Grain size reduction for perpendicular magnetic recording media using an Ar-ion etched Ru seedlayer

Hua Yuan[a)] and David E. Laughlin
Data Storage Systems Center, Department of Materials Science and Engineering, Carnegie Mellon University, Pittsburgh, Pennsylvania 15213, USA

(Received 3 July 2008; accepted 16 August 2008; published online 11 September 2008)

We report an approach to reduce grain size for perpendicular media by using an Ar-ion etched Ru seedlayer. The surface etching affects the microstructure of the subsequent high pressure deposited Ru2+SiO_2 interlayer and magnetic layer, whose grain size can be controlled. SiO_2 in the interlayer plays a more important role in grain size reduction on the etched seedlayer than the as-grown seedlayer. Small Ru2 grains ~3.9 nm and magnetic grains ~5.3 nm with uniform distribution have been obtained. Thinner Ru grain boundaries with reduced size could be the cause of nonideal one-to-one grain growth and degraded magnetic properties. © *2008 American Institute of Physics*.
[DOI: 10.1063/1.2979685]

High density magnetic recording requires granular perpendicular media of fine grain size, uniform distribution, high anisotropy, good exchange decoupling, narrow *c*-axis alignment, and small distance from the soft magnetic underlayer to the hard magnetic recording layer. An amorphous oxide phase is added to the sputtered polycrystalline interlayer or magnetic layer to control the grain size and grain segregation.[1–5] The grain size reduction of the interlayer is known to decrease the grain size of the magnetic layer as the heteroepitaxial growth forms between the thin films. Current media uses two layers of Ru, one to obtain strong (0002) texture and one to be a template for isolated grains. These are denoted as Ru1 and Ru2, respectively. In our previous study,[6] ultrafine columnar Ru2 grains (~2 nm) intermixed with different oxide phases were produced by means of oxide additions to the Ru2 interlayer of the dual Ru structure. Due to the nongranular Ru microstructure, the CoPt magnetic grains (~5.8 nm) could not follow the interlayer grain size and consequently grew on top of several Ru grains. Therefore, the formation of a well-defined granular Ru2 interlayer template is very important to produce small magnetic grains with a columnar microstructure and good recording properties.

In the present study, the Ru2 grain size has been well controlled by means of increasing the nucleation rate of the Ru2 nuclei when the film starts to form. It was found that by utilizing both the Ru1 seedlayer with a rough surface and a Ru2+SiO_2 interlayer, a well-isolated sub-4-nm interlayer film was produced. How the microstructure of the interlayers affects the magnetic layers was investigated in detail.

The film structures of our granular media are listed in Table I. All the films were sputter deposited on naturally oxidized 1 in. silicon substrates at room temperature by using a Leybold–Heraeus Z400 sputtering system with radio frequency sputtering method. The Ru1 seedlayer in samples c, d, C, and D was etched *in situ* with Ar ions after deposition. This etching process was performed under 15 mTorr pressure and 50 W power sputtering conditions. The etching rate of Ru is ~0.01 nm/s and the time for the surface etching was varied from 0 to 20 min. The magnetic layer of all the samples was kept the same. The *M*-*H* hysteresis loops were measured by the alternating gradient force magnetometer. The crystallographic texture of thin films was characterized by a Philips X'pert diffractometer using Cu $K\alpha$ radiation. The microstructures and surface morphology of the samples were analyzed on the JOEL 2000, Tecnai F20 transmission electron microscope (TEM) and a commercial Nanoscope Dimension 3100 atomic force microscope (AFM).

The surface condition of the Ru1 seedlayer was investigated by AFM technique. The Ru1 surface was bombarded by Ar ions for 0, 5, 10, and 20 min, respectively, after deposition. The final thickness of these samples was kept to be 15 nm. From the 1×1 μm^2 area scan, the average roughness value (R_a) of the samples increases slightly from 0.237 to 0.244 nm with increasing etching time from 0 to 20 min. A much more uniformly rough surface morphology appears with increasing etching time.

Figure 1 is a schematic illustration of layer structure of the media and the high resolution cross sectional TEM image of sample Si substrate\Ta(3 nm)\Ru1(15 nm, etched)\Ru2+SiO_2(3 nm)\CoPt+SiO_2(8 nm) (similar to sample D, but different thickness). Figure 2 is the plan-view TEM images

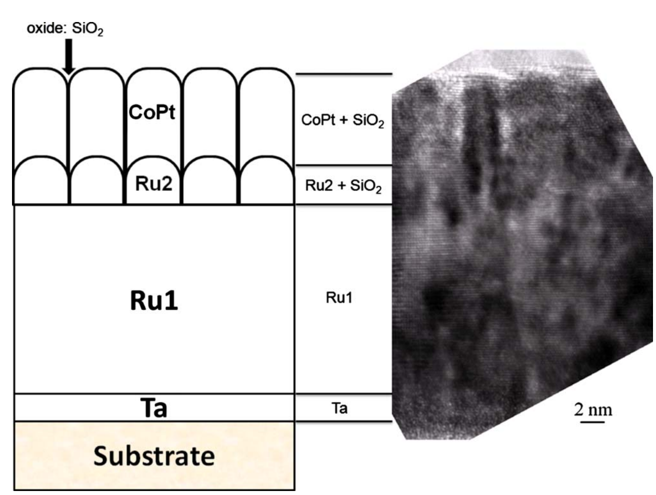

FIG. 1. (Color online) Schematic illustration of media structure and high resolution cross sectional TEM image of the sample Si substrate\Ta(3 nm)\Ru1(15 nm, etched)\Ru2+SiO_2(3 nm)\CoPt+SiO_2(8 nm).

[a)]Electronic mail: huay@andrew.cmu.edu.

TABLE I. List of samples sputter deposited on 1 in. silicon wafer.

Sample	Layer 1, adhesion layer	Layer 2, seedlayer	Layer 3, interlayer	Layer 4, magnetic layer
a	Ta (3 nm)	Ru1 (15 nm)	Ru2 (5 nm)	⋯
b	Ta (3 nm)	Ru1 (15 nm)	Ru2+10%SiO_2(5 nm)	⋯
c	Ta (3 nm)	Ru1 (15 nm) (etched)	Ru2 (5 nm)	⋯
d	Ta (3 nm)	Ru1 (15 nm) (etched)	Ru2+10%SiO_2 (5 nm)	⋯
A	Ta (3 nm)	Ru1 (15 nm)	Ru2 (5 nm)	CoPt+SiO_2 (13 nm)
B	Ta (3 nm)	Ru1 (15 nm)	Ru2+10%SiO_2 (5 nm)	CoPt+SiO_2 (13 nm)
C	Ta (3 nm)	Ru1 (15 nm) (etched)	Ru2 (5 nm)	CoPt+SiO_2 (13 nm)
D	Ta (3 nm)	Ru1 (15 nm) (etched)	Ru2+10%SiO_2 (5 nm)	CoPt+SiO_2 (13 nm)

of Ru2 based interlayers in samples a, b, c, and d. Comparing samples a and b [Figs. 2(a) and 2(b)], the 10 vol % SiO_2 addition is seen to greatly enhance the granular morphology and form clearer grain boundaries, which is consistent with a previous study.[1] The mean Ru grain size is as large as 7.5 nm. After depositing on the rough Ru1 seedlayer, it is clear that the mean grain size of the pure Ru2 layer decreases to ~6.2 nm [Fig. 2(c)]. Recalling the AFM results, the induced surface steps reduce the grain size by increasing nucleation sites and desorption energy of the adatoms or molecular species. Moreover, the lateral grain growth and coalescence process are limited by larger surface diffusion activation energy. Sample d [Fig. 2(d)] shows that the Ru size reduction effect is stronger when 10 vol % SiO_2 is codeposited within the Ru2 interlayer on top of the rough Ru1 layer. This secondary SiO_2 addition seems to help further limit the surface diffusion length due to the coexistence of immiscible metal and oxide phases with rough seedlayer surfaces. Therefore, the resultant Ru mean grain size is as small as ~3.9 nm with ~17.9% standard deviation, which is about the same as the ~17.3% grain size distribution in the conventional Ru2 interlayer.

Following this, a CoPt+SiO_2 layer was deposited on these four different interlayers a, b, c, and d, corresponding to samples A, B, C, and D, respectively (see Fig. 3). It was found that the CoPt grain size follows the size of Ru2 grains, implying that the two adjacent films almost have a one-to-one epitaxial growth. However, the smallest magnetic grain size is ~5.3 nm with ~15.1% distribution [Fig. 3(d)], which is ~1.4 nm larger than the Ru2 grain size. On the other hand, it was found that the Ru2 grain boundary thickness decreases from ~1.8 to ~0.5 nm with the decreasing grain size [see Fig. 2(d)]. It is most likely that due to the thinner Ru2 grain boundary, the grain size difference between the Ru2 and magnetic grains increases.

The x-ray diffraction $\theta/2\theta$ spectra and Ru (0002) rocking curve patterns of sample d with 15 nm Ru1 and a similar sample with 8 nm Ru1 indicate that the *c*-axis dispersion of the dual Ru layers is narrower with increasing Ru1 layer thickness. The full width at half maximum (FWHM) value for overall Ru (0002) is 3.9° when the Ru1 thickness is 8 nm. It is greatly reduced to 2.7° when the Ru1 thickness is 15 nm. It drops from 4.2° to 3.1° for CoPt (0002) accordingly, indicating an improved texture of the magnetic layer with a thick seedlayer. However, in both cases, the data suggest a well-maintained texture even though the Ru1 surface has been etched and an oxide has been added into the Ru2 interlayer during the fabrication process. Thus, the overall seed-

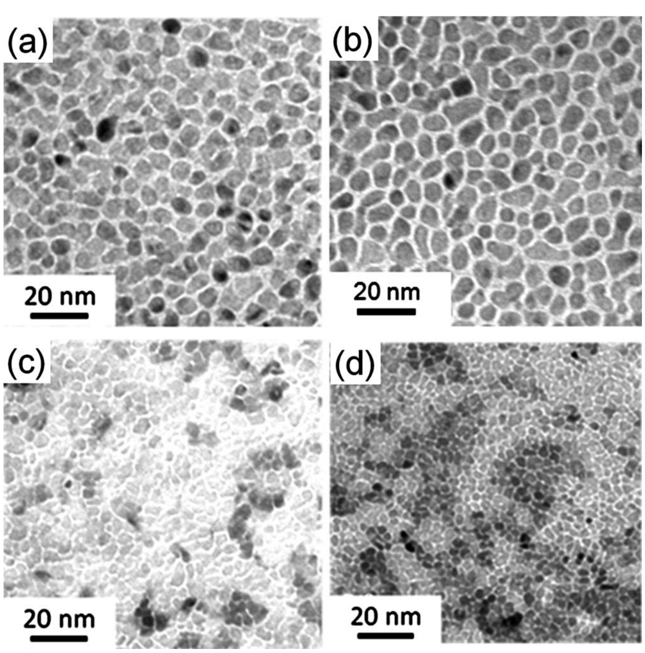

FIG. 2. Plan-view TEM images of the Ru2 based interlayers of (a) sample a, (b) sample b, (c) sample c, and (d) sample d.

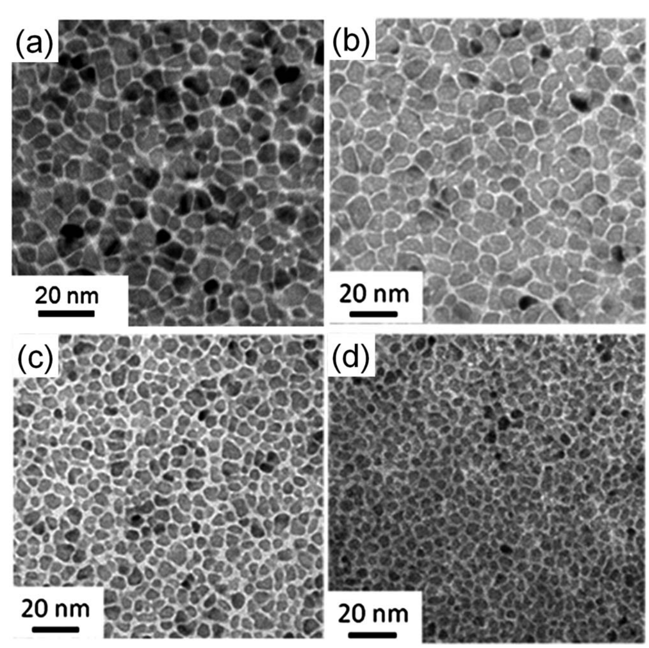

FIG. 3. Plan-view TEM images of the magnetic layers of (a) sample A, (b) sample B, (c) sample C, and (d) sample D.

layer and interlayer have a thickness as small as 16 nm (Ta: 3 nm; Ru1: 8 nm; Ru2+(SiO_2): 5 nm). Meanwhile, small grain size (~4 nm) and low FWHM (~4°) of the films have been obtained.

Figure 4 shows the coercivity (H_c) of the CoPt+SiO_2 layer in each sample with two interlayer thicknesses (5 and 10 nm). For each case, both oxide additions and Ru1 surface etching play an important role in reducing grain size, while the latter seems to be a strong factor. It is clear that H_c decreases with decreasing grain size. This could be due to the potential magnetocrystalline anisotropy energy (K_u) drop, indicating a possible grain size dependence on K_u. On the other hand, the smaller grain size interlayer has a much narrower grain boundary. This might cause a stronger intergranular exchange coupling and decrease H_c of the magnetic layer.

In the present work, the control of the grain size and grain size distribution in both the Ru2+(SiO_2) and CoPt +SiO_2 layers by means of *in situ* etching the surface of the as-deposited Ru1 seedlayer using Ar ions has been developed. A Ru2 grain size reduction from 7.5 ± 1.3 to 3.9 ± 0.7 nm and a CoPt grain size reduction from 7.6 ± 1.3 to 5.3 ± 0.8 nm have been obtained. The Ru2 grain boundary thickness reduces from ~1.8 to ~0.5 nm with decreasing grain size, which forms a nonideal one-to-one grain growth between the adjacent thin films. It was found that the surface treatment and oxide additions do not have a negative effect on the texture of the thin films. In comparison to the approach of adding large volume fraction of oxide in Ru interlayer and many other methods, this approach by means of fabricating rough Ru1 seedlayer is very promising in decreasing the Ru2 and Co alloy magnetic layer grain size and grain size distribution without deterioration of crystallographic orientation, while maintaining a very thin overall seedlayer and interlayer thickness. However, obtaining good magnetic properties of small grain size media on top of interlayers with a small grain size and thin grain boundary will be a challenge.

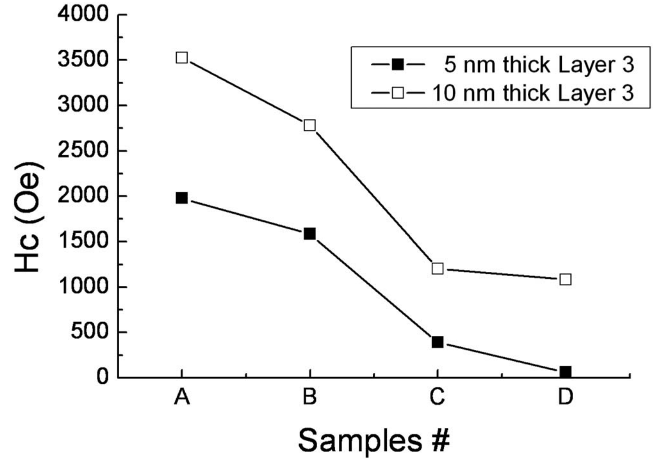

FIG. 4. Coercivity (H_c) for 13 nm thick CoPt+SiO_2 film in samples A, B, C, and D with two interlayer thicknesses of 5 and 10 nm.

The authors would like to thank Professor Jian-Gang Zhu at Carnegie Mellon University for helpful discussions and also the Seagate Technology, Pittsburgh, and Data Storage Systems Center of Carnegie Mellon University for financial support.

[1]U. Kwon, R. Sinclair, E. M. T. Velu, S. Malhotra, and G. Bertero, IEEE Trans. Magn. **41**, 3193 (2005).
[2]I. Takekuma, R. Araki, M. Igarashi, H. Nemoto, I. Tamai, Y. Hirayama, and Y. Hosoe, J. Appl. Phys. **99**, 08E713 (2006).
[3]S. N. Piramanayagam, J. Z. Shi, H. B. Zhao, C. K. Pock, C. S. Mah, C. Y. Ong, J. M. Zhao, J. Zhang, Y. S. Kay, and L. Lu, IEEE Trans. Magn. **43**, 633 (2007).
[4]S. N. Piramanayagam and K. Srinivasan, Appl. Phys. Lett. **91**, 142508 (2007).
[5]K. Srinivasan, S. N. Piramanayagam, S. K. Wong, and Y. S. Kay, J. Appl. Phys. **103**, 07F512 (2008).
[6]H. Yuan, D. E. Laughlin, X. Zhu and B. Lu, J. Appl. Phys. **103**, 07F513 (2008).