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Enhancement of coercivity in nanometer-size CoPt crystallites

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In this study, we showed that a magnetic coercivity (H_c) as high as 37 kOe was obtained in a CoPt thin film that contains separated nanometer-size CoPt crystallites. We prepared CoPt thin films with thicknesses of 5 and 175 nm by magnetron sputtering. After annealing in an Ar/H_2 atmosphere at temperatures from 650 to 750 °C for 3–12 h, we measured the magnetic properties and found that magnetic H_c relates to annealing temperature, annealing time and film thickness. From atomic force microscopy and magnetic force microscopy studies, the magnetic single domain size of CoPt is in the range of 100–200 nm. The high H_c is likely due to the well-separated nanometer-size crystallites and the well-ordered fct phase of CoPt alloy. © *1999 American Institute of Physics.* [S0021-8979(99)51108-0]

I. INTRODUCTION

Many magnetic devices are required to have high magnetic coercivity (H_c), such as magnetic bias films of magneto-resistive elements, magnetic tips for magnetic force microscopy, high-density magnetic recording media, and magneto-optic recording media.[1–7] The CoPt binary alloy is an excellent system because of its chemical stability and high magnetic anisotropy. The anisotropy of CoPt compounds is as high as 4×10^7 ergs/cm^3 and the saturation magnetization is about 800 emu/cm^3. From the calculation of Stoner–Wohlfarth theory, $H_c=0.98K/M_s$, where K is the anisotropy constant and M_s is the saturation magnetization. Thus, an H_c around 49 kOe is expected.[8,9]

It has been shown that the ordered fct structure can be obtained in the temperature range of 600–850 °C. The transmission electron microscopy (TEM) result also established that CoPt samples with ordered fct structure were achieved after being annealed at 750 °C for 30 min.[5]

As shown in the previous article, the high coercivity value of about 30 kOe was observed in a CoPt film with nanometer-size crystallites. For this article, we directly measured the magnetic domain structure using magnetic force microscopy (MFM). We also show that a CoPt film with a much higher coercivity value was prepared by a two-step annealing process.

II. EXPERIMENT

The CoPt alloy films were prepared on fused quartz substrates by direct current magnetron sputtering. The target was made by mixing high-purity Co and Pt powder with a 1:1 atomic ratio and sintered at 1050 °C for 24 h. The base pressure before introducing the Ar gas was 2×10^{-8} Torr. The chamber pressure during sputtering was 10^{-2} Torr. The film thickness was in the range of 5–175 nm. To improve the ordering and to control the separation of crystallites, the CoPt films were annealed under an Ar/H_2 atmosphere in the temperature range of 650–750 °C, and the range of annealing time was from 3 to 12 h. The magnetic properties of the films were measured by a superconducting quanturn interference device magnetometer. The morphology and the magnetic domain structure of the CoPt films were examined by atomic force microscopy (AFM) and MFM.

III. RESULTS AND DISCUSSIONS

The observed high H_c in the CoPt film can be explained by the presence of the well ordered nanometer-size crystallites. These crystallites are noninteracting and form a random single-domain system.[5] However, there is no direct evidence from early studies. In this article, we present the direct measurements of the magnetic domain structure of these high H_c CoPt films from MFM and the comparison of the magnetic properties of the CoPt films with different annealing conditions.

Hysteresis loops of CoPt films with different thicknesses on quartz substrates, measured at 300 K, are shown in Fig. 1. Both of the samples were annealed at 750 °C for 3 h. Figure 1(a) is the sample with the thickness of 175 nm. An H_c of 12 kOe and a saturation magnetization of 734 emu/cm^3 were observed. Figure 1(b) is the sample with the thickness of 5 nm. An H_c of 16 kOe and a saturation magnetization of 680 emu/cm^3 were observed. The small variation of the saturation magnetization may be due to the difficulty in the thickness measurement for the 5-nm-thick film. The major difference between these two films is their H_c values. We observed some variation of H_c values from samples that were not prepared in the same sputtering run. However, the H_c value of the 175-nm-thick film was always much less than that of the 5-nm-thick film when they were annealed in the same conditions.

Figures 2 and 3 are the AFM and MFM images of the same films (annealed at 750 °C for 3 h, with thicknesses of 5 and 175 nm. As shown in Fig. 2(a), the 5-nm-thick film contains well-separated nanometer-size crystallites as indi-

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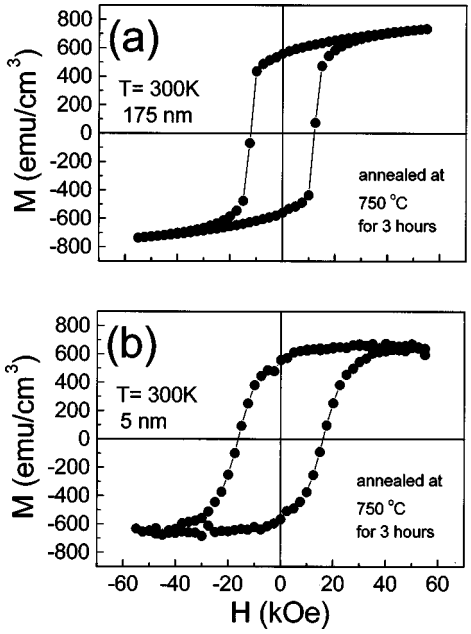

FIG. 1. Hysteresis loops of CoPt films on quartz substrates and annealed at 750 °C for 3 h and measured at 300 K. The film thicknesses are (a) 175 and (b) 5 nm.

cated in the AFM image. The sizes of the crystallites are in the range of 100–400 nm. The height of crystallites is in the range of 20–80 nm. The MFM image, Fig. 2(b), was obtained using a CoPt MFM tip magnetized parallel to the sample surface. The light and dark contrast corresponds to the strength of the stray-field gradient on the sample surface. The lighter color represents a frequency shift in the MFM tip when the magnetization of the sample and that of the MFM tip are repulsive. As shown in Fig. 2(b), crystallites with one light and dark area are single-domain (as indicated by ''*S*''); the grain that may contain a few crystallites with two or more light and dark areas are multidomain (as indicated by ''*M*''). The size of a single-domain crystallite is between 100 and 200 nm. Figure 3 shows the AFM and MFM images of

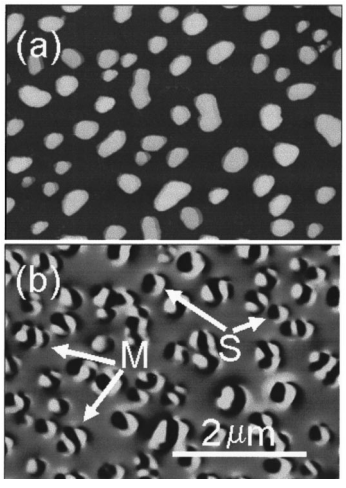

FIG. 2. The topographic (a) and magnetic (b) images of a 5-nm-thick CoPt film on a quartz substrate, annealed at 750 °C for 3 h. *M* indicates a multidomain grain. *S* indicates a single-domain grain.

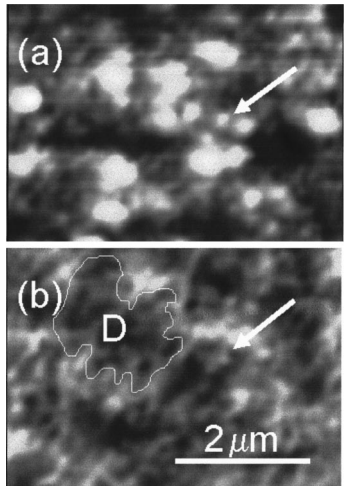

FIG. 3. The topographic (a) and magnetic (b) images of a 175-nm-thick CoPt film on a quartz substrate, annealed at 750 °C for 3 h. The arrows indicate the typical crystallite size and its corresponding magnetic domain. A region of coupled magnetic domains is marked by *D*.

the sample with the film thickness of 175 nm. The roughness of the film surface is in the range of 50–100 nm. The crystallites of the 175-nm-thick film are not separated. However, the size of the crystallites is in the range of 100–400 nm, as indicated by the arrow in Fig. 3(a). This is also consistent with the results from the TEM study that was reported previously.[5] As shown in Fig. 3(b), the magnetic domains of the crystallites with about the same direction are coupled together so that they form much larger white and dark area. The size of the magnetic domain, as indicated by an arrow in Fig. 3(b), is about a few hundred nanometers. The major difference between these two films is that the 5-nm-thick film contains many noninteracting nanometer-size magnetic domain and the 175-nm-thick film has more magnetic inter-

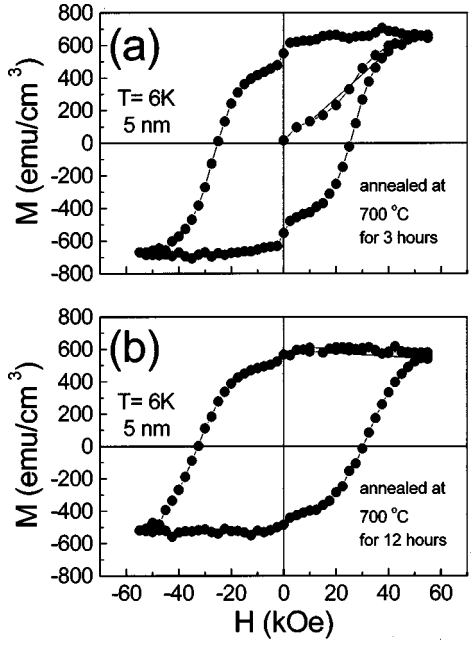

FIG. 4. Hysteresis loops of 5 nm CoPt films on quartz substrates, annealed for 3 h (a) and 12 h (b) at 700 °C, measured at 6 K.

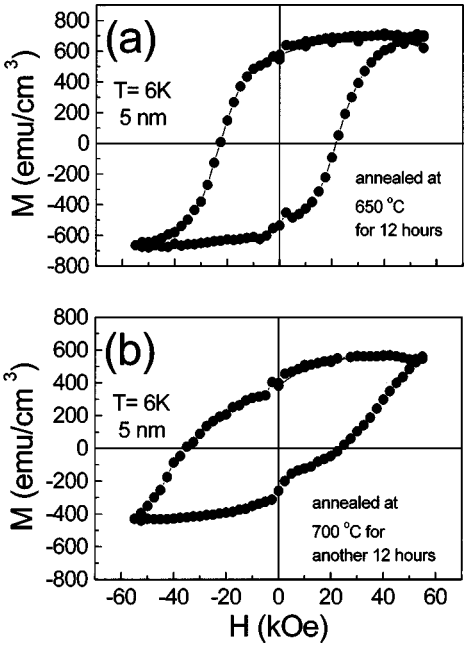

FIG. 5. Hysteresis loops of a 5 nm CoPt film on a quartz substrate measured at 6 K. (a) The film was annealed at 650 °C for 12 h. (b) The same film was annealed at 750 °C for another 12 h.

actions between the crystallites. The magnetic interaction is likely to reduce the H_c value. The coupling magnetic domains may have incoherent domain rotations.

In order to reduce the size of the crystallites in the film and study the effect of the ordering on the H_c value, we compare 5-nm-thick samples that were annealed at 700 °C for 3 and 12 h. Hysteresis loops of CoPt films measured at 6 K are shown in Fig. 4. An H_c value of 25 kOe and a saturation magnetization of 668 emu/cm^3 were observed in the sample annealed at 700 °C for 3 h. An H_c value of 33 kOe and a saturation magnetization of 586 emu/cm^3 were observed in the sample annealed at 700 °C for 12 h. The variation of the saturation magnetization may be due to the order and disorder of the CoPt phase in the sample. The saturation magnetization of the as-prepared film with a face-centered-cubic structure (Co and Pt atoms are disorderly distributed) are higher than those with the ordered CoPt fct phase. As shown in Fig. 4(a), the disorder phase with a has low H_c value may cause the drop of the magnetization near zero applied magnetic field. We have noted that the hysteresis loop is not symmetrical (it is a minor loop). The variation of the saturation magnetization may be also due to the fact that the applied magnetic field (55 kOe) was not high enough to saturate this sample.

As shown in Fig. 5, this sample was first annealed at 650 °C for 12 h. It was observed with an H_c of 23 kOe and the saturation magnetization of 712 emu/cm^3. Then it was annealed at 700 °C for another 12 h. The H_c value was as high as 37 kOe. It is very clear that the hysteresis loop is not symmetrical. For a higher applied magnetic field, a larger H_c value is expected. Through two-step annealing, the H_c value was improved by about 4 kOe as compared to one-step annealing. This may be due to the formation of smaller crystallites in the films at lower temperature and an ordered structure after annealing at 700 °C for 12 h. However, the reason needs further research.

IV. SUMMARY

We showed that CoPt thin films with well-separated crystallites and the well-ordered fct structure were achieved by controlling the annealing time and annealing temperature. As observed by MFM, noninteracting magnetic domains are important in achieving high H_c values. We also studied two-step annealing. From the experimental results, we have enhanced the magnetic coercivity of a CoPt film to 37 kOe.

ACKNOWLEDGMENT

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