Tailoring magnetic domains with hard x-ray free-electron laser-printed gratings

Victor Ukleev,1 Max Burian,1,2 Sebastian Gliga,1 C.A.F. Vaz,1 Benedikt Rösner,2 Danny Fainozzi,3 Gediminas Senutiunas,4 Adam Kubec,4 Roman Mankowsky,5 Henrik T. Lemke,5 Ethan R. Rosenberg,6 Caroline A. Ross,5 Elisabeth Müller,7 Christian David,4 Cristian Svetina,5 and Urs Staub1
1) Swiss Light Source, Paul Scherrer Institute, 5232 Villigen PSI, Switzerland
2) Laboratory for Non Linear Optics, Paul Scherrer Institute, 5232 Villigen PSI, Switzerland
3) Elettra - Sincrotrone Trieste S.C.p.A., 34149 Basovizza, Trieste, Italy
4) Laboratory for X-ray Nanoscience and Technologies, Paul Scherrer Institute, 5232 Villigen PSI, Switzerland
5) SwissFEL, Paul Scherrer Institute, 5232 Villigen PSI, Switzerland
6) Department of Materials Science and Engineering, Massachusetts Institute of Technology, Cambridge, MA 02139, USA
7) Electron Microscopy Facility, Paul Scherrer Institute, 5232 Villigen PSI, Switzerland

(*Electronic mail: victor.ukleev@psi.ch, urs.staub@psi.ch)

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The direct control of magnetic domain patterns on nanometer length and femtosecond time scales is of great interest, both fundamentally and for potential applications in spintronic devices. Here, we demonstrate how magnetic domain patterns in a Tm-doped yttrium iron garnet (Tm:YIG) thin film with perpendicular magnetic anisotropy can be permanently imprinted by the high intensity photon pulses of a hard x-ray transient grating (XTG). The XTG technique allows for the effective ultrafast manipulation of magnetic domain patterns, leading to characteristic changes in the magnetic structure.

Ultrafast magnetization dynamics have been intensely investigated using visible light, extreme ultraviolet and soft x-ray transient grating spectroscopy over the past decade. Complementary spatial and temporal studies of the spin dynamics are extremely valuable for the understanding of light-matter interactions as well as of intrinsic processes in strongly correlated systems. Such knowledge is essential for the development of potential applications in ultrafast spintronics at the nanoscale.

X-ray transient gratings (XTG) are formed by interfering coherent beams at the sample to generate spatially periodic excitation patterns. These patterns can equally display temporal structure, e.g., by using periodic laser pulses. XTGs offer unique opportunities to manipulate the structural and electronic properties of materials at the femtosecond timescale down to spatial scales of a few nanometers. In optical and XUV spectroscopy, splitting and crossing two laser pulses by a set of mirrors is a standard method to generate XTGs; in contrast, this is non-trivial in the soft x-ray and hard x-ray regimes. However, recent developments in x-ray free-electron lasers (XFEL) and x-ray optics have enabled an extension of the XTG technique towards hard x-ray energies.

In the present study we report the successful manipulation of the periodicity and of the spatial orientation of magnetic domains within a thin magnetic film by imprinting a hard x-ray grating that causes permanent structural changes of the material, leading to characteristic changes in the magnetic structure.

A 24 nm-thick thulium-substituted yttrium iron garnet \(Y_{0.51}Tm_{0.49}Fe_{5}O_{12}\) (Tm:YIG) film displaying perpendicular magnetic anisotropy (PMA) was grown on a (111) gadolinium gallium garnet \((Gd_{3}Ga_{5}O_{12}, \text{GGG})\) substrate by means of pulsed laser deposition (PLD). Details of the PLD synthesis and characterization of the sample are given in Refs. 22 and 23. This composition yields a film with lower perpendicular magnetic anisotropy than that of TmIG but with similar magnetization.

Highly-intense XFEL pulses with 40 fs duration and 50 Hz repetition rate were delivered by SwissFEL at the Bernina beamline24 to imprint the grating onto the Tm:YIG sample in the same setup as in Ref. 18. The energy of the incoming XFEL beam was 7.1 keV with a bandwidth of 0.3%. In total, 1000 XFEL pulses with duration of ~40 fs each were utilized to permanently imprint the gratings onto the sample. The x-ray beam fluence was controlled by a set of attenuators, varying from 3.5 mJ/cm\(^2\) to 70 mJ/cm\(^2\) at the sample, corresponding to an average of 5% and 100% of peak fluence of the full beam, respectively. A schematic illustration of the XTG experiment is shown in Fig. 1. The XTG pattern was generated by diffracting the incoming hard x-ray beam on the transmission phase grating. The quasi-one-dimensional grating with a spatial repetition period of \(\Lambda = 1650 \text{ nm}\) was made of polycrystalline chemical-vapour-deposited diamond. More details on the grating fabrication can be found in Refs. 18 and 25.

The grating was placed at the distance of 150 mm upstream of the sample (Fig. 1a). The sample-to-grating distance and the real-space periodicity of the phase grating \(\Lambda\) determine the...
periodicity of the XTG pattern at the sample position, which can either be smaller or larger than \( \Lambda \) depending on the convergence or divergence of the incident photon beam. In the present case, the period of the phase grating and its distance to the sample were chosen so that the real-space XTG period is of the order of the width of magnetic domains in Tm:YIG, of a few \( \mu \)m. The dark field image recorded with an optical microscope presented in Fig. 1 shows the contrast generated by the permanent XTG imprint due to damage of the Tm:YIG film. To simplify the navigation on the sample surface in the experiments, Pt markers have been deposited on Tm:YIG by means of focused ion beam (FIB) as shown in Fig. 1.

The magnetic domain structure of the film was measured by photoemission electron microscopy at the SIM beamline of the Swiss Light Source (PSI, Switzerland) by exploiting the x-ray magnetic circular dichroism effect (XMCD-PEEM). In this technique, x-ray light (here tuned to the Fe \( L_3 \) absorption edge at 710.6 eV) uniformly illuminates the sample and the intensity of photoemitted secondary electrons is imaged to obtain local maps of the x-ray absorption of the sample with a spatial resolution down to 50 nm. By averaging PEEM images measured with opposite circular polarizations \( C^+ \) and \( C^- \) one obtains a map of the local electron photoemission, which is very sensitive to the local surface potential and to the sample morphology. The sum \( (C^+ + C^-) \) image is shown in Fig. 2a. The stripes visible in the surface topography correspond well to the optical microscopy result shown in Fig. 1a, and is due to modification of the material structure induced by the XTG exposure.

Magnetic XMCD images \((C^+ - C^-)/(C^+ + C^-)\) are shown in Figs. 2b-d for regions exposed to the XTGs at different fluences. Figure 2b shows the XMCD contrast for the same area as Fig. 2a. Domain patterns typical for YIG-based systems with out-of-plane anisotropy are present in the pristine regions of the sample. We note that the pristine regions exhibit an asymmetry between the widths of the out-of-plane domains pointing parallel and antiparallel to the film normal (areas of the bright and dark contrast). We observe a width \( \sim 20 \mu \)m of the bright domains, as compared to a width of \( \sim 3 \mu \)m the dark domains (Figs. 2b-d). We attribute this to the presence of a small magnetic bias field present, or, alternatively, non-zero remanent magnetization of the film. The magnetic domain patterns are clearly different in the irradiated areas. For the maximal XTG fluence (Fig. 2b), the orientation of the magnetic domain stripes is visibly modified and aligned with the regions exposed to the XTG, creating parallel band domains in the irradiated region. In the exposed regions where the XTG fluence is lower, more random domain patterns are observed (Figures 2c,d). In the XTG-imprinted area, the amplitude of the magnetic contrast remains comparable or even stronger to the unexposed one, as seen from the line profiles given in Fig. 2c. This indicates that the magnitude of the magnetic moment was largely unaffected by the imprint.

In the case of the permanently imprinted gratings with fluences 70 mJ/cm\(^2\) and 17.5 mJ/cm\(^2\) (Figures 2c,d) the periodicity and the size of magnetic domains differ from those in the pristine area. This is clearly seen in the extracted line profiles of the XMCD-PEEM intensities (Fig. 2e) in the highlighted regions of interest for 70 mJ/cm\(^2\). We find that the average distance between magnetic domains is of \( 4.8 \pm 0.3 \) \( \mu \)m in the exposed region and of \( l = 6.3 \pm 0.3 \) \( \mu \)m in a nearby pristine region, as extracted from the red and blue regions in 2e, respectively.

In the XTG irradiated regions, the presence of closely spaced domains is likely due to two processes: 1) local demagnetization, and 2) pinning within the damaged areas. The former explains the varying degrees of modification of the domain pattern as a function of fluence. The latter is supported by the fact that domain pinning takes place exactly at the permanently imprinted grating as seen in Figures 2f,g, which respectively show the magnetic contrast and surface topography in the area exposed to 70 mJ/cm\(^2\) XTG. In the case of the intermediate fluence of 17.5 mJ/cm\(^2\), lower pinning contributes to the the more randomly altered magnetic domain pattern, as compared to that at 70 mJ/cm\(^2\).

To better understand the qualitative changes of the magnetic domain pattern induced by the XTG, the effect of the spatially periodic demagnetization on Tm:YIG system was investigated...
FIG. 2. (a) Topography of the sample area exposed with the XTG with the maximum beam fluence of 70 mJ/cm$^2$ and the corresponding XMCD-PEEM image of the magnetic contrast (b). (c,d) XMCD contrast in the areas of the sample exposed to XTG with the lower fluences of 17.5 mJ/cm$^2$ and 3.5 mJ/cm$^2$, respectively. (e) Line profiles of the XMCD intensity taken along the pristine (blue) and XTG-exposed (red) areas of the film, highlighted in the panel (d) by the corresponding colour. The blue curve is shifted by +0.15 for clarity. (f,g) Magnified images of the XMCD and topography contrasts shown in panels (b) and (a), respectively. White scale bars correspond to 20 $\mu$m.

by micromagnetic simulations. The simulated geometry consisted in a thin film with dimensions of 9000 $\times$ 9000 $\times$ 24 nm$^3$ with a cell size of $6 \times 6 \times 6$ nm$^3$. The exchange stiffness $A_{ex} = 2.3$ pJ/m, first-order uniaxial out-of-plane anisotropy constant $K_{u1} = 18$ kJ/m$^3$, and saturation magnetization $M_s = 140$ kA/m typical for Tm:YIG were used as material parameters. The Gilbert damping constant was taken to be $\alpha = 1$ to accelerate convergence of the simulations. The present simulations did not take into account material damage due to the x-ray irradiation nor ultrafast processes taking place at the sub-ns time scale.

An initial magnetic configuration resulting from the competition between the magnetostatic, exchange and anisotropy energies, and displaying a maze domain was used (Fig. 3a). Note that this configuration exhibits equal regions with dark and bright contrasts. The effects of the XTG were simulated by assuming the total quenching of the magnetization due to the transient grating, i.e., by locally setting $A_{ex}$, $M_s$ and $K_{u1}$ to zero within rectangular regions (Fig. 3b). The width of the modified regions was 400 nm. This quenching of the magnetization leads to a redistribution of the magnetization along the boundaries of the exposed regions. The magnetization is subsequently restored with the same material parameters, giving rise to a modified pattern (Fig. 3c) made of band domains that closely match the position of the transient gratings, reflecting the experiments.

To explain the observed change in domain periodicity in the irradiated regions, we suggest that magnetization pinning occurs in regions where the material’s magnetic parameters have been strongly altered. Although the surrounding magnetization may have recovered, the persistent pinning may be due to physical changes of the sample.

Reduction of the irradiation dose was mimicked by narrowing the width of demagnetized regions to 100 nm in the simulation. The result is a more disordered imprinted pattern (Fig. 3d), which does not lead to the formation of parallel band domains, in qualitative agreement with the patterns observed in the sample areas exposed with XTGs with the beam fluences of 17.5 mJ/cm$^2$ and 3.5 mJ/cm$^2$ (Figures 2c,d). Moreover, the simulations show that in the absence of physical pinning, the imprint does not affect the width of magnetic domains which is determined by the competition between the exchange, magnetostatic and anisotropy energies. Instead, the grating geometry affects the orientation of the domains in the irradiated area. It is remarkable that despite the simplicity of the model, the simulations reproduce the main effects of the XTG. The fact that the simulations display features that are not experimentally observed (bubble states in Fig. 3c) or do not reproduce certain experimentally-observed features (such as domains that are perfectly parallel to the XTG) points to the role played by physical defects and changes in material parameters.

Indeed, aside from physical changes in the sample, the domain wall pinning could be induced by the local modification of uniaxial anisotropy in the exposed regions. Reduction of the anisotropy constant by $\sim 35\%$ ($K_{u1} = 12$ kJ/m$^3$)
FIG. 3. Micromagnetic simulations of the XTG effect on the magnetic domain pattern. Starting with a pristine domain pattern (a), the effects of the XTG are simulated by defining regions of the sample (green) where the magnetization is transiently suppressed (b). A modified equilibrium magnetization pattern emerges after transient exposure with (c) 400 nm-wide and (d) 100 nm-wide gratings. The blue scale bar corresponds to 1 μm.

results in a spin-reorientation transition leading to in-plane magnetization within the affected areas and pinned domain walls at the interfaces with the out-of-plane domains. It is also known that while maze patterns form in the presence of uniaxial out-of-plane anisotropy, the formation of parallel band domains requires the presence of additional, secondary anisotropy contributions superimposed to the fundamental out-of-plane anisotropy. Moreover, the stronger contrast in the exposed areas seen in Fig. 2 hints towards the in-plane magnetization component.

While intense optical pulses and ion irradiation can result in structural and associated magnetic changes in iron garnet, the effect of hard x-rays is less explored. X-ray induced damage has so far also been observed in other magnetic oxides. In the case of Tm:YIG, Fe$^{3+}$ and Fe$^{2+}$ as well as oxygen vacancies may be present in the film. Their redistribution across inequivalent octahedral sites by the x-ray irradiation may modify the magnetocrystalline anisotropy and create a pinning potential in the irradiated areas.

In conclusion, we have investigated magnetic domain structures imprinted in a Tm:YIG PMA film by an XFEL hard x-ray transient grating. By measuring the resulting magnetic patterns with XMCD-PEEM, we observed modifications of the domain patterns in the exposed regions that correlate with permanent changes in the sample. Particularly, the observed decrease of the magnetic domain spacing and their orientation suggests a pinning of the domain walls to the defects imprinted by the x-rays. XFELs allow the generation of gratings with periods down to a few nanometers and durations of tens of femtoseconds, offering a possible route for the ultrafast manipulation of magnetic structures by x-rays down to the nanometer scale in suitable materials. Although the currently studied material only supports micrometer-scale magnetic domains, further investigations could lead to promising pathways to imprint magnetic textures, such as bubble domains or topological magnetic skyrmions of a smaller size. Taking advantage of the periodic spatial patterns induced by radiation as used to produce the XTG, these textures could also be arranged into artificial arrays using x-ray gratings with a custom shape, imprinting for example, one-dimensional chains, or two-dimensional hexagonal or square lattices. The XTG approach is more technologically promising than the generation of magnetic skyrmions by using a focused x-ray beam. Furthermore, our study extends the XTG approach towards the hard x-ray range, allowing one to manipulate and probe bulk specimens and reach resonant edges of a broad range of elements.

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AUTHOR DECLARATIONS

Conflict of Interest

The authors have no conflicts to disclose.

Author Contributions

V.U. and M.B. analyzed the data and wrote the paper, M.B., C.A.F.V., B.R., D.F., G.S., F.D., F.K., R.M., H.T.L., C.S., U.S. performed XFEL and PEEM experiments, E.R and C.A.R. synthesized the sample, C.A.F.V., M.B. and E.A.M. prepared the sample for PEEM and characterized it with scanning electron microscopy, C.D. prepared gratings, S.G. performed micromagnetic simulations, M.B., C.D., C.S. and U.S. jointly conceived the project.
DATA AVAILABILITY STATEMENT

The data is available from the PSI repository.

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