The formation and magnetism of iron nanostructures on ordered polymer templates

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Abstract. We have followed the self-assembly of a magnetic antidot array during Fe sputter deposition onto a highly ordered, nanostructured polymer template by using a new kind of synchrotron radiation three-dimensional x-ray microscopy. The structural growth, chemical state (the formation of oxides) and the magnetic transition were determined in situ with 0.1 nm spatial resolution, revealing the excellent properties of polymer templates for a simple self-assembled large-scale fabrication of nanomagnetic systems tunable in size and spatial arrangement. Our method allows us to establish stability limits of novel magnetic nanostructures for high-density magnetic recording in a quantitative manner.
1. Introduction

Large-scale nanopatterning is one of the major issues in nanotechnology and nanoscience, but conventional methods such as optical lithography or focused ion beam etching increasingly lack the desired spatial resolution or suffer from long processing times. The realization of block copolymer films with unit cells down to a few nanometers and their alignment into long-range crystalline order over arbitrary large length scales [1–5] constitutes a new category of fabrication methods based on the hierarchical bottom-up approach. It has been shown that highly ordered metallic nanostructures can be realized by thermal evaporation of small numbers of metal atoms onto these templates due to a strong selective wetting of the different polymer blocks [6].

Particular attention has been given to magnetic nanostructures forming on block copolymer templates due to their potential for high-density magnetic recording. Tailoring magnetic nanostructures into this direction requires a precise knowledge of their structure, chemistry and magnetism. Unfortunately, there is a lack of characterization tools capable of tracking all these properties with the required spatial resolution, especially for three-dimensional (3D) objects. To gain a comprehensive knowledge of the sensitive interplay of these three parameters, all of them should be continuously recorded during formation.

Magnetic antidot arrays, the inverse structure of dot arrays, have attracted particular attention as candidate materials for data storage technology [7–10]. Their internal magnetic shape anisotropy caused by the size and arrangement of nanoholes induces a dramatic increase in the coercivity of magnetic materials, which can reach values up to two orders of magnitude higher than that of its parent continuous thin films [11]. Because antidot arrays are characterized by the absence of isolated material, bit cells in the lattice should not be influenced by the superparamagnetic effect. This qualifies them as candidates to beat the superparamagnetic bit size limit. In addition, experimental and theoretical investigations currently focus on magnetic antidot arrays as magnonic crystals with a variety of standing spin waves pattern [12–15], on their domain structure and on their magnetic hysteretic switching behavior [16–20].

In this work, we investigate in situ the formation of a magnetic antidot array by iron sputter deposition onto a diblock copolymer (PS-b-PMMA) template with a 3D surface morphology. Grazing-incidence small angle x-ray scattering (GISAXS), nuclear resonant x-ray reflectometry...
Figure 1. Scheme of the experimental setup for synchrotron radiation 3D microscopy on the self-assembled growth of the iron nanostructure. $^{57}$Fe is sputter deposited onto a highly ordered polymer template with a 3D surface morphology as sketched in figure 2. GISAXS and NFS are combined to study the process of self-assembly and to determine the chemical state of the iron nanostructure as well as its magnetic transition to an ordered ferromagnetic state. In addition, NRXR measurements reveal the depth profile of iron in the template with 0.1 nm spatial resolution. The notation of the scattering geometry is shown below.

(NRXR) and nuclear forward scattering (NFS) of synchrotron radiation at 14.4 keV resonance of the $^{57}$Fe Mössbauer isotope were combined to follow the growth of the iron nanostructure, to detect its chemical state (the formation of oxides) and to investigate the magnetic transition from the nonmagnetic to an ordered ferromagnetic state. This new approach offers sensitivity to all the key parameters over a wide dynamic range from sub-nm to $\mu$m length scales, providing comprehensive information not accessible with conventional microscopy.

2. The experimental setup and preparation of the polymer template

The experiment was performed at the nuclear resonance beam line ID18 [21] of the European Synchrotron Radiation Facility in the 16-bunch mode of operation. A combined in situ NFS/GISAXS setup (figure 1) was implemented using a fully remote controlled UHV sputter deposition system [22]. The direct beam was focused to a cross section of 300 $\mu$m $\times$ 150 $\mu$m onto a MarCCD-165 camera with a focal length of 15 m and a sample-to-detector distance of 4 m. This enables a suitable GISAXS resolution in $q$-space with $0.003$ nm$^{-1} < \Delta q_y < 2$ nm$^{-1}$. The scattering vectors in the horizontal and vertical directions $q_y$ and $q_z$ (see figure 1) are given by $q_y = 2\pi/\lambda (\sin \Psi)$ and $q_z = 2\pi/\lambda (\sin \alpha_{in} + \sin \alpha_{out})$, respectively. Here $\lambda$ is the wavelength of the x-ray. To detect the nuclear reflectivity, a stack of four avalanche photo diodes was
used. For the GISAXS measurements this detector was replaced by a beam stop to block the specular reflected beam. The linearly polarized synchrotron x-ray beam was monochromatized to a bandwidth of 1 meV and tuned to the 14.41 keV transition of $^{57}$Fe nuclei.

2.1. The polymer template

The nanostructured template was made of a poly(styrene)-block-poly(methyl methacrylate) (PS-b-PMMA) copolymer that was spin coated on a pre-treated silicon (100) wafer. The 90 nm thick film was annealed for 72 h at 165°C in vacuum to form a hexagonal arrangement of vertical PMMA cylinders in the PS matrix. The template was afterwards exposed to UV light (wavelength 254 nm) for 4 h to break the polymer chains in the PMMA and crosslink the PS block [23]. The cylinders were then partially removed from the matrix by treating with glacial acetic acid and distilled water. This way we realize the arrangement of ordered vertical nanopores embedded in a flat PS matrix.

Figure 2(e) shows ex situ magneto-optic Kerr effect (MOKE) magnetization curves of iron which was sputter deposited onto such a nanoporous polymer template and capped with a 5 nm thick Ta layer to prevent oxidation. The iron undergoes a hard magnetic transition typical of magnetic antidot arrays. The in situ sputter experiment will provide detailed information about the extent to which such an antidot array can be prepared with this method. It will help clarify the accumulation of iron on the template, it will help identify its chemical state on the organic template and, finally, it will allow us to detect its intrinsic magnetic transition.

3. Results

3.1. Grazing-incidence small angle x-ray scattering (GISAXS) on the virgin polymer template

The in situ experiment started with the characterization of the virgin nanoporous polymer template using GISAXS. The sample was illuminated with an x-ray beam at an angle of incidence of 0.2°, slightly above the critical angle of the polymer. A full 2D simulation of the intensity distributions of the diffusely scattered x-rays was performed with the data evaluation software IsGISAXS [24] to obtain a comprehensive picture of the template morphology. The simulation was done within the distorted wave Born approximation for the polymer layer and for the form factor of the empty cylinders. The size spacing coupling was treated within the local monodisperse approximation. Representing the pores by a lateral arrangement of hexagonally oriented empty cylinders, an average distance of 47.2 nm was found to reproduce the position of the first side wings in the horizontal ($q_y$) intensity distribution (figure 2(a), red line). These holes are symmetrically ordered within domains of 500 nm in width and the domains are laterally averaged over all in-plane orientations (figure 2(b)). The diameter of the empty cylinders was determined to be 28.2±0.2 nm. This quantifies the relative surface area of the holes to about 30%. These findings are in good agreement with atomic force microscopy (AFM) measurements (figure 2(d)), which locally probe the template. In contrast to this direct imaging technique, GISAXS provides access to buried vertical structures. A vertical line cut through the first side wing is shown in figure 2(a) (blue line). The intensity distribution depends on the same parameters as conventional x-ray reflectivity curves and on the vertical correlation of the holes. Here we only give the size distribution of the depth of the holes. Two equally populated depths
Figure 2. Investigation of the nanoporous polymer template. (a) 2D-GISAXS image of the sample. The line cuts (black lines) show the scattering intensity caused by structural correlations along the horizontal and vertical directions (through the Yoneda peak at $\alpha_{\text{out}} = 0.10^\circ$ and along the first side wing at $\Psi = 0.12^\circ$). The continuous red and blue lines show a consistent high-quality fit of both cuts. (b) and (c) Structural parameters of the nanoporous film derived from the simulation. Here a cross section through the polymer template is shown. GISAXS reveals information about the sample morphology averaged over the footprint of the x-ray beam (in this case 40 mm $\times$ 0.3 mm). (d) AFM topography that probes the surface morphology locally. (e) Ex situ MOKE magnetization curves of capped iron which was sputter deposited onto such a nanoporous polymer template. The iron undergoes a hard-magnetic transition typical of magnetic antidot arrays. The in situ sputter experiment will give detailed information about the extent to which such an antidot array can be prepared with this method. It will clarify the accumulation of iron on the template (on the surface and/or inside the holes), its chemical state on the organic template (metallic iron or oxide) and its intrinsic magnetic transition in this 3D system.

were found at values of 6.4 and 22.5 nm, both with a size distribution of $\pm 5\%$. This precise information on the template morphology constitutes the basis for the later reconstruction of the 3D self-assembled iron nanostructure.
3.2. GISAXS on the iron sputter deposited template

Iron was stepwise sputter deposited at room temperature with a deposition rate of 0.2 nm min$^{-1}$ onto the template. The applied voltage was 250 V and the sample-to-target distance 8 cm. After certain deposition steps the sample was investigated using GISAXS, NRXR and NFS until the magnetically ordered ferromagnetic state was reached. A relatively high argon pressure of 2 $\times$ 10$^{-2}$ mbar was used to reduce the thermal energy of the sputtered iron atoms to avoid implantation of the atoms into the polymer. Two of the GISAXS patterns taken during the sputter deposition process are plotted in figure 3 to show the early and final deposition stages (nominal Fe deposition 1.0 and 5.1 nm, respectively). The angle of incidence of 0.4$^\circ$ was used to separate the specular reflection from the Yoneda peak originating from the iron layer. The intensity distribution along $q_z$ of the final deposition stage is characterized by additional long-period diffuse Kiessig fringes typical of a vertical growth with a small distribution in height. In contrast with this, almost no change is seen along $q_y$. Also, in the early stage of growth no significant change of the horizontal GISAXS intensity distribution can be seen, especially at high $q_y$-values. Thus GISAXS reveals that the iron homogeneously wets and reproduces the lateral surface structure of the polymer template right from the beginning. The formation and agglomeration of separated iron islands during the deposition run can be excluded.

For the simulation of the GISAXS patterns (within the capabilities of the IsGISAXS software), the antidot array was modeled as an arrangement of hexagonally ordered and shaped prisms placed on top of the polymer. Remarkably, no metal inclusions inside the holes had to be taken into account. In particular, the shape of the Yoneda peak which sensitively varies with the iron depth profile is very well reproduced within this simulation. The evaluation of the GISAXS pattern reveals an increase of the relative iron surface coverage during deposition from 70% (which coincides with the relative surface area of the pure template excluding holes) to about 80%. This corresponds to a decrease of 14% in the diameter of the holes with increasing film thickness. The vertical thickness $d$ of the iron antidot array is 1.7 nm after iron deposition of 1.0 and 5.7 nm at the final stage of 5.1 nm nominal iron layer thickness. The thickness variation in both cases is below 25% of the nominal value.

Until now, GISAXS has revealed a dominating vertical growth on top of the template but important questions regarding the structural properties remain unanswered. How much iron goes into the holes and is there any chemical interaction with the template in general? Finally, how does the magnetic order in the array evolve during growth? These questions are answered via NRXR and time resolved NFS measurements.

3.3. Nuclear resonance scattering on the growing iron nanostructure

3.3.1. Experimental procedure. The resonant reflectivity curves were taken simultaneously with the conventional (nonresonant) x-ray reflectivity curves. The resolution of the iron depth profile determined with this method is enhanced to 0.1 nm using x-ray interference effects. The chemical and magnetic state of the growing iron nanostructure can be derived from the temporal evolution of the delayed nuclear signal after excitation by the synchrotron radiation pulses [25–27]. The coherent de-excitation of the hyperfine-split nuclear level leads to a beat pattern in the temporal evolution of the resonantly scattered wave field (referred to as the time spectrum in the following) from which the magnitude and orientation of the magnetic and electric hyperfine fields can be determined with high precision.
Figure 3. Evolution of the polymer surface morphology during iron sputter deposition. 2D-GISAXS data and simulation of the early and final deposition stages (1.0 and 5.1 nm of the deposited iron). The investigation shows that the iron homogeneously wets the polystyrene surface and the diameter of the holes decreases by about 14% during the deposition process. The thickness of the antidot array was determined to be 1.7 and 5.7 nm after deposition of 1.0 and 5.1 nm of iron, respectively.
3.3.2. Origin of the 0.1 nm sensitivity of the nuclear reflectometry to the iron depth profile.

Due to the 141 ns lifetime of the nuclear transition, the resonantly reflected photons can be discriminated via time gating from the electronically reflected ones (prompt signal of the reflection channel). The delayed signal that is collected between the x-ray synchrotron pulses is of pure nuclear origin, resulting in a very high sensitivity to the iron depth profile. The interference of the incoming and reflected radiation forms a standing wave above the sample with a spatially oscillating modulation perpendicular to the surface. The position of the resulting nodes and antinodes of this standing wave can be adjusted by the angle of incidence. The field intensity distribution inside the sample is strongly affected by the metallized polymer surface, which causes a strong x-ray wave guiding effect and multiple reflections of the x-rays inside the polymer x-ray guiding layer [28]. These reflections inside the polymer layer interfere constructively at selected angular positions below the critical angle of the iron capping layer. The formation of these so-called waveguide modes results in a significant increase of the field intensity (see the calculated field intensity for the first waveguide mode at 1.0 nm deposition in figure 4). The nuclear signal will therefore be drastically boosted at angular positions where waveguide modes are excited and the vertical position of the iron coincides with one of the antinodes of the standing wave field [29, 30]. Only a small (or zero) nuclear contribution will originate from the iron placed in the nodes of the standing wave field. The simulation of the nuclear reflectivity around the critical angle thus offers 0.1 nm sensitivity to the iron located within the layer system, especially inside the holes.

The time spectra during the sputter deposition process were taken at the angular position of the first waveguide mode \(\alpha_{in} = 1.6 \text{ mrad}\) up to a film thickness of \(d = 2.4 \text{ nm}\) and at the position of the third waveguide mode \(\alpha_{in} = 2.0 \text{ mrad}\) for the following deposition steps. At these angular positions, the sensitivity to the iron located inside the holes is strongly enhanced, but allows maintenance of a strong contribution from the antidot array on top of the template. The nuclear reflectivity data and time spectra were simulated with the program package CONUSS [31] where the structural parameters from the GISAXS measurements and from the conventional x-ray reflectivity measurements were taken into account. A consistent four-layer model was established for the iron depth profile that will be discussed for the deposition steps illustrated in figure 4.

3.3.3. The 3D structure and chemical state of the growing iron nanostructure.

The evaluation shows that 45% of the first 1 nm iron deposited on top of the template oxidizes due to chemical interaction with the polymer surface. This explains the rather high thickness of the antidot array detected with GISAXS (1.7 nm), which is due to the volume expansion during oxidation of about 100% relative to the metal volume. Subsequently, sputter deposited iron forms a metallic iron film on top of this oxide layer.

Interestingly, the \(^{57}\text{Fe}\) depth profile obtained from the simulation excludes a significant accumulation of material at the bottom of the nanopores. Instead, a homogeneous nonmagnetic iron oxide film is formed at their inner walls, which grows linearly with the amount of deposited iron. This oxide contributes to the long-period beating of the time spectra in figure 4, reflected by an isomer shift of 0.45 mm s\(^{-1}\) and a quadrupole splitting of 0.7 mm s\(^{-1}\). Therefore, the iron atoms reaching the bottom of both types of holes do not wet the PMMA but stay on the PS to form a homogeneous film at the inner wall of the empty cylinders (figure 4). Such a remarkable strong selective wetting has already been found for metal atoms deposited onto a PS-b-PMMA copolymer with a flat surface morphology [6], but has not been (or could not be) seen on a
Figure 4. Nuclear reflectivity curves (left column) and time spectra (right column) recorded at selected sputter deposition steps (1.0, 2.1, 3.0 and 5.1 nm). Solid lines are simulations. The reflectivity oscillations are very sensitive to the vertical distribution of $^{57}$Fe, thus revealing the incorporation of iron into the nanoholes with 0.1 nm accuracy. The time dependence of the nuclear signal is a fingerprint of the hyperfine fields and is used to determine the chemical and magnetic states. The schematic drawings in the central column present cross sections of the chemical structure of the sputter deposited nanoporous template at the corresponding deposition stages.

polymer template with a 3D structured surface. This effect is probably due to a mobility contrast for the deposited atoms between the copolymer domains and could additionally be affected by the different surface tensions.

As no iron could be found below the bottom of the pores, significant metal diffusion into the polymer can be excluded. An evaluation of the time spectra shows that the iron deposited inside the holes is completely oxidized and stays nonmagnetic. Using the precise GISAXS information about the morphology of the template, the surface wall area of the empty cylinders
Figure 5. (a) Evolution of the magnetization state of the iron antidot array determined from the time spectra (see the selected data sets in figure 4). $B_{\text{HF}}$ is the width of the magnetic hyperfine field used in the simulations. The upper data set shows the magnetization signal from the oxide formed in contact with the polymer surface. (b) Ex situ field emission scanning electron microscope (FESEM) image of the antidot array showing the material-specific contrast using secondary electrons for detection. The lower schematic drawing represents the 3D chemical state after nominal 5.1 nm deposited iron revealed by the in situ experiment. An iron thickness of the magnetic antidot array (*) of 4.8 nm was found on top of a FeO buffer of 0.9 nm (**). The oxide wall thickness inside the pores (*** is 4.4 and 1.3 nm for the two types of holes.

and thus the thickness of the oxide coating was calculated. The NRXR depth profile at the end of the deposition process reveals homogeneous oxide wall thicknesses of about 4.4 and 1.3 nm inside the holes with a depth of 6 and 22 nm (figure 5(b)), respectively. The strong oxidation effects involved inside the holes can be deduced either from the oxygen-containing surface of the PMMA cylinders on which most of the iron atoms are deposited first or from the remaining fractions of PMMA chains inside the holes.

3.3.4. Magnetization of the growing iron nanostructure. The evolution of the magnetic properties of the iron antidot array is monitored via the evolution of the magnitude of the magnetic hyperfine field $B_{\text{HF}}$, which is to a very good approximation proportional to the magnetization. The results are presented in figure 5(a) (green data set). The ordered ferromagnetic state in the antidot array is reached at a critical thickness $d_c$, which lies between the $d_c$ of a continuous film and the critical radius of spherical particles. A continuous iron film (depending on the substrate) becomes ferromagnetic at room temperature between a few monolayers and a thickness of 3 nm [32–34]. Magnetic single-domain iron nanoparticles become magnetically stabilized against room-temperature thermal excitation at a diameter of about 15 nm (the experimental reference for spherical iron nanoparticles does not exist; this value is the calculated one for spherical single-crystalline particles with an anisotropy constant of bulk bcc iron; the diameter for polycrystalline iron nanoparticle will be increased

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significantly). Here already at a thickness of 5 nm the hyperfine field reaches the $\alpha$-iron bulk value of about 33.3 T, as revealed by the pronounced high-frequency oscillations in the time spectra (figure 4). This behavior reflects the reduced dimensionality of the system, as well as the kind of magnetic transition itself. The onset of ferromagnetic order is observed at a thickness of 2.1 nm with a sudden increase of the magnetic hyperfine field. With further increasing the thickness a slow transition into an ordered ferromagnetic state takes place. The gradual transition very likely reflects the slow change of the ratio between surface and bulk atoms, which is slower than that for a growing continuous film and growing nanoparticles.

3.3.5. Impact of the oxide buffer on the field-dependant behavior of the iron antidot array. The upper curve in figure 5(a) describes the magnetic state of the ultra-thin oxide layer which is formed below the antidot array in contact with the top polymer surface. The evolution of its hyperfine field shows that this ultra-thin spin ensemble becomes magnetically stabilized by the adjacent iron layer. The final hyperfine field value identifies this oxide to be rather pure FeO (wüstite) [35]. Since FeO is known to order antiferromagnetically, such a magnetic oxide layer should significantly influence the magnetic properties of the iron antidot array. The contact between a ferromagnetic layer and an antiferromagnet usually causes an anisotropy component which is transferred to the ferromagnet (exchange bias effect [37]). However, Couet et al [35] showed recently that for an ultra-thin antiferromagnetic oxide layer embedded between Fe layers these effects are shifted to low temperatures, even below 50 K. In this case the oxide’s blocking temperature is significantly reduced in the ultra-thin film limit because the thermal energy overcomes the antiferromagnets’ own anisotropy [35, 36]. This brings us to the conclusion that at room temperature there is no significant influence of the oxide on the magnetic properties of the iron antidot array.

4. Conclusion

In conclusion, we have studied in situ the self-assembled growth of sputter deposited iron on top of a polymer template with an ordered arrangement of nanopores. A new kind of structural, chemical and magnetic high-resolution 3D microscopy was applied by combining complementary synchrotron radiation x-ray scattering techniques. We wish to point out that only this in situ study with the continuous monitoring of key parameters during growth enables one to obtain and correlate reliably information even for the smallest changes in the parameters during formation in a rather complicated structure.

A strong selective 3D-wetting of iron on the polymer domains was found. This emphasizes the excellent suitability of ordered diblock copolymer templates for a simple realization of sputter deposited nanomagnetic systems. By depositions of Fe on this template antidot arrays were formed with an evolution of the magnetic state that differs considerably from that of its parent continuous thin film. An ultra-thin single-phase oxide layer (FeO) in contact with the polymer film is formed, but it does not modify the field-dependant magnetic switching behavior of magnetic cells in the antidot array.

This study shows a recipe and an advanced tool for characterizing nanomagnetic structures that can also be applied, with an appropriate template, for the realization of magnetic nanostripes and dot arrays. The remarkable progress made recently in the fabrication of polymer templates, which now allows the arrangement of polymer nanodomains of a few nanometers in
size into arbitrary large single-crystalline lattices [1], supports the approach presented to realize self-assembled magnetic nanostructure.

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