Ultrafast Single Shot X-Ray Imaging of Magnetic Phase Transitions

J. Lüning, W. Schlotter, J. Stöhr, H. C. Siegmann
Stanford Synchrotron Radiation Laboratory, Stanford, CA
S. Eisebitt
BESSY GmbH, Berlin, Germany

We propose an experiment that makes full use of the unique capabilities offered by LCLS to image the critical fluctuations in a ferromagnetic film in the vicinity of its Curie temperature. The high intensity, femtosecond time structure, and coherence of the LCLS x-ray pulses will enable us to record ultrafast consecutive images of critical fluctuations using a newly developed lensless imaging technique, which is based on Fourier transform holography. The initial LCLS pulse length of 230 fs is sufficient for our experiment.
The present letter of intent (LOI) falls under 'Category C'. It is based on the implementation of a soft x-ray beam line (~ 800 eV) with 10:1 demagnification optics on LCLS. The small inherent energy band width of the LCLS x-ray pulses renders a monochromator unnecessary for initial experiments but it will be beneficial for later experiments. Furthermore, a beam splitter with variable time delay in the femto- to nanosecond range is needed for resolving the phase transition dynamics. The funding for a suitable sample chamber is presently being sought as part of a Stanford Ultrafast Science Center proposal.

Contact information:

Jan Lüning
SSRL, Staff Scientist, luning@slac.stanford.edu, 650 926 4539

W. Schlotter
SSRL, PhD student, wschlott@slac.stanford.edu, 650 926 2218

J. Stöhr
SSRL, Professor, stohr@slac.stanford.edu, 650 926 2570

Hans C. Siegmann
SSRL, Visiting Professor, siegmann@slac.stanford.edu, 650 926 3842

Stanford Synchrotron Radiation Laboratory
2575 Sand Hill Road, Mail Stop 69
Menlo Park, CA 94025
fax: 650 926 4100

Stefan Eisebitt
BESSY Staff Scientist, eisebitt@bessy.de, ++49 30 6392 4884
BESSY GmbH,
Albert-Einstein-Str.15,
12489 Berlin, Germany
fax: +49 30 6392-2990

Introduction

One of the central topics in physics is the understanding of phase transitions in matter. These can occur as structural changes or as changes in the electronic or magnetic properties of a material. For the description of such phase transitions powerful symmetry and statistical concepts have been developed in theoretical physics. One might argue that such theoretical approaches are satisfactory. This, however, would underestimate the importance that simple pictures of physical processes have played in the development of physics. It is through simple pictures that we conceptualize difficult processes. And in many cases a full understanding of a phenomenon is not realized until it has been reduced to a simple picture. The verification of such a simple real space picture of a phase transitions is the goal of the present proposal.

We have chosen a particularly simple yet technologically important phase transition for our study: the ferromagnetic to paramagnetic phase transition that occurs at the Curie temperature T_c of the transition metals Fe, Co and Ni. The basic question is simple: What does the magnetic domain structure of a ferromagnet look like at the Currie temperature?

Mean field theory predicts that the long range magnetic order collapses at the Curie temperature leading to a random orientation of the atomic spins. However, it turns out that the mean field theory cannot describe what really happens in the vicinity of the critical temperature. The modern theory of continuous phase transitions has shown that the observations made in a number of indirect experiments can be explained by the occurrence of “critical fluctuations” which may extend over many thousands of atomic distances. For magnetic phase transitions this implies that the magnetic correlation length diverges for an ideal material as one approaches the transition temperature. Magnetic domains of very large size and very short life time should be forming and dissolving. This has, however, never been confirmed by a direct imaging experiment.

It is proposed here to directly observe such critical fluctuations of the magnetization in a new experiment that combines the latest developments in x-ray imaging with the uniquely matching capabilities of XFEL sources. We propose to image the elusive spin blocks and their dynamics. The first step is to prove the existence of spin blocks by recording a single shot picture on a timescale that is faster than the fluctuations. The second step is the study of spin block dynamics by recording a series of such pictures at well-defined time intervals.

Scientific Background

Most studies of magnetic critical phenomena have been done on 3-dimensional (3D) objects. The fluctuations manifest themselves in a tail of the magnetization-dependent properties such as electrical resistivity and specific heat to temperatures above T_c, and in a cusp of the magnetic susceptibility at T_c. Theoretical work has shown that these anomalies might be explained by the existence of spin blocks, consisting of spontaneously magnetized regions, that form and dissolve randomly as the temperature approaches T_c, as illustrated in Fig. 1.

In 3D, the diameter D and the lifetime τ of the spin blocks is believed to lie in the range $10 < D < 100$ nm and $10 < \tau < 100$ ps, respectively, as the temperature closely approaches T_c to between $1\% > (T_c - T) / T_c > 0.001\%$. The spin blocks may be envisioned as randomly oriented macrospins with a very large magnetic moment that can be aligned in an external field against the action of thermal motion. In this way, the cusp of the magnetic susceptibility χ, the tail of the magnetization dependent properties to temperatures above T_c, as well as other phenomena such as the critical exponent with which the magnetization disappears at T_c can be explained in a natural way. Yet as mentioned earlier, nobody has yet been able to observe the spin blocks directly.

One of the major reasons for this lack of experimental confirmation is that the lifetime and diameter of the spin blocks predicted by the universal theory of phase transitions are such that it has not been possible to image them in any of the conventional ways. It is important to realize that critical fluctuations are very sensitive to magnetic fields. The theory can only be applied if the sample is shielded from external magnetic fields as small as the earth magnetic field. Magneto-optics has generated the possibility to measure the spontaneous magnetization without applying any magnetic field and at a time scale solely determined by the length of the photon pulse. Magneto-optics thus establishes the basis for the proposed experiment. X-rays have to be used rather than optical lasers because only X-rays can resolve the expected small magnetic structures.

Furthermore, the theoretical predictions depend very much on the exact nature of the magnetic interactions and on impurities and defects. Our approach is to study the fluctuations in 2-dimensional ultra-thin films. Such ferromagnetic films can be prepared nearly defect free by epitaxial growth with thicknesses of only a few monolayers. Also, in 2D systems such as ferromagnetic thin films consisting of few atomic layers only, the critical fluctuations are more prominent as compared to 3D-systems. Back *et al.* [Bac94] find an extremely sharp decrease of the spontaneous magnetization at T_c in a two layer thick Fe film on W(110). This is consistent with the magnetic correlation length reaching 10^4 atomic distances, which corresponds to several micrometers. Evidently, morphological defects such as monoatomic steps arising from the imperfect substrate at a separation of 50 – 200 nm and the steps due to patch formation during the growth of the Fe-overlayer are not able to break the correlation length since it greatly exceeds the spacing between defects. This is the key to observing true 2D phase transitions.

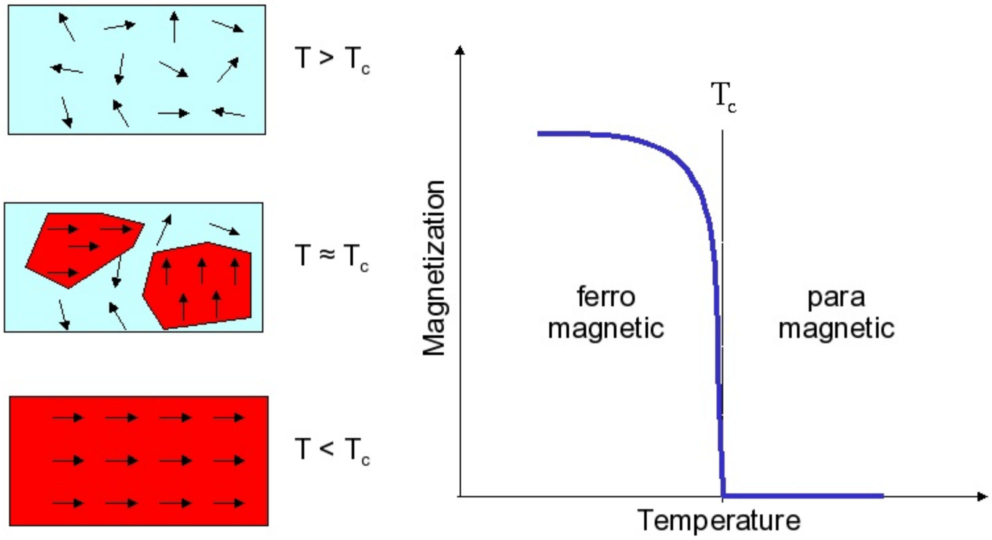

Figure 1: Temperature dependence of the magnetic domain structure in a ferromagnet: Below the Curie temperature T_c the magnetic interaction dominates over the thermal energy, and the material exhibits a static domain structure. Above T_c the material is paramagnetic, the magnetic moments are free to rotate. In the vicinity of the ferro- to paramagnetic phase transition at T_c the balance of magnetic interaction and thermal activation leads to magnetization fluctuations: Within the overall disorder, areas with ordered magnetic moments exist momentary. Theory predicts that the size of these ordered regions diverges at the phase transition.

How long does it take these 2D spin blocks to dissolve? Naturally, larger objects take more time to dissolve. Following Patashinsky and Prokrovsky [Pat79], the lifetime τ is proportional to the magnetic susceptibility χ. As the cusp of χ in 3D-systems is of the order of 100, but may be as large as 10^6 in 2D as found by Back *et al.* [Bac94] for both, Co and Fe films, the lifetime of the

2D spin blocks may be as long as 100 ns in close proximity to T_c. A systematic study of the behavior near T_c therefore requires a large dynamic range from femtoseconds to nanoseconds and a spatial resolution from near atomic dimensions to about a micrometer.

More generally, the direct detection of spin blocks and their lifetime would mark a major breakthrough and may lead to significant progress in the understanding of the mechanism leading to second order phase transitions. Due to the universal nature of these transitions, any progress may impact many phenomena such as order/disorder transitions and unmixing in binary alloys and thus have an impact reaching far beyond magnetism.

Summary of proposed LCLS experiments

We propose to investigate the critical fluctuations occurring at magnetic phase transitions. Our initial LCLS experiment will demonstrate the existence of magnetic spin blocks by taking snap shot pictures on a time scale faster than the fluctuations. The next step will be to study the spin block dynamics by recording a series of such pictures at well-defined delay times. For imaging of the magnetic spin blocks we will use a newly developed lensless imaging technique described in detail in the next section. These experiments, however, would not be possible without LCLS's ultra-bright, ultra-short, and fully coherent x-ray pulses. Key properties and implications of the proposed experiment are:

Single x-ray pulse imaging: A single LCLS pulse will be sufficient to obtain an image of the instantaneous magnetic domain structure which is expected to be static on the time scale set by the ultrashort x-ray pulse length (230 fs), even close to the phase transition.

50 nm spatial resolution or better: We have demonstrated 50 nm spatial resolution with our newly developed lensless x-ray imaging technique which will take full advantage of the unprecedented full coherence of LCLS x-ray pulses. As discussed in the next section subsequent phase retrieval may improve this to near-wavelength limited spatial resolution.

Dynamics of critical fluctuations: To image the fluctuation dynamics we will split the beam and produce consecutive x-ray pulses with a well defined time separation at the sample. From each pulse we will obtain an image of the magnetic domain structure, using a scheme discussed below, and thus resolve the dynamics occurring on a femto- to nanosecond time scale.

Ultrafast, non-deterministic dynamics: It is important to realize that the proposed experiments significantly differ from today's ultrafast pump-probe experiments. Such experiments rely on reversibility of the sample to a well defined state before each pump-probe cycle. In contrast, the critical fluctuations that are the subject of our study are non-deterministic and their study requires complete images to be recorded in a single shot.

Ultrafast relaxation dynamics: It is clear that once we have demonstrated the feasibility of ultrafast single shot imaging a whole class of new experiments will become possible. Besides single shot imaging of spontaneously occurring fluctuations we also envision to study ultrafast relaxation dynamics in pump-probe experiments.

In this LOI we propose the development of the x-ray optics infrastructure necessary to carry out these experiments at LCLS. This comprises the development of x-ray optics specifically designed to preserve coherence and time structure of the x-ray pulses for transport, focusing, splitting, and redirection of the x-ray beam. A soft x-ray monochromator is not needed initially, since the inherent energy resolution of LCLS results in sufficient temporal coherence for the first experiments. For future experiments a higher energy resolution for resonant scattering may be required. We therefore propose to also start the development of a time-structure and coherence preserving soft x-ray monochromator.

Within the scope of this LOI we only propose the development of the LCLS infrastructure summarized above and outlined in more detail in the last section of this document. This infrastructure can be shared by other experiments (e.g. AMO) that require coherent soft x-ray pulses. As stated earlier, we have requested funds for the development and construction of the experimental sample chamber in a separate proposal to DOE-BES.

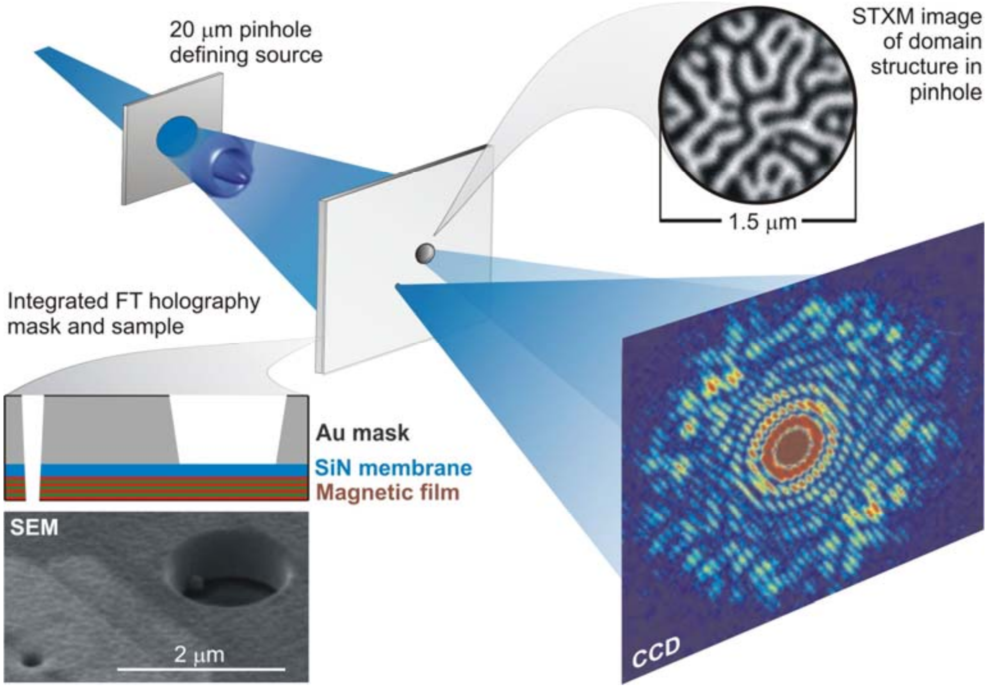

Figure 2: Illustration of our x-ray Fourier transform holography experiment. The x-ray beam from an undulator source with variable polarization is incident on a pinhole that redefines the source. The central part of the Fraunhofer pattern of the pinhole then illuminates a mask that consist of a “sample hole” and a “reference hole”. The SEM image on the lower left shows a close-up of the two holes which were drilled into a Au film by a focused ion beam. In our case the mask and sample were integrated, as illustrated above the SEM image. The magnetic domain structure within the pinhole opening, recorded by a scanning transmission x-ray microscope (STXM), is shown on the right top. The experimentally recorded hologram of the sample is reproduced in false color on the lower right.

Current status of the lensless imaging technique development

Over the last two years we have been developing a lensless magnetic imaging technique that is ideally suited for LCLS since it is based on scattering of spatially and temporally coherent x-rays. Such a coherent beam can be obtained today by selecting the coherent fraction of a monochromatic x-ray beam with a pinhole placed far from the x-ray source. The central Airy disk of the Fraunhofer pattern formed by the transmitted beam can then be used to coherently illuminate a sample. The interference pattern from the scattering of the sample, called the speckle pattern, is detected with a suitable area detector such as a CCD camera. To obtain a real space image of the sample from this speckle pattern the phase of the scattering amplitudes has to be reconstructed. This has been attempted by phase retrieval algorithms that rely on oversampling of the speckle pattern [Say52,Ger72,Fie82]. This approach has been successfully demonstrated for samples with simple test geometries by others [Mia99,He03] and by us [Eis04]. For real-world samples like a magnetic domain structure with complex scattering amplitudes, however, this approach has so far failed.

We therefore have developed a new approach that is based on the unique image reconstruction afforded by holographic methods [Gab71,Gab48]. Our x-ray version of the Fourier transform holography technique [Str65] is illustrated in Fig. 2 .The figure caption explains the key components of the experiment. In essence, two coherent beams are obtained from a gold mask with two holes with respective diameters of 1.5 μm and 100 nm. The beam from the larger hole is transmitted and diffracted by the sample and interferes on the detector with the reference beam transmitted through the small pinhole. The incident photon energy was tuned to the Co L_3 edge (785 eV) for optimum magnetic contrast. The real space image of the magnetic domain structure can be easily obtained by a single Fourier transformation of the recorded scattering intensities [Str65]. This yields the intensity auto-correlation of the sample shown in figure 3a. The large, saturated area in the center contains the sample/sample and reference/reference hole auto-correlations. Offset to opposite directions with respect to the image center are the respective sample/reference hole cross-correlations. These give the desired real space image of the magnetic domain structure within the sample pinhole area. To derive the spatial resolution of the holographic image we have plotted the indicated intensity line profile in figure 3c. The contrast and signal to noise ratio is remarkable, and the image is comparable to that recorded by means of state-of-the-art transmission microscopy (see Fig. 2).

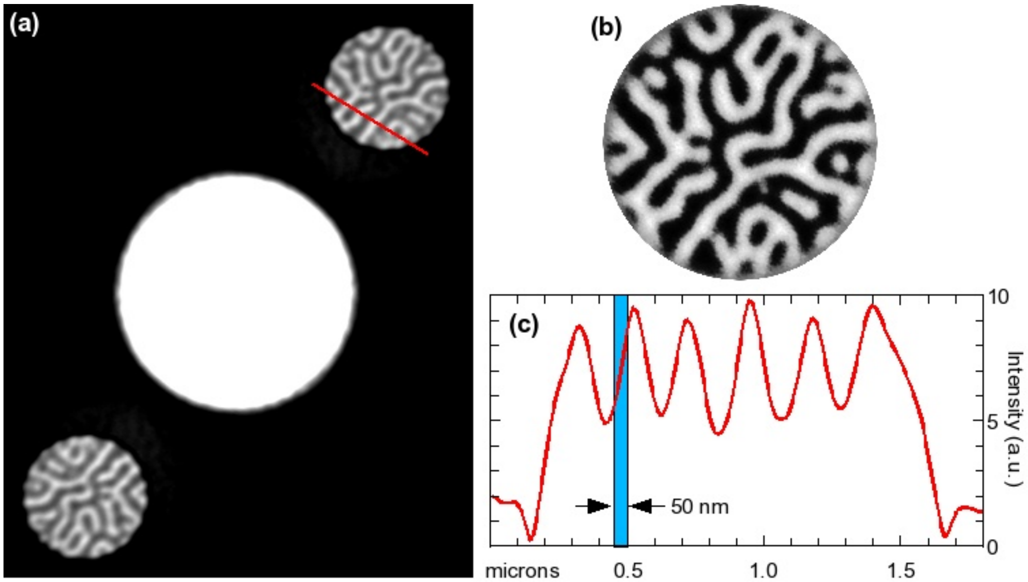

Figure 3: Real space image of the magnetic domain structure as retrieved from the hologram shown in figure 2. (a) A single discrete fast Fourier transformation of the Fourier transform hologram intensities yields the autocorrelation of the sample. (b) The upper one of the twin images is enlarged in the top right. (c) Intensity profile along the red line in (a).

One of the unique properties of this imaging approach is that no focusing or alignment is required. While this is convenient for imaging at a synchrotron radiation storage ring, it is essential for the envisioned single x-ray pulse imaging at LCLS. It is important to note that lensless imaging by Fourier Transform holography is a true imaging technique. No iterative algorithm is required to obtain the real space structure. From the experimental conditions used to obtain the hologram shown in figure 2, and thus the holographic image shown in figure 3, we can estimate that a single LCLS x-ray pulse will be more than adequate to record an equivalent x-ray hologram.

When thinking about the ultimate spatial resolution it is important to bear in mind that the recorded x-ray hologram can also be interpreted as a speckle pattern. Hence, when successfully applying an iterative algorithm to retrieve the scattering phases, the achievable spatial resolution is only limited by the maximum momentum transfer, i.e., ultimately by the wavelength, which is of order 2 nm at the transition metal L edges. Since iterative phase retrieval algorithms are the more effective the closer the initial input is to the real space structure [Fie78], it appears possible to employ a two step analysis. In the first step the Fourier transform provides a resolution that is of the order of the reference hole diameter. In the second analysis step, iterative phase retrieval algorithms are used to obtain higher resolution. The ultimate resolution in the second step is determined by the angular range and signal to noise of the measured scattering intensities. From an intensity point of view, a focused LCLS beam is capable of yielding near wavelength limited spatial resolution in a single shot.

Preparation of LCLS experiments

Our experimental program for the next four years is focused on preparing the single shot imaging experiments at LCLS. Key areas will be: improvement of our lensless imaging technique, implementation of a detection apparatus suitable for the LCLS flux, and development of a suitable sample mask. In addition we will develop the preparation of ultra-thin defect-free magnetic films and perform preliminary investigations into magnetic critical phenomena on SPEAR3.

We expect to improve the spatial resolution of our lensless imaging technique notably by manufacturing masks with smaller effective reference apertures [Ber03]. In addition we will explore mathematical procedures like wave propagation and phase retrieval algorithms to further increase the spatial resolution beyond the limit set by the diameter of the reference hole.

An intense area of research will be the development of a sample/mask structure that can withstand the intense x-ray pulses and minimize the energy deposited into the magnetic film. To minimize the energy deposited by absorption in the film we will explore tuning of the photon energy below the dichroic L_3 absorption edge. Since the absorption decreases faster than the dichroism of the scattering phases, this will make the film effectively a phase contrast sample [Kor01]. How much this can be exploited to reduce absorption remains to be investigated, since with decreasing dichroism of the scattering phases the scattering contrast decreases. This will be addressed through experiments on SPEAR3, where we will also investigate the feasibility of Fourier transform holography in a reflective geometry. In this geometry the amount of deposited energy can be reduced by orders of magnitude in comparison with a transmission geometry.

The single most limiting factor in lensless imaging experiments is the small dynamical range of today's signal integrating imaging detectors, like CCD cameras. The reason for this is that the scattering intensity decreases by orders of magnitude with increasing momentum transfer $\mathbf{q}$. Hence, a CCD camera needs to be read out repeatedly to record a complete scattering pattern. Since this is not an option for single shot imaging, the dynamic range of the detector needs to be matched to the experiment. To achieve this we currently foresee the following two options which may also be combined. First, the central beam can be attenuated by a specifically designed micro-structured detector mask. Second, the weak scattering at high $\mathbf{q}$ and the intense scattering at low $\mathbf{q}$ can be recorded with separate detectors. The low $\mathbf{q}$ detector could then be placed at a larger distance to disperse the intensity over more detector pixels. These schemes will be developed on SPEAR3. Note that even pixel detectors under development today - and anticipated to become available during the next years - will not provide the required dynamical range. Hence, we expect a wide variety of experiments to benefit from our detector development.

On the experimental side, we will use resonant small angle incoherent x-ray scattering (SAXS) at SPEAR3 [Gui55,Kor01] to obtain new information on critical magnetic phenomena. For these experiments we will develop the preparation of defect free epitaxially grown ferromagnetic transition metal films of only a few monolayers in thickness. These will be the same films needed for the LCLS experiments. From these experiments we expect to obtain statistical information about the average domain size, shape, and characteristic correlation length of magnetic fluctuations in the vicinity of the critical temperature. Recording such resonant SAXS pattern for a series of temperatures across the Curie temperature may enable us to obtain an indication of the presence of spin blocks and their size. However, even if fluctuations are observed, the obtained information is incomplete since it is only of statistical nature. A complete experiment consists of resolving individual spin block fluctuations to verify that they are true critical fluctuations occurring spontaneously at random locations and times, rather than fluctuating magnetic domains nucleated or pinned by defects or impurities. Such pinned or stimulated fluctuations may mask or falsify the dynamics of the true critical fluctuations.

Envisioned experiments at LCLS

The proof that the observed fluctuations are truly spontaneous and occur at random locations requires true time dependent images and therefore LCLS. Single shot measurements will also overcome experimental limitations in sample temperature stabilization, which is expected to be on the order of ±0.01° C. At LCLS we will let the temperature drift slowly through the phase transition temperature, while recording (at the LCLS pulse rate) snap shot images that reveal the momentary domain structure. From a series of such images the temperature dependence of the size of the magnetic fluctuations can then be extracted unambiguously.

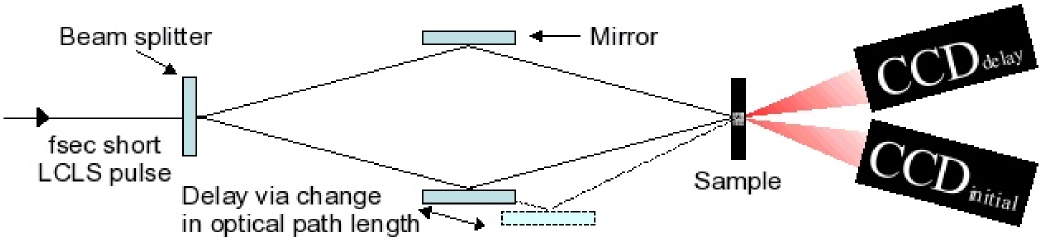

Figure 4: To image ultrafast dynamics a single femtosecond short x-ray pulse is split into two separate pulses. By varying the path length for one of the pulses the sample can be illuminated with a variable time delay between the two pulses. The scattering of these two pulses is spatially separated due to the difference in the incidence angle of the x-ray pulses.

LCLS will also enable us to study fluctuation dynamics on time scales down to the pulse width itself. Using a beam splitter with a variable path length for one of the two x-ray pulses as sketched in figure 4, it will be possible to obtain two x-ray pulses with a tunable time delay. In our case time delays in the picosecond/nanosecond range appear most important. The two consecutive pulses will traverse the sample under slightly different angles so that their speckle patterns will be separated in space and can be recorded independently either by two detectors or a single larger detector. By spatially restricting the sample to an area smaller than the footprint of the x-ray pulses, this experiment only requires that the pulses overlap on the sample, i.e., that the 'active' sample area of a few microns in diameter is illuminated by both pulses. Using both these pulses for lensless imaging of the magnetic domain structure will enable us to resolve ultrafast dynamics of the magnetization fluctuations. Note that due to the essentially two-dimensional domain structure the difference in x-ray incidence angle is insignificant.

One major challenge in this experiment will be to understand how much the energy deposited by the first x-ray pulse influences the magnetization dynamics. In fact, it may be that the interaction cannot be made negligible. This, however, would constitute interesting science by itself, since we would observe the ultrafast relaxation dynamics of a system pushed out of its equilibrium. This points to a unique strength of the two pulse imaging method, namely, its capability to study the non-repeatable, chaotic component of relaxation dynamics which is not accessible by traditional pump-probe experiments that rely on accumulating statistics by repeating the same process over and over again.

Required LCLS infrastructure

To realize the proposed experiment we will need a pink soft x-ray beam in the photon energy range around 800 eV. This energy range is needed, since it contains the $L_{3,2}$ edges of the magnetic transition metals Fe (710 eV), Co (780 eV) and Ni (850 eV). Together with the rare earth metal Gd ($M_{4,5}$ edge at 1190 eV) these ferromagnets are the materials of choice for the proposed experiment. The reason for this is that the transition metal $L_{3,2}$ edges (2p to 3d transitions) and rare earth $M_{4,5}$ edges (3d to 4f transitions) exhibit the strongest magnetic dichroism. Furthermore, it is possible to grow stable, defect free, ultra-thin transition metal films that can be characterized ex-situ in preparation of the LCLS experiments.

The beam line will need focusing optics that demagnify the source by about 10:1 (horizontally and vertically). This particular demagnification ratio follows from the photon beam source size of 93 µm and divergence of 8.1 µrad for 800 eV FEL radiation. Since the sample aperture will only be about 5 µm in diameter, the demagnification should be at least 10:1. Note that for larger samples a homogeneous illumination can always be achieved by placing the sample behind the focus. On the other hand, the demagnification should not exceed 10:1, since the scattering experiment requires the beam divergence to be less than 100 µrad.

Our first experiments do not require a monochromator, since the expected FEL pulse energy bandwidth of 0.1% is sufficient. At 800 eV this corresponds to 800 meV, which is sufficiently low with respect to both, our spectroscopy and scattering needs. In particular, 0.1% band width at 800 eV determines the longitudinal coherence length to be about 1 µm. This will limit the achievable spatial resolution to about 5 nm in our experiment, which is significantly better than what is needed to study critical magnetic fluctuations. Furthermore, our estimates indicate that we may not have sufficient intensity in a single shot to achieve significantly higher spatial resolution.

The availability of a soft x-ray monochromator would enable experiments where high energy resolution is required for isolating specific near edge peaks. For example, to image the antiferromagnetic domain structures in transition metal oxides it is necessary to tune to specific near-edge multiplet structures. This requires an energy resolution of about 250 meV around 800 eV, corresponding to an energy band width of about 0.03%.

In summary, the required infrastructure for the proposed ***single shot imaging*** experiment is rather simple and initially consists of beam transport pipes and focusing optics only. It is worth noting that this experiment is not affected by shot-to-shot intensity fluctuations. Also, spatial beam stability is not critical, since the amplitude of the beam motions at the sample position are only one tenth of the source.

The investigation of magnetic fluctuation dynamics by ***probe-probe imaging techniques***, on the other hand, requires additional x-ray optics for splitting and redirecting of the FEL pulses. A possible layout of such a beam splitter is sketched in figure 4. Since each of the two split beams is detected independently, recombination of the x-ray pulse path is not required. This simplifies the design and implementation and also increases the overall efficiency. Using multilayer mirrors for splitting and redirection of the x-ray pulses, a 23% reflectivity around 800 eV can be achieved for each optical element. The overall efficiency of the beam splitter should thus be 5%. Furthermore, since we will use masked samples, it is only necessary that the two pulses overlap on the sample aperture, which relaxes the positional requirements for the delay line optics. An analysis of this design, done in context of the technical design report of the BESSY FEL, showed that for a 20° angle difference between the two x-ray path ways it is possible to image dynamics occurring on lateral length scales down to about 10 nm. The off-axis illumination of the sample introduces a time gradient on the sample as the wavefront reaches different parts of the sample surface at different times. This gradient, however, amounts for 10° off-axis illumination to only 1.7 fs per 1 μm lateral distance, and can thus be neglected on the time scale of interest in our experiments.

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