Microstructure and magnetic properties of perpendicular media with reduced grain size

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A new approach to control the grain size of Co alloy oxide perpendicular media by means of Ar-ion etching is presented. Ar plasma was applied to the surface of a continuous Ru1 layer, on top of which a granular Ru2+SiO_2 layer was subsequently deposited. It was found that etching time and high Ar-ion energy have a significant effect on the surface roughness of the seedlayer, which in turn have a great effect on microstructure and magnetic properties of the media. A granular Ru2 +SiO_2 layer with mean grain size of 3.9 ± 0.7 nm was obtained, and its oxide boundary thickness decreased with the decreasing Ru grain size. As a result, the mean grain size of the magnetic layer was greatly reduced to 5.3 ± 0.8 nm from 7.6 ± 1.3 nm of a controlled sample without Ar-ion etching. The coercivity (H_c) of the media is 2.7 kOe, while H_c of the controlled sample is 4.8 kOe. The small CoPt grain size, the possible oxidization of metal grains, and the resultant increasing c-axis dispersion of the magnetic layer can explain the decreasing H_c and remanent magnetization (M_r) and increasing (H_n-H_c) values of the perpendicular MH loops. © *2009 American Institute of Physics*. [DOI: 10.1063/1.3070586]

I. INTRODUCTION

In the pursuit of high area density of hard disk drives, obtaining small grain size and narrow grain size distribution characteristics of the recording media is crucial for sharp transition and high signal to noise ratio.[1] Utilizing Ru +oxide interlayers under the magnetic layer for the purpose of grain size control is not a new idea;[2–4] however, the development of the ideal well-segregated small columnar grain microstructure of both interlayer and magnetic layer remains a challenge. Also, the question of how the grain size in the microstructure is related to the magnetic properties is of great interest. In our earlier study,[5] the Ru2 grain size was greatly reduced by means of increasing the nucleation rate of the Ru2 nuclei when the film forms on top of an Ar-ion etched Ru1 seedlayer. The media structure is schematically shown in Fig. 1. A granular Ru2+SiO_2 layer with average grain size of 3.9 ± 0.7 nm was obtained. As a result, the mean grain size of the magnetic layer was greatly reduced to 5.3 ± 0.8 nm. The microstructures of these two layers are shown in Fig. 2. Additionally, another study[6] showing that using Ar-ion etched Pt seedlayers effectively helped in reducing the grain size of FePt thin films indicates an important relationship between the significant surface roughening and smaller grain size characteristics as well.

In the present study, the relationship between the microstructure and magnetic properties of the perpendicular media with reduced grain size has been investigated. Analytical transmission electron microscopy (TEM) analysis of oxide (e.g., SiO_x) distribution in different media was performed, which explains the different shapes of the perpendicular MH hysteresis loops measured by vibrating sample magnetometer (VSM). It was found that lower coercivity (H_c), lower remanent magnetization (M_r), and increasing (H_n-H_c) values of media (H_n: nucleation field) could be the result of small grain size, the possible oxidization processing of metal grains, and the larger c-axis dispersion of CoPt grains in the magnetic layer, which cannot be easily determined by the regular bright field TEM imaging and x-ray diffraction (XRD) experiments. Discussion of this is the major focus of the following text.

II. EXPERIMENT

A controlled as-deposited thin film sample A: Ta\Ru1\Ru2+SiO_2\CoPt+SiO_2 and the other thin film

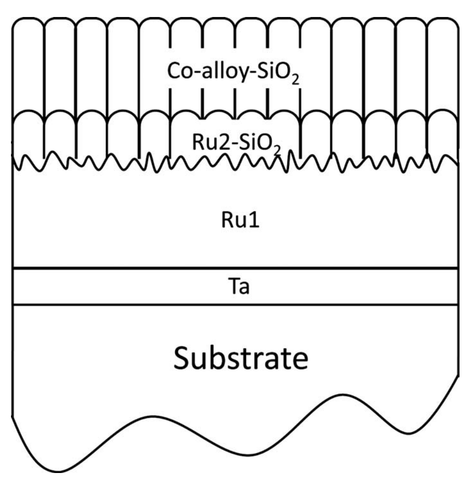

FIG. 1. The schematic illustration of perpendicular media structure (sample B).

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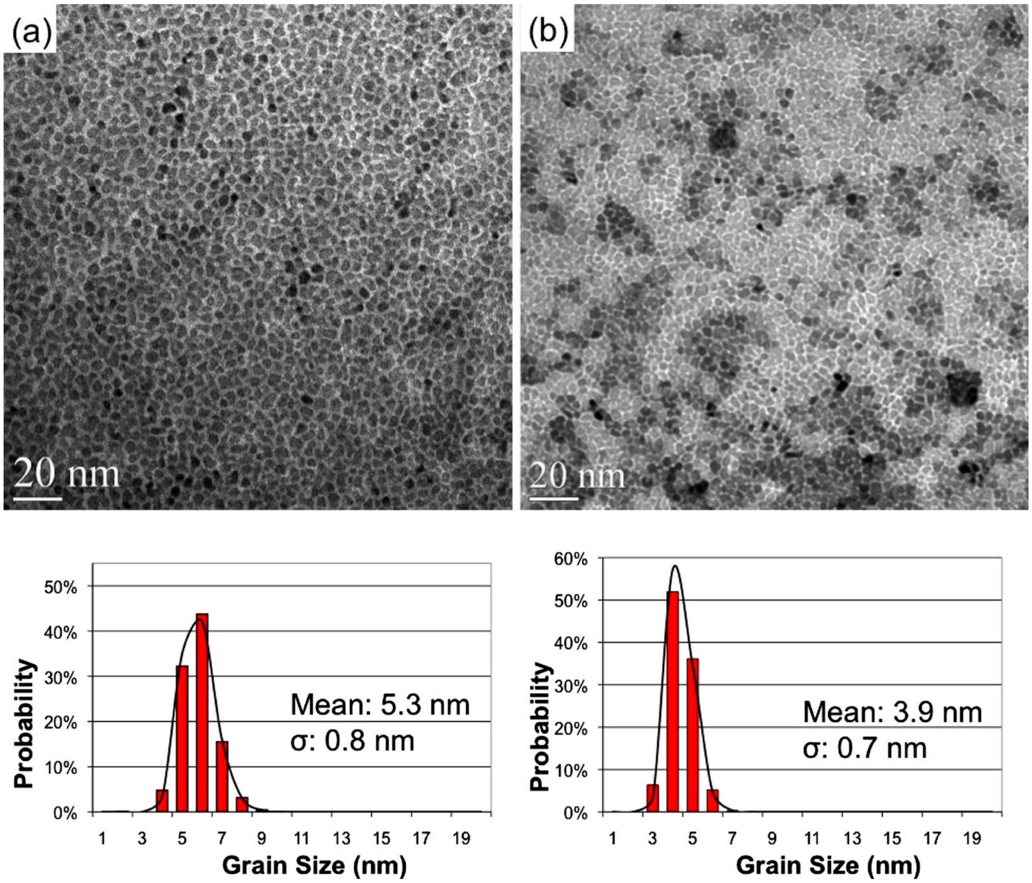

FIG. 2. (Color online) TEM plan-view images of (a) CoPt+SiO_2 layer and (b) Ru2+SiO_2 layer on Ar-ion etched Ru1 seedlayer in sample B.

sample B: Ta\Ru1 (Ar-ion etched)\Ru2+SiO_2\CoPt+SiO_2 (see Fig. 1) were deposited on the naturally oxidized 1 in. Si substrates by radio frequency diode sputtering on the Leybold-Heraeus Z-400 system. The base pressure was about 5×10^{-7} Torr and the argon pressure for the magnetic layer and Ru2+SiO_2 interlayer deposition was fixed at 45 mTorr under the low adatom mobility condition. The effect of surface etching with high Ar-ion energy on the magnetic properties of the media was investigated. The etching time was fixed at 20 min. JOEL 2000 TEM was utilized to study the microstructure of the thin films. Tecnai high resolution TEM was used to study the Si and O electron energy loss spectroscopy (EELS) elemental map in the CoPt+SiO_2 magnetic layer. A VSM was utilized to measure the perpendicular *MH* hysteresis loops.

III. RESULTS AND DISCUSSION

Figure 3 is the TEM EELS elemental mapping of the O *K* edge [Fig. 3(a)] and Si $L_{2,3}$ edge [Fig. 3(b)] of CoPt+SiO_2 magnetic layer in a controlled sample A and the O *K* edge [Fig. 3(c)] and Si $L_{2,3}$ edge [Fig. 3(d)] of CoPt+SiO_2 magnetic layer in a smaller grain size sample B, respectively. It can be seen that oxide segregates to the grain boundaries in both cases, which forms the well-known granular columnar microstructure in the films. Due to the immiscibility of Si with either Co or Pt elements, the EELS Si mapping usually delineates the metal grain morphology, while the white regions represent the locations of Si atoms in the grain boundary. More often, oxygen is seen to be located along with Si. However, the oxygen is more broadly distributed, which indicates a gradient of oxygen distribution from the center of the grain boundary toward the inner magnetic grain and the formation of an oxygen rich shell of the metal grains. In the controlled sample A [Figs. 3(a) and 3(b)], oxygen seems to have high concentration in the grain boundary as expected.

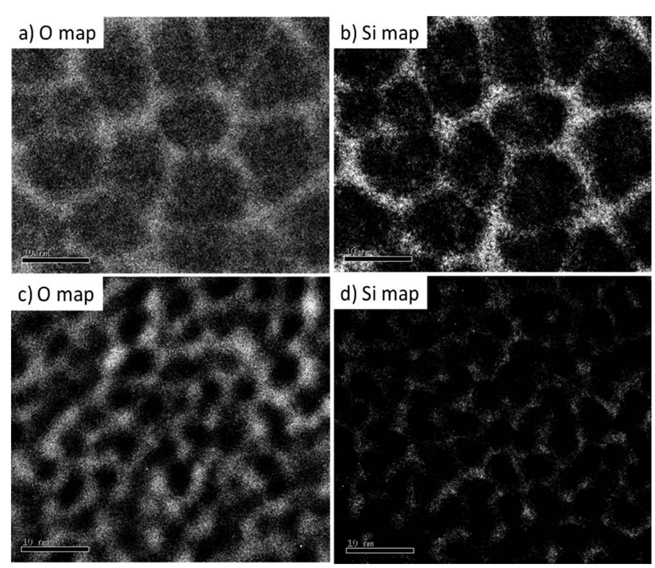

FIG. 3. TEM EELS elemental map of (a) O *K* edge and (b) Si $L_{2,3}$ edge of CoPt+SiO_2 magnetic layer in sample A; (c) O *K* edge and (d) Si $L_{2,3}$ edge of CoPt+SiO_2 magnetic layer in sample B.

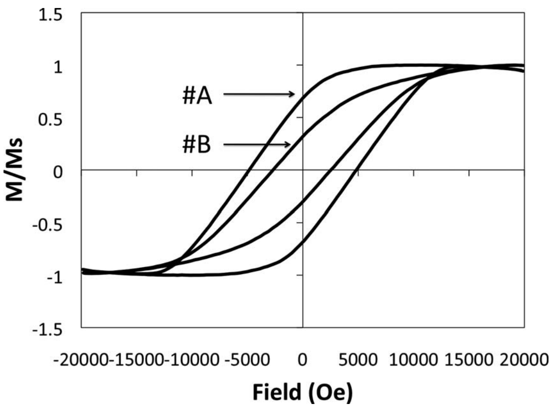

FIG. 4. Normalized perpendicular *MH* hysteresis loops of samples A and B measured by VSM.

However, it has very uniformly lower counts evenly in the inner grain region with very narrow variation from grain to grain. Si is mainly contained in the grain boundary as previously stated; however a few Si counts could be detected in the inner grain regions where the oxygen is relatively deficient as determined from the statistics. Compared with the controlled sample, the small grain sample B has a similar distribution of Si and O mainly in the grain boundary, but with noticeable differences as well. Due to the small grain size of this sample, the Si counts in the grain can hardly be detected since most of the Si atoms are confined in the grain boundary in this case. This is possibly due to the enhanced segregation induced by the smaller diffusion length would be needed when the metal grain size is smaller. There is much stronger oxygen contrast along the grain boundaries, which is possibly originated from the greater probability of adsorption/attachment of oxygen atoms to the grain surface due to the smaller grain size and increasing grain surface area. In such a way, less portion of oxygen is pumped away during deposition compared with the larger grain size sample. Additionally, the oxygen distribution variation inside the grain from grain to grain degrades very quickly. Although ~50% grains have very low oxygen content and well defined granular form, the rest of the grains have high oxygen content and resultant high density of Co–O bonds, which contribute to the presence of a low anisotropy and nonthermally stable grains. On the other hand, due to the small size of CoPt grains and similar gradient of oxygen distribution from the center of the grain boundary to the inner grain region, the area of net CoPt core in the grain is significantly reduced, which makes the situation even worse.

Figure 4 shows the normalized perpendicular *MH* hysteresis loops of controlled sample A (large grain size, as-grown Ru1 seedlayer) and sample B (small grain size, etched Ru1 seedlayer) by VSM. With decreasing grain size, the H_c drops from 4.8 kOe in the controlled sample toward 2.7 kOe in a smaller grain size sample. The $(H_n - H_c)$ value increases and the squareness decreases when the magnetic grain size decreases, which could be explained by both the high oxygen content in the CoPt grains and poorer grain orientation due to the etching treating to the seedlayer surface. Such a CoPt grain orientation degradation cannot be accurately determined from XRD rocking curve scans of its (00.2) peak since CoPt sits on the tail of (00.2) Ru peak for all the samples and they cannot be deconvoluted from each other very well. On the other hand, we know that the magnetic layer is fixed in these two samples due to the same sputter processing. Therefore, another cause is probably due to the higher density of Co–O bonds in the magnetic layer in a nonuniformly distributed manner. The origin of the possible increasing probability of Co–O may be from the increasing elemental distribution variation from grain to grain due to the small grain size referring to the EELS mapping results. The fact that H_c decreases with decreasing grain size could be more or less either related to the decreasing effective crystalline anisotropy energy (K_u) value judging from the low net grain area due to the small grain size or the increasing grain-to-grain elemental distribution variation as stated above or the possible increase in surface area and surface anisotropy, as discussed elsewhere.[7] On the other hand, since the interlayer Ru grain boundary thickness reduces from ~1.8 to ~0.5 nm, its effect on enhancing magnetic intergranular exchange decoupling may be significantly reduced, which might be the cause of nonideal heteroepitaxial grain growth and increasing grain-to-grain variation in terms of grain size distribution, elemental distribution, anisotropy distribution, switching field distribution, and resultant poorer overall magnetic properties.

IV. CONCLUSIONS

In the present work, we studied the relationship between the microstructure and magnetic properties of the media as a function of CoPt grain size in the magnetic layer. The variation in magnetic grain size was obtained by utilizing an Ar-ion etched seedlayer discussed elsewhere.[5] It was found that the degraded magnetic properties of smaller grain size media judging from the hysteresis loops could be due to the increasing nonuniform distribution of oxygen, larger grain-to-grain variation in Co–O bonds density, low K_u, and larger *c*-axis dispersion of the magnetic grains.

ACKNOWLEDGMENTS

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