Dependence of Co anisotropy constants on temperature, processing, and underlayer

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The dependence of the Co anisotropy constants on the measurement temperature, deposition conditions, and underlayer materials was studied using unicrystal Co($10\overline{10}$)/Cr (or NiAl)(112)/Ag(110) films sputter deposited on hydrofluoric acid etched Si(110) substrates. The single, in-plane easy axis orientation in these films allows the direct determination of the anisotropy constants. The anisotropy constants of unicrystal Co films are smaller than those of a bulk Co single crystal, and the temperature dependence of the anisotropy constants is stronger. The K_1 value drops by 50% as the temperature is increased from 25 °C to 75 °C, and then becomes negative at 135 °C. This zero-crossing temperature is considerably lower than the 250 °C at which K_1 of a bulk Co single crystal decreases to zero. The anisotropy constants also vary with the film preparation substrate temperature. Applying a substrate bias during the Co deposition effectively increases K_1 to near bulk material values. Unicrystal Co films grown on NiAl/Ag/HF–Si(110) show smaller K_1 as compared to those on Cr underlayers. The addition of a thin Cr intermediate layer on the NiAl underlayer, however, restores K_1 to the larger value obtained on Cr/Ag/HF–Si(110). © 2000 American Institute of Physics. [S0021-8979(00)69908-5]

The magnetocrystalline anisotropy is an important property of Co-alloy materials. In magnetic recording, the anisotropy field determines the maximum achievable coercivity,^{1,2} and the product of the anisotropy constant and the particle volume dictates the thermal stability.³ Hence, knowledge and control of the anisotropy constants are essential for the design of Co-alloy media. We have previously reported on a technique to epitaxially grow unicrystal Co-alloy(1010)/ Cr(112)/Ag(110) films by sputter deposition on hydrofluoric acid (HF)-etched Si(110) single crystal substrates.⁴ These films exhibit a single, in-plane easy axis orientation and thus allow the direct determination of the anisotropy constants by torque and hard axis hysteresis loop measurements. While single crystal MgO(110) substrates were also used to grow unicrystal Co-alloy films for studies on the compositional and temperature dependence of anisotropy constants,⁵ Si substrates are of interest since the wafers are readily available and inexpensive. Hence, this epitaxial unicrystal film structure provides a pathway for a systematic fundamental study of the magnetocrystalline anisotropy. This article will focus on pure Co anisotropy constants as functions of the measurement temperature, the processing conditions such as growth temperature and substrate bias, and different underlayer materials.

The thin-film fabrication and characterization details have been described in our previous publications.^{4,6} While the anisotropy constants can be determined from either torque curves or hard axis hysteresis loops, for this study the use of hard axis loops is emphasized because they can be

conveniently obtained and the loop shape directly reveals important qualitative characteristics of the anisotropy constants. In this method, the anisotropy constants K_1 and K_2 can be determined by fitting the hard axis m(H) loops to⁷

$$\frac{m_s H}{V} = 2K_1 \left(\frac{m}{m_s}\right) + 4K_2 \left(\frac{m}{m_s}\right)^3,$$

 $0 \le m \le m_s, \text{ and } H > 0.$ (1)

Here, V is the sample volume and m_s is the saturation moment of the sample. Figure 1 shows the schematic plots of the hard axis m(H) loops for various K_1 and K_2 . They are calculated with H decreasing from the saturation field to zero, which is the same as the measurement procedure. When $K_2=0$, the hard axis loop is a straight line. The curvature in the loop results from a nonzero K_2 (assume K_2



FIG. 1. Schematic hard axis hysteresis loops calculated for different K_1 and K_2 . Here, $H_K = 2K_1/M_s$. The field *H* decreases from the saturation field to zero.

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FIG. 2. Hard axis hysteresis loop of a typical Co(500 Å)/Cr(500 Å)/Ag(750 Å)/HF–Si(110) film.

>0). K_1 determines the characteristics of the loop at the origin. When K_1 >0, the loop will go through the origin with a finite positive slope

$$\frac{d}{dH}\left(\frac{m}{m_s}\right)_{H=0} = \frac{m_s}{2K_1V} = \frac{1}{H_K},\tag{2}$$

and thus a steeper slope indicates a smaller K_1 . When $K_1 = 0$, the loop will be tangent to the vertical axis at the origin. When $K_1 < 0$, the loop will intersect with the vertical axis, corresponding to a remanence squareness

$$S = \frac{m_r}{m_s} = \sqrt{\frac{|K_1|}{2K_2}}.$$
 (3)

The in-plane hard axis hysteresis loop of a typical unicrystal Co/Cr/Ag/HF–Si(110) film is shown in Fig. 2. It shows a curve with virtually zero openness; the remanence squareness S < 0.02. This confirms the almost perfect easy axis alignment. The anisotropy constants determined from the hard axis loop are $K_1=1.55\times10^6$ erg/cm³ and K_2 $=1.30\times10^6$ erg/cm³. This K_1 is much smaller than the value reported for a bulk Co single crystal ($K_1=4.5$ $\times10^6$ erg/cm³ and $K_2=1.5\times10^6$ erg/cm³).⁷ Given the good alignment of the Co grain easy axes, the small K_1 cannot be explained by assuming an easy axis orientation dispersion. It is likely that stacking faults may develop during the growth of the hcp Co film and the resulting fcc stacking sequence may be responsible for this decrease.

The temperature dependences of the saturation magnetization (M_s), K_1 , and K_2 of the unicrystal Co/Cr/Ag/HF– Si(110) sample are shown in Fig. 3, and the corresponding hard axis hysteresis loops at selected temperatures are shown



FIG. 3. Saturation magnetization and anisotropy constants of a Co(500 Å)/Cr(500 Å)/Ag(750 Å)/HF–Si(110) film at different measurement temperatures.



FIG. 4. Hard axis hysteresis loops of a Co(500 Å)/Cr(500 Å)/Ag(750 Å)/HF-Si(110) film at different measurement temperatures.

in Fig. 4. The measurement spans from room temperature (25 °C) to 200 °C. M_S remains almost constant, without any observable decrease. K_1 shows a very strong temperature dependence. When the temperature is increased to 75 °C, K_1 has already dropped by 50%, from $1.55 \times 10^6 \text{ erg/cm}^3$ to 0.75×10^6 erg/cm³, as is also indicated by the hard axis loop showing a larger slope at the origin. K_1 continues to decrease with increasing temperature and crosses zero at 135 °C. The hard axis loops at 150 °C and 200 °C intersect with the vertical axis, confirming negative K_1 values. This zero-crossing temperature of 135 °C is considerably lower than the 250 °C at which K_1 of a bulk Co single crystal decreases to zero.⁷ K_2 , on the other hand, decreases slowly and remains positive at 200 °C. Both K_1 and K_2 measured at 25 °C after the sample was cooled back down from 200 °C are in excellent agreement with the values obtained before the heat treatment. This suggests that an internal film stress is unlikely to explain the small anisotropy values and the strong temperature dependence. The rapid decrease in K_1 is probably related to a high concentration of crystal defects in the films.

Anisotropy constants are usually considered to be intrinsic parameters of the materials. However, the observation of the unusually low K_1 values and the strong temperature dependence suggest that the anisotropy constants may be dependent on the thin film microstructure. Hence, processing conditions that modify the film microstructure are very likely to alter the anisotropy constants.

The anisotropy constants of unicrystal Co films were found to vary with the substrate temperature during the deposition. As shown in Fig. 5, K_1 is increased when the substrate temperature is increased from 250 °C to 300 °C. When the substrate temperature is further increased to 350 °C, how-



FIG. 5. Anisotropy constants of unicrystal Co(500 Å)/Cr(500 Å)/Ag(750 Å)/HF–Si(110) films grown with different substrate temperatures.



FIG. 6. Anisotropy constants of unicrystal Co(500 Å)/Cr(500 Å)/Ag(750 Å)/HF–Si(110) films with the Co layer deposited at different substrate bias voltages. The applied bias values are negative. There was no substrate bias during the deposition of the Cr and Ag layers.

ever, K_1 drops again. K_2 shows a similar dependence on the substrate temperature, only with smaller variation. At low temperatures the mobility of the Co atoms as they are being deposited is small. This may result in a larger concentration of crystal defects, and thus smaller anisotropy constants. On the other hand, Co is known to have a stable fcc phase above 422 °C.⁸ Therefore, as the temperature approaches 422 °C, although the overall structure is still hcp, the fcc stacking may become increasingly thermodynamically favored. This would result in a higher density of stacking faults, thereby decreasing the anisotropy constants.

Applying a substrate bias was found very effective in increasing K_1 . As shown in Fig. 6, K_1 increases as the applied substrate bias becomes more negative. Its value is almost doubled at a substrate bias of -300 V. Applying a substrate bias tends to remove those atoms loosely bound to the surface during the film growth, and leave those tightly bound ones to form more compact films with a higher degree of crystal perfection.⁹ Unicrystal Co films deposited with a substrate bias may contain fewer defects, and hence larger anisotropy constants.

NiAl has a crystal structure and lattice constant similar to those of Cr. NiAl underlayers have been used for Co-alloy media, and compared to Cr underlayers, the smaller and more uniform grain size in NiAl underlayers are preferable in order to achieve low noise media.¹⁰ Unicrystal $Co(10\overline{10})$ films were also successfully grown on NiAl(112), Cr(112)/ NiAl(112), and NiAl(112)/Cr(112) layers deposited on Ag(110)/HF-Si(110). The epitaxial relationships and the single easy axis orientation were confirmed using x-ray and electron diffraction. The anisotropy constants of the unicrystal Co films grown on four different underlayer structures are compared in Table I. Unicrystal Co films grown on Cr underlayers show larger K_1 values than those obtained on NiAl underlayers. The smaller K_1 values are undesirable since they compromise the thermal stability of hard disk media. However, when a 100 Å Cr intermediate layer is grown between the Co layer and the NiAl underlayer, K_1 is almost fully restored to the larger value obtained on Cr. Similarly, a NiAl intermediate layer on the Cr underlayer decreases K_1 to about the same value as that obtained on NiAl underlayers. Lee et al. reported that Cr intermediate layers on NiAl underlayers enhance the coercivities of the subsequently depos-

TABLE I. Comparison of the anisotropy constants of the unicrystal Co films grown on different underlayer structures. These underlayers were all epitaxially grown on Ag(750 Å)/HF–Si(110).

	<i>K</i> ₁	<i>K</i> ₂
Underlayer	(10^6 erg/cm^3)	
Cr(500 Å)	1.55	1.30
NiAl(500 Å)	1.24	1.20
Cr(100 Å)/NiAl(400 Å)	1.50	1.36
NiAl(100 Å)/Cr(400 Å)	1.27	1.15

ited Co-alloy films, while NiAl intermediate layers on Cr underlayers decrease the coercivities.¹¹ The results shown in Table I suggest that a possible cause of the coercivity enhancement and degradation may be the respective increase and decrease in the anisotropy constants. The comparison also suggests that the Co anisotropy constants are largely determined by the material directly underneath. Although NiAl(112) and Cr(112) surfaces show similar atomic spacings and both induce the epitaxial growth of unicrystal Co films, the difference in surface atoms has a significant effect on the anisotropy constants of the Co films.

In summary, we have studied the dependence of Co anisotropy constants on the measurement temperature, various processing conditions, and underlayer structures. The anisotropy constants are smaller than those for a bulk single crystal, and show very strong temperature dependence. Varying the growth temperature and substrate bias during the deposition can effectively change the anisotropy constants. While NiAl underlayers result in a smaller K_1 , the addition of Cr intermediate layers can restore K_1 to the larger value obtained on Cr underlayers. These results suggest that the anisotropy constants may be dependent on the thin film microstructure. The relationship between the anisotropy constants and the microstructure will be the topic of further investigation.

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