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Manipulating the switching in modulated iron nanowires grown by focused electron beam induced deposition

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ABSTRACT

The future technological impact of smaller, faster and more efficient spintronic devices compared to current technologies inspires the quest of new approaches and strategies. Emerging non-conventional nanofabrication tools are required for this purpose. One attractive technique is focused electron beam induced deposition, a direct-writing process of ferromagnetic nano-objects. Here, we report the fabrication of highly pure iron wires with one-dimensional thickness modulation using diiron nonacarbonyl, $\text{Fe}_2(\text{CO})_9$ as a starting material. For that purpose, we employ a strategy for the electron beam scanning method, in which the beam spots are separated a certain distance from each other in one direction during the deposition process. Magnetic properties of the wires have been experimentally studied by magneto-optical-Kerr microscopy and supported by micromagnetic simulations. Our results suggest that the thickness modulation induces a local magnetic anisotropy along the short axis on the iron wire, which is not present in wires with a uniform thickness. By the proposed writing strategy, the switching field in such modulated wires could be controlled due to changes in magnetostatic interactions. Based on our outcomes, we conjecture that this procedure can be valuable in research on the impact of one-dimensional thickness modulation in nano-objects and future spintronic devices.

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1. Introduction

The promising impact of smaller, faster and more energy efficient spintronic schemes in the current implemented technology based on semiconductor devices motivates the research of novel materials and architectures. Thus, the manipulation of magnetization is crucial for future applications in spintronics and nanomagnetism. Particularly, the magnetic switching of nanomagnets, as characterized by the coercive field (H_c), has been widely studied in the last decades [1–3]. In monodomain nanostructures, the magnetization reversal process basically consists of the nucleation of a domain wall (DW) and of its propagation, which are governed by magnetic properties of materials as well as structural parameters of the nano-objects. When considering larger nanostructured magnetic objects, such as in a wire-like geometry, one can manipulate different domains of opposite magnetization. This way, magnetic data can be stored in the nanowire and can be moved through it by a spin-polarized current, forming a so-called racetrack memory [4].

Exploring the magnetization reversal process may profit from emerging methods for patterning structures at the nanoscale using focused electron or ion beams (FEB or FIB) [5,6]. Focused electron beam induced deposition (FEBID) is one example of the available focused beam induced processing techniques. In general, it consists the direct-

writing of materials on a substrate using the FEB as a ‘pencil’. The primary beam, the secondary electrons and the backscattered ones interact with precursor molecules adsorbed on a substrate surface, dissociating them into non-volatile and volatile compounds. The non-volatile ones are attached to the surface whereas the volatile ones are pumped away. FEBID has unique advantages to fabricate nanoelements with respect to conventional techniques (such as electron beam lithography): it is a mask-less and single-step process, and it has the ability to grow ‘real’ 3D nano-objects [7]. Moreover, functionally engineered nanostructures could be grown on any surface in a routinely run [8], in particular magnetic deposits based on cobalt [9–11] and iron [12–16]. In the fields of spintronics and nanomagnetism, valuable breakthroughs of using FIB in magnetic nanostructures have been carried out to control of magnetization reversal and domain wall conduit in planar nanowires [17–19], the fabrication of 3D nanowires [7,20,21], the synthesis of nanomagnet logic [22,23] and the growth of densely packed magnetic arrays [24]. Thus, FEBID is highlighted as an alternative nanolithography method for applications in magnetic logic [25] and non-volatile magnetic storage [26].

In the present manuscript, we propose a novel strategy of using FEBID to produce magnetic thin film elements of which the thickness is modulated in a stripe-like pattern in order to tune the switching behaviour. We report on the magnetic reversal behaviour in magnetic Fe FEBID wires prepared using this approach. The magnetization reversal is investigated by magneto-optical Kerr microscopy, where the hysteresis loops are obtained by measuring the intensity of the wire image as a

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function of the applied magnetic field. Our approach consists varying the ‘scanning strategy’ of this direct-write technique (see Fig. 1). More specifically, by changing the beam overlap in one direction during the e-beam writing process, we introduce a subtle thickness modulation along the long axis of the wires, enabling a new way to control the local anisotropy and the switching fields of nanomagnets. By using micromagnetic simulations together with experimental results, we demonstrate that the coercive field dependence in such structures is governed by the magnetostatic energy. This energy term promotes the formation of a multi-domain state in which neighbouring domains are aligned in an antiparallel configuration. That magnetic domain configuration could be utilized in magnetostatic surface waves [27,28]. We envision that our e-beam writing strategy could have a great potential in fundamental investigations and for applications in spintronics and magnonics.

2. Experimental details

2.1. Iron wires with thickness modulation

Fe wires were grown in a FEI Nova Nanolab 600 dual beam system using $\text{Fe}_2(\text{CO})_9$ as a precursor material. The Fe gas injection system (GIS) uses a small needle of 160 μm in diameter positioned at an angle of $\sim 16.5^\circ$ with the horizontal sample, 40 μm from the area of interest in the horizontal direction and 40 μm from the substrate in the vertical direction. The deposits were grown on a boron-doped Si substrate with a few nanometres of native SiO_2 on top.

Typical deposition parameters used for the FEBID process were: electron beam voltage = 5 kV, electron beam current = 6.3 nA, electron beam diameter (w) ~ 33 nm, electron beam overlapping in y -direction = 0%, electron scanning mode = raster, time that electron

beam stays at the same spot (dwell time) = 30 μs , time the electron beam is deflected to permit the replenishment of precursor molecules (refresh time) = 50 ms, electron beam dose ~ 238 nC/ μm^2 , precursor material temperature ($T_{\text{precursor}}$) = 27 $^\circ\text{C}$, base pressure inside the deposition chamber (p_{base}) $\sim 6 \times 10^{-7}$ mbar, and pressure inside the deposition chamber during the deposition (p_{process}) $\sim 4 \times 10^{-6}$ mbar (since we cannot give real data of the effective pump speed for $\text{Fe}_2(\text{CO})_9$ molecules in our equipment, the molecular flux (particles/ cm^2s) that reaches the surface has not been estimated). The dimensions of wires are $10 \times 0.5 \times 0.02$ μm^3 , keeping aspect ratio ($\text{AR} = \text{length}/\text{width}$) equal to 20 to have a single domain structure in remanence. To investigate the effect of the thickness modulation in the magnetic switching of wires, we create a subtle thickness modulation in the Fe wires modifying the e-beam scanning strategy (see Fig. 1(a)). We vary the distance (d) between the centres of the beam spot (indicated as circles) to the next one along its long axis (x -axis), from 5 up to 100 nm. As a result one can obtain a wire with a one-dimensional thickness modulation as represented in Fig. 1(b).

The composition of wires was studied in-situ by energy dispersive X-ray spectroscopy (EDS) using a detector with ~ 135 eV energy resolution calibrated for Si $K\alpha$ line, and EDAX Genesis software. The spectra were collected at an acceleration voltage of 5 kV and electron beam current of 0.80 nA.

The profile and thickness of the wires were analysed by an atomic force microscope (AFM) in non-contact mode. Wires grown under the same conditions are equivalent in shape, except for thickness, where differences in the precursor pressure and focusing produce maximum changes in thickness of around the 20%. Moreover, the profile of thickness modulation in the wires was investigated by standard FIB cross-sectional inspection.

2.2. Magnetization reversal of modulated wires

The magnetic characterization of Fe wires at room-temperature has been studied by magneto-optical Kerr microscopy. Since the shape anisotropy and in-plane magnetization govern the magnetization reversal in Fe wires, a longitudinal Kerr effect configuration and an in-plane magnetic field oriented parallel to the easy axis (x -axis) were used. The hysteresis loops are obtained by measuring the intensity of the image of the wires. First, a background intensity of the wire at zero field was measured. After that, the wire was saturated in a negative field, and the background intensity was subtracted to collect intensity values with appreciable changes in the magnetization with respect to the saturated state. Then, the magnetic field was swept to positive values in small steps of 0.02 mT and after each step the intensity was measured. When the magnetization changes in the wire, a change in the intensity was observed at a certain field, called the switching field. This procedure was repeated to sweep the field from positive to negative values to make a complete hysteresis loop. The typical linear slope caused by the Faraday effect was subtracted for the data. The measured intensity was normalized to have a value of zero at zero magnetic field. The hysteresis loops were collected from 50 averaged intensity values of the images in Fe wires providing their magnetic information. The noise in the measured intensity is typically around 20% of its maximum value.

2.3. Three-dimensional micromagnetic simulations of modulated wires

To evaluate the influence of the thickness profile to the magnetization reversal of modulated Fe wires, micromagnetic simulations using the 3-dimensional Object Oriented Micromagnetic Framework (OOMMF) code [29] were performed. The material parameters used in the simulations are: saturation magnetization $M_s = 1700$ emu/ cm^3 , exchange stiffness $A = 16 \times 10^{-12}$ J/m and magnetocrystalline anisotropy $K = 0$ J/ cm^3 . Here, we assume that the magnetocrystalline anisotropy is averaged out because of the random orientation of grains, as revealed by

Fig. 1. Sketch of the growth of modulated iron wires by focused electron beam induced deposition. (a) Electron beam scanning strategy on the substrate surface. (b) Top panel: deposit fabricated using the previous strategy on the substrate surface. Bottom panel: Cross-sectional view of an area of the deposit indicated with the black line in the top panel. d indicates the distance between centres of the beam spots along their long axis (x -axis) and w the beam diameter.

transmission electron microscopy studies [15,19]. Since the Gaussian-shaped profile of the wires shows the same general dependence of H_c with thickness as a rectangle-shaped profile [19], we simplify the geometry of our wires in order to speed up simulations. We simulate a rectangular-shaped wire with a rectangular-shaped profile of the thickness modulation with the same aspect ratio as the experimental wires ($1.248 \mu\text{m} \times 60 \text{ nm} \times 1\text{--}2 \text{ nm}$). A unit cell size of $4 \times 4 \times 1 \text{ nm}^3$ is used in the x , y and z -direction. Magnetic hysteresis loops and field evolution of magnetic configurations in the wires are calculated under a static magnetic field applied in the long axis (x -axis) with a constant damping coefficient $\alpha = 0.5$ (to speed up the calculation and avoid precessional effects), and an equilibrium criteria of $dm/dt < 0.1 \text{ deg./ns}$.

3. Results and discussion

3.1. Magnetic switching in modulated wires

Recent studies have shown for iron FEBID deposits [19,22] the dependence of coercive field with thickness does not follow the Stoner–Wohlfarth model for a single domain particle [30,31]. The latter predicts that structures with the same AR show a coercive field trend proportional to the thickness, whereas experiments on FEBID structures display the opposite trend for thicknesses larger than 20 nm.

However, the dependence of H_c in wires with modulated thickness has not been investigated so far. In these modulated Fe wires (see experimental section for details), we keep a fixed $AR = 20$, whereas we vary the distance (d) between beam spots along their long axis, creating a 1D thickness modulation along their long axis. SEM images of a small area of several modulated wires as a function of d/w are displayed in Fig. 2. The inset of Figure S1(a) in the Supplementary data shows a typical SEM image of one of the studied wires with pointed end. This shape is usually utilized to prevent the easy nucleation of domain walls at the end of the wire and consequently low coercive field values. The composition of the iron wires analysed by EDS reveals that they contain a composition of 75–80 at.% in Fe, 10–15 at.% in C and 5–10 at.% in O, which corresponds to the optimized values reported in the past which guaranteed the magnetic behaviour [15] (See Figure S1 in Supplementary data). The thickness and profile of Fe wires are investigated by means of AFM measurements (see Figures S2–5 in Supplementary data). From the profiles in x and y direction, as extracted from the AFM images, it is found that the maximum thickness of the wires corresponds to $\sim 20 \text{ nm}$ and that our method was successful in creating a thickness modulation. Although we cannot extract the real thickness in the modulated areas from the images due to several experimental reasons listed in the following. We used AFM in the non-contact mode, the tip had a typical curvature radius of 10 nm, the lateral resolution of the technique in the ideal case could be 10 nm and, the non-uniform profile characteristic of our modulated wires. For this purpose, high-resolution SEM images of FIB cross-sectional view of wires are taken. As an example

the inset of Fig. 3(d) shows the SEM image for a modulated wire of $d/w = 3.07$.

Fig. 3 shows examples of hysteresis loops for modulated Fe wires, (a) $d/w = 0.15$, (b) $d/w = 0.31$ and (c) $d/w = 1.53$. For all cases, we observe square-shaped loops, typical for easy axis (x -axis) in ferromagnetic structures. Moreover, the switching field decreases as a function of d/w (see Fig. 3(d)). Such a dependence could be explained by an induced local anisotropy along the short axis (y -axis) of the wire which is absent in the uniform thickness wires. In modulated wires, one should consider two competing magnetic anisotropies, one along the long axis (x -axis) of the wire resulting from the shape anisotropy and the other one is along the short axis (y -axis) of the wire due to the created thickness modulation [32,33]. Since the artificially created anisotropy is getting more pronounced as a function of d/w , it could cause a non-uniform switching field in the different regions of the wires and different domain structures.

3.2. Micromagnetic study of the influence of the profile in the magnetization reversal of modulated wires

In order to understand the switching behaviours of the experimental data, we performed micromagnetic simulations on the modulated wires with, $d/w = 1.33$, $d/w = 2$ and $d/w = 3$, where the thickness in the modulated areas corresponds to a half of the total thickness (see the bottom inset of Fig. 4(a)). The switching field versus d/w is depicted in Fig. 4. Here the switching field is selected from the value where M_x/M_0 corresponds to zero in each case, as marked in the top inset of Fig. 4(a). Although the values of the switching fields from the simulations are relatively larger than those from experiments, which might be due to the fact that our micromagnetic simulations at $T = 0 \text{ K}$ prevent thermal activation in a switching process, the general trend is qualitatively in good agreement with the experimental results in Fig. 3(d). We note that the hysteresis loop in the modulated wires, here i.e., for $d/w = 2$ (see the inset of Fig. 4(a)), shows a mixture of an S-shaped and a square-shaped loop, contrast to the experimental data (see Fig. 3(a–c)) where only square-shaped loops are observed. In a uniform thickness wire, the easy axis is x -axis but this scenario is modified by introducing a thickness modulation. Having a look at a single modulated area, the x -axis is now the hard axis and y -axis turns into an easy axis. Therefore by measuring magnetization along the x -axis, those loops should have a completely S-like shape. Since each modulated area is however both exchange- and dipolar-coupled to each other via a non-modulated area, the loops should display a mixture of both hard and easy axis behaviours as observed in the simulations. As already mentioned, this behaviour is absent in hysteresis loops of experimental wires. This different behaviour could be caused by several reasons such as the Kerr microscope resolution and the clear difference in the experimental and simulated profile. We speculate that since the resolution of our microscope is around $\sim 200 \text{ nm}$, it could be highly improbable

Fig. 2. SEM images of modulated Fe wires varying d/w taken at a viewing angle of 52° with the substrate normal.

Fig. 3. (a–c) Examples of magnetization MOKE loops for modulated Fe wires. All the magnetization refers to the x-direction (easy magnetic axis). (a) $d/w = 0.15$, (b) $d/w = 0.31$, and (c) $d/w = 1.53$. (d) Dependence of switching field with d/w . The line is a visual guide for the reader. Inset shows a SEM image of a cross-sectional view of the wire with $d/w = 3.07$.

to measure differences in the intensity for each modulated area (~ 50 nm in length for x-axis) from the measurement of a complete wire (10 μm in length). On the other hand, the simulated wires have a rectangular shaped profile, which is a simplified version of the real Gaussian-shaped profile of experimental ones. This simplification could cause that the competition between both exchange- and dipolar-interactions would be stronger in the simulated wires. Simulations however aim to explain qualitatively the switching field dependence of the experimental wires, so detailed analysis of the shape of hysteresis loops is beyond the scope of this work.

To get a closer insight of underlying physics, we further extracted magnetic energies from the simulations. In our model system, the switching field is determined by a compromise among Zeeman energy (E_{Zeeman}), exchange energy (E_{Exchange}) and demagnetization energy (E_{Demag}), which strongly depend on local magnetic configurations. To get rid of the complexity of the problem, we compared each energy term normalized by the volume of each microstructure, under the very high in-plane field, i.e., $H_x = -500$ mT, where the magnetization in all cases of microstructure is fully saturated in the $-x$ -direction (see one example in Figure S7 of the Supplementary data). Fig. 4(b) displays the dependence of each energy term on d/w . Both E_{Zeeman} and E_{Exchange} are almost constant irrespectively of d/w , whereas E_{Demag} decreases with d/w . The dependence of E_{Demag} on d/w is in accordance with the dependence of the switching field on d/w as observed in both experiments and simulations, inferring that demagnetization energy plays a dominant role in determining the switching field.

The magnetic configuration in the domain structure of the wires also provides us important clues to understand magnetic behaviour in our thickness-modulated system. Fig. 5(a) shows in the top panel the magnetization components at the switching field ($M_x/M_0 \sim 0$) for positive and negative applied magnetic fields, for example, in the wire with $d/w = 2$. Fig. 5(b) displays two regions of two magnetization

vector colour maps (M_x/M_0 at positive and negative applied fields) indicated by the blue coloured area in (a). First of all attending to the $+x$ ($\mu_0 H_x^+$) and $-x$ ($\mu_0 H_x^-$) components in (a), we clearly observe that their sign is opposite to each other. This indicates that the energy barrier to overcome the switching field at positives and negative fields is mainly due to this component in the domain structure. This energy is caused by the manipulation of the shape anisotropy by the local anisotropy introduced by the thickness modulation. This conclusion agrees well with our previous argument in which the demagnetization energy is the main contribution for the switching field dependence in our simulations and experiments. Second, focussing on the $+y$ ($\mu_0 H_y^+$) and $-y$ ($\mu_0 H_y^-$) components, we identify a multi-domain structure with an antiparallel configuration (from -1 to 1 , and from 1 to -1 and so on) whereas a mono-domain state except for the edges is observed in uniform thickness wires (see Figure S6 in the Supplementary data). In the first case, these magnetic domains are formed by a competition of the energy terms [32,34,35]. Since our wires experience a one-dimensional thickness modulation at the surface, the magnetization in each modulated area is exchange coupled via the non-modulated area as well (see Fig. 5(b)). Thus each area does not play a role as an individual domain or nano-object. Instead of this the magnetic dipolar interaction between the adjacent thick and thin areas creates energetically more favourable large domains, forming a multi-domain structure antiferromagnetic coupled. In modulated wires, the domain size is mainly determined by that energy term.

Taking the y components of the magnetization for the different modulated wires (see the inset of Fig. 5(c)), one could extract the maximum value of its derivative, which properly follows the same trend as the demagnetization energy with d/w (see Fig. 5(c)).

Thus, from the micromagnetic simulations one can conclude that for such wires a decrease of the demagnetization energy with d/w causes a similar qualitatively trend in the switching field with d/w , as

Fig. 4. Micromagnetic simulations of modulated Fe wires. (a) Switching field dependence with d/w . Inset shows hysteresis loop for wire $d/w = 2$. (b) Normalized energy terms as a function of d/w . Lines are a visual guides for the reader.

experimentally observed in Fig. 3(d) and the formation of multi-domain structure with antiparallel configuration.

4. Conclusions and outlook

In this work, we have proposed a “novel strategy” to fabricate nano-objects with a one-dimensional thickness modulation by FEBID. In this approach, we modify the e-beam sweeping by varying the beam overlap in one direction during the writing process. This procedure has been successfully applied to fabricate a novel set of highly pure iron wires with a one-dimensional thickness modulation. This method allows one to manipulate the local anisotropy and the switching fields of nanomagnets.

We have systematically characterized the magnetic reversal behaviour of modulated wires in a broad range of distance/width ratio. Above all, we have experimentally demonstrated the manipulation of switching fields as a function of the thickness modulation. Additionally, by using micromagnetic simulations we have explained that this coercive field dependence mainly depends on the magnetostatic energy. This energy contribution produces large domains in such wires with an antiparallel configuration. Such a peculiar magnetic domain configuration could be utilized in the propagation of magnetostatic surface waves. The proposed e-beam writing strategy could be utilized in research on the effect of 1-D thickness modulation in nanomagnets and forthcoming spintronic devices.

Notes

The authors declare no competing financial interest.

Fig. 5. (a) Magnetization components (x, y, z) at the switching fields (positive and negative applied fields) for a region of the simulated wire $d/w = 2$. (b) Snapshots of two magnetization vector colour maps (M_x/M_0 at positive and negative applied fields, x, z plane) indicated by the blue coloured area in (a). (c) Dependence of the maximum value of the dM_y/M_0 with d/w . Inset shows the domain structure of the y component of the magnetization in the modulated wire. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

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Supplementary data

Supplementary data to this article can be found online at <http://dx.doi.org/10.1016/j.mee.2016.01.032>.

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