Stacking Faults in Smaller Grain Size Perpendicular Media

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The effect of stacking faults (SFs) on the uniaxial magnetocrystalline anisotropy (K_u) of perpendicular magnetic recording media is an important factor when the media grain size becomes smaller. In this study, the relationship between grain size, SFs, and the magnetic properties of the media has been evaluated. It was found that when the SiO₂ content in the Co-alloy granular thin film increases, the grain size and K_u decrease, and the amount of SFs increases. Keeping the same oxide volume fraction and decreasing the magnetic grain size does not further increase the amount of SFs much; however, the K_u value further decreases. This is likely due to the larger volume content of the possible presence of a deadlayer in the outermost shell of the smaller magnetic grains. The measured thermal energy barrier (E_B) and intrinsic switching field (H_0) decrease for smaller grain size media, determined by fitting the DC demagnetization (DCD) measurements with Sharrock's formula. Improving the structure and microstructure characteristics of the smaller grain size media will be important to obtain good magnetic and thermal properties.

Index Terms—Grain size, magnetocrystalline anisotropy, perpendicular magnetic recording media, stacking faults.

I. INTRODUCTION

■ HE thermal stability factor is determined by the uniaxial magnetocrystalline anisotropy (K_u) and grain volume (V) characteristics of the granular magnetic thin film media. In order to obtain high areal density and high signal-to-noise ratio, good properties such as large K_u , small grain size, and high thermal stability of media are required [1]. K_u of granular Co-alloy perpendicular magnetic recording media strongly depends on its crystallographic texture and microstructure. An ideal hexagonal-close-packed (hcp) structure is shown in Fig. 1. When stacking faults (SFs) are present in a thin film with hcp structure, they result in a lower K_u media, due to the lower anisotropy nature of the present face-centered-cubic (fcc) phase. This will lead to the nonthermally stable grains. An earlier study [2] pointed out the importance of the role of SiO₂ in the fcc phase increases at 30 at.% Pt addition and the degradation of K_u of CoPtCr–SiO₂ media compared with that of CoPtCr media. On the other hand, when the media has smaller grain size, the thermal fluctuation increases which aids the irreversible switching of magnetization overcoming the thermal energy barrier. Thus, magnetic viscosity phenomena can be often observed. This becomes worse when the grain size and K_u decrease at the same time. How the amount of SFs varies with grain size is important to understand the cause of K_u degradation. In this study, the relationship between SFs, grain size, K_u properties and magnetic after effect [3], [4] of the media will be discussed in detail.

II. EXPERIMENTAL METHOD AND DISCUSSIONS

Samples were sputter deposited by using a four-target Leybold–Heraeus Z-400 system with RF diode mode. The base pressure for deposition was $\sim 5 * 10^{-7}$ torr or better. A controlled sample: a continuous CoPt alloy thin film, a

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•••A •••B •••A HRTEM

hcp stacking

plane

Fig. 1. High-resolution TEM and schematic illustration of the hcp structure.

granular CoPt + oxide media of different grain sizes were subsequently prepared without a soft magnetic underlayer. The details of the sample structure are described in Table I. The composition for the magnetic alloy thin film is Co₇₃Pt₂₇. DC demagnetization (DCD) remanent curves were measured by a LakeShore vibrating sample magnetometer (VSM) to analyze the thermal energy barrier and intrinsic switching field in the magnetic media samples. [5] The maximum field was 18 kOe. The time duration at different applied field is 25, 50, 100, 200, 400, and 800 s. K_u was measured by the hard axis method using VSM as well and then corrected by the demagnetization factor. X-ray in-plane $\theta/2\theta$ scan was used to quantify the amount of SFs by means of detecting the integrated peak intensity ratio of $I(10.0)_{hcp}/[I(11.0)_{hcp} + I(220)_{fcc}]$ using an X'Pert diffractometer with Cu K α radiation. JOEL 2000 transmission electron microscopy (TEM) was utilized to study the microstructures of the thin films.

Table I shows the sample structures, grain sizes (defined as the mean grain-to-grain distance), $I(10.0)_{hcp}/[I(11.0)_{hcp} + I(220)_{fcc}]$ intensity ratios, SFs contents, K_u and M_s values for all the samples in this study. In order to quantify the amount of SFs in the CoPt grains, Lorentz-polarization factor [shown in (1)] has been utilized for geometrical correction. The SFs content can be calculated by using (1)–(4) and the measured $I(10.0)_{hcp}/[I(11.0)_{hcp} + I(220)_{fcc}]$ intensity ratio by the X-ray in-plane scans. After the calculation, sample 1: a continuous CoPt alloy thin film is found to have almost perfect hcp

TABLE IMAGNETIC GRAIN SIZE (DEFINED AS THE AVERAGE GRAIN-TO-GRAINDISTANCE), $I(10.0)_{hcp}/[I(11.0)_{hcp} + I(220)_{fcc}]$ INTENSITYRATIO, SFS CONTENT, K_u and M_s ValuesOF FOUR SAMPLES, RESPECTIVELY

Samples	CoPt Grain Size (nm)	CoPt Reflections	2 0 (deg)	Lorentz Factor	Measured I(10.0) _{hep} / (I(11.0) _{hep} +I(220) _{fee})	SFs Content (vol %)	K _u (erg/cm³)	Ms (emu/cm³)
<u>CoPt</u> \dual Ru\Ta\Si Sub.	22.0	(10.0) _{hcp}	39.37	14.93	1.85	0%	9.0 × 10 ⁶	679
		(11.0) _{hep} + (220) _{fee}	71.64	3.96				
<u>CoPt + 11 % SiO</u> \dual Ru\Ta\Si Sub.	9.8	(10.0) _{hep}	40.38	14.12	1.67	2.6 %	3.2 × 10 ⁶	635
		(11.0) _{hcp} + (220) _{fce}	73.23	3.80				
<u>CoPt + 17 % SiQ</u> \dual Ru\Ta\Si Sub.	7.5	(10.0) _{hep}	39.09	15.27	0.87	22.0 %	2.9 × 10 ⁶	660
		$(11.0)_{hcp} + (220)_{fee}$	71.89	3.94				
CoPt + 17 % SiQ ₂ \Ru + 10 % SiQ ₂ \Ru (low pressure, Ar-ion etched)\Ta\Si Sub.	5.3	(10.0) _{hcp}	39.77	14.60	0.70	29.1 %	1.4 × 10 ⁶	534
		(11.0) _{hcp} + (220) _{fcc}	72.13	3.92				

stacking and zero amount of fcc content. As 11 vol% oxide is cosputtered in the CoPt film, SFs content increases up to only 2.6%. However, as 17 vol% oxide is included in the thin film, the SFs content increases dramatically up to 22.0% and above depending on the grain size. These calculated SFs volume fractions are quite consistent with the observed SFs quantity directly determined from the high-resolution TEM images

Lorentz-polarization factor =
$$\frac{1 + \cos^2 2\theta}{\sin^2 \theta \cos \theta}$$
 (1)

measured-ratio_{CoPt} =
$$\left(\frac{I(10.0)_{hcp}}{I(11.0)_{hcp} + I(220)_{fcc}}\right)_{CoPt}$$
 (2)

$$\left(\frac{I(11.0)_{\rm hcp}}{I(220)_{\rm fcc}}\right)_{\rm CoPt} = \left(\frac{L_{(10.0)_{\rm hcp}}^p \times |F|_{(10.0)_{\rm hcp}}^2 \times V_{\rm hcp}}{L_{(220)_{\rm fcc}}^p \times |F|_{(220)_{\rm fcc}}^2 \times V_{\rm fcc}}\right)_{\rm CoPt}$$
(3)

$$\left(\frac{I(10.0)_{\rm hcp}}{I(11.0)_{\rm hcp}}\right)_{\rm CoPt} = \left(\frac{I(10.0)_{\rm hcp}}{I(11.0)_{\rm hcp}}\right)_{\rm Ru}.$$
(4)

Data in Table I generally suggests the fact that the presence of large amount of SFs is harmful to obtain high K_u granular oxide CoPt thin films. In the continuous CoPt thin film sputter deposited at room temperature, the grain size is as large as around 22 nm determined by plan-view TEM image. However, it has a very low SFs content, which results in a very high K_u property of 9.0 $\times 10^6$ erg/cm³. When 11 vol.% of an amorphous oxide phase such as SiO₂ is added into the CoPt magnetic alloy thin film, there is no significant increase in SFs content. However, the K_u drops quite a lot from 9.04 $\times 10^6$ to 3.17 $\times 10^6$ erg/cm³. The average CoPt grain size reduces from 22.0 to 9.8 nm as observed in the TEM images. Such K_u degradation does not seem to be strongly SFs related. With further increase of oxide volume fraction up to 17%, the noticeable increase in the amount of SFs has been observed. This could possibly explain the dramatic K_u drop from 3.2 $\times 10^6$ erg/cm³ to 2.9 $\times 10^6$ erg/cm³ or so. The grain size does further decrease to 7.5 nm. The X-ray diffracted peaks have broader width and reduced maximum intensity, which implies that the grain size is smaller as well. It seems that the addition of the excess 6 vol% SiO₂ greatly contributes to the high probability of faulted stacking of the atomic layers in CoPt grains comparing sample 2 and 3. In addition, when the grain size in the last sample further decreases to 5.3 nm by using Ar-ion etched Ru seedlayer method [8], the amount of SFs increases up to 29.1% and K_u value further drops quickly to 1.4 $\times 10^6$ erg/cm³. The slight increase in SFs content could not explain the big K_u difference from sample 3 to sample 4. Therefore, SFs does not seem to be the primary source of K_u decay when the magnetic grain size becomes smaller.

The size-dependent effect in the magnetic media is of great importance when the grain size becomes smaller and the surface to volume ratio increases. Assuming that at the outmost shell of the cylindrical shaped grain (at the interface between metal grain and oxide boundary), there is an effective thin deadlayer, which usually has a discontinuity of crystal grain structure, a high density of broken metal bonds, high oxygen content, strong surface anisotropy effect, etc. [9]. This thin deadlayer is assumed to have very low K_u property. Comparing sample 3 (~7.5-nm grain-to-grain distance and ~1.5-nm grain boundary thickness) with sample 4 (5.3-nm grain-to-grain distance and \sim 1.0-nm grain boundary thickness), there is an approximate 1.4 $\times 10^6$ erg/cm³ K_u loss when the effective deadlayer thicknesses in large grain size sample 3 and small grain size sample 4 are assumed to be 0.5 and 0.8 nm, respectively. Therefore, on top of such assumption and calculation, it is not surprising that the grain size effect leads to a significant K_u reduction, in addition to the slight increase of SFs content in the thin film. In order to justify the previous assumptions, the analytical TEM electron energy loss spectroscopy (EELS) cobalt and oxygen elemental mapping study of a cross-sectional sample of the Co-alloy + oxide recording layer in a typical perpendicular media was performed. The results are shown in Fig. 2. By means of carefully aligning the same sample area in different images, the contrast clearly shows that grain interior is Co rich and O deficient. The inverted contrast observed in the grain boundary region suggests the O richness and Co deficiency. In addition, there is a statistically wider distribution of the O element across the grain boundary compared with the nominal grain boundary width determined from the bright field image, although the magnetic grains can possibly be uniformly oxidized during sample preparation process. The relationship between the microstructure and K_u of the samples implies that enhanced segregation of oxide within the grain boundaries will be helpful to maintain high K_u of granular magnetic thin films of smaller grain size.

The thermal stability of the magnetic grains in the perpendicular magnetic recording media depends strongly on K_u . The following experiments have been performed to evaluate the magnetic viscosity effect on media of different grain sizes in sample 3 and sample 4. The remanent hysteresis loops of the granular oxide CoPt thin films samples 3 and 4 are measured at room temperature (298 K). The average coercivity (H_c) and remanent coercivity $(H_{c,r})$ of sample 3 measured at room temperature (298) K) are 4.8 and 5.0 kOe, respectively, whereas these values for sample 4 are 2.2 and 4.6 kOe, respectively. For both types of coercivities, sample 4 (small grain size) shows much lower values, indicating a much lower thermal stability. Nevertheless, in both cases, the remanent coercivity slightly decreases with increasing time duration during the measurement due to the greater effect of thermal agitation with longer time. It is also quite clear that the gap between the remanent loop and hysteresis loop is much larger for sample 4 compared to sample 3.



Fig. 2. EFTEM BF image of Co-alloy + oxide media and EELS elemental maps of Co and O elements (the same area).

The dynamic remanent coercivities can be determined by the Sharrock's formula [6] shown as follows:

$$H = H_0 \left\{ 1 - \left[\frac{k_B T}{K_u V} \ln \left(\frac{f_0 t}{\ln 2} \right) \right]^{1/2} \right\}$$
(5)

where the attempt frequency f_0 was assumed to be 10^{10} s⁻¹, k_B is the Boltzmann constant (1.38 × 10^{-23} J/K), T is absolute temperature, V_{act} is the activation volume for the magnetization switching, and H_0 is the intrinsic switching field [7].

In order to eliminate the effect of unquantifiable demagnetization factor in the thin film, the thermal effect is evaluated under the condition where 50% of the magnetization is reversed, whereas the net magnetization is equal to zero. The relationship between the applied reverse field H versus $\ln(f_0 t)^{1/2}$ of granular oxide CoPt thin film samples 3 and 4 under the condition whereas 50% of the magnetization is reversed after saturation (zero net magnetization) has been obtained. The data follows a linear fitting. The good agreement between linearity line fitting and the experimental data of the samples implies the fact that Sharrock's formula is a fair approximation. It can be utilized to obtain the intrinsic switching field H_0 and thermal energy barrier E_B for both samples. It was also found that the small grain size sample has a much smaller switching field, since the grains can be more easily switched under the same conditions. By fitting these curves with Sharrock's formula, the intrinsic switching field H_0 reduces from 6.7 to 3.8 kOe at 50% switching point when the grain size decreases. At the same time, the average thermal energy barrier drops from 6.8 to 4.2 eV. Therefore, the thermal stability factors decrease from 263 to 162 subsequently. The activation volume $V_{\rm act}$ has been calculated from the known thermal energy barrier and K_u values. It is 3791 nm³ for sample 3 and 4942 nm³ for sample 4. Assuming a cylindrical

shape of the magnetic grain with thickness of 13 nm, the activation diameter of magnetization switching is calculated to be 19.3 nm for sample 3 and 22.0 nm for sample 4. The difference between the activation diameter and grain size indicates that there is strong agglomeration between the few neighboring grains. When the grain size is smaller, this effect is more significant. The thermal energy barrier distribution or switching field distribution will not be further discussed here since the demagnetization factor cannot be accurately determined experimentally.

In conclusion, the high oxide volume fraction in granular Co-alloy + oxide perpendicular magnetic thin film media has been found to be a cause of large amount of present SFs. Reducing magnetic grain size with the same oxide content does not seem to further induce much more structural defects such as SFs. However, the increasing surface-to-volume ratio due to the smaller grain size can significantly increase the effective deadlayer amount and thickness in the outermost shell of the magnetic grains. This is likely to be related to the K_u loss. In addition, it has been found that the intrinsic switching field H_0 and thermal energy barrier E_B decrease with decreasing magnetic grain size. Such a grain-size-dependent effect is detrimental to the magnetic properties of the media. Sharrock's formula has been utilized as a good approximation to the experimental results. It could be utilized to characterize media of different magnetic properties.

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