt:C cluster films with 5 vol % FePt annealed at 700 °C for 60 min: (a) measured at room temperature and (b) at 10 K; (c) hysteresis loop obtained by numerical simulation based on Landau–Lifshtz–Gilbert equation.

cating that most ordering was completed at 700 °C annealing. The coercivity increased slowly with further increase of T_A for the film with 5 vol % FePt. However, it tends towards saturation for a film with 30 vol % FePt. This result suggests that high anisotropy $L1_0$ ordered FePt clusters are well isolated and decoupled in dilute films with 5 vol % FePt, which is in agreement with TEM observations shown in Fig. 3(a).

Figure 5 shows the in-plane and perpendicular hysteresis loops of the FePt:C film with 5 vol % FePt annealed at 700 °C for 60 min, measured at room temperature (a) and 10 K (b). Both in-plane and perpendicular loops are similar, indicating that the easy axes of the FePt clusters are distributed randomly, which is in agreement with the XRD measurement. A perpendicular coercivitity of about 29 kOe at room temperature and 40 kOe at 10 K are achieved. This result indicates the high degree of $L1_0$ ordering after annealing for a relatively long time (>10 min). The loops are not saturated at an applied field of 70 kOe. Assuming these isolated clusters are noninteracting and the formula $H_c = 0.48H_k$ can be applied,[9,10] an anisotropy field of 83 kOe is estimated. A numerical simulation is performed by running the NIST OOMMF code based on Landau–Lifshtz–Gilbert equation.[11] The $L1_0$ FePt spheres are assumed having diameter of 4.5 nm, and randomly orientated in a nonmagnetic matrix without intergranular exchange interactions. The parameters are chosen as anisotropy constant $K_u = 4 \times 10^7$ erg/cm^3, exchange constant $A = 1 \times 10^{-6}$ erg/cm, and saturation magnetization $M_s = 900$ emu/cm^3. The FePt spheres occupy 5% of the total volume. The simulation result is shown in Fig. 5(c), which shows a Stoner–Wohlfarth-like hysteresis for randomly oriented particles. The simulation yields a coercivity of 42.7 kOe, which is close to the experimental result of 40 kOe at 10 K. This indicates that the 5% volume fraction FePt clusters in a C matrix behave essentially as noninteractive Stoner–Wohlfarth particles.

IV. SUMMARY AND CONCLUSIONS

Magnetic properties and nanostructure of $L1_0$ ordered dilute FePt:C nanocluster films have been investigated. Single-crystal features of $L1_0$ FePt clusters that are well isolated by the C matrix were observed in the annealed film with 5 vol % FePt. A sintering effect was observed with increase of FePt volume fraction due to some clusters in contact during deposition. The room-temperature coercivity of the films can be readily tailored in a large range by controlling the annealing temperature and annealing time, and C isolation. Simulation result suggests a Stoner–Wohlfarth-like behavior in 5 vol % FePt cluster film. Such system may show more interesting nanomagnetism if the orientation of the clusters can be controlled, which needs to be further investigated.

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