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Nanostructure and Magnetic Properties of FePt : C Cluster Films

Yingfan Xu, M. L. Yan, and D. J. Sellmyer, Member, IEEE

Abstract-Magnetic properties and nanostructure of FePt:C cluster-deposited films with C volume fraction of 7%, 14%, 33%, and 45% have been studied. As-deposited FePt : C films were prepared by a multilayer method in which FePt layers were deposited from a cluster source employing a gas-aggregation technique and C layers from a normal sputtering gun. In the as-deposited films, FePt clusters with fcc structure are embedded in the C matrix. The high anisotropy FePt L1₀ cluster structure was realized in the films via post-deposition annealing and the nanostructure of the films was observed by high-resolution transmission electron microscope (TEM). The results for a film with 45 vol. % C showed that FePt clusters are well separated by C matrix and the cluster diameter is about 4.5 nm. The coercivity increases with increase of annealing temperature; coercivities larger than 9 kOe were achieved in the films after annealing at a temperature of 700 °C and above. Magnetization reversal of the films was studied by moment-decay measurements and the data were fitted with the Sharrock formula. For the film with 45 vol. % C annealed at 625 °C, the thermal stability factor $K_u V^*/k_B, T$ activation volume V^* , and anisotropy constant K_u are 231, 0.83 × 10⁻¹⁸ cm³ and 1.2 × 10⁷ erg/cm³, respectively.

Index Terms—FePt, high anisotropy, L1₀ structure, nanocluster, thermal stability.

I. INTRODUCTION

 \mathbf{T} e FePt nanoparticles with the high-anisotropy L1₀ ordered phase have significant potential for extremely high-density magnetic recording (EHDR) defined as approaching an areal density of 1 Tb/in². This follows from the favorable properties of FePt including its magnetic crystalline anisotropy constant (about 7×10^7 erg/cm³) and good chemical stability [1], [2]. In order to improve the signal-to-noise ratio, the medium for EHDR requires magnetically decoupled or weakly coupled particles having sizes below 10 nm with an extremely uniform size distribution and thermal stability at room temperature [3]. We have prepared FePt and CoPt nanoclusters with small cluster size (ranging from 3 to 6 nm in diameter) by using a gas-aggregation technique, in which magnetron sputtering is employed in the cluster source [4], [5]. Fig. 1 shows an example of highresolution TEM image of FePt nanoclusters with narrow size distribution (standard deviation $\sigma/d \sim 0.1$, where d is mean diameter).

In this study, FePt: C nanocluster films with different C volume fraction have been prepared. Carbon is used as matrix

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Fig. 1. TEM image of FePt nanoclusters deposited at a power of 200 W. Shown below is corresponding cluster size distribution.

for the purpose of isolating the FePt clusters to decrease the exchange interaction, and to reduce the cluster growth during high-temperature annealing.

II. EXPERIMENT

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 $[\text{FePt}(x \text{ nm})/C(y \text{ nm})]_{\times 6}$, where $x \approx 1 \text{ nm}$ and 0.075 < y < 0.825 nm, corresponding to C volume fraction changing from 7% to 45%. Since the FePt clusters are approximately spherical with $d \sim 4.5$ nm, prepared with a power of 100 W, $x \approx 1$ nm implies only a partial occupancy of the cluster layers. The high-anisotropy FePt L10 structure was obtained with post-deposition annealing using a rapid-thermal-annealing oven. Crystal structure and nanostructure of the FePt: C films were examined with a Rigaku X-ray diffractometer (XRD) using CuK_{α} radiation and a JEOL 2010 transmission electron microscope (TEM). Magnetic properties and moment-decay measurements were carried out with an alternative gradient force magnetometer (AGFM).



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Fig. 2. XRD $(\theta - 2\theta)$ scans of FePt : C cluster films with 14 vol. % C annealed at different temperature for 10 min.



Fig. 3. XRD $(\theta - 2\theta)$ scans of FePt: C films with different C volume fraction annealed at 750 °C for 10 min.

III. RESULTS AND DISCUSSION

XRD measurements confirmed the ordering of the L1₀ structure. Fig. 2 shows the XRD ($\theta - 2\theta$) scans of FePt : C cluster films with 14 vol. % C annealed at 650 °C, 700 °C, and 750 °C for 10 min, respectively. The intensity of the L1₀-structure peaks increases with annealing temperature, indicating that the L1₀ ordering started to develop at 650 °C, and trended toward a completion at temperatures higher than 700 °C. The peak width decreased clearly after annealing at 750 °C, suggesting the cluster growth in the films annealed at high temperature. Fig. 3 shows XRD spectra of the FePt : C cluster films with different C volume fraction annealed at 750 °C for 10 min. The intensity of the L1₀-structure peaks decreases with increase of C volume fraction, while the peak width increases with the increase of C content, suggesting that increase of carbon content reduced the FePt cluster growth.

Fig. 4 shows the dependence of in-plane and perpendicular coercivities of FePt : C films with 45 vol. % C on the annealing



Fig. 4. Effect of annealing temperature on in-plane and out-of-plane coercivities of FePt : C cluster films with 45 vol. % C. Annealing time: 10 min.



Fig. 5. (a) TEM bright field image of FePt : C cluster film with 45 vol. % C and (b) the high-resolution image. The film was annealed at $650 \text{ }^{\circ}\text{C}$ for 10 min.

temperature T_A . Both in-plane (H// film plane) and out-ofplane $(H\perp$ film plane) coercivities increased with increase of annealing temperature. The coercivities increased rapidly with T_A between 600 °C and 650 °C, then increased slowly with T_A above 650 °C. The in-plane coercivity is about 2 kOe higher than the out-of-plane coercivity. The in-plane coercivity larger than 9 kOe was achieved in the films after annealing at a temperature of 700 °C and above. Since the maximum magnetic field applied to the sample was 13 kOe during AGFM measurement, it is possible that magnetization was not saturated, which might result in smaller measured value of coercivity for films annealed at higher temperature.

Fig. 5 shows the TEM plan-view images for the FePt: C film with 45 vol. % C annealed at 650 °C for 10 min. FePt clusters are embedded in C matrix. Shown in Fig. 5(b) is the high-resolution TEM image. It shows that single FePt clusters with diameter about 4.5 nm were well separated by amorphous C matrix; the cluster growth during high temperature annealing has been remarkably reduced with addition of higher volume fraction of C.



Fig. 6. Effect of annealing temperature on thermal stability and magnetic anisotropy field of FePt:C cluster films with 45 vol. % C. Annealing time: 10 min.

As grain size gets smaller and smaller with increased recording areal density, the magnetic moment in grain may reverse spontaneously via thermal activation. Therefore, understanding the magnetization reversal behavior of media is of essential importance to control and design new media with desired properties and good thermal stability. We carried out a set of moment decay measurements with the AGFM for FePt: C films with 45 vol. % C. The data were fitted with Sharrock formula [6] and the thermal stability factor and anisotropy constant were extracted from the fitting parameters. The detailed experiment and fitting procedures can be found in [7]. Fig. 6 shows the annealing effect on the thermal stability factor $K_u V^* / k_B T$ and anisotropy field H_k for the FePt:C films with 45 vol. % C. H_k was calculated from $H_0 = 0.48 H_k$, assuming that the clusters were decoupled by C isolation. H_0 was obtained from the Sharrock fitting parameter. H_k increases rapidly with annealing temperature T_A between 600 °C and 625 °C, then increases slowly and to saturation for $T_A > 625$ °C. $K_u V^*/k_B T$ increases linearly with T_A except for the point at 675 °C that might be caused by either experimental error or V^* being unusually small. M_s decreases with the increase of T_A , e.g., M_s is about 654 emu/cm³ for $T_A = 650 \,^{\circ}\text{C}$ and 621 emu/cm³ for 700 $^{\circ}\text{C}$. Since K_u would be constant after the completion of L10 ordering, the further increase of $K_u V^*/k_B T$ with T_A is mainly due to the increase of V^{*}. As shown in Fig. 7, K_u is about 1.2×10^7 erg/cm³ for $T_A > 625 \,^{\circ}\text{C}$; V* increases slowly initially and then increases more rapidly with T_A , which results in the quasi-linear increase of $K_u V^* / k_B$ T.



Fig. 7. Effect of annealing temperature on magnetic anisotropy constant and activation volume of FePt : C cluster films with 45 vol. % C.

IV. CONCLUSION

L1₀ structure ordered FePt: C nanocluster films have been prepared by the cluster-deposition technique and subsequent thermal annealing. Magnetic and nanostructural properties of the cluster films have been systematically investigated. The thermal stability factor $K_u V^*/k_B T$, activation volume V^* , and anisotropy constant K_u are obtained from the moment-decay measurements and Sharrock formula fitting. The FePt nanoclusters studied are thermally stable, have a narrow size distribution and controllable coercivities. Their properties are similar to those required for magnetic recording at extremely high densities, i.e., approaching an areal density of 1 Tb/in² [3]. For practical application, the cluster orientation and film roughness must be further investigated and controlled.

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