Characterization of CoPt-C Nanocomposite Films
Prepared by Pulsed Filtered Vacuum Arc Deposition

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ABSTRACT

CoPt-C nanocomposite thin films of various carbon compositions were prepared by a pulsed filtered vacuum arc deposition technique. Annealing was performed in vacuum for one hour at various temperatures. The dependence of the magnetic properties on the carbon composition and annealing temperature was studied. Both x-ray diffraction and magnetic force microscopy analyses confirmed the formation of nano-crystallites of face-centered-tetragonal CoPt phase in the carbon matrix after annealing at a sufficiently high temperature, depending on the carbon composition. For the film with a particular composition of Co_{24}Pt_{31}C_{45}, the coercivity and the grain size were observed to increase with increasing annealing temperature, up to a value of 7 kOe at an annealing temperature of 750°C, and the grain size increased from 9 to 20 nm.

Key words: high-density magnetic recording, CoPt-C granular films, ferromagnetic, magnetic domain structure, MFM
1. Introduction

In recent years, the storage density in magnetic recording media has drastically increased. For the extremely high-density recording, an areal density of more than 100 Gb/in² has been proposed [1]. To achieve such high density, the bit cell dimension should be shrinking. This requires the reduction of grain sizes to 10-12nm. However, due to the superparamagnetic behavior, the magnetization of the bit cell would become destabilized. To avoid the occurring of thermal instability, high uniaxial magnetocrystalline anisotropy (K_u) materials would be required for the recording medium. Face-centered tetragonal (fct) CoPt has a large K_u value of about 5x10^7 erg/cm³ and high coercivity. Therefore it is a good candidate material for ultra high recording medium [2-4].

Recently, carbon encapsulated magnetic nanograins have been intensively studied [5-10]. The most important advantage of carbon encapsulation is the increase of the effective distance of neighboring magnetic grains so that the inter-grains exchange coupling can be weakened or eliminated. This is an important issue for the reduction of media noise. On the other hand, both CoPt and C phases have excellent chemical stability and good corrosion resistance. Carbon encapsulation can also provide protection for air-sensitive grains against degradation. In this work, we have employed the pulsed filtered vacuum arc deposition technique to prepare CoPt-C nanocomposite films of various carbon compositions and studied their properties.

2. Experiments

CoPt-C nanocomposite thin films about 40 nm to 50 nm thick of various composition were prepared by a 3-source pulsed filtered vacuum arc deposition system. The details of the system have been described elsewhere [11]. Thermally grown SiO₂ films about 100 nm on Si (100) wafers were used as substrate. Three adjacent sources with pure graphite, platinum and cobalt as the cathode materials were operated simultaneously in a pulse mode with a pulse duration of 2.5 ms and frequency of 4 pulses/s. Of the three sources, the platinum source was positioned at the center. The substrate was placed at the center of the chamber facing the platinum source and a negative bias voltage of -80V was applied. The composition of the films was varied by adjusting the arc discharge conditions and was monitored by the integrated charges arriving at the sample holder from the respective arc sources. Thermal annealing was performed in a vacuum furnace (<10⁻³ Pa) for one hour at various temperatures, respectively.

The composition and thickness of the CoPt-C films were determined by non-Rutherford backscattering spectrometry (NRBS). And the structural properties were analyzed by x-ray diffraction (XRD). The magnetic properties were characterized by a vibrating sample magnetometer (VSM). The morphology and magnetic domain structures were investigated by atomic force microscopy (AFM) and magnetic force microscopy (MFM). AFM and MFM imaging were performed using tapping and lift modes under ambient conditions, using a Nanoscope III scanning probe microscope with a vertically magnetized hard Co-alloy-coated silicon tips.

3. Results and discussion

There are two phases of CoPt, namely, face centered-cubic (fcc) CoPt and face-centered-tetragonal (fct) CoPt. The fct phase is known to have a larger K_u value [2-4]. In order to obtain the ordered fct phase from disordered as-deposited fcc phase, high-temperature annealing is required to overcome the energy barrier for superlattice ordering [2-5]. Figure 1 shows the XRD patterns of Co₃Pt₄C₁₄ films annealed at various temperatures. The XRD patterns for fcc CoPt and nano-crystallites of fct CoPt are quite similar except that there are several additional peaks such as, fct (001) and fct (110) peaks for the fct phase [2]. The other peaks in the XRD patterns
can be contributed by both the fcc and fct phases. As shown in Fig. 1, broad fct (001) and fct (110) peaks first emerged in the spectrum of the sample annealed at 600°C. This is an indication of the formation of the fct CoPt phase. These two peaks became sharper as the annealing temperature increased to 650°C, indicating larger fct CoPt grains were formed.

Shown in Fig. 2 are the MFM images of the film with a composition of Co$_{24}$Pt$_{31}$C$_{45}$ after annealing at various temperatures. The bright and dark regions in the MFM images are associated with magnetization parallel or anti-parallel to the film normal. Weak and dispersed magnetic domains are observed in the MFM images of films annealed at 500°C and 550°C, showing that these films exhibit a low perpendicular anisotropy. This indicates that the films mainly consist of disordered fcc CoPt. For samples annealed at 600°C and 650°C, the MFM images showed good contrast, indicating that these films exhibit a larger perpendicular anisotropy. The sharp change in the domain structures as revealed by the MFM images is an indication of the transition from fcc CoPt phase to the high Ku value fct CoPt phase in the films, which is consistent with the XRD results.

Fig. 1. XRD patterns of Co$_{24}$Pt$_{31}$C$_{45}$ films annealed at various temperatures in vacuum for 1 h.

Fig. 2. MFM images of Co$_{24}$Pt$_{31}$C$_{45}$ films annealed at various temperatures. (a) 500°C, (b) 550°C, (c) 600°C and (d) 650°C.

Fig. 3. Magnetic hysteresis loops measured at 300 K for the Co$_{24}$Pt$_{31}$C$_{45}$ films after annealing at the temperature as indicated.

Figure 3 shows the in-plane magnetic hysteresis loops of the Co$_{24}$Pt$_{31}$C$_{45}$ films after annealing at various temperatures in vacuum for 1 hour. It is seen from Fig. 3 that as the annealing temperature increases, there is no significant change of the saturation magnetization of the films at a value of about 300 emu/cm$^3$. The film showed a soft ferromagnetic characteristic after annealing at 550°C with a coercivity of about 100 Oe and a
saturation magnetic field of about 200 Oe. After annealing at a higher temperature of 600°C, the film became magnetically harder exhibiting a coercivity of 760 Oe. The increase in coercivity is attributed to the formation of the fct CoPt phase. The coercivity increased further with increasing annealing temperature, up to a value of 7 kOe at an annealing temperature of 750°C. This is also understandable to be the result of fct CoPt grain growth and more complete fcc to fct CoPt phase transformation. The coercivity as a function of annealing temperature is plotted in Fig. 4.

![Figure 4: Hc of Co34Pt31C15 films annealed at various temperature.](image)

The grain size is estimated by Scherrer’s formula using the (111) peak width from the XRD patterns. Shown in Fig. 5 is the grain size d against annealing temperature for CoPtC films of various compositions as indicated. The grain size of CoPt generally increases with annealing temperature due to thermal growth of the grains. It is also seen that higher carbon content will result in smaller as-deposited grain size as well as suppression of grain growth upon annealing. In fact, XRD results also showed that for the film with the highest carbon content, Co8Pt23C6, the fct phase were observed only after annealing at temperatures equal or higher than 700°C. Figure 6 shows the magnetic hysteresis loops measured at 300 K for the 650°C annealed films with various compositions as indicated. Among this batch of films in the present study, the Co37Pt31C12 film showed the highest magnetic moment of about 470 emu/cm³ because it had the highest CoPt concentration. However, the coercivity of this film was measured to be only about 500 Oe, smaller than that of the Co34Pt31C15 film which exhibited a coercivity of about 2100 Oe. Though the XRD patterns of both films showed the formation of fct CoPt phase. This is probably because the carbon content of the Co34Pt31C15 film is too small to provide sufficient and effective encapsulation of the CoPt grains. Hence, the grain size grew rapidly with increasing annealing temperature, up to 21nm at 650°C, causing the formation of multi-domain structures in the film. In contrast, the film with the highest carbon content, Co16Pt54C60, exhibited the smallest coercivity. This is obviously correlated with the smallest grain size and the absence of effective fct CoPt phase formation in this sample. These results show that the grain size and coercivity strongly depend on the carbon content as well as thermal treatment conditions.

It was estimated that for applications as a ultra-high density recording medium, a material should have a coercivity of about 5k Oe and

![Figure 5: Grain size d against annealing temperature for samples of various compositions as indicated.](image)
isolated grains with a size smaller than that of about 10-12 nm [1]. From the preliminary results obtained so far in the present study, the grain size is about 17nm to achieve such a high coercivity value. Continued effort to further optimize the carbon content and annealing time is on-going in our laboratory aiming at increasing the coercivity and reducing the grain size of the films.

4. Conclusion

In summary, we have prepared CoPt-C nanocomposite films of various carbon compositions by pulsed filtered vacuum arc deposition and studied their properties using various characterization techniques. Diversified magnetic properties were observed in these films, depending on the composition and thermal annealing conditions. Both x-ray diffraction and magnetic force microscopy analyses confirmed the formation of nano-crystallites of fct CoPt phase in the carbon matrix after annealing at a sufficiently high temperature, depending on the carbon composition. The dependence of the CoPt grain size on the composition and annealing temperature, and their correlation with the coercivity have also been studied. For example, the coercivity and the grain size of the Co$_{55}$Pt$_{45}$ film were observed to increase with increasing annealing temperature, up to a value of 7 kOe at an annealing temperature of 750$^\circ$C, and the grain size increased from 9 to 20 nm. Further optimization in the carbon composition and thermal annealing processes is in progress aiming at improving the properties of CoPt-C thin films for applications as ultrahigh density recording media.

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References