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Magnetic memory in discrete media observed by coherent soft x-ray resonant scattering

G Beutier^{1,5,9}, A Marty¹, F Livet², A Haznar³, E Dudzik^{3,6}, S Stanescu^{4,7}, V Chamard^{2,8} and G van der Laan^{3,5}

¹ CEA, INAC, SP2M, 17 rue des martyrs, F-38054 Grenoble, France
² SIMaP, 1130 rue de la Piscine, BP 75, 38402 St Martin d'Hères, France
³ Daresbury Laboratory, Warrington WA4 4AD, UK
⁴ ESRF, 6 rue Jules Horowitz, 38000 Grenoble, France
⁵ now at Diamond Light Source, Harwell Science and Innovation Campus, Chilton OX11 0DE, UK
⁶ now at Helmholtz-Zentrum Berlin, Glienicker Strasse 100, Berlin D-14109, Germany
⁷ now at SOLEIL, L'Orme des Merisiers Saint-Aubin, BP 48, 91192 Gif-sur-Yvette CEDEX, France
⁸ now at IM2NP, Aix-Marseille University, FST Saint Jérôme, 13397 Marseille Cedex 20, France
E-mail: guillaume.beutier@simap.grenoble-inp.fr

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Abstract. Using soft x-ray resonant magnetic scattering with a coherent beam, we find evidence for memory effects in the magnetic configuration of a periodic array of submicron ferromagnetic lines under external magnetic field. The memory effect is explained by the dipolar coupling between the lines which is lost when the sample is saturated by the external magnetic field.

⁹ Author to whom any correspondence should be addressed.

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1. Introduction

In the race for terabit per square inch data storage densities, all conventional magnetic media will sooner or later encounter the stumbling block of the superparamagnetic limit, which makes the data unstable at room temperature and restricts further scale reduction of magnetic materials. Discrete media with perpendicular magnetization can offer a promising way to overcome this limit [1]. The stability of the magnetic configuration for such materials is not yet well characterized, because the size reduction of the bits pushes traditional characterization techniques to their limits of spatial resolution and sensitivity. Moreover, the magnetic stability of nanoscale devices requires a characterization by a non-invasive technique, capable of probing the fine details of the magnetic configuration of the device at the nanometric length scale.

With the advent of third-generation synchrotron radiation sources, coherent x-ray scattering has taken off as a useful tool to characterize the detailed structural evolution of materials under external constraints such as temperature as well as equilibrium fluctuations [2, 3]. This method has also been adapted to the study of magnetic materials in order to probe antiferromagnetic domain fluctuations [4] or ferromagnetic domain evolution under applied magnetic field [5]. Conventional x-ray scattering measures the square modulus of the Fourier transform of the atomic scattering amplitude, but incoherent beams average the intensity over a large number of coherence volumes, which means that only the average properties of the material are measured. Instead, a coherent beam is scattered by a single coherence volume, yielding a grainy scattering pattern, referred to as a speckle pattern [6]. Such a pattern contains detailed information about the nanoscopic configuration of the coherently illuminated area. In ferromagnetic materials, x-ray resonant magnetic scattering using a coherent beam yields a speckle pattern that is directly related to the magnetic domain configuration [7]. Since a speckle pattern corresponds to a unique two-dimensional domain structure, monitoring the changes in the speckle pattern provides a convenient way to study any change in the domain structure under applied field and hence to characterize the magnetic memory of materials [8].

In Co/Pt multilayers, the perpendicular magnetic anisotropy leads to the formation at remanence (i.e. zero field) of a maze of ferromagnetic domains with alternating up and down perpendicular magnetization. The magnetic memory of such a domain configuration increases with the amount of structural disorder because magnetic domains are pinned to structural defaults [9]. In patterned media, such as arrays of lines or dots, the multidomain structure cannot occur if the size of the bit (a single line or dot) is small compared to the equilibrium domain size, so that each element carries a uniform perpendicular magnetization that switches



Figure 1. Left and middle: AFM/MFM measurement of a $10 \times 10 \,\mu\text{m}^2$ area of the Co/Pt sample. The left image shows the topological data (the lines appear in yellow and the gaps in black) and the middle image shows the magnetic data of the same area (red and green stripes represent opposite directions of the perpendicular magnetization). The gap between the lines is not visible on the magnetic image. The magnetic state was prepared by rotating the sample about the line direction in a gradually decreasing magnetic field, starting from a saturating field. Right: magnetization loops of the sample measured by magneto-optical Kerr effect in polar geometry (the spot size was smaller than the $2 \times 2 \,\text{mm}^2$ patterned area). The coercivity of the Co/Pt multilayer is much higher on the patterned media (red curves) than on the continuous wafer (blue curve). The minor loops measured on the patterned sample demonstrate the remanence of the net magnetization after any applied field, from which we infer the remanence of the magnetic state.

as a single domain. The entire array forms then an artificial magnetic structure with properties that are determined by its geometrical parameters. The magnetic memory of such a system has not been characterized so far.

Here, we report the magnetic properties of a grating of nanometric lines etched into silicon and covered with a Co/Pt multilayer having perpendicular magnetization. It has been shown for this type of system with well chosen geometric parameters that the lines possess a distribution of switching fields [10] and that the magnetization reversal occurs in collective modes [11]. Using soft x-ray resonant magnetic scattering (SXRMS) with a fully coherent beam at the Co L_3 absorption edge, we evidence a cooperative memory that is erased by saturating the entire array structure.

2. Description of the sample and experimental set-up

The sample was prepared at the Commissariat à l'Énergie Atomique (CEA) in Grenoble. A $2 \times 2 \text{ mm}^2$ grating of nanolines was etched into a silicon wafer by electron beam lithography and reactive ion etching. The lines are 300 nm high, 100 nm wide and have a gap of 75 nm between them. The periodicity of the array across the lines is thus 175 nm. In the other direction, along the 2 mm length of the etched area, the lines are continuous. A Pt/(Co_{6A}/Pt_{25A})₁₂ multilayer was sputter deposited in normal incidence on the patterned sample. Further details of the sample preparation can be found in [12]. Such a multilayer, when deposited on a continuous film, has a magnetic remanence of 100% (see figure 1). This ensures that each line can host a single

stable domain with magnetization either up or down. Here, such a magnetic state has been obtained by rotating the sample about the line direction in a decreasing magnetic field, starting from a saturating field. Magnetic force microscopy (MFM) confirms that almost all lines are monodomain, except for a few isolated defect lines (see figure 1). In this demagnetized state, the nanolines display a prevailing antiferromagnetic order due to the dipolar coupling, with only a few defects in the antiferromagnetic alternation. Further MFM measurements showed that the lines remain mainly monodomain after application of a perpendicular magnetic field.

Coherent SXRMS measurements were performed on beamline ID08 at the European Synchrotron Radiation Facility (ESRF), in a vacuum chamber dedicated for soft x-ray diffraction. The scattering was measured in reflection geometry with the magnetic nanolines parallel to the scattering plane. The diffraction patterns were recorded on a back-illuminated CCD camera which was mounted onto a fixed flange of the vacuum chamber, at 30° from the incident beam. The beam delivered by the undulator was circularly polarized. Magnetic contrast in the scattering was obtained by tuning the x-ray energy to the Co L_3 edge, at ~778 eV photon energy [13]. The corresponding wavelength, $\lambda \approx 1.6$ nm, is ideally suited for diffraction from nanostructures, such as the grating sample. The structural periodicity of the Co/Pt multilayer was chosen to match the incidence angle, imposed by the experimental set-up, with the Bragg angle of the multilayer at 778 keV. The incident x-ray beam was filtered by a 10 μ m diameter pinhole, in order to illuminate the sample with an almost fully coherent beam. The footprint of the beam on the sample, 7 mm behind the 10 μ m pinhole, is ~10 × 40 μ m², covering about 58 lines. However, near-field calculations of the wavefront suggest that the amplitude is spread over more lines, roughly 100. The oversampling ratio in the detection for this experiment was evaluated to be about 8, i.e. the smaller relevant details of the scattering pattern were eight times larger than the pixel size of the detector (13.5 μ m), allowing for sufficient redundancy in the measured data.

A perpendicular magnetic field at the sample could be applied using an electromagnet mounted inside the vacuum chamber. Its position was manually adjustable and the distance between the pole and the sample was known with sufficient accuracy to evaluate the strength of the magnetic field with $\sim 25\%$ accuracy (the magnetic field was characterized off-line as a function of the electric current and pole distance). This was acceptable for the purpose of this experiment. The field could not be applied continuously without overheating the coil and was therefore delivered in 1 s pulses. The diffraction patterns were measured after the pulses. The full magnetic remanence of the sample, demonstrated in figure 1, is therefore a key requirement of the experiment. The experimental setup is described in more detail in [14].

3. Results and discussion

In x-ray scattering, a perfect structural periodicity of the nanograting results in a Dirac comb of the so-called superlattice peaks, and a perfect antiferromagnetic order results in sharp half-order peaks between the superlattice peaks. With an incoherent beam, defects in the antiferromagnetic order can contribute to the diffuse scattering of the magnetic peaks, which is difficult to separate from the background and contains only information about the average correlation lengths such as the structural and magnetic roughness. However, in coherent x-ray diffraction, these defects will split the antiferromagnetic peaks into several speckles that are directly related to the local configuration.



Figure 2. SXRMS image from the Co/Pt magnetic nanograting in a demagnetized state. The logarithmic colour bar indicates the intensity scale in photons. The image (2048×298 pixels) was obtained by summing 1000 frames of 0.8 s. The specular peak is the second peak from the left. The red box between the specular reflection and the left first-order superlattice peak is the region of interest, shown on an expanded scale in figure 3.

Figure 2 displays a typical coherent diffraction pattern. The Dirac comb of superlattice peaks are located along an elliptical line on the image plane due to the curvature of the Ewald sphere. Each diffraction spot is surrounded by a series of periodic rings caused by the scattering from the pinhole. The superlattice peaks, except for the zeroth-order (specular reflectivity), are split into speckles because of structural irregularities in the nanograting. The speckle structure changes drastically when the sample is translated by a few microns, because structural defects move in and out of the illuminated area, but we found they are perfectly stable when applying the magnetic field pulses. This demonstrates the stability of our experimental setup with respect to the applied magnetic field. In the saturated state, the magnetic order is the same as the structural order (barring defects), and there are no additional features on the diffraction pattern (except for some low level background speckles due to the surface roughness). However, in any non-saturated state, the sample scatters speckles around the superlattice peaks, as seen in figure 2. These speckles are much more intense than those caused by the sample roughness and we can therefore assume that the speckle pattern between the Bragg peaks is of magnetic origin. Moreover, this speckle pattern disappears when moving the energy off the Co L_3 resonance, as is characteristic for magnetic diffraction peaks. They appear along the curvature containing the superlattice peaks, which confirms that the lines are mostly monodomain, as found by MFM, because the illumination is fully coherent. A few domain walls in some of the lines would be sufficient to scatter magnetic speckles away from the main curvature line. The speckles between each of the superlattice peaks show essentially the same pattern because they correspond to the same magnetic domain structure of the sample. Note that all speckles, including those related to the structural roughness, have the same size and shape, as determined in our case by the pinhole. It is the smallest relevant feature of the pattern, since it corresponds to the maximal correlation length probed by the beam, i.e. the spot size of the illuminated area. Only the distribution of the speckles is a property of the sample, and in particular the distribution of the magnetic speckles depends on the magnetic configuration of the sample. The series of speckles observed here between the structural peaks are therefore completely different from a harmonic spectrum of magnetic peaks obtained from a periodic magnetic order measured with a partially coherent beam. The latter peaks are related to the main magnetic period and their width is given by the dispersion of the magnetic period [15].



Figure 3. Top: the region of interest (red box of figure 2) $(160 \times 32 \text{ pixels})$ showing a typical speckle pattern for a demagnetized state acquired with 1000 frames of 0.8 s. Linear colour scale of intensities. Bottom: horizontal slice through the speckle pattern.

Typical images are obtained by summing over a few hundred frames of ~ 1 s exposure time. In the following, an 'image' will refer to a sum over several frames (100 in most cases). The exact exposure time per frame was varied to remain as close as possible to—but below—the saturation of the specular reflection on the CCD. The intensity of the magnetic speckles was about 1 photon s⁻¹, thus only a few hundred photons for an image of 100 frames of 0.8 s. Such a low signal could be extracted from the background by treating the frames using the droplet algorithm, which enables us to use the CCD as a photon counter [16, 17].

The resemblance between two different magnetic configurations of the ~ 100 illuminated lines is directly related to the resemblance between their corresponding speckle patterns. In the following, we will focus on a region of interest of 160×32 pixels, covering the area between the specular reflection and the left first-order superlattice peak (cf figure 2). This region is shown on an expanded scale in figure 3 for a demagnetized state. The pattern is roughly symmetric with respect to the half-order position (the middle of the pattern) because the magnetic state can be described by a real function (with values of +1/-1 for up/down magnetized lines). The deviation from the perfect symmetry arises mainly due to the non-planar wave illumination of the sample.

3.1. Quantitative analysis of the images

The resemblance between two different speckle patterns, $I_1(i, j)$ and $I_2(i, j)$, can be quantified using a correlation coefficient defined as

$$\rho(I_1, I_2) \equiv \frac{\max_{\Delta i, \Delta j} \left[\sum_{i,j} I_1(i, j) I_2(i + \Delta i, j + \Delta j) \right]}{\sqrt{\sum_{i,j} [I_1(i, j)]^2 \sum_{i,j} [I_2(i, j)]^2}},$$
(1)

where the sums are taken over the pixel indices i, j in the region of interest, as e.g. displayed in figure 3. Periodic boundaries were applied, such that $\sum_{i,j} [I_2(i + \Delta i, j + \Delta j)]^2 = \sum_{i,j} [I_2(i, j)]^2$, which holds well if the background intensity is low.

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The definition in (1) enables a drift correction for the pattern on the CCD. The operator 'max' indicates the maximum value of the expression in the brackets obtained by varying the integers Δi and Δj over a small range. If the experiment is completely stable, $\Delta i = \Delta j = 0$, however, we allow for a small drift of the pattern that never exceeded more than 2–3 pixels in each direction, which is smaller than the speckle size ($\sim 7-8$ pixels). Using a simpler expression for the correlation, with $\Delta i = \Delta j = 0$, results in an artificial loss of correlation over time due to drift, which is, however, not very large. Note that this effect differs from a drift of the beam on the sample, which instead modifies the speckle pattern.

 $\rho(I_1, I_2) = 1$, if and only if $I_1 = I_2$ for each pixel. Poisson noise, in reality, prevents a complete resemblance between different images of the same configuration, and the correlation coefficient is limited by $\rho(I_1, I_2) \leq \rho_{\text{poisson}}(I_1, I_2)$. If $I_1(i, j)$ and $I_2(i, j)$ are two different images measured from the same magnetic configuration, the correlation coefficient is given as

$$\rho_{\text{poisson}}(I_1, I_2) \equiv \frac{\langle \sum_{i,j} I_1(i, j) I_2(i, j) \rangle}{\langle \sum_{i,j} [I_1(i, j)]^2 \rangle} = 1 - \frac{\sum_{i,j} \langle I(i, j) \rangle}{\sum_{i,j} \langle I(i, j) \rangle^2},\tag{2}$$

where the angle brackets mean an ensemble average over a large number of measurements. The correction term $\sum \langle I \rangle / \sum \langle I^2 \rangle$ in (2) depends on the number of counts. For images of 1000 frames, such as shown in figure 3, $\rho_{\text{poisson}} \approx 0.995$. For typical images of 100 frames of 1 s, with about 100 photons in the pixel of maximum intensity, $\rho_{\text{poisson}} \approx 0.97$. For instance, we found that the mutual correlations of 9 images of 100 frames from the same demagnetized state, taken sequentially without applying any magnetic field, have coefficients between 0.93 and 0.98. We can conclude that the effect of the Poisson noise on our correlation coefficient is small compared to the induced changes in the speckle pattern. This test measurement also demonstrates the stability of both the sample and the experimental setup.

Incoherent scattering gives a lower boundary of the correlation $\rho(I_1, I_2)$ between two independent images. Although the beam is almost fully coherent, some partial incoherence scatters a broad diffuse intensity. It is small compared to the contrasted speckles, as seen in figure 3, but it is responsible for some 'artificial' correlation in the calculation of $\rho(I_1, I_2)$. The reason is that the broad diffuse intensity covers the entire area over which the calculation of the correlation is performed, whereas the magnetic speckles occupy only part of it. Therefore, the correlation coefficient ρ barely drops below 0.4 for very different speckle patterns. All in all, our correlation coefficient ρ varies in practice from ~0.4 for very different speckle patterns to ~0.97 for very similar speckle patterns.

3.2. Evolution of the speckle pattern as a function of field

The evolution of the speckle pattern can be nicely followed as a function of applied magnetic field. Figure 4 shows the evolution of the intensities of the specular, superlattice and magnetic peaks along a magnetization loop. The circular polarization of the incident beam allows for an interference between the charge scattering and magnetic scattering [13]. This is the reason that the superlattice peaks vary linearly with the magnetization and they reproduce typical magnetization curves of the sample [10]. The so-called magnetic peaks appear around half way between superstructure peaks and require an antiferromagnetic ordering. Their intensity reaches a maximum value when the net magnetization vanishes, i.e. at the coercive field. At the saturated state, the magnetic speckles are completely absent. Close to the coercive field, the speckles



Figure 4. Evolution of the diffraction pattern along a magnetization loop. The perpendicular field was applied by pulses of 1 s and released between each point of the curve. Top left: integrated intensity of the superlattice peaks, scaled to the specular peak (or zeroth-order). The labels refer to the order of the superlattice peaks (from -1 to 4 from left to right in figure 2). Top right: integrated intensity of the speckles around the half-order positions (with same labeling convention). Bottom: several states of the $-\frac{1}{2}$ order speckle pattern. A movie of the entire loop is available as supplementary material; see stacks.iop.org/NJP/11/113026/ mmedia.

start to appear along the entire line. This distribution shows that, at the initial stage of the reversal, no particular order exists. The first lines that reverse are on average too far away from each other to be coupled by dipolar field, and an isolated reversed line is not able to influence its neighbours to create long-range order. When the number of reversed lines is increasing, they tend to induce an antiferromagnetic order, i.e. they prevent the neighbouring lines from reversing to the same direction. The speckles then merge and the middle ones, representing correlations close to a perfect antiferromagnetic order, become stronger while those between

the half-order and the superlattice peak tend to disappear. Once half of all lines are reversed, the speckles split again, showing that the last lines to return are not selected by the magnetic coupling, since they are too far apart to interact. The last remaining speckles disappear when the sample is saturated. Note that during the entire reversal process, the magnetic scattering remains concentrated in speckles along the main diffraction line. This means that the reversal of the lines does not proceed by fragmentation of the magnetic domain or creation of multiple domain walls inside the lines. If numerous domain walls were formed along the length of the lines within the footprint of the beam (40 μ m longitudinal), many weak speckles would appear away from the main diffraction line, exactly as those we observe all along the main diffraction line in the early stages of the reversal process, when no particular order prevails. This suggests indeed that each line is reversed as a single block or in large blocks with a length of the order of several tens of microns.

In order to investigate whether the lines are always reversed in the same sequence, we have repeated this process several times and compared the speckle patterns obtained at the same field of successive loops. Here, we focus on the demagnetized state, obtained at the coercive field, which we identify as the maximum of the intensity between the superlattice peaks. In this state, the antiferromagnetic order is at its maximum but not perfect, such that only a few speckles remain close to the half-order position in between the superlattice peaks. Figure 5 shows different speckle patterns obtained for the same pulse of the coercive field. The sample was saturated in the direction opposite to the coercive field between two successive measurements. Some of the speckle patterns are very similar to each other, while others are very different. The histogram of mutual resemblance reflects this wide range of cases. We conclude that the flipping process does not always go through the same state of demagnetization, but that several states can be reached. The process is not completely random either, since in these few measurements some very similar images were recorded.

To ensure that the different configurations are not due to some inconsistency of the pulsed field, we collected a series of 10 images, each of them taken after a pulse of the same coercive field but without saturating them in between. The first three and the last image are shown in figure 6. The sample exhibits some evolution under repeated pulses of magnetic field, and converges toward a stable configuration. However, this evolution is small compared to the drastic variations in the speckle patterns observed after saturation of the sample (figure 5).

The different magnetic configurations shown in figure 5 were obtained from the saturated state, in which case the magnetic coupling has no influence. The first lines to flip are determined by the distribution of the individual flipping fields [10]. In order to investigate the importance of the magnetic coupling, we have recorded speckle patterns taken after successive pulses of various magnetic field intensity without ever saturating the sample. Starting from the negative saturated state, a pulse of positive coercive field was applied to demagnetize the sample. Then, field pulses of alternating sign and increasing intensity were applied. History of the magnetic field applied is too weak to change the magnetic state of the sample, resulting in a speckle pattern similar to the first one ($\rho(1, n) \approx 0.89$). Speckle patterns recorded after a strong enough field are different from the first ones, but the initial state is recovered by applying the positive coercive field ($\rho(n, n-2)$) in figure 7) remains above 0.85, almost as high as of images taken after application of the same coercive field (figure 6). The negative coercive field was applied to the last three images taken after a negative field. They also show high mutual correlations



Figure 5. (Top) Various speckle patterns observed at the coercive field after saturation of the sample. It is seen that the variation in the patterns is quite large, indicating that the magnetic memory is lost after saturation. (Bottom) Histogram of the correlation coefficient.

 $(\rho(n, n-2) \approx 0.84)$. The above demonstrates that at this stage the flipping of the lines is determined by the magnetic coupling between the lines instead of by the inhomogeneity of the individual flipping fields.

The last six images were taken after switching from one coercive field to the opposite, and the strong variations of the total intensity of the 'magnetic' speckles show that each pulse modifies the magnetic structure (figure 7, bottom left). Interestingly, the correlation between one image to the next remains fairly high ($\rho(n, n-1) \ge 0.8$) and continuously increases up to more than 0.85 (figure 7, bottom right). This suggests that the magnetic configuration converges toward two almost demagnetized states that require as few lines as possible to flip from one state to the other. We also note that the first relevant change of intensity occurs between patterns 11 and 12, when a pulse of -0.975 kOe is applied, but the correlation coefficient already starts decreasing between patterns 7 and 8, with a pulse of -0.875 kOe, and drops dramatically between patterns 9 and 10, with a pulse of -0.95 kOe. It shows that magnetic fields close enough to the coercive field can reverse a couple of lines, which triggers a reorganization of the



Figure 6. Evolution of the speckle pattern after 10 successive pulses of the coercive field. Only the first three and the last patterns are shown in the upper part. The sample was saturated in the opposite direction prior to the first pulse. The resemblance between the first and the last image is quantified by a correlation coefficient $\rho = 0.86$. Bottom left: the evolution of the resemblance between successive images. After several iterations, the resemblance reaches values comparable to those of images taken without applying any field in between. Bottom right: the histogram with the cross-correlation coefficients of the 10 images.

magnetic configuration via dipolar coupling, leaving the net magnetization unchanged. Such a field is indeed enough to reverse the magnetization of the same multilayer on a continuous medium (figure 1), and may thus be able to reverse individual lines where the mean field of dipole interaction is weaker. We suspect that the reorganization of the magnetic structure occurs in a cascade of events that only time-resolved measurements with a free-electron laser could reveal.

4. Conclusion

Using coherent SXRMS, we have investigated the detailed flipping processes of a magnetic nanograting under applied field and evidenced details that cannot be seen with global magnetization techniques. Starting from a saturated state, the grating was able to reach several different magnetic configurations, which implies that the distribution of the individual flipping fields of the lines is mixed with some random process that determines the choice of the first lines that flip. Starting from a non-saturated magnetized state and applying the opposite coercive field, the sample always retrieved a similar magnetic configuration, which shows that the magnetic coupling between the lines is the dominant factor in the flipping process when the sample is not saturated. This study suggests that the detailed order of the flipping, from one saturated state



Figure 7. Bottom right: correlation of images taken after non-saturating pulses of increasing magnetic field. The sequence of magnetic field pulses is shown in the bottom left plot, together with the evolution of the total intensity of the 'magnetic' speckles (in red). The general slope of the intensity curve is attributed to a slow drift of the beam with respect to the pinhole, that was frequently observed during the experiment. The images corresponding to the last ten fields are shown above.

to the other, is determined as soon as the first lines have been flipped, but that the sequential order of flipping can be modified by pulses of magnetic field close to the coercive field. An interesting prospect of coherent soft x-ray scattering is that this technique can be extended to time resolved measurements using pump–probe techniques. This would allow the study of the collective magnetization dynamics of discrete media on a picosecond timescale [18].

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