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Patterning of permalloy thin films by means of electron-beam lithography and focused ion-beam milling

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Abstract

Focused ion-beam milling has been employed to structure magnetic nanoelements from 20 nm thick films of permalloy (Ni₈₁Fe₁₉). Rectangles are patterned into permalloy thin films grown on Si substrates by means of electron-beam lithography and focused ion-beam (FIB) milling down to 100 nm dimensions. In this study, we analyse the effect of the FIB milling parameters (ion current, spot size, dose) on the resulting magnetic domain structures. The ion currents have been varied between 10 pA and 10 000 pA; the dose of the ion beam used for milling was varied in order to achieve the best definition for the milled areas. The resulting edges of the permalloy structures are characterized by means of AFM. We find that a small ion dose does not affect the resulting magnetic domain patterns in the structures, so FIB milling can be applied to create high-quality permalloy nanostructures.

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Keywords: Permalloy; Focused ion-beam milling; Magnetic properties

1. Introduction

Structuring magnetic samples is very important for basic research as well as for applications in e.g. magneto-electronics [1]. Focused ion-beam (FIB) irradiation and milling are very versatile

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S. Getlawi et al. / Superlattices and Microstructures 44 (2008) 699-704

and rapid laboratory techniques for laterally structuring materials into devices [2–4]. In some cases, fabrication rates may even be high enough to additionally consider using FIB for the commercial production of nanostructures. Recently, FIB milling has been demonstrated to be capable of producing high quality magnetic nanostructures from magnetically *hard* thin films [5]. However, for *magnetically soft* materials, such as those used in the emerging technology of magnetoelectronic devices [6,7], the degree to which the magnetic properties of the structured devices suffer due to FIB milling remains unclear. Ion implantation, the introduction of magnetic pinning defects, and nanometer-scale displacements are all ion-induced mechanisms which have recently been shown to strongly influence soft magnetic properties [6]. Therefore, it is essential to study the effects of various ion currents (doses) on the magnetic properties of permalloy samples.

In this contribution, we structured permalloy thin films into rectangles by means of electronbeam lithography, and then subsequently cut the elements further into squares, circles and ellipses by means of focused ion-beam milling. The resulting structures are analysed by atomic force microscopy (AFM) and magnetic force microscopy (MFM).

2. Experimental procedure

Thin permalloy (Py) films with a nominal thickness of 20 nm or 50 nm were produced using RF sputtering on Si substrates and with a 5 nm thick Ti cap layer. Electron-beam lithography was carried out using a Hitachi SEM equipped with a Raith nanolithography unit, followed by a lift-off process. The FIB system employed here consists of a FEI dual beam workstation (Strata DB 235) [8], which is equipped with a FIB column employing a Ga liquid metal ion source. The ion beam currents employed ranged from 1 pA to 20 000 pA; the dwell time was 1 μ s, and a overlap of 50% was chosen. This corresponds to ion doses ranging between 0.01 nC μ m⁻² and 0.3 nC μ m⁻². The ion beam of a 10 pA results in a spot size of 10 nm; an ion beam of 10 000 pA has a corresponding spot size of 300 nm. More details about the FIB milling process can be found in Ref. [9].

AFM and MFM was performed using a Nanoscope IV (Veeco/DI) employing micromachined Si cantilevers (Nanoworld Services GmbH, type PPP, 2–3 Nm⁻¹, resonance frequency $f_{\rm res} \approx$ 60–70 kHz, [10]). For MFM, these cantilevers were coated with 20 nm thick CoCr film ("standard cantilever") or with 20 nm films of NiCo (low coercivity tip; coercivity ~1 Oe) and NiFe (permalloy).

3. Results and discussion

Fig. 1(a) presents a permalloy rectangle produced by electron-beam lithography. The two inner cuts in form of a cross are created by focused ion-beam milling. The depth of the trenches is chosen in order to ensure that all magnetic material is removed. The ion current for FIB milling was chosen as 10 pA, following Refs. [11,12]. Here, it is visible that the trenches created by FIB are irregular, due to the preferential milling occurring in certain grain orientations in the trenches. As the FIB milling is performed within two separate milling rectangles, the depth of the trenches in the overlap is much larger. This first FIB cutting was done creating larger trenches as in the following experiments. This figure enables a comparison of the edges created by EBL and FIB. For the present case of a 50 nm thick permalloy film, the resulting edge roughness of FIB-cut and EBL-cut edges is nearly similar. Fig. 1(b) shows a detail of the resulting Py rectangles after the second FIB cutting process, together with the SEM-measured edge lengths. The small squares have dimensions of about $500 \times 500 \text{ nm}^2$. Fig. 1(c) gives a topography and magnetic image of a

S. Getlawi et al. / Superlattices and Microstructures 44 (2008) 699-704



Fig. 1. (a) SEM image of a 50 nm thick permalloy square made by EBL with two subsequent FIB cuts (ion current 10 pA). (b) Details of the $500 \times 500 \text{ nm}^2$ permalloy rectangles created by FIB cutting. (c) Topography and magnetic image of the permalloy square after the first FIB cutting step.



Fig. 2. Dependence of the magnetic structure on the applied ion-beam current ranging from 10 pA to 20 000 pA.

permalloy square (same sample as in (a)) after the first FIB cutting step. This image was measured with a standard CoCr cantilever. The resulting magnetic domain pattern is a clear Landau domain structure.

In Fig. 2(a)–(f), we present the effect of various ion-beam doses on the magnetic properties of the permalloy film as measured by means of MFM. Here, we focus mainly on the magnetic domain patterns in the resulting small squares after the second FIB cutting. The FIB cuts are made by an automated process routine for rectangles; the rectangles themselves are positioned

S. Getlawi et al. / Superlattices and Microstructures 44 (2008) 699-704





Fig. 3. (a) Topography and magnetic image of a FIB-milled circle, 10 pA ion-beam current. The arrows point to a defect in the sample edge. (b) Topography and magnetic image of four FIB-milled ellipses, 10 pA ion-beam current. The upper right ellipse exhibits defects in the upper right part, causing an alteration of the magnetic domain pattern.

manually, so some small deviations may occur between the different images. Here, we employ a 20 nm thick permalloy film. At low ion beam currents [10 pA (a), 50 pA (b)], the resulting sample edges are homogeneous, resulting in well reproduced Landau domain structures. An increase of the ion current [100 pA (c), 1000 pA (d)] causes a much wider gap between the elements due to the increase in beam spot size, and also a small amount of defects along the edges, which may alter the domain structure. Increasing the ion beam current even further [5000 pA, (e)] causes more artefacts in the magnetic images. Finally, at 20 000 pA (f), the irradiation damage is so big that the magnetic properties of the permalloy elements are altered. Here, we do have a severe damage of the microstructure of the permalloy.

Fig. 3(a) shows the topography and a magnetic image of a FIB-milled permalloy disk. Also for the circles, there is a standard FIB processing routine available. The dose for the FIB milling was kept low at 0.03 nC μ m⁻² (ion current of 100 pA) in order to ensure no significant damage caused by the ion milling. The MFM image reveals a vortex structure in the centre of the disk, but also a defect at the bottom of the circle, which causes a domain wall to be pinned.

Fig. 3(b) presents a topography image of a Py ellipse, and the corresponding MFM image. The cutting routine for this purpose had to be created by means of a CAD program. Three of the four created ellipses show a homogenous magnetic pattern, but the patterned ellipse is slightly tilted (see also the topography view). On the right side, one can see the effect of an imperfectly cut edge on the resulting magnetic structure, causing the presence of four domains instead of two. Otherwise, the FIB milling produced well defined shapes of the resulting nanostructures, which are magnetically intact.

The present FIB-milled structures (squares, rectangles, circles, ellipses) are in principle well defined if the ion-beam current is not higher than 300 pA. The quality of the sample edges is found to be comparable to that of EBL-prepared permalloy elements as seen in Figs. 1 and

S. Getlawi et al. / Superlattices and Microstructures 44 (2008) 699-704



Fig. 4. Comparison of MFM images obtained with three different MFM cantilever coatings, CoCr (a), NiFe (b) and NiCo (c). The MFM images obtained by NiFe and by the NiCo low coercivity tip (c) reveal no image disturbation by negative tip-sample interactions.

2; employing AFM measurements and image analysis on the SEM images. In Ref. [11] the FIB-created edges were found to be more rough. However, we have not yet performed MFM domain imaging in applied fields as was done in Ref. [11], which will give further information about the influence of the element edges on the magnetic properties. This will be carried out in a following experiment. Here it is important to note that the ion-beam imaging within the FIB process should be avoided, as this may create unwanted defects to the permalloy elements. Control measurements using electron backscatter diffraction (EBSD) revealed that ion imaging can completely obscure the Kikuchi patterns of permalloy, indicating severe damage to the crystallinity. This could be the reason for the problems found in Ref. [11], whereas other groups [12,13] found the magnetic properties of the FIB-milled permalloy samples useful for their experiments. With this result, it becomes possible to create permalloy nanostructures using FIB milling.

MFM domain imaging of permalloy samples is still a complicated procedure, as permalloy is a soft magnetic material. This may cause interactions between the tip and sample, which can obscure the resulting magnetic images [14,15]. Fig. 4 presents three different experimental runs on a permalloy square with FIB-milled cuts, but using different magnetic coatings on the MFM cantilevers. The lift height was kept at 50 nm for all images shown here. The MFM images

S. Getlawi et al. / Superlattices and Microstructures 44 (2008) 699-704

of the resulting domain structures were obtained using (a) a standard cantilever with 20 nm CoCr coating, (b) a permalloy-coated cantilever (20 nm permalloy film), and (c) a NiCo-coated cantilever (low coercivity cantilever, coercivity \sim 1 Oe). All MFM cantilevers employed here were of the PPP type. Note the reduction in MFM signal strength in (b), which otherwise gives a well-reproduced magnetic image. For both the NiCo and NiFe coatings, there is no negative effect on the observed domain structures. The NiFe tips are, however, not stable in ambient conditions and the obtained *z*-range is only 2°, while the z-range for NiCo is 4°. Therefore, this is a clear advantage for the low-coercivity NiCo tip, where no extra cap layer is required.

4. Conclusions

FIB milling is employed to structure permalloy thin films. We find that small ion doses (=small ion currents) do not affect the resulting magnetic domain patterns in the nanostructures created, whereas higher doses lead to the destruction of the magnetic structures. Therefore, FIB milling at low ion-beam currents can be applied to create high-quality permalloy nanostructures [5]. Different MFM cantilevers were tested to image the domain patterns in the resulting nanostructures. The low-coercivity NiCo cantilevers prove very successful to image the domain patterns of permalloy without negative effects.

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