Nanopatterned hard/soft bilayer magnetic antidot arrays with long-range periodicity

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Abstract

A top-down approach using focused ion beam has been employed to fabricate Co/Permalloy hard-soft bilayer magnetic antidot arrays. These nanopatterned films are studied with particular emphasis on magnetic coercivity. The antidots have a diameter of 40 nm and the studied antidot symmetries are square and hexagonal. A dependence of magnetic coercivity on the relative thicknesses of the magnetically hard (Co) and soft (Permalloy) layers is found; increasing Permalloy thickness results in lower magnetic coercivity. Furthermore, the long-range periodicity of top-down nanopatterned antidots results in higher magnetic coercivity and a stronger magnetic domain-wall pinning, compared to identical hard/soft bilayers of short-range order deposited on porous anodic alumina. The combination of antidot symmetry and hard/soft thickness, allow for efficient tailoring of the magnetic properties of nanopatterned thin films. Finally, magnetic force microscopy imaging of the antidot array magnetic configuration shows striking qualitative differences between the two array symmetries: square symmetry arrays have inhomogeneous magnetic state and a high density of immobile super-domain walls, whereas hexagonal symmetry arrays show a homogeneous magnetic configuration.

Keywords: hard/soft bilayers; antidot arrays; focused ion beam; MOKE magnetometry; AFM; MFM

1. Introduction

The magnetic properties of thin films can be tailored by means of nm-scale patterning, resulting in unique properties for advanced applications (1; 2). An established way to pattern magnetic thin films is to create groups of ordered holes, usually dubbed “magnetic antidot arrays” (MAAs) (3; 4; 5). Such magnetic nanostructures are being intensively studied as magnonic crystals for microwave devices applications (6; 7), magnetically-active plasmonic media (8), magnetic bio-sensing (9), microfluidics applications (10), anisotropic magneto-resistance sensors (11), and lately as magnetic diodes for low-power electronics (12).

In the past, both bottom-up (13; 14; 15; 16) and top-down (17; 18; 19; 20) nanopatterning approaches have been employed for MAA fabrication. The advantage of the top-down approach is that long-range order may be obtained up to mm-wide areas, and the ordering parameters of the hole arrays, as well as the hole size and shape, may be tailored at will. This allows for studying magnetic nanostructures unobtainable using bottom-up approaches, which exhibit defect-free lattices within an area that ranges from a few μm^2 to about 20 μm^2 at best (21), in addition to the fact that only hexagonal symmetry may be obtained. Furthermore, top-down nanofabricated arrays may be laterally confined at will and complex combinations of different MAA symmetries and sizes may be patterned at the same device – each serving a unique purpose – allowing for the realization of magnonic circuits (22).

The three main parameters affecting MAA properties are the material used, the antidot shape and size, and the array symmetry. Until now, the effects of antidot shape and size, as well as array symmetry, have been extensively studied. However, the actual materials investigated are mainly Permalloy ($\text{Ni}_{20}\text{Fe}_{80}$) – a widely-studied soft magnetic material – or Co-rich alloys, materials with relatively hard magnetic properties, having uniaxial magnetic anisotropy derived mostly from their hcp crystallographic symmetry. The effect of hard/soft bilayers on the properties of MAAs has only been recently the focus of some studies, e.g. Co/NiFe (13) and TbCo/NiFe (23), fabricated in both cases using porous anodic alumina and a bottom-up approach.

In this work we make use of a top-down approach using the focused ion beam (FIB) technique for nanopatterning Co/permalloy hard-soft bilayer MAAs. The antidots have a diameter of 40 nm with square and hexagonal symmetries; the lattice constant is 240 nm or 360 nm for both cases. Our study addresses the dependence of magnetic coercivity on the relative thicknesses of the magnetically hard Co and soft permalloy (Py) layers. Furthermore, the long-range periodicity of top-down nanopatterned antidots results in higher magnetic coercivity and a stronger magnetic domain-wall pinning, compared to identical hard/soft bilayers deposited on top of porous anodic alumina. Finally, magnetic imaging reveals the effect of array symmetry on the magnetic nanostructure of the MAAs.

2. Experimental Details

All layers were deposited at room temperature using an ultra-high vacuum (base pressure 1.5×10^{-9} Torr) magnetron sputtering system (AJA ATC-2200V). Si(100) wafers with 500 nm amorphous thermal oxide were used as substrates. Metallic Pt, Co, Ni, and Fe targets were used as source materials (purity 99.99%). Co-sputtering of Ni and Fe resulted in the deposition of $\text{Ni}_{77}\text{Fe}_{23}$ (Permalloy) films as determined by EDX measurements. DC power was applied to the targets (approximately 2.5 W/cm^2 at Co and Fe and 6 W/cm^2 at Ni), while the working gas was Ar at 3 mTorr pressure. The capping Pt layer, 5 nm thick, was deposited using 1 W/cm^2 DC power. The deposition rate was measured using a quartz crystal microbalance sensor, calibrated against x-ray reflectivity (XRR) measurements of test samples. The substrate was rotated during deposition (80 revolutions per minute) to ensure thickness homogeneity and avoid the development of any magnetic anisotropy axes. Two types of bilayers were fabricated: Co(20 nm)/Py(9 nm) and Co(20 nm)/Py(27 nm), labeled Co/Py-thin and Co/Py-thick, respectively; for comparison purposes, single layers of Co(20 nm) and Py(27 nm) were also prepared.

The nanopatterning of the MAAs was done by directly etching the continuous film using an IonLine FIB machine with 30 keV Ga ions and the following fixed parameters: $10 \mu\text{m}$ opening and 6.6 pA ion current. Different doses were used depending on the material: 30, 35 and 40 mC/cm^2 for Co, Py and Co/Py bilayers, respectively. The dwell time was also modified such that the ion beam completely perforated the film and that the hole diameter was consistent, so at least 20 nm of the substrate was etched in each case.

A commercial magneto-optical Kerr effect, MOKE, set-up (Nanomoke2 from Durham Magnetooptics Ltd.) was used to measure the hysteresis loops at room temperature, using a 635 nm wavelength laser. The laser spot was focused to $3 \mu\text{m}$ diameter and placed at the center of each antidot array by means of a robotic stage, which allows sample movement with about $1 \mu\text{m}$ precision. X-ray diffraction (XRD) structural analysis was performed using a Siemens D500 diffractometer with Cu-K α radiation, in steps of 0.03° and a counting time of 6 s/step. Surface morphology and the magnetic domain structure were studied by atomic/magnetic force microscopy (AFM/MFM) using a Bruker Dimension Icon microscope operating in non-contact mode with commercial probes from Nanosensors (types PPP-FM for AFM and MESP-LM for MFM).

3. Results and Discussion

3.1 Non-patterned films

The crystallographic and magnetostatic properties of non-patterned films were studied first. X-ray diffractograms indicate that the Py film has (111) oriented fcc crystallites (figure 1). The Co sample shows a broad peak between 42° and 52° approximately, which can be ascribed to the convolution of small hcp (002) and (101) peaks, and fcc (200) oriented crystallites. For the bilayer samples, the strong Py (111) Bragg peak dominates the diffractograms, due to the higher crystallinity of the Py layer compared to the Co layer, in addition to the fact that the Py layer is the upper layer. Finally, all the diffractograms show a broad peak corresponding to the thin fcc Pt capping layer, as well as the substrate Si(110) Bragg peak.

Figure 1. θ -2 θ diffractograms of the studied samples. From left to right: fcc Pt(111) and fcc Py(111) Bragg reflections (solid lines); hcp Co(002), hcp Co(101), and fcc Co(200) Bragg reflections (dashed lines).

Magnetostatic characterization shows that all the non-patterned films and bilayers have a small in-plane uniaxial anisotropy (figure 2), despite the substrate rotation during deposition. The pure Co film has an easy axis oriented along the 90° direction (not shown), whereas the Py film has easy axis along 0° (reference axes are defined in figure 3). The Co/Py-thin bilayer has uniaxial anisotropy similar to that of pure Co film; in contrast, the Co/Py-thick bilayer has its easy axis along the 0° direction. Magnetic coercivity is below 20 Oe along all directions and decreases as the Py content increases. Similar behavior has been

observed in the past for thin polycrystalline Co (24), as well as Py films (25); i.e. a planar uniaxial anisotropy was observed, with easy and hard axes separated by 90°. It has been shown that domain wall motion is the main process of magnetization switching in these films. Since the films investigated in this work were deposited onto amorphous SiO₂, a substrate-induced orientation of crystallites or film strain is excluded.

A possible explanation could be interface-induced magnetic anisotropy due to the Pt capping layer present in all samples, since Pt is a polarizable material (26). It is worth noting that interface-induced magnetism is a wide and interesting field of research, since film interfaces break translational and inversion symmetry and a variety of effects appear (27), but this subject is beyond the scope of this paper.

Figure 2. MOKE hysteresis loops of the two non-patterned bilayer films along various directions; (left) Co/Py-thin, (right) Co/Py-thick

3.2 Morphology

Figure 3 shows the surface morphology of the MAAs imaged using atomic force microscopy (AFM). The studied symmetries are square and hexagonal (see figure 3) with lattice constants of 240 nm and 360 nm. The orientation axes used throughout this study are also shown. Taking into account such axes, the first neighbor directions for square arrays are 0° and 90°, whilst the second neighbor directions are 45° and 135°. In the case of hexagonal arrays, the first neighbor directions for square arrays are 0°, 60° and 120°, whilst the second neighbor directions are 30°, 90° and 150°. Disk-shaped MAAs are patterned to avoid any macroscopic shape anisotropy (17); their diameter is 15 µm and they are surrounded by a 0.5 µm-wide circular trench to magnetically decouple the MAAs from the rest of the continuous multilayer. Non-patterned regions confined by a trench were also fabricated for the sake of comparison. Antidots can be considered as defects acting as domain-wall (DW) pinning centers during magnetization reversal (17), thus, the defect volume density (DVD) of the MAAs in correlation to the hole diameter is of great importance to their study. Taking

into account the MAA geometry (17) and considering an antidot diameter of 40 nm (as measured in figure 3 at half depth), the DVD is given by the formula $\frac{\pi(d/a)^2}{4}$ for the square symmetry and $\frac{\pi}{2\sqrt{3}}\left(\frac{d}{a}\right)^2$ for the hexagonal symmetry arrays, where a is the nearest neighbor center-to-center distance and d is the antidot diameter; the values are calculated to be 0.0218 and 0.0097 for the 240 nm and 360 nm square lattices, respectively; similarly, the DVD is 0.0251 and 0.0112 for the 240 nm and 360 nm hexagonal lattices, respectively.

Figure 3 AFM images displaying the morphology of the MAAs. A high-resolution image obtained at a single antidot in the Co film shows that its diameter is approximately 40 nm.

It is worth mentioning that FIB etched antidots always have a local alteration of the film around each hole due to the tail of the Gaussian-like section of the ion beam, which produces Ga implantation and intermixing with adjacent layers (17; 18). If neighboring nanoholes are too close, the modified areas around each dot overlap and the antidot array can even behave magnetically as an array of isolated dots (18). However, since in our samples the distance between neighbors is at least 4 times the hole diameter, this effect is not present.

3.3 Magnetostatic properties

Figure 4 shows representative MOKE hysteresis loops for fields applied along first and second-neighbor directions for the square and hexagonal 240 nm Co(20 nm)/NiFe(9 nm) bilayers, respectively. In all the studied MAAs, a striking increase of coercivity is observed with respect to the non-patterned films, from a few Oe to several 10s of Oe, depending on material, DVD, and direction. The small “jump” apparent at low applied magnetic fields is related to un-obstructed DW motion between neighboring anti-dots and depends on the magnetic properties of the non-patterned films, as can be seen in figure 2.

Figure 4 Representative MOKE hysteresis loops of MAAs. Square (left) and hexagonal (right) symmetries of the Co/Py-thin bilayer; the lattice constant is 240 nm; first and second-neighbor directions, denoted by the angles, are shown: 0° and 45° , respectively, for the square array, and 0° and 30° , respectively, for the hexagonal array. Loops obtained at non-patterned bilayers (TF) are also shown for comparison purposes.

Figure 5 displays the coercivity values of all the studied samples, including non-patterned films. For the sake of simplicity, only two directions are shown in each case, a first neighbor direction (0° for both square and hexagonal arrays) and a second neighbor direction (45° for square arrays and 30° for hexagonal ones). Only subtle differences of around 10% were obtained in the coercivity for equivalent directions, i.e. between 0° and 90° for square arrays and between 0° and 60° for hexagonal ones. The results, summarized in figure 5, are in agreement with previous studies showing a monotonic dependence of coercivity on the DVD of the MAAs (17), whereas coercivity values of more than 700 Oe in Co MAAs having 0.58 DVD and more than 500 Oe in Py MAAs having 0.69 DVD, are reported in the literature (21). Furthermore, it is worth noting that the coercivity values obtained in this study are much higher than those obtained for MAAs fabricated under the same conditions (identical equipment and sputtering parameters) but onto porous anodic alumina, which were reported in our previous publication (13). This shows that, for comparable DVD values, the long range order obtained in top-down fabricated MAAs induces more efficient domain wall pinning than that obtained with bottom-up fabrication.

Figure 5 Summary of the magnetic coercivity values of all the MAAs, as obtained by MOKE magnetometry.

From the data, it becomes apparent that in-plane magnetic anisotropy arises in certain MAAs. In the high DVD cases (lattice constant 240 nm), square symmetry MAAs show the highest coercivity along the direction of the second antidot neighbors, that is along 45°; in contrast, for hexagonal symmetry MAAs, the highest anisotropy is obtained along 0°, the direction of the first antidot neighbors. For square symmetry MAAs these results are in agreement with our previous study of FIB nanopatterned MAAs of pure Co (17). However, the opposite behavior is observed for hexagonal symmetry MAAs; we attribute this to the subtle coercivity differences introduced by the Py layers. Moreover, the two studies were performed on MAA samples deposited using different sputtering systems and most importantly, different substrates; as a result, the properties of the films are not necessarily identical. In the low DVD cases (lattice constant 360 nm), no particular systematic dependence of coercivity to anisotropy is observed; this is also in agreement with our previous work on a single Co film (17): as the DVD increases, the array symmetry becomes more important. Furthermore, it should be noted that bottom-up fabricated MAAs onto porous anodic alumina substrates – deposited simultaneously to the ones of this study – do not show any preferred in-plane orientation, as a result of the short-range order of the MAAs in the bottom-up approach (13).

Finally, the high DVD square symmetry MAAs show coercivity differences between the 1st and 2nd neighbor directions that greatly depend on the material; it appears that single layers have greater difference and the maximum value is obtained for the Co film. Bilayers have smaller differences and the thicker NiFe bilayer has smaller coercivity difference between the two directions. This is not the case for the rest of the MAAs where minor differences between in-plane directions are observed.

3.4 Magnetic microstructure

Magnetic Force Microscopy, MFM, imaging is employed for studying the magnetic nanostructure of the MAAs (figures 6 and 7). Prior to taking the images, the samples were demagnetized along the 0° direction. Commercial low-moment MFM tips were used (Bruker, MESP-LM); we have observed that higher moment tips create perturbations in the MAAs, especially those patterned in Permalloy. The tips were magnetized along the pyramid axis, thus, the obtained contrast corresponds to the magnetic charges. The MAAs have principally in-plane magnetic anisotropy, as expected from the magnetic shape anisotropy of the parent multilayers. However, out-of-plane magnetization arises in the presence of antidots, yielding perpendicular magnetic moment components (15; 28). It is evident that the magnetic nanostructure of the MAAs is correlated with the array symmetry.

Figure 6 Representative MFM image of the Co/Py-thick MAA; the magnetic microstructure of a hexagonal symmetry MAA with 240 nm lattice constant is shown (MFM lift height: 30 nm). (a) Low magnification image; (b) focus on image (a); (c) schematic showing the local magnetization direction that corresponds to the boxed area in (b).

As was shown in the previous section, magnetic coercivity depends strongly on the DVD and material; however, although the MAAs shown in figures 6 and 7 have similar magnetic coercivity, striking differences are observed to their magnetic nanostructure. In the hexagonal MAAs, although magnetization has a texture at the local nm-scale, magnetic moments homogeneously depend on the array symmetry due to the pinning effect of the holes (see figure 6(a)). Magnetic poles of opposite sign accumulate at each side of the holes along the 0° direction and such a domain structure has been reported several times in the literature for hexagonal antidot arrays made of Ni (28), Py (4), and Fe/Ni/Fe (29) (a schematic is depicted in figure 6(c)).

In contrast, square symmetry MAAs (figure 7) show texture in the local scale, as well as inhomogeneities in the superstructure scale; the magnetic structure observed corresponds with super-domains – regions with uniform magnetization texture – separated by regions with different texture by super domain-walls (SDWs). Such magnetic structures have been studied in the past in Py (30; 31) and Ni (15) MAAs and classified according to their magnetic nanostructure. Two kinds of SDWs are observed: low stray field energy (LE-SDWs) that are linear and expand over several lattice constants, and high stray field energy (HE-SDWs) that occupy only a few lattice constants and are situated at kinks of LE-SDWs.

Figure 6 Representative MFM image of the Co/Py-thick MAA; the magnetic microstructure of a square symmetry MAA with 240 nm lattice constant is shown (MFM lift height: 30 nm). (a) Low magnification image: vertical dashed lines mark LE-SDWs; (b) focus on image (a); the

square boxes mark HE-SDWs; (c) and (d) schematics showing the local magnetization direction that corresponds to boxed areas in (b).

5. Conclusion

From the data above, it becomes clear that MAAs provide an efficient way to tailor the magnetic properties of thin films; their magnetic coercivity, as well as in-plane anisotropy, may be tailored by appropriate selection of the bilayer materials and MAA symmetry. Our study shows that top-down nanofabricated MAAs show higher coercivity than simultaneously deposited bottom-up ones; it appears that long-range order results in more efficient DW pinning and allows for tailoring of the MAA magnetic properties. In addition, high DVD square symmetry MAAs show high directionality of the in-plane anisotropy; a smaller effect is observed for other symmetries and DVD values. However, MAAs with disordered antidot distributions, or MAAs having short-range order, do not show any anisotropy directionality. Finally, the study of the MAA magnetic nanostructure shows that long-range inhomogeneities appear at the square symmetry MAAs, as opposed to the homogeneous nanostructure of the hexagonal symmetry MAAs. From the above, it becomes clear that top-down nanofabricated MAAs are more suitable for magnonic circuit applications, compared to bottom-up nanofabricated ones. Furthermore, the local magnetic inhomogeneities of square symmetry MAAs in the form of super-domain walls could make them inappropriate for magnonic circuit applications.

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The authors declare that there is no known conflict of interest