Fabrication, Microstructure, Magnetic, and Recording Properties of Percolated Perpendicular Media

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In this paper, a new type of perpendicular magnetic media which we have termed *percolated perpendicular media* is discussed. We present the method we used to fabricate the media as well as an energetic rationale for the driving force to produce the desired microstructure. The microstructures of samples with various amounts of oxide material are presented and the optimum one for our investigation is presented. We also present some preliminary drag test data that can be used to evaluate the recording properties of this media.

Index Terms—Domain wall pinning, microstructure, percolated media, perpendicular magnetic media, recording properties.

I. INTRODUCTION

T HE magnetic recording media that is currently in use, whether it be longitudinal or perpendicular, consists of closely packed Co alloy grains that are exchanged decoupled by either chemical segregation (e.g., Cr) in longitudinal media or an oxide phase (e.g., SiO₂) in perpendicular media. The grain size of such media has continued to decrease. Such media microstructure with isolated or nearly isolated magnetic grains has enabled the rapid area recording density growth over the past 15 years. However, the size of the magnetic grains is approaching the superparamagnetic limit and this granular microstructure is estimated to have an area density limitation less than 500 Gb/in² for today's hard disk drive systems.

To be able to keep increasing the areal density of media, a new paradigm for magnetic recording is needed. Research is progressing on patterned media [1] as well as heat assisted magnetic recording (HAMR) [2], [3]. Both of these methods will entail a new hard disk drive platform. In this paper we will discuss a novel medium microstructure, which we have denoted as percolated perpendicular media [4], [5] which does not necessitate a new hard disk drive platform. Percolated perpendicular media consists of a polycrystalline thin film of magnetic material with dense, evenly distributed nonmagnetic entities (see Fig. 1). The magnetic grains are percolated and hence magnetically coupled and have strong perpendicular magnetic anisotropy due to crystallographic orientation. The nonmagnetic entities (white regions) are small (\sim 3 to 5 nm) and act as pinning sites for domain walls. The distance between the nonmagnetic entities is also small, which essentially determines the magnitude of the transition noise. These domain wall pinning sites can be either nonmagnetic materials such as oxides, or physical



Fig. 1. Schematic of the ideal microstructure of percolated perpendicular media. The top is a plan view of the ideal microstructure and the bottom is a cross section view of the ideal microstructure. The white regions represent columns of the nonmagnetic constituent.

voids. The magnetic material in which the nonmagnetic pinning sites reside can be polycrystalline, but should have strong perpendicular magnetic anisotropy. The ferromagnetic exchange coupling between adjacent grains is maintained through proper film microstructure.

An important property of percolated perpendicular media is that the media enables a much lower transition noise since the separation between the adjacent pinning sites can be much smaller than the grain size in today's conventional perpendicular media, while the exchange coupling in the medium enables sufficient thermal magnetic stability. The coercivity of the medium can be engineered so that sufficient overwrite

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Fig. 2. (a) Schematic of the sample stacking layers: $substrate/Ta(5 nm)/Ru(30 nm)/CoPt-SiO_2(5 nm)$ and (b) alternate sputtering.

capability can be achieved. An advantage of this medium is that it maintains sufficient thermal magnetic stability without requiring very high medium coercivity. This is because the medium is continuous and hence all the grains are exchange coupled. For perpendicular media, the strongest magnetization decay arises from a fully saturated region where the demagnetization field is the strongest. That is also the place the nucleation of a domain is the most difficult. In another words, the thermal magnetic stability of the medium has to do with the nucleation field which could be relatively independent of the medium coercivity.

A comprehensive micromagnetic simulation study on such media is reported in this proceedings [5] in addition to the preliminary micromagnetic calculations that have been reported earlier [6], [7]. In this research, we present the method used to fabricate the media and the preliminary results on the microstructure and magnetic properties of $Co-Pt/SiO_2$ films.

II. METHOD OF FABRICATION OF PERCOLATED PERPENDICULAR MEDIA

Co–Pt/SiO₂ films and Ru/Ta underlayers were deposited on Si substrates by RF magnetron sputtering. The Co–Pt/SiO₂ films were fabricated by the alternate sputtering of Co–16%Pt and SiO₂ targets as shown in Fig. 2. The Ar pressure was maintained at approximately 10 mTorr for all the samples. X-ray diffraction (XRD) and transmission electron microscopy (TEM) were used to study the microstructure of the films. A polar magnetooptical Kerr effect (MOKE) looper was used to study the magnetic properties.

The magnetic layer of current perpendicular media consists of small *hcp* Co alloy columnar grains that are surrounded by an amorphous oxide such as Silicon oxide or Titanium oxide. The plan view microstructure of such media looks similar to that shown in Fig. 3(a). The oxide acts to magnetically isolate the grains from each other. Current media have grains of about 6-10 nm and oxide thickness less than about 1 nm.

To obtain the desired microstructure for percolated perpendicular media, we first produce the microstructure shown in Fig. 3(a) by successive sputtering of oxide and *hcp* Co–Pt films. The volume fraction of oxide can be controlled by the relative sputtering times and hence the thickness of the two materials. The microstructure of this media shows a grain size of about 8 nm and an oxide thickness of about 1.2 nm.

The micrograph shown in Fig. 3(a) has about 17% SiO₂ and the rest is Co-Pt. The magnetic film with this microstructure has a low coercivity, likely due to incomplete segregation of SiO₂ towards grain boundaries, which could result in insufficient intergranular exchange decoupling [8] and lessened crystalline anisotropy strength. After annealing at 600 °C for one minute, the coercivity increases dramatically to a value of about 4 kOe. See Fig. 3(c). The magnetic grains shown in Fig. 3(c)are magnetically interconnected, that is, they are magnetically percolated. In the microstructure shown in Fig. 3(c), the oxide particles, which are columnar shaped rods, have a diameter of about 4 nm and a spacing of about 13 nm. The hcp Co-Pt grains remain highly textured crystallographically, with their hexagonal c-axes perpendicular to the film. The grain size of the annealed films is slightly larger than the as deposited films. The oxide phase is in the shape of columns, and they pin the magnetic domain walls, hindering their motion and hence producing the increased coercivity.

Heating the sample at 550 °C for one minute [Fig. 3(b)] does not change the microstructure enough to cause complete interconnectivity of the magnetic grains. Hence, the coercivity of this film does not increase. Annealing for longer times at 550 °C would cause the microstructure (and hence coercivity) to approach that of the film shown in Fig. 3(c), but we want to minimize the annealing time so as not to cause interdiffusion of the various layers of the media structure. X-ray diffraction patterns displaying the (0002) peaks of the Ru underlayer and Co–Pt hcp film as a function of annealing temperatures are shown in Fig. 4. The films show a highly textured microstructure which remained after the annealing. The *c*-axes of the films became smaller and closer to their bulk values after the annealing, as shown of the peak shift in the inset of Fig. 4.

Schematic plan view representations of the two microstructures of Figs. 3(a) and (c) are shown in Fig. 5. The oxide/metal interface has a high specific energy and the total surface energy of the composite oxide/Co-Pt alloy film can be reduced by lowering the interfacial area. One way that this can be done is by changing the microstructure shown in Fig. 5(a) (that is the microstructure in which the oxide surrounds the Co-Pt grains) to the microstructure shown in Fig. 5(b), the one in which the magnetic hcp Co-Pt is percolated. Calculations show that the total oxide/Co-Pt interface area is less for the microstructure shown in Fig. 5(b) than for that of the microstructure shown in Fig. 5(a)when the oxide volume fraction is less than about 35.5%. For volume fractions of oxide higher than this, the microstructure of Fig. 5(a) with the oxide surrounding the Co–Pt grains is more stable than the one shown in Fig. 5(b). Details of the calculations are shown in the Appendix.

It should be emphasized that the calculation in the Appendix is valid only for the specific microstructures shown in Fig. 5. If there are fewer columns of oxide per grain of magnetic material produced, the maximum amount of oxide possible for a percolated magnetic grain structure will be higher. Also, if the samples are heated for extended times, the columns of the microstructure simulated in Fig. 5(b) will break up by the Rayleigh instability [9], [10] into spherical regions, giving rise to more,



Fig. 3. Microstructure and MOKE Hysteresis loops of Co-Pt-17% SiO₂ thin films: (a) as deposited, (b) after annealing at 550 °C for one minute and (c) after annealing at 600 °C for one minute.



Fig. 4. Conventional $2\theta/\theta$ XRD spectra for a 5 nm CoPt–17%SiO₂ media layer on a 30 nm Ru underlayer. The inset shows the magnified area around 42° .



Fig. 5. Schematic microstructure of thin films showing (a) percolated oxide and (b) percolated magnetic grains.

but weaker pinning sites. Such spheres of the oxide are difficult to distinguish from columns by plan view transmission electron microscopy, as the projected images of both look the same, namely as circular regions. It is necessary to utilize cross sectional transmission electron microscopy to observe if the columnar rods have broken into spheres. The annealing process is one in which lower energy microstructures are obtained. With higher annealing temperatures and longer annealing times lower energy microstructures will be obtained. It should be pointed out that the optimum microstructure for percolated perpendicular media is not the one with the lowest energy (that is not the equilibrium microstructure), so care must be taken to stop the evolution of the oxide microstructure at the point when columns occur.

The larger the volume fraction of the oxide, the larger will be the radius of the oxide column. See (3) in the Appendix. Since the size and spacing of the oxide columns determines the extrinsic magnetic properties of the magnetic film, there will be an optimum amount of oxide. To obtain the optimum value of the oxide and hence the optimum microstructure, and magnetic properties, for percolated perpendicular media we fabricated films of oxide amounts varying from 7% through 50% oxide. These films were annealed at temperatures varying from 500 °C to 700 °C for various times. As indicated above, the best temperature for annealing was found to be 600 °C. Fig. 6 shows the annealed microstructure of samples with 9%, 20%, and 33% oxide, annealed at 600 °C for one minute. Clearly the sample with 20% oxide yields the highest coercivity after annealing. It can be seen in this figure that the sample with 33% oxide does not change to the columnar arrangement of the oxide after annealing for one minute at 600 °C. Also, note, that the sample with 9% oxide has smaller and fewer columns of oxide in the annealed microstructure [Fig. 6(a)].

0.10 0.10 0.10 0.05 0.05 0.05 Kerr Rotation [a.u.] Kerr Rotation [a.u.] Kerr Rotation [a.u.] 0.00 0.00 0.00 -0.05 -0.05 -0.05 -0.10 -0.10 -0.10 -12 -10 -8 -6 -4 -2 0 2 6 8 10 12 -12 -10 -8 10 12 -4 -2 0 2 8 4 -12-10-8-6-4-202 6 10 12 -6 4 6 4 8 Applied Field [kOe] Applied Field [kOe] Applied Field [kOe]

Fig. 6. Microstructure and MOKE Hysteresis loops of Co–Pt thin films after annealing at 600 $^{\circ}$ C for one minute, with a SiO₂ volume fraction of 9% (a), 20% (b), and 33% (c).

Recording testing was performed using a scanning contact recording tester. A perpendicular write head with trailing shield and a current-in-plane spin valve head were used for recording and read back, respectively. Note the percolated perpendicular media samples are without a soft magnetic underlayer. The samples are mounted on an x-y piezo stage, which has a position accuracy of 0.5 nm. The head is in contact with the medium and bipolarity current pulses are supplied to the recording head. Fig. 7 shows a two-dimensional read-back image of recorded tracks at 80 nm and 100 nm transition spacing. Well written transitions are observed at these densities. Since the spacing between adjacent pinning sites in this medium is around 11 nm, similar to grain size, we did not expect the performance of this particular example of this medium to surpass the conventional granular perpendicular thin film media.

For the fabrication process described in this paper, the density of the pinning sites, i.e., the separation between adjacent pinning sites, can be controlled by controlling the grain size. It is also possible to produce the same final microstructure with desirable pinning sites via cosputtering and possible substrate heating.

III. CONCLUSION

We have presented our work to date on the fabrication of percolated perpendicular media. A range of oxide percentages, annealing temperatures and times were investigated and the optimum values for the fabrication of percolated perpendicular media were determined. A model to aid us in setting various parameters was developed. To date, the percolated perpendicular media is very promising, and with more experimental work we hope to continue to improve the magnetic recording properties of the media.



Fig. 7. Two-dimensional read-back image of tracks recorded at various transition spacings.

Appendix Details of the Calculation of the Microstructural Stability

In Fig. 5(a), we have assumed that the Co–Pt grains are hexagonal columns, with SiO₂ as the matrix. The Co–Pt grains have an average size of h, and the thickness of the SiO₂ between the Co–Pt grains is denoted as t. The volume of a Co–Pt grain and the volume of the SiO₂ phase that is associated with it are given as

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$$V_{\rm Co-Pt} = 2\sqrt{3}h^2 \tag{1}$$

and

$$V_{\rm SiO_2} = 2\sqrt{3}(ht + t^2/4).$$
 (2)

After annealing, we assume that a cylinder of SiO_2 of radius r is formed at each of the grain vortices. Assuming constant volume of SiO_2 we obtain

$$V_{\rm SiO_2} = 2\sqrt{3}(ht + t^2/4) = 2\pi r^2.$$
 (3)

Therefore, the radius of SiO_2 pinning site in Fig. 5(b) is

$$r = \left[\sqrt{3}/\pi (ht + t^2/4)\right]^{1/2}.$$
(4)

The metal/oxide interface area for a Co–Pt grain before annealing [Fig. 5(a)] is given as

$$S_a = 4\sqrt{3}h.$$
 (5)

And after annealing [Fig. 5(b)], it is given as

$$S_b = 4\pi r. \tag{6}$$

Since each column of oxide is shared by three grains, there are two SiO₂ pinning sites per Co–Pt grain.

The microstructure in Fig. 5(b) will be more stable than the one in 5(a) if $S_a > S_b$, or

$$4\sqrt{3h} > 4\pi r.$$
 (7)

Replacing r in (7) by (4) yields

$$t/h < 2[(1+\sqrt{3}/\pi)^{1/2} - 1] \cong 0.491.$$
 (8)

The volume ratio of SiO₂ to Co-Pt is

$$\frac{V_{\rm SiO2}}{V_{\rm Co-Pt}} = t/h + 1/4(t/h)^2 < 0.55$$
(9)

or

$$V_{\rm SiO_2}\% < 35.5\%.$$
 (10)

In this calculation, only change of the metal/oxide interface energy is considered. If the metal/metal boundaries in Fig. 5(b) are taken into account, the volume fraction of SiO₂ will be even smaller. Also, surface curvature was not taken into account. It should be also noted that Fig. 5(b) is only an intermittent stage, upon further annealing, SiO₂ will continue to grow in order to reduce interfacial area and hence energy. Thus, the annealing temperature and time must be carefully controlled to obtain the optimum microstructure.

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