

# Effects of Cr and SiN Contents on the Microstructure and Magnetic Grain Interactions of Nanocomposite FePtCr–SiN Thin Films

S. C. Chen, P. C. Kuo, A. C. Sun, C. T. Lie, and C. C. Chiang

**Abstract**—We have fabricated granular nanocomposite  $[(\text{FePt})_{100-x}\text{Cr}_x]_{100-\delta}-(\text{SiN})_\delta$  thin films with  $x = 0\text{--}25$  at.% and  $\delta = 0\text{--}30$  vol.% on natural-oxidized silicon substrate by dc and radio-frequency magnetron cosputtering of FePt, Cr, and  $\text{Si}_3\text{N}_4$  targets. We annealed the as-deposited film in vacuum at  $600^\circ\text{C}$  and then quenched it with ice water in order to transform the soft magnetic face-centered cubic  $\gamma$ -FePt phase to the hard magnetic face-centered tetragonal  $\gamma_1$ -FePt phase ( $\text{L}_{10}$  phase). The transmission-electron-microscopy observation indicated that: 1) Cr and SiN can restrain the grain growth of magnetic grains and 2) the structure of the film is nonmagnetic SiN matrix with FePtCr particles dispersed in it. Average grain size of the magnetic particles decreased as Cr or SiN content of the film was increased. Energy dispersed spectrum (EDS) analysis showed that Cr exists mainly in the grain surface area (between the grain boundary and the inner grain) of the magnetic grains. The magnetic grains are isolated by SiN and magnetic grain interactions are reduced as Cr or SiN content of the film is increased. Increasing SiN volume fraction of the magnetic film will increase the thermal stability of the film. We found the  $[(\text{FePt})_{90}\text{Cr}_{10}]_{85}-(\text{SiN})_{15}$  film, annealed at  $600^\circ\text{C}$  for 30 min, is suitable for high-density magnetic recording. Average grain size of the FePtCr in this film is about 9.5 nm. Its in-plane coercivity  $H_{c//}$  is 3.7 kOe, saturation magnetization  $M_s$  is  $450\text{ emu/cm}^3$ , and in-plane squareness  $S_{//}$  is about 0.75.

**Index Terms**—FePtCr–SiN granular film, magnetron cosputtering, thermal stability of coercivity.

## I. INTRODUCTION

MAGNETIC thin films of CoPt and FePt have both high coercivity and high magnetocrystalline anisotropy constant  $K_u$  (The  $K_u$  of CoPt is about  $5 \times 10^7\text{ erg/cm}^3$  and it is about  $7 \times 10^7\text{ erg/cm}^3$  for FePt). These characteristics make them potential materials for high-density magnetic recording media [1]–[3]. However, thermal stability of the film and the medium noise caused by the magnetic grain interactions have to be solved [4], [5]. For the reduction of medium noise, recent research is inclined to use nonmagnetic materials, which can separate magnetic grains to decrease intergranular magneto-static interaction and exchange coupling. Therefore, nanocom-

posite magnetic films with isolated magnetic grains dispersed in a nonmagnetic matrix are expected to be more suitable for high-density magnetic recording media in the future. Currently, nanocomposite films with composite materials of CoPt–C [6], CoPt–BN [7], FePt–SiN [8], and FePt–Zr [9] have been verified that they have potential for application to high-density magnetic recording media.

While recording density of the magnetic film is increased, the magnetic particle in the film is smaller. Previous investigations have shown that the grain size of the FePt can be reduced by adding Cr into FePt film [10], and medium noise can be minimized by dispersing magnetic FePt grains into an amorphous nonmagnetic SiN matrix [8]. In this paper, we have prepared FePtCr–SiN film and discuss the effects of Cr and SiN compositions on the microstructure, exchange coupling, and thermal stability of the coercivity of the magnetic film.

## II. EXPERIMENT

The  $[(\text{Fe}_{50}\text{Pt}_{50})_{100-x}\text{Cr}_x]_{100-\delta}-(\text{SiN})_\delta$  nanocomposite films (where  $x = 0 \sim 25$  at.%,  $\delta = 0 \sim 30$  vol.%) are fabricated on natural-oxidized silicon wafer by dc and radio-frequency magnetron cosputter of  $\text{Fe}_{50}\text{Pt}_{50}$ , Cr, and  $\text{Si}_3\text{N}_4$  targets at room temperature. The substrate is rotated at 75 r/min in order to make the film composition uniform across the wafer. The film thickness is fixed at 10 nm in order to examine the possibility for applying in high-density magnetic recording media, and 5 nm of SiN cap layer is used to prevent oxidation.

The base pressure of the sputter chamber is controlled at  $3 \times 10^{-7}$  torr and the argon pressure is fixed at 7 mtorr. The adjustment of the power supplies of FePt, Cr, and  $\text{Si}_3\text{N}_4$  guns provides a wide range of Cr content and SiN volume fractions of the magnetic layer. The as-deposited film is encapsulated in a quartz tube, annealed in vacuum at  $600^\circ\text{C}$  for 30 min, and then quenched in ice water.

The structure of the film is identified by an X-ray diffractometer (XRD) with  $\text{Cu-K}_\alpha$  radiation, and microstructures of the film are observed by transmission electron microscopy (TEM). Magnetic properties of the film are measured by vibrating sample magnetometer (VSM) and superconducting quantum interference device (SQUID) with maximum applied fields of 13 and 50 kOe, respectively. Average grain size of the film is calculated from TEM bright field image. Composition and the homogeneity of the film are determined by energy dispersed spectrum (EDS), and the film thickness is measured by atomic force microscope (AFM).

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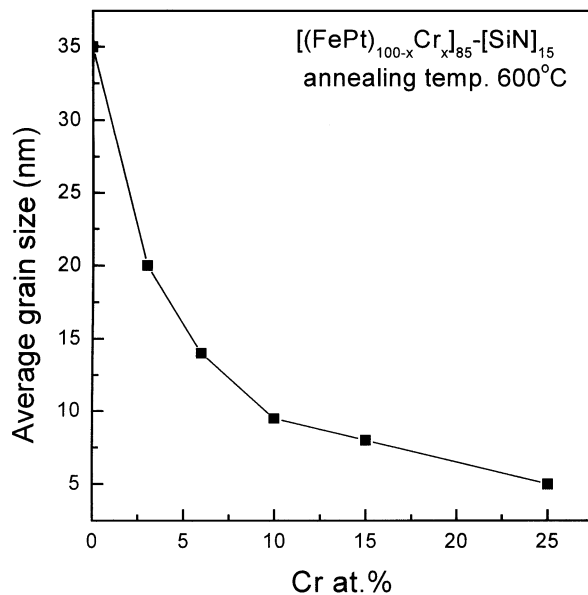


Fig. 1. Variation of average grain size with Cr content of the annealed  $[(\text{FePt})_{100-x}\text{Cr}_x]_{85}-(\text{SiN})_{15}$  films.

### III. RESULTS AND DISCUSSION

Fig. 1 shows the variation of average grain size with Cr content of the annealed  $[(\text{FePt})_{100-x}\text{Cr}_x]_{85}-(\text{SiN})_{15}$  films. SiN volume fraction of the film is fixed at 15 vol.%. The average grain size ( $d$ ) is calculated from the TEM bright field image. It is obvious that average grain size of the film is decreased as Cr content of the film is increased. For the annealed  $[\text{FePt}]_{85}-(\text{SiN})_{15}$  film (Cr = 0 at.%),  $d$  is about 35 nm, but it decreases to about 9.5 nm as Cr content increases to 10 at.%. Fig. 2(a) and (b) shows the TEM bright field images of the annealed  $[\text{FePt}]_{85}-(\text{SiN})_{15}$  and  $[(\text{FePt})_{85}\text{Cr}_{15}]_{85}-(\text{SiN})_{15}$  films, respectively. Average grain size of Fig. 2(a) is about 35 and 8 nm for Fig. 2(b). From Figs. 1 and 2, we can see that the magnetic particles become smaller and the interparticle distance is increased as Cr content of the film increases. In order to understand the distribution of Cr in the film, we use high annealing temperatures and long annealing time to obtain large magnetic particles for TEM-EDS analysis. Fig. 3(a) is a bright field image of the  $(\text{FePt})_{90}\text{Cr}_{10}$  film which annealed at 700 °C for 1 h. EDS elemental analyses of the pointed areas of I, II, and III illustrated in the TEM image of Fig. 3(a) are shown in Fig. 3(b), 3(c) and 3(d), respectively. The EDS analyses indicate that the composition of inner grain (position I) consists of Fe and Pt, the composition of grain surface area II consists of Fe, Pt as well as Cr, and the composition of grain boundary III consists of only purely Cr. These results reveal that most of Cr exists in the grain boundary and it restrains the grain growth during annealing (see Fig. 1). It is, therefore, proposed that the magnetic grains become smaller with increasing Cr content. This has resulted in increased intergrain distance as well as the reduced interaction strength of the magnetic grains.

Mayo *et al.* [11] modified the Wohlfarth relation by using interaction-based deviation parameter  $\delta M$  to examine the interaction of magnetic particles as follows:

$$\delta M = I_d(H) - [1 - 2I_r(H)]$$

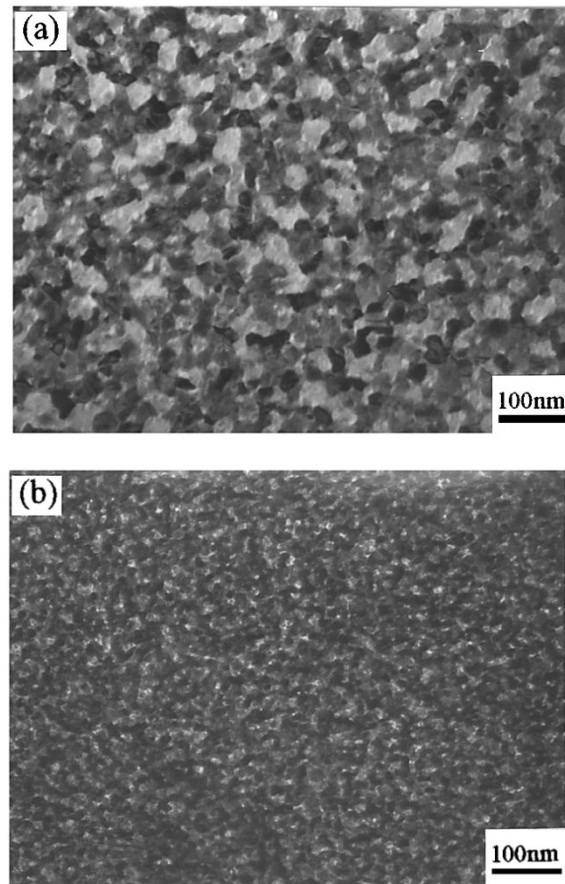


Fig. 2. TEM bright field images of the  $[(\text{FePt})_{100-x}\text{Cr}_x]_{85}-(\text{SiN})_{15}$  films which annealed at 600 °C with (a) Cr = 0 at.% and (b) Cr = 15 at.%.

where  $I_d(H)$  is the dc demagnetization remanence curve and  $I_r(H)$  is the isothermal remanence curve. Positive  $\delta M$  shows strong interaction of magnetic particles due to exchange coupling. Negative  $\delta M$  shows weak magnetic particle interactions resulting from magnetic dipole interaction. In practice, medium noise is expected as low as possible. Therefore, negative  $\delta M$  of the magnetic film is preferable for magnetic recording media application.

Fig. 4 shows the relations between  $\delta M$  and applied field  $H_a$  of the  $[(\text{FePt})_{100-x}\text{Cr}_x]_{85}-(\text{SiN})_{15}$  films with different Cr contents. SiN volume fraction is fixed at 15 vol.%. The  $\delta M$  value of the  $[\text{FePt}]_{85}-(\text{SiN})_{15}$  film (Cr = 0 at.%) is positive under applied field, so the magnetic particle interactions in this film is from exchange coupling. As Cr content increases, the FePt content in the  $[(\text{FePt})_{100-x}\text{Cr}_x]_{85}-(\text{SiN})_{15}$  film is decreased and the distance between FePt particles becomes larger and the exchange coupling is reduced. Hence,  $\delta M$  of the film decreases as Cr content increased, as shown in Fig. 4. As Cr content is increased to 25 at.%,  $\delta M$  is decreased to slightly negative and the interparticle interactions become dipole interaction.

Fig. 5 shows the variation of average grain size with SiN volume fraction of the annealed  $[(\text{FePt})_{90}\text{Cr}_{10}]_{100-\delta}-(\text{SiN})_{\delta}$  film. The Cr content is fixed at 10 at.%. The average grain size for the annealed  $(\text{FePt})_{90}\text{Cr}_{10}$  film (SiN = 0 vol.%) is about 18 nm, but it has decreased to about 9.5 nm as SiN volume

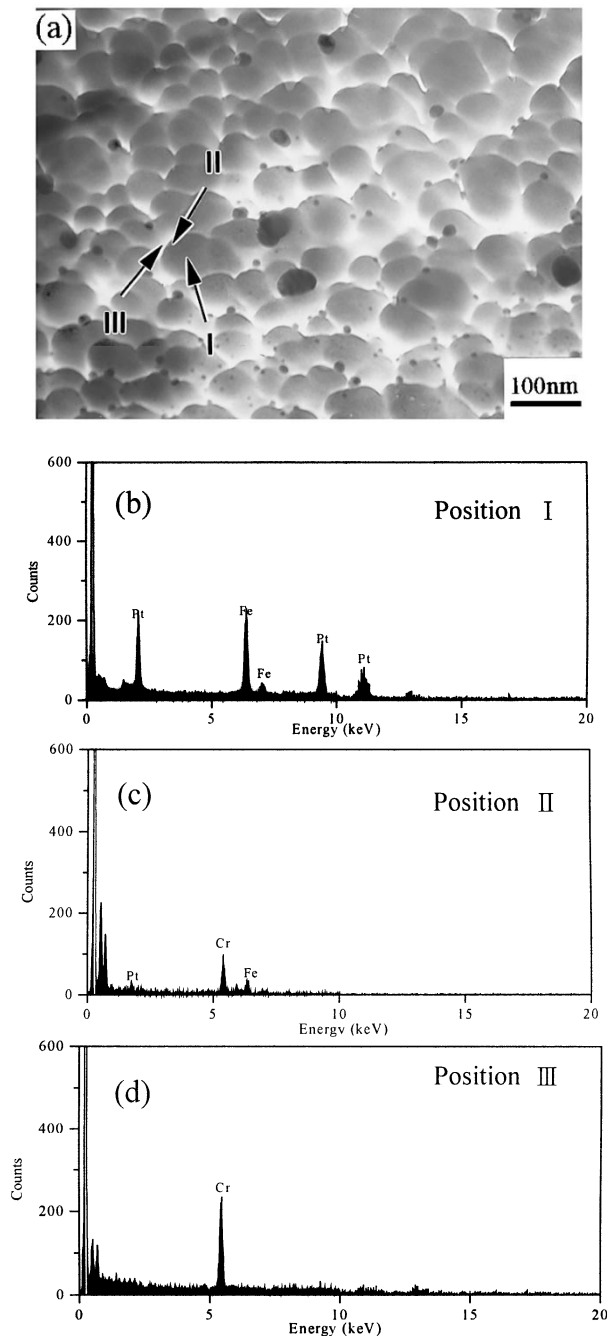


Fig. 3. (a) TEM bright field image of the  $(\text{FePt})_{90}\text{Cr}_{10}$  film annealed at  $700^\circ\text{C}$  for 1 h. (b) shows the composition of the  $(\text{FePt})_{90}\text{Cr}_{10}$  grain shown in position I, (c) the composition of the grain surface area shown in position II, and (d) the composition of grain boundary shown in position III.

fraction increases to 15 vol.%. Fig. 6(a) and (b) has shown the TEM bright field images of annealed  $(\text{FePt})_{90}\text{Cr}_{10}$  and  $[(\text{FePt})_{90}\text{Cr}_{10}]_{80}-(\text{SiN})_{20}$  films, respectively. It can be seen that average grain size of Fig. 6(a) is about 18 nm and it is about 8 nm for Fig. 6(b). From Figs. 5 and 6, we have observed that the interparticles distance has increased and the magnetic particle size has reduced with increasing SiN volume fraction. Fig. 7 shows the relationship between  $\delta M$  and  $H_a$  of various annealed  $[(\text{FePt})_{90}\text{Cr}_{10}]_{100-\delta}-(\text{SiN})_{\delta}$  films with different SiN volume fractions. The Cr content is fixed at 10 at.%. It has indicated that the  $\delta M$  value of the  $(\text{FePt})_{90}\text{Cr}_{10}$  film

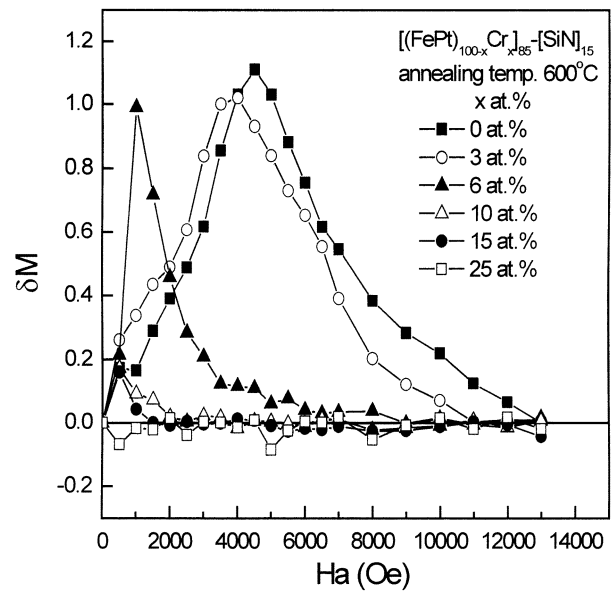


Fig. 4. Relations between  $\delta M$  and applied field of the annealed  $[(\text{FePt})_{100-x}\text{Cr}_x]_{85}-(\text{SiN})_{15}$  films with different Cr contents.

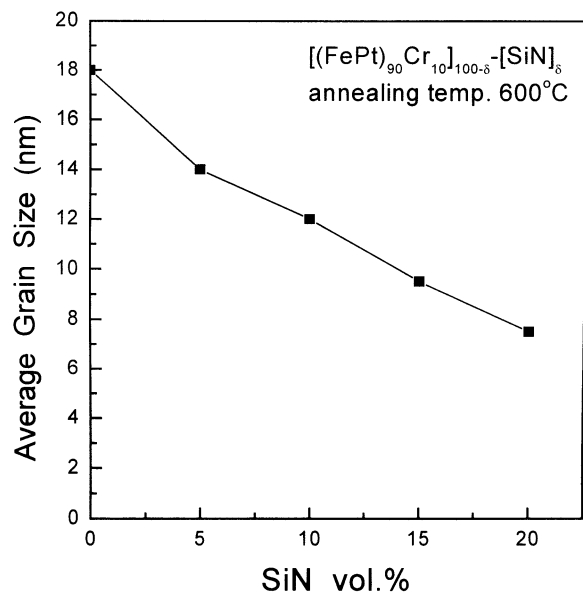


Fig. 5. Variation of average grain size with SiN volume fraction of the annealed  $[(\text{FePt})_{90}\text{Cr}_{10}]_{100-\delta}-(\text{SiN})_{\delta}$  film.

(SiN = 0 vol.%) is positive and the interaction of magnetic grains in this film is due to exchange coupling.  $\delta M$  of the film decreases to about zero as SiN volume fraction of the magnetic film increases to about 20 vol.% and becomes negative as SiN volume fraction increases further. As SiN volume fraction of the magnetic film reaches 30 vol.%,  $\delta M$  becomes negative and the type of interparticle interactions is dipole interaction. It has shown that increasing SiN volume fraction of the magnetic film decreases the strength of interparticle interactions. This is because high SiN volume fraction expands the distance among magnetic particles. The TEM images of Figs. 2 and 6 also confirm the  $\delta M$ - $H_a$  curves of Figs. 4 and 7, respectively, i.e., increased Cr or SiN content of the film will reduce the strength of magnetic particle interactions.

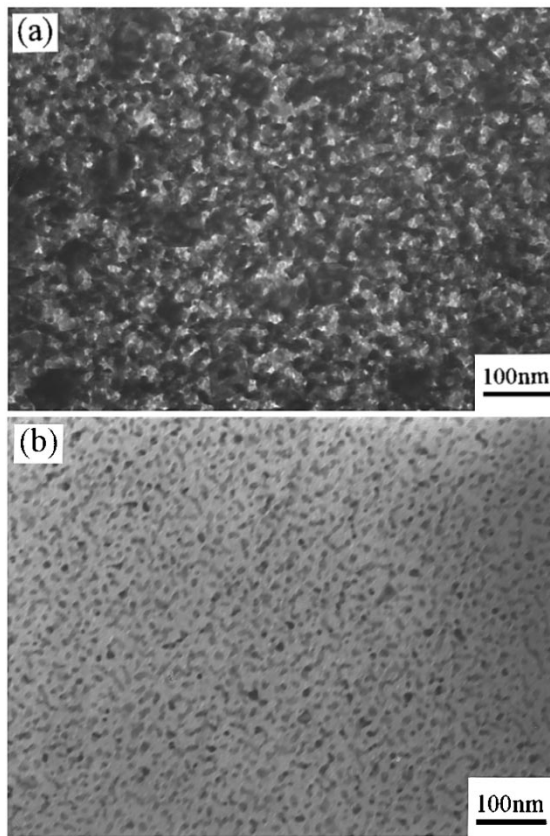


Fig. 6. TEM bright field images of annealed  $[(\text{FePt})_{90}\text{Cr}_{10}]_{100-\delta}-(\text{SiN})_{\delta}$  films with SiN volume fractions of (a) 0 vol.% and (b) 20 vol.%.

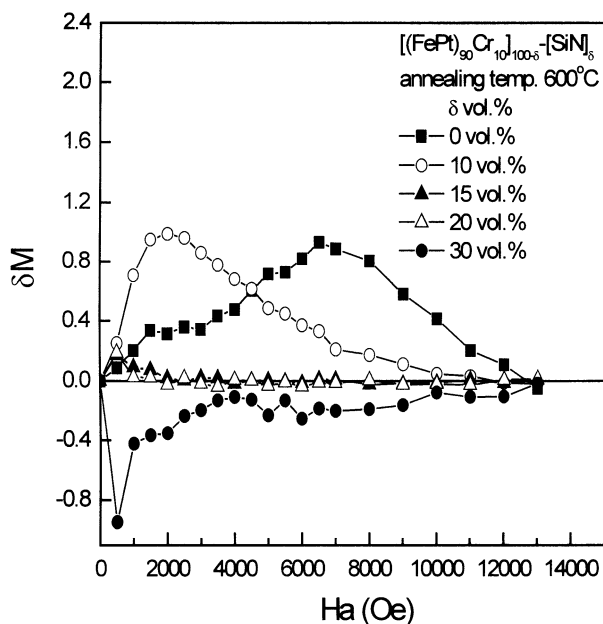


Fig. 7. Relationship between  $\delta M$  and  $H_a$  of the annealed  $[(\text{FePt})_{90}\text{Cr}_{10}]_{100-\delta}-(\text{SiN})_{\delta}$  films with varying SiN volume fractions.

In addition, it has been shown by Karanasos *et al.* that decreasing the strength of interparticle interactions reduces the squareness ( $M_r/M_s$ ) of magnetic film [12]. According to the Stoner–Wohlfarth model, the squareness of randomly oriented

and noninteracting particles is about 0.5 [13]. The relationship between in-plane squareness  $S_{//}$  and Cr content of the annealed  $[(\text{FePt})_{100-x}\text{Cr}_x]_{85}-(\text{SiN})_{15}$  film is shown in Fig. 8(a). SiN volume fraction of the film is fixed at 15 vol.%. Fig. 8(b) shows that the variation of  $S_{//}$  with SiN volume fraction of the annealed  $[(\text{FePt})_{90}\text{Cr}_{10}]_{100-\delta}-(\text{SiN})_{\delta}$  film with the Cr content fixed at 10 at.%. It can be seen that  $S_{//}$  value in Fig. 8(a) drops as Cr content increases.  $S_{//}$  is 0.81 when Cr = 0 at.%.  $S_{//}$  reduces to about 0.53 as Cr content increased to 15 at.%. Likely,  $S_{//}$  value of Fig. 8(b) goes down as SiN content increases.  $S_{//}$  is 0.8 when SiN = 0 vol.%.  $S_{//}$  is down to about 0.48 as SiN volume fraction of the magnetic film is up to 30 vol.%. These results suggest that the magnetic FePtCr particles become randomly oriented and magnetically isolated as Cr or SiN content is increased, which in turn reduces the strength of interparticle interactions. In addition, these results have shown to agree with the  $\delta M-H_a$  curves in Figs. 4 and 7 as well as the TEM microstructures in Figs. 2 and 6.

It is shown in Fig. 9 that an increase of either Cr or SiN content decreases the in-plane coercivity  $H_{c//}$  of the annealed  $[(\text{FePt})_{100-x}\text{Cr}_x]_{100-\delta}-(\text{SiN})_{\delta}$  film. When SiN volume fraction is fixed at 15 vol.%, the  $H_{c//}$  value of the annealed  $[\text{FePt}]_{85}-(\text{SiN})_{15}$  film (Cr = 0 at.%) is about 8000 Oe. However, it decreases to about 3700 Oe with Cr content increasing to 10 at.%, shown in Fig. 9(a). Likely, when Cr content fix at 10 at.%,  $H_{c//}$  value of the annealed  $(\text{FePt})_{90}\text{Cr}_{10}$  film (SiN = 0 vol.%) is about 5600 Oe. It decreases to about 350 Oe as SiN volume fraction of the film increases to 30 vol.%, as shown in Fig. 9(b). It is hypothesized that increasing Cr or SiN content of the magnetic film inhibits the magnetic grain growth during annealing, making the grain size deviated from single domain size. Hence, it is possible that some magnetic grains become superparamagnetic particles. Moreover, the diffusion of Cr into FePt grain surface area decreases the crystal anisotropy constant of FePt. Hence,  $H_{c//}$  value decreases as Cr or SiN content increases.

On the other hand, Cr is nonmagnetic material. Increasing Cr content dilutes the  $M_s$  value of the magnetic film. When SiN volume fraction is fixed at 15 vol.%,  $M_s$  value of the annealed  $[\text{FePt}]_{85}-(\text{SiN})_{15}$  film (Cr = 0 at.%) is about 490 emu/cm<sup>3</sup>. However, it decreases to about 450 emu/cm<sup>3</sup> as Cr content increases to 10 at.%, shown in Fig. 9(a). Owing to the reaction of magnetic grains with Si substrate [8],  $M_s$  value of the annealed  $(\text{FePt})_{90}\text{Cr}_{10}$  film (SiN = 0 vol.%) is only about 275 emu/cm<sup>3</sup>. It increases to about 480 emu/cm<sup>3</sup> as SiN volume fraction increases to 5 vol.%, shown in Fig. 9(b). This suggests the protective effect of SiN on the magnetic particles from reaction with Si substrate is good. However, the  $M_s$  value decreases as SiN volume fraction is higher than about 5 vol.%. Since SiN is also a nonmagnetic substance, it dilutes the  $M_s$  value of the magnetic film. Hence, the  $M_s$  value decreases to about 180 emu/cm<sup>3</sup> as SiN volume fraction increases to 30 vol.%, shown in Fig. 9(b).

From the above experiment, we find that the granular  $[(\text{FePt})_{90}\text{Cr}_{10}]_{85}-(\text{SiN})_{15}$  film (Cr = 10 at.%, SiN = 15 vol.%) after annealing at 600 °C for 30 min has magnetic properties of  $H_{c//} = 3.7$  kOe,  $M_s = 450$  emu/cm<sup>3</sup>, and  $S_{//} \cong 0.75$ . The particle size of this film is about 9.5 nm. It is suitable for high-density magnetic recording media application.

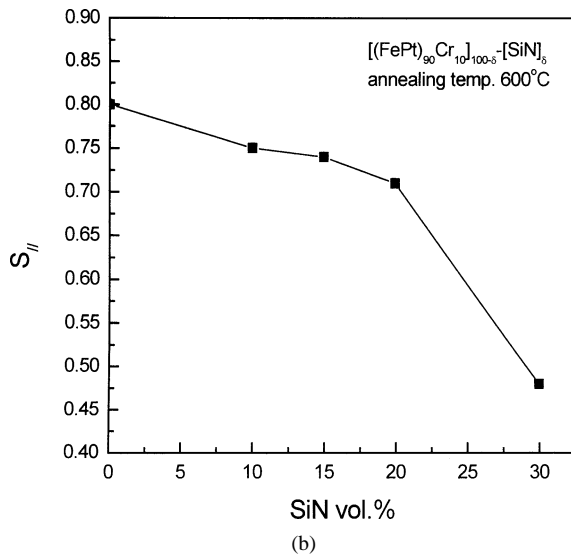
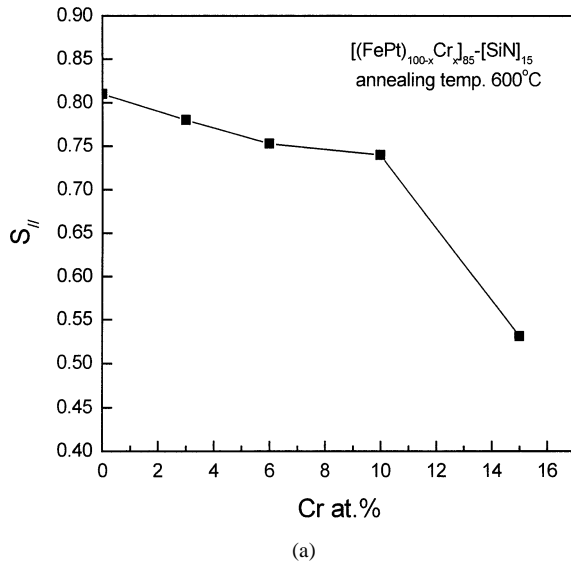


Fig. 8. (a) Relationship between  $S_{||}$  and Cr content of annealed  $[(\text{FePt})_{100-x}\text{Cr}_x]_{85}-(\text{SiN})_{15}$  films. (b) Relationship between  $S_{||}$  and SiN volume fractions of the annealed  $[(\text{FePt})_{90}\text{Cr}_{10}]_{100-\delta}-(\text{SiN})_{\delta}$  film.

The temperature coefficient of coercivity, defined by  $dH_{c||}/dT$ , was determined as  $dH_{c||}/dT = [H_{c||}(100^\circ\text{C}) - H_{c||}(25^\circ\text{C})]/75^\circ\text{C}$ , where  $H_{c||}(100^\circ\text{C})$  and  $H_{c||}(25^\circ\text{C})$  are the in-plane coercivities at 100 °C and 25 °C, respectively. The values of  $dH_{c||}/dT$  as a function of the SiN volume fraction of the  $[(\text{FePt})_{90}\text{Cr}_{10}]_{100-\delta}-(\text{SiN})_{\delta}$  film are plotted in Fig. 10. The dashed line indicates that the coercivity is not affected by the temperature. It is obvious in Fig. 10 that the absolute value of  $dH_{c||}/dT$  becomes smaller as SiN volume fraction of the film increases. When SiN = 0 vol.%,  $dH_{c||}/dT$  is measured to be  $-3.970 \text{ Oe}/^\circ\text{C}$ .  $dH_{c||}/dT$  is increased to  $-0.762 \text{ Oe}/^\circ\text{C}$  as SiN volume fraction of the film increases to 20 vol.%. The deviation of  $dH_{c||}/dT$  value from dashed line is smaller as SiN volume fraction of the film is increased. This means that thermal stability of the coercivity is improved as SiN volume fraction is increased. Coercivity of the film due to stress anisotropy is increased as temperature or SiN volume fraction

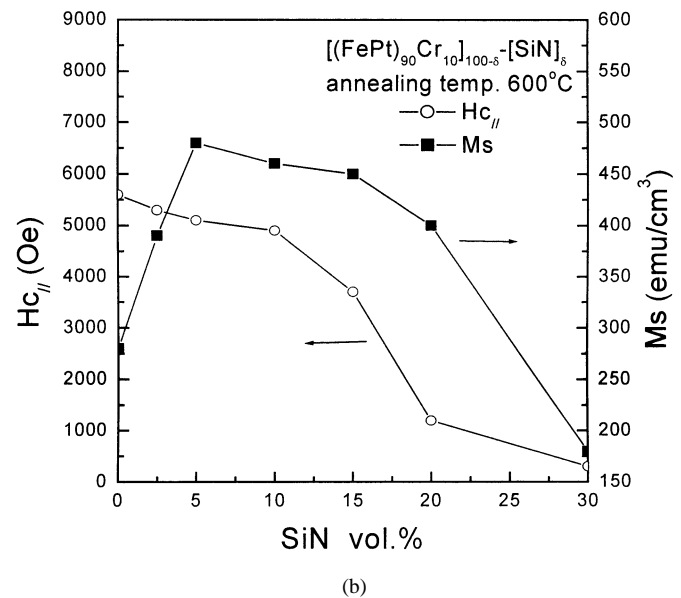
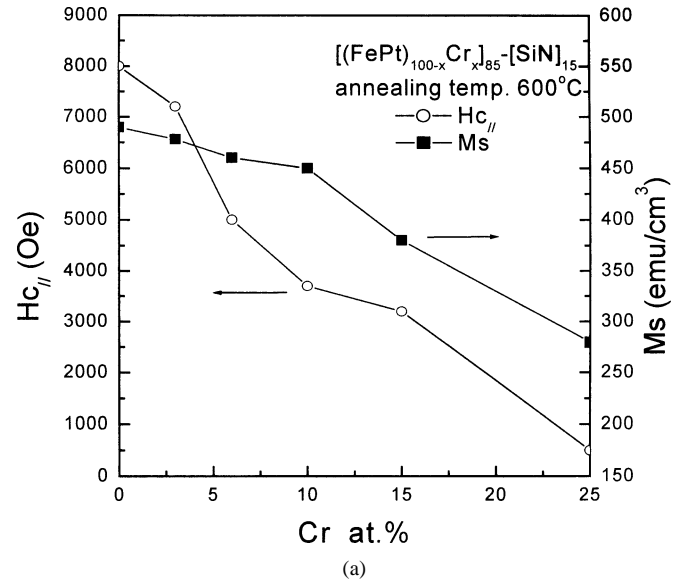


Fig. 9. (a) Variations of  $H_{c||}$  and  $M_s$  with varying Cr content of the annealed  $[(\text{FePt})_{100-x}\text{Cr}_x]_{85}-(\text{SiN})_{15}$  film. (b) Variations of  $H_{c||}$  and  $M_s$  with varying SiN volume fraction of the annealed  $[(\text{FePt})_{90}\text{Cr}_{10}]_{100-\delta}-(\text{SiN})_{\delta}$  film.

of the film is raised, because the magnetic particle FePtCr is a metallic material while SiN matrix is a ceramic material. Since the thermal expansion coefficient of FePtCr is much larger than that of SiN, the compressive stress on the FePtCr grains increases with increasing temperature. The compressive stress on the FePtCr grains also increases as SiN volume fraction increases. On the other hand, coercivity of the film due to crystal anisotropy is decreased as temperature increases. This is because crystal anisotropy constant  $K_u$  of the FePtCr grains decreases at high temperature. Due to the cancellation of the variations of stress anisotropy and crystal anisotropy with temperature, the absolute value of  $dH_{c||}/dT$  become smaller as the SiN volume fraction increases. This means that increasing SiN volume fraction is able to enhance thermal stability of the coercivity of the film.

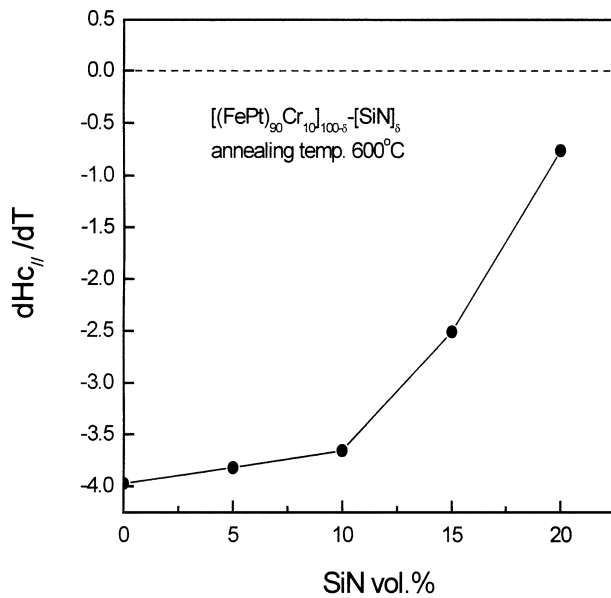


Fig. 10. Relation between  $dH_{c_{//}}/dT$  and SiN volume fraction of annealed  $[(\text{FePt})_{90}\text{Cr}_{10}]_{100-\delta}-(\text{SiN})_{\delta}$  films.

#### IV. CONCLUSION

We have shown that  $\delta M$  and  $S_{//}$  of the FePtCr-SiN film decrease as Cr or SiN content of the film is increased. This indicates that the magnetic particle interactions of the film are reduced as Cr or SiN content of the film is increased. The magnetic FePtCr particles become randomly oriented and magnetically isolated by nonmagnetic materials as Cr content of the annealed  $[(\text{FePt})_{100-x}\text{Cr}_x]_{85}-(\text{SiN})_{15}$  film is higher than 15 at.% or the SiN content is larger than 28 vol.%. Thermal stability of the coercivity is improved as SiN volume fraction of the film is increased.

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