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Multiple oxide content media for columnar grain growth in L1₀ FePt thin films

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An approach to enhance the height-to-diameter ratio of FePt grains in heat-assisted magnetic recording media is proposed. The FePt-SiO_x thin films are deposited with a decrease of the SiO_x percentage along the film growth direction. When bi-layer and tri-layer media are sputtered at 410 °C, we observe discontinuities in the FePt grains at interfaces between layers, which lead to poor epitaxial growth. Due to increased atomic diffusion, the bi-layer media sputtered at 450 °C is shown to (1) grow into continuous columnar grains with similar size as single-layer media but much higher aspect ratio, (2) have better L1₀ ordering and larger coercivity. © 2013 American Institute of Physics. [<http://dx.doi.org/10.1063/1.4798295>]

Considerable attention has been given to L1₀ FePt as the most probable material for the next generation magnetic recording media due to its high magnetocrystalline anisotropy of $\sim 5\text{--}7 \times 10^7 \text{ erg/cm}^3$. Achieving good grain morphology, strong perpendicular (001) texture, and high chemical ordering still remain challenging for its commercial applications to be realized. Control of grain morphology including small grain size with a narrow size distribution, good boundary isolation, and exchange decoupling has been attempted by adding segregants into the FePt magnetic thin films. Various segregants such as C (Refs. 1 and 2), B (Ref. 3), MgO (Ref. 4), TiO₂ (Ref. 5), Ta₂O₅ (Ref. 6), and SiO_x (Ref. 7) have been studied. FePt-C granular systems have been reported to bring about small, uniform, and isolated grains, but the FePt grains are sphere-like.^{2,8} For media in the tetrabit/in² regime, a columnar morphology is more desirable to gain thermal stability. In our previous work, the SiO_x segregant was shown to promote columnar growth for FePt grains.⁹ Small columnar grains with a large SiO_x percentage, however, become unstable with respect to their columnar shape. For instance, when the oxide volume fraction increases from 37% to 45%, 16 nm thick FePt films deposited at 410 °C will break up into double layers to minimize the total surface energy [Figures 1(a) and 1(b)]. The film thickness has to be reduced to 9 nm for the FePt grains to remain columnar [Figure 1(c)]. It has been found⁹ that the formation of the secondary layer of FePt grains results in poor perpendicular texture and hence negatively affects the magnetic properties of the media. In this paper, we present an approach for the solution of this problem, namely a multiple oxide content media, to control the columnar grain growth of L1₀ FePt thin films. The media are composed of a stack of FePt-SiO_x layers, with the oxide volume fraction in each layer decreased from bottom to top. Our objective is to demonstrate the columnar growth condition for 16 nm FePt-45%SiO_x via the proposed fabrication technique.

Films were fabricated by rf sputtering, using a Leybold Heraeus Z400 sputtering system at a base pressure

$< 5 \times 10^{-7}$ Torr and an Ar sputtering pressure of 10 mTorr. FePt-SiO_x media were multi-deposited from Fe₅₅Pt₄₅ and SiO_x targets onto naturally oxidized 1 in. Si(001) wafers. The thickness of FePt in each step was 0.76 nm and that of SiO_x was varied for different oxide volume fractions. MgO (15 nm) underlayers were sputtered at room temperature and

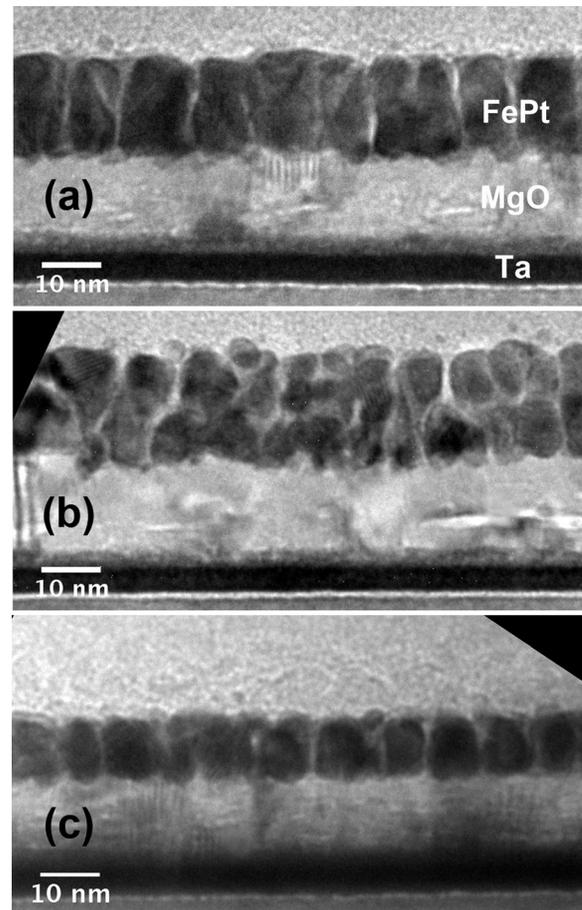


FIG. 1. Cross-section TEM images of (a) 16 nm FePt-37vol. %SiO_x, (b) 16 nm FePt-45vol. %SiO_x, and (c) 9 nm FePt-45 vol. %SiO_x.

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TABLE I. List of samples sputter deposited onto 1 in. Si wafers.

Sample	FePt-SiO _x film stack	Deposition temperature	Overall media thickness	Overall SiO _x vol. %
Single-layer S	9 nm – 45%SiO _x	410 °C	9 nm	45%
Tri-layer T	5 nm – 37%SiO _x , 5 nm – 45%SiO _x , 6 nm – 55%SiO _x	410 °C	16 nm	46%
Bi-layer B1	10 nm – 37%SiO _x , 6 nm – 55%SiO _x	410 °C	16 nm	44%
Bi-layer B2	10 nm – 37%SiO _x , 6 nm – 55%SiO _x	450 °C	16 nm	44%

then heated to 410 °C or 450 °C prior to deposition of FePt-SiO_x. The deposition rates of FePt, SiO_x, and MgO were 0.710 Å/s, 0.263 Å/s, and 0.147 Å/s, respectively. The film texture and microstructure were characterized by X-ray diffraction (XRD) with CuK α radiation and transmission electron microscopy (TEM). The magnetic properties were tested at room temperature by a vibrating sample magnetometer (VSM) with a maximum applied field of 9 T.

To obtain 16 nm thick multiple oxide content media, tri-layer FePt-37%SiO_x(5 nm)/FePt-45%SiO_x(5 nm)/FePt-55%SiO_x(6 nm)/MgO and bi-layer FePt-37%SiO_x(10 nm)/FePt-55%SiO_x(6 nm)/MgO media were prepared. The SiO_x volume percentage was estimated from the relative thickness ratio of SiO_x and FePt. The thickness of each FePt-SiO_x layer is chosen to ensure columnar growth conditions at the corresponding oxide volume fraction.⁹ The overall volume fractions in those two media were 46% and 44%, respectively. A 9 nm thick single layer FePt-45%SiO_x was also fabricated for comparison. The film structures of our samples are summarized in Table I.

At a deposition temperature of 410 °C, the single layer S media reveals a smooth columnar granular microstructure [Figure 2(a)]. In the tri-layer T and bi-layer B1 media, discontinuities at the interfaces between the FePt-SiO_x layers are observed as shown in the cross-section TEM images [Figures 2(b) and 2(c)]. The appearance of FePt (111) peaks from both tri-layer and bi-layer structures in their XRD patterns [Figures 3(b) and 3(c)] suggests that epitaxial growth between FePt-SiO_x layers is not perfect, especially in the tri-layer media. The XRD results are consistent with the observed microstructure. The L1₀ order parameter, *S*, of the magnetic films is fairly low: 0.48, 0.36, and 0.45 for the single, tri-, and bi-layer media, respectively. *S* is calculated from the integral intensity ratio of FePt (001) and FePt (002) peaks with absorption and Lorentz correction factors included.¹⁰ The low chemical order in the films results in low coercivities, *H_c* in the perpendicular M-H loops, which are listed in Table II. Since there are more interfaces or discontinuities in the tri-layer media, this deteriorates the epitaxial growth of FePt more and hence makes *S* and *H_c* smaller than the bi-layer media.

Further raising the deposition temperature of the bi-layer media to 450 °C was found to effectively enhance the atomic diffusion, which assists in minimizing the discontinuities, promoting epitaxial growth between layers, and improving L1₀ atomic ordering. Better diffusion of SiO_x to the top surface during sputtering homogenizes the oxide along the FePt grain thickness. As displayed in Figure 2(d), the columnar grains become more continuous. The increase of *S* to 0.65 and a shifting of FePt (002) peak relative to FePt (001) peak to a much higher angle in the XRD pattern of the

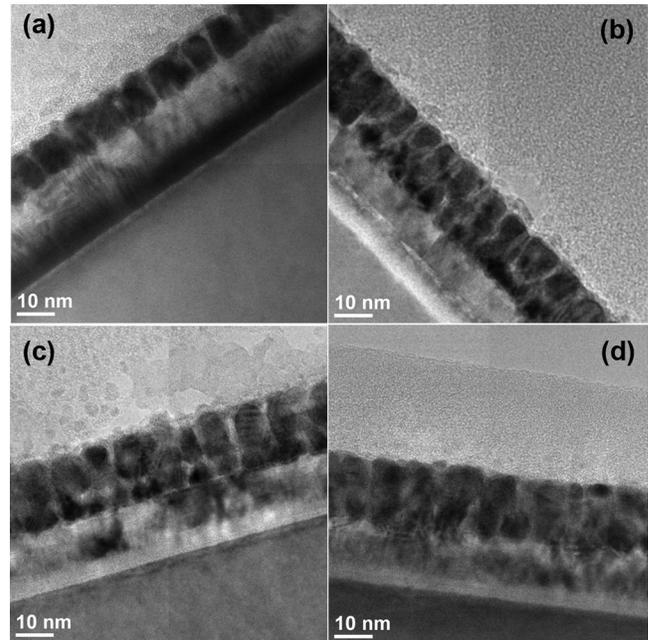


FIG. 2. Cross-section TEM images of (a) S, (b) T, (c) B1, and (d) B2.

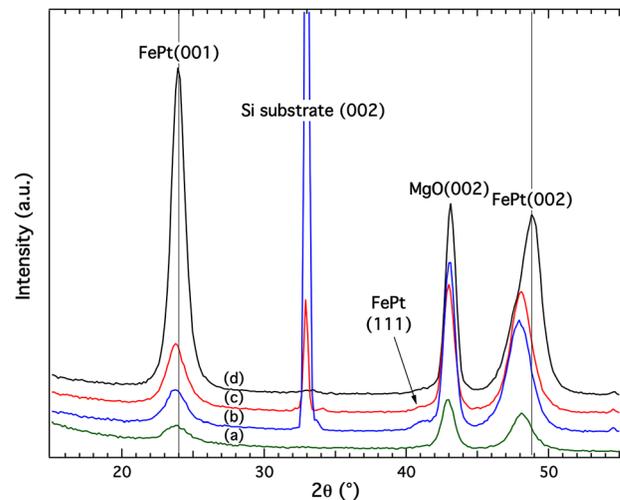


FIG. 3. XRD spectra of sample (a) S, (b) T, (c) B1, and (d) B2.

TABLE II. Summary of *I*₀₀₁/*I*₀₀₂ ratio, order parameter *S*, average FePt grain size, and *H_c* of the samples.

Sample	Integral <i>I</i> ₀₀₁ / <i>I</i> ₀₀₂	Order parameter <i>S</i>	Average FePt grain Size	Coercivity <i>H_c</i>
S	0.56	0.48	9.7 nm	0.9 kOe
T	0.3	0.36	9.1 nm	1.4 kOe
B1	0.48	0.45	10.4 nm	2.0 kOe
B2	1.03	0.65	9.9 nm	7.9 kOe

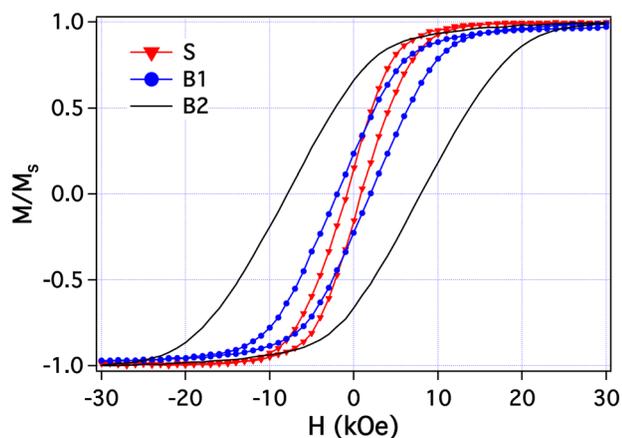


FIG. 4. M-H loops of sample S, B1, and B2, respectively.

film B2 [Figure 3(d)] indicate a significant improvement in $L1_0$ ordering. The coercivity of the sample is around 8 kOe [Figure 4].

Since the overall oxide amount in the four samples was kept approximately the same, the average grain sizes measured from the plane-view TEM images do not show significant differences among the samples and around 10 nm (Table II). The “ghost images” observed in the TEM images of T and B1 [Figures 5(b) and 5(c)] come from the misalignment of the grains at the layer interfaces and the different grain sizes between FePt-SiO_x layers. These are not due to out of focus conditions. The “ghost images” are greatly reduced in the TEM image of B2 [Figure 5(d)], which confirms the presence of more homogeneous columnar grains along the film thickness. It is also worth noting that the FePt grain sizes seen in the in-plane images (~ 10 nm) are larger than those in the cross-section images (~ 5 nm) due to the serpentine microstructure of the FePt-SiO_x systems.⁹

In summary, we attempted to maintain the FePt columnar grain height at 45 vol. % SiO_x by introducing the tri-layer and bi-layer media. The bi-layer media deposited at 450 °C shows a remarkable improvement of grain aspect ratio, $L1_0$ ordering, and H_c . Even though SiO_x is utilized as a segregant in our demonstration, this method can be applied to other segregants, especially C. C is known for its strong tendency to diffuse onto the film surface.¹¹ Therefore, we believe the multiple oxide (or segregant) fraction structure is a promising technique to achieve columnar grain shape for FePt-based media.

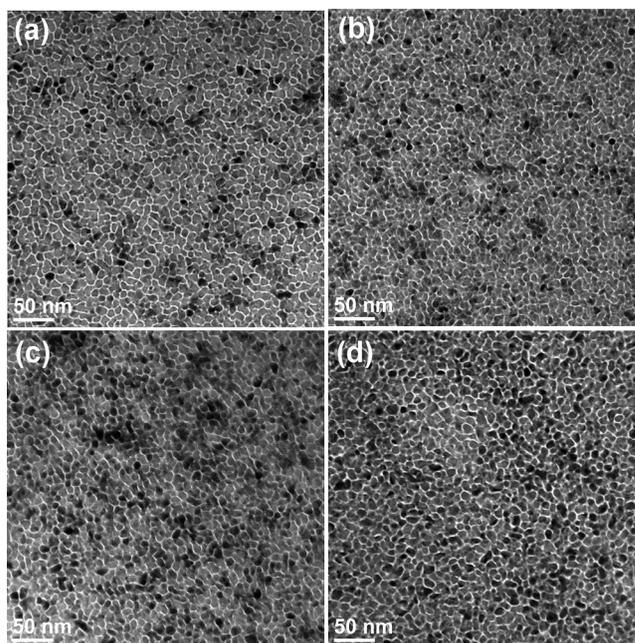


FIG. 5. Plane-view TEM images of sample (a) S, (b) T, (c) B1, and (d) B2.

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