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Switching field distribution and magnetization reversal process of FePt dot patterns

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ABSTRACT

The fabrication of FePt nanodots with a high structural quality and the control of their switching fields are key issues in realizing high density bit pattern recording. We have prepared FePt dot patterns for dots with 15–300 nm diameters by electron beam lithography and re-annealing, and studied the relation between magnetization reversal process and structure of FePt nanodots. The switching field (H_{sw}) of dot patterns re-annealed at 710 °C for 240 min showed a bimodal distribution, where a higher peak was found at 5–6 T, and a lower peak was found at ~2 T. It was revealed by cross-sectional TEM analysis that the structure of dots in the pattern can be classified into two groups. One group has a high degree of order with well-defined [0 0 1] crystalline growth, and the other group includes structurally-disturbed dots like [1 1 1] growth and twin crystals. This structural inhomogeneity causes the magnetic switching field distribution observed.

1. Introduction

$L1_0$ FePt is expected to be the most suitable material for bit pattern media because of its large uniaxial magnetic anisotropy. For an assumed coherent rotation of magnetization in an $L1_0$ FePt dot, the coercivity at room temperature is calculated to be over 10 T at uni-axial magnetic anisotropy constant K_u of $\sim 7 \times 10^7$ erg/cm^3, with a saturation magnetization M_s of ~1100 emu/cm^3 [1–4]. $L1_0$ FePt films have been fabricated by epitaxial methods [2–6], and by annealing with additives like Cu, Ag, and oxide [7–10]. Such films show excellent magnetic properties with a high order parameter (S) for an $L1_0$ structure and [0 0 1] crystalline growth perpendicular to the film plane. Patterning $L1_0$ FePt films into dot arrays has been done mostly by electron-beam lithography and ion-milling using the high quality films mentioned above. Self-organized resist patterns [11] can result in dot patterns on full disk substrates. The fabrication of FePt nanodots, with a practical dot size less than 20 nm and high structural quality, has become the focal issue in efforts to make high density bit pattern recording possible.

The coercivity of FePt particles and patterned dots has been reported to be 2–4 T, and generally shows a wide distribution of switching field strengths [4,9,12–16]. These coercivities may be high enough for bit patterned media and thermally assisted magnetic recording, but their distribution must be controlled. Magnetization switching may take place through a magnetization reversal of nucleation sites and successive domain expansion to the whole dot [17]. Structural defects may become nucleation sites for magnetization reversal, and could then be the cause of the observed degradation of H_{sw} and its wide distribution of measured values [12,13].

In this paper, FePt dot patterns with 15–300 nm diameter dots were prepared by electron-beam lithography, ion-milling, and re-annealing, and the correlation between the switching field distribution and the structure of patterned dots has been studied.

2. Experimental details

2.1. Fabrication of films and patterning

FePt was sputtered on thermally oxidized Si substrates. 10% SiO_2 was added during sputtering to promote $L1_0$ ordering at low temperatures [9–11]. For practical application to bit-patterned

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media, a film with less than 10 nm is required. Thus, we used an FePt thickness of 6.1 nm in this experiment. Since FePt films on Si substrate become a particulate film when heated over 600 °C [10], sputtered films were first annealed by rapid thermal annealing (RTA) at about 570 °C for 10 min. The crystal structure of films was studied by X-ray diffraction (XRD) using Cu $K\alpha$ radiation. The degree of order of films annealed at 570 °C for 10 min was found to be $S \sim 0.89$ by fitting the θ–2θ diffraction patterns. The films showed a [0 0 1] crystalline growth, and [1 1 1] and [1 1 0] diffraction lines were not observed in the film. After the annealing, patterns were formed in 100 μm squares by electron-beam lithography and ion-milling [18,19]. We used a negative resist of *p*-chloromethyl-methoxy-calix[4]arene (TEBN-1 provided by Tokuyama Corp.). The concentration of resist solutions was controlled so that the thickness of resist became 15 nm. The resist films were prepared by spin coating the resist solution on a substrate at 2000 rpm, and prebaking at 110 °C for 1 min. The resist films were patterned by e-beam writing system (ELS7500, ELIONIX Inc.) operated at 50 kV and 50 Pa. After exposure, the resist patterns were developed in *o*-xylene for 30 s and isopropanol for 30 s. FePt dot patterns were transferred through the resist mask by using Ar ion milling at 200 keV and 60 mA with rotating substrate. A grazing incident angle of Ar ions was set at 60 degrees to film surface. Dot diameters were designed to be 15–300 nm.

The pattern prepared by the above-mentioned process is called pre-annealed patterns hereafter. Finally, patterns were annealed again at 710 °C to eliminate structural damage resulting from nanofabrication and to promote an fcc to $L1_0$ transformation. These patterns are called re-annealed patterns. The schematic diagram for the film preparation and patterning is shown in Fig. 1.

2.2. Structural analyses and magnetic properties

Geometric characterization of patterns was done by scanning electron microscopy (SEM) and atomic force microscopy (AFM). The SEM and AFM images of the pre-annealed pattern (0.71 T bit/in.^{2}) with a 15 nm dot diameter and 30 nm pitches are shown in Fig. 2a and b, respectively. The dispersion in dot location is shown in Fig. 2c, and was found to be 5 nm, with a statistical position variance (σ/p) of ~10%.

The structure of the pre-annealed patterns was studied by micro X-ray diffraction (μ-XRD) at BL13XU beamline [20] at SPring-8, a synchrotron radiation facility. Width of micro X-ray beam is $1.2 \times 0.3\ \mu m^2$. The crystal structure of the re-annealed patterns was analyzed by cross-sectional transmission electron microscopy (TEM).

Hysteresis curves of dot patterns were measured by X-ray magnetic circular dichroism (XMCD) at the Pt L_3 edge, with static magnetic fields up to 10 T at BL39XU beamline [20] at SPring-8. Remanent magnetization curves of the dot pattern was also measured using the Kerr effect by applying a pulse field of up to 7 T to consider a thermal fluctuation effect. The time-domain half width of the pulse field was 1 ms.

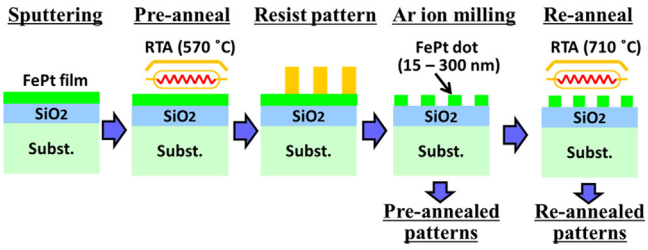

Fig. 1. Schematic diagram of sample preparation.

3. Experimental results and discussion

3.1. Crystalline structure of dot patterns

The results of micro-XRD for the pre-annealed pattern are shown in Fig. 3, where the diffraction intensity was normalized by the (0 0 1) diffraction intensity of the pre-annealed film and corrected by taking account of a change of an areal density of FePt dots. Both (0 0 1) superlattice line and (0 0 2) fundamental lines were clearly observed for all samples. However the (0 0 1) line gradually decreases with reducing dot size to 30 nm diameter and decreases extremely with a further reduction of dot size to 15 nm. At the same time, a decrease of the relative ratio $I_{(0\,0\,1)}/I_{(0\,0\,2)}$ is observed, which suggests a lowering of degree of order (S). The intensity of the fundamental (0 0 2) line also shows a decrease with a reduction of dot size, which is typically observed at d = 15 nm. Namely, it is considered that the film structure with the [0 0 1] growth perpendicular to the film plane collapsed, or even a crystallinity itself was lost during ion-milling. The structure of the disturbed area may be a fcc (or amorphous) state [12,13,18,21], but a coherent lattice relation between the $L1_0$ phase and the induced fcc phase is quite small. When such structurally degradation is assumed to be formed at sidewall of patterned dots, a thickness of the disturbed layer is roughly estimated to be 3–5 nm from the change of the (0 0 1) and (0 0 2) intensity as a function of dot size.

The crystalline structure of dots in the re-annealed pattern was analyzed by cross-sectional TEM. Cross-sectional TEM analysis was focused on the 30 nm dot patterns re-annealed at 710 °C for 240 min. The structures of 15 individual dots were chosen for analysis. The typical bright field images are shown in Fig. 4. The structure of these dots can be classified into two groups. The first group has a high degree of order with well-defined [0 0 1] crystalline growth, which can be clearly seen in the layered bright and dark lattice image in Fig. 4b. In this group, the structural degradation, like an fcc or amorphous phase resulting from ion milling, was not detected within the resolution of the TEM images. The second group includes mis-oriented dots like [1 1 1] growth (Fig. 4c), twin crystals (Fig. 4d). In total, 8 of the 15 dots observed belong to the first group and the rest belong to the second group.

From the micro-XRD and TEM studies for the pre-annealed patterns and the re-annealed patterns, a structural change in dots is supposed to take place in the following way;

(1) In the pre-annealed pattern, each dot consists of a mixed structure of the $L1_0$ ordered phase and the structurally-disturbed (fcc or amorphous) phase. This structurally -disturbed area is considered to arise from ion-milling of the patterning process and to be formed at sidewall of dots.
(2) By re-annealing process, a structure disturbed phase induced by ion-milling re-transforms into an $L1_0$ phase to some extent. Accordingly, some mis-oriented dots and twins were formed through re-crystallization, and the dots make two groups with regards to a structural quality. The first group has a high degree of order with well-defined [0 0 1] crystalline growth. The second group includes mis-oriented dots like [1 1 1] growth, twin crystals, and structurally unclear dots. These two groups are expected to have different magnetic properties, which will be shown in the next paragraph.

3.2. Magnetic properties of patterned dots

Hysteresis curves measured by XMCD are shown in Fig. 5. Coercivity (H_c) depends on both the sample preparation process and the dot size. The maximum H_c for the pre-annealed pattern was about 1.5 T, and the coercivity was increased by re-annealing.