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Probing nanoscale behavior of magnetic materials with soft X-ray spectromicroscopy

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Abstract

The magnetic properties of matter continue to be a vibrant research area driven both by scientific curiosity to unravel the basic physical processes which govern magnetism and the vast and diverse utilization of magnetic materials in current and future devices, e.g., in information and sensor technologies. Relevant length and time scales approach fundamental limits of magnetism and with state-of-the-art synthesis approaches we are able to create and tailor unprecedented properties. Novel analytical tools are required to match these advances and soft X-ray probes are among the most promising ones. Strong and element-specific magnetic X-ray dichroism effects as well as the nanometer wavelength of photons and the availability of fsec short and intense X-ray pulses at upcoming X-ray sources enable unique experimental opportunities for the study of magnetic behavior. This article provides an overview of recent achievements and future perspectives in magnetic soft X-ray spectromicroscopies which permit us to gain spatially resolved insight into the ultrafast spin dynamics and the magnetic properties of buried interfaces of advanced magnetic nanostructures.

Keywords: interfaces; nanomagnetism; spin dynamics; X-ray microscopy; X-ray spectroscopy.

1. Introduction

The magnetic properties of matter have attracted our curiosity since ancient times. Even though we have no direct sense for magnetism, magnetic materials were technologically used, e.g., for navigation already in ancient China.

Electric power, which since the industrial revolution in the 19th century has been one of the major energy resources, relies heavily on the implementation of magnetic materials in essential components of, e.g., electric motors and transformers. And the concept of storing or handling information in current information technologies is again based on magnetic materials, e.g., in magnetic hard disks or magnetic sensor devices.

Scientists have always been "mesmerized" to unravel the very fundamental origin of magnetism. During the classical period of physics James Clark Maxwell and André-Marie Ampère established the connection between electricity and magnetism, Hans Christian Oersted discovered that electric currents create magnetic fields and Michael Faraday discovered electromagnetic induction, diamagnetism and the law of electrolysis, which laid foundation for technological inventions such as AC power systems by Nikola Tesla. John Kerr and again Michael Faraday studied the interaction of light polarization with magnetic fields. With the beginning of the quantum era of physics at the beginning of the 20th century Wolfgang Pauli postulated the existence of the electron spin [1], which was soon discovered by George Uhlenbeck and Samuel Goudsmit [2], and formulated the exclusion principle for spins, which are the two key features in our current understanding of the origin of magnetic phenomena. Werner Heisenberg developed a model to explain ferromagnetism using Pauli's exclusion principle. It was realized that since the spin of the electron exhibits the property of angular momentum, there are two different magnetic moments, the spin and the orbital part, where the latter relates to magnetic anisotropies, which in turn plays an important role in the technological functionality of many modern magnetic devices. Finally, it is the spin-orbit interaction which establishes the link between these two quantities.

Today, we are witnessing the emerging of a new era in electronics, spin electronics or spintronics [3], where in addition to the charge of the electrons the spin of the individual electron will be taken into account. The importance of spintronics was recognized by the Nobel Prize in physics in 2007 to Albert Fert [4] and Peter Gruenberg [5] for their discovery of Giant magnetoresistance (GMR), which can be seen as one of the first spintronic effects. Very soon after its discovery GMR was already utilized in read head technology of magnetic hard disks and has enabled the rapid increase in storage density since then.

length, has typical values up to a few hundred nm. A special situation occurs in the so-called water window around 2.4 nm, where the absorption in water drops significantly to allow for large penetration depth of aqueous samples.

This short wavelength of soft X-rays has an immediate consequence for soft X-ray imaging. As, in general, the diffraction limited spatial resolution in microscopies scales with the wavelength of the probe, any X-ray based microscopy technique is in principal capable of providing nanometer spatial resolution.

Synchrotron radiation (SR), which is currently generated at numerous dedicated large-scale facilities around the world such as the Advanced Light Source in Berkeley, CA, is the primary source for soft X-rays of high intensity, broad and tunable wavelength regime and controllable polarization. These multiuser facilities provide a large portfolio of instrumentation, where up to hundreds of experiments can be performed simultaneously using the unique properties of SR. The light is generated by electrons when they are accelerated in magnetic fields, which either keeps them on the circular track in the storage ring (bending magnet radiation) or which are deliberately wiggled in arrays of linearly arranged permanent magnets. The latter devices, so-called undulators or wigglers, are the characteristic sources for third generation SR facilities. They produce much enhanced photon intensities compared to a bending magnet source and they also provide largely coherent X-rays with controllable polarization. The electrons in the storage ring with a typical energy of several GeV, which makes them run close to the speed of light, are packaged in short bunches, which means that the X-rays emitted also have an inherent time structure. These X-ray flashes are typically around 100 ps long and can be used for stroboscopic X-ray studies. The next generation of X-ray sources will be X-ray free electron lasers. They will provide not only fully coherent light, both spatially and temporally, but also single bursts of X-rays at fs time scales and very high peak intensities. They will allow performing unprecedented X-ray studies, such as single shot fs dynamics of solid state materials.

For the study of magnetic properties the polarization of X-rays is very important. The magneto-optical Kerr or Faraday effect, which detects the interaction of polarized optical light with the magnetization in a ferromagnetic sample as a rotation of the light polarization after reflection or transmission, serves as a magnetic contrast mechanism. Soft X-ray magnetic circular dichroism (XMCD), which is a similar effect in the X-ray regime, has been discovered at the nickel $L_{2,3}$ edges in the late 1980s [18]. It describes the observation that the absorption of circularly polarized X-rays depends strongly on the relative orientation between the photon helicity and the photon propagation direction. The XMCD effect occurs predominantly in the vicinity of resonant X-ray absorption edges, such as the spin-orbit coupled L_2 and L_3 edges, which reflect the element specific binding energies of inner core electrons. Large XMCD effects with values up to 25% occur particularly for 3d transition metals such as Fe, Co, Ni, which are the most prominent materials for magnetic specimens.

Magnetic X-ray spectroscopies provide a wealth of important information particularly for magnetic systems. Owing to angular momentum conservation in the X-ray absorption process, the photoelectron carries spin and orbital momentum, which is different for the spin-orbit split $p_{3/2}$ and $p_{1/2}$ electronic levels, i.e., for the L_3 and L_2 absorption edges. Using the so-called magneto-optical sum rules, XMCD spectroscopy can be used to determine quantitatively spin and orbital moments in ferro- and ferromagnetic systems [19, 20].

The magnetization direction particularly in antiferromagnets can be studied using linearly polarized X-rays. The effect of X-ray magnetic linear dichroism (XMLD) detects the different absorption of the electric field vector of the photons for parallel and orthogonal orientation of the spin axis [21].

There are various ways to experimentally detect photoabsorption. The most direct measurement is the transmission method, which basically counts the incoming and the transmitted photon intensity and relates them through an exponential law. However, owing to the limited penetration length of soft X-rays this method can only be applied for X-ray transparent specimens, such as thin films on X-ray transparent substrates. During the absorption process secondary electrons are generated and detecting the electron yield, i.e., measuring the current associated with that is another commonly used method. Another alternative is to record the fluorescence yield which occurs after photoabsorption, which is a very sensitive detection method; however, it also exhibits a dichroism effect, which therefore convolutes with the original XMCD effects of interest.

The basic idea of magnetic X-ray microscopies is to record the spectroscopic magnetic response, such as XMCD, in a laterally resolving microscopic technique. So far, both transmission [22, 23] and photoemission [24] approaches have been realized and are widely used, whereas fluorescence mode detection for magnetic imaging has not yet been demonstrated experimentally.

The concept for transmission soft X-ray microscopy follows the optical design of optical microscopes [17]. However, as the refractive index of X-rays is close to one in the soft X-ray regime, conventional lenses cannot be used. The lack of appropriate optics has prevented soft X-ray microscopes until the mid-1980s when Fresnel zone plates (FZPs), which are circular gratings with a radially increasing line density have been established as high resolution optical elements [25, 26]. The fabrication of the FZP for the soft X-ray regime was enabled by the maturity of nanotechnology tools such as e-beam lithography which has then enabled the fabrication of high quality diffractive X-ray lenses. FZPs can be designed and customized for specific purposes and applications. Varying a few parameters, such as Δr, which is the outermost ring diameter, N, the number of zones, and λ, the photon wavelength at which the FZP is operating, one obtains a spatial resolution which is proportional to Δr, a focal length, which is $\sim 4N(\Delta r)^2/\lambda$ and a spectral bandwidth which is $\sim 1/N$. The most advanced FZPs for soft X-ray microscopy have achieved a spatial resolution better than 10 nm [27] (W. Chao et al. 2011, private communication), and current developments seem to make the single digit nanometer spatial resolution regime become feasible in the near future. The scheme of the optical setup of the full-field soft X-ray microscope XM-1, located at the Advanced Light Source in Berkeley, CA, where

superconducting materials, where the vortices play a crucial role to allow superconducting wires to be used in future power grids. Vortex structures and their associate structures such as antivortices are also very interesting topological objects for fundamental studies.

Magnetic vortex structures occur in soft ferromagnetic films and patterned elements, such as thin disks of the soft $Ni_{80}Fe_{20}$ alloy, as a result of the balance between exchange and dipolar energies [32]. They are characterized by a curling magnetization in the plane of the disk with a vortex core (VC) in the center, where the magnetization points perpendicular to the plane of the disk. Two binary properties are commonly used to describe this structure: the chirality, i.e., the counter-clockwise or clockwise curling of the in-plane magnetization, and the polarity, i.e., the up or down direction of the vortex core's magnetization. Both the static and dynamic properties of these objects have recently attracted an increased scientific interest both for fundamental and applied reasons [33–37]. For example, magnetic vortex structures were suggested as potential future high-density and non-volatile recording systems, as the size of the vortex core is proportional to the magnetic exchange length Λ, which can extend into the sub-10 nm regime, and the magnetic core represents a very stable spin configuration, in fact protected by topology.

The first experimental images of magnetic vortex cores have been obtained by magnetic force microscopy (MFM) [33] (Figure 2A) and Lorentz transmission electron microscopy (L-TEM) [38] (Figure 2B). The static internal structure of a vortex has been studied at almost atomic spatial resolution by spin-polarized scanning tunneling microscopy (SP-STM) (Figure 2C) [39]. Figure 2D shows a typical image of magnetic vortex cores in the center of permalloy (PY) disks obtained with M-TXM [40]. The difference to, e.g., MFM or L-TEM imaging is that M-TXM images directly component of the element-specific magnetization along the photon propagation direction instead of imaging the stray field emanating from the VC as in MFM or an increased/decreased electron intensity due to the Lorentz force when the electrons travel through the object in L-TEM. M-TXM images can therefore be used to measure the actual M_z profile of the vortex core limited only by the spatial resolution of the instruments. Fine details such as a small negative dip close to the vortex core, which originates from the dipolar field of the VC closing the field lines back into the PY disks could be observed in a recent study and were in full agreement with 3 dim micromagnetic simulations [40].

To understand the functionality of potential technological devices based on magnetic vortex structures, studies of the vortex dynamics are of paramount importance. Ultrafast dynamics of the vortex structure has been investigated by time-resolved Kerr microscopy [41], revealing the rich spectrum of excitations and eigen modes of the vortex [41–43]. However, owing to the diffraction limits of spatial resolution in optical microscopies these techniques are insufficient for smaller structures.

Time-resolved studies of spin dynamics with soft X-ray microscopy combine the high spatial resolution with the inherent time structure of current synchrotron storage rings which is given by the length of the electron bunches circulating the storage ring corresponding to <100 ps. However, the fact that the number of photons per electron bunch at current third generation synchrotrons is rather low, typically only a few tens of photons, a stroboscopic pump-probe scheme has to be used. To accumulate sufficient photons, e.g., for a single image, approximately 10^{10} photons are required; therefore, 10^{8-9} pump-probe cycles are needed per image. As a consequence, only fully reproducible processes can be studied or, in other words, only the fully reproducible part of the magnetization dynamics can be investigated.

The results shown in Figure 3 were obtained at the Advanced Light Source in Berkeley, CA, operating in the so-called two-bunch mode operation. Two electron bunches, each 70 ps in length circulate at a 3-MHz frequency, i.e., separated by 328 ns. The clock signal of the synchrotron triggers a fast electronic pulser, which launches fast electronic pulses into a waveguide structure. These pump pulses create either a local Oersted field pulse or, if the current is sent directly through the magnetic element creates a spin torque onto the magnetic spin structure. To follow the time development of the excited magnetic domain pattern the pump pulses are delayed relative to the X-ray probing pulse. Time zero, i.e., the time when the X-rays arrive at the sample is monitored by a fast avalanche photodiode close to the sample.

Figure 3 shows a sequence of images taken at varying delay times of several ns in steps of 100 ps between the pump and the probe-pulse [44]. One can clearly see that the vortex center, i.e., the vortex core performs a gyrotropic motion. The high spatial resolution allows to measure the gyration radius as a function of excitation frequency. Solving the Thiele equation with these measured radii as experimental input, one can derive quantitatively the polarization P of the currents in PY, which is an important quantity describing the spin-torque strength. These studies revealed a value of p=0.67, which was the first direct and unambiguous determination.
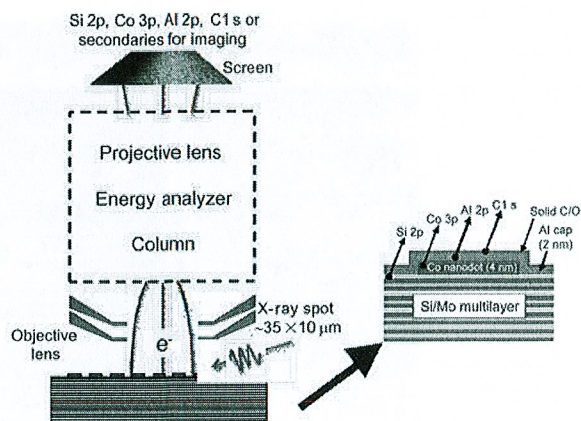
3.2. Depth resolved magnetic soft X-ray microscopy

In addition to elemental composition new functionalities in nanoscale magnetic devices can also be tailored through confinement and proximity effects. Whereas the former can be achieved, e.g., by artificial nanopatterning, typical examples for the latter are the properties of interfaces, which occur, e.g., in magnetic multilayered systems. The study of magnetism at surfaces is a particular case and numerous experimental techniques with enhanced surface sensitivities have been developed and are widely available. Scanning probe techniques sensing various forces in the proximity of surfaces or techniques which detect electrons emanating from the topological structure of the surfaces are just a few examples. However, rather often, interfaces, which are deeply buried in the bulk underneath a stack of several other magnetic and non-magnetic layers are of greater technological importance and a depth resolved analysis of magnetic properties, e.g., in multilayered structures is therefore highly required.
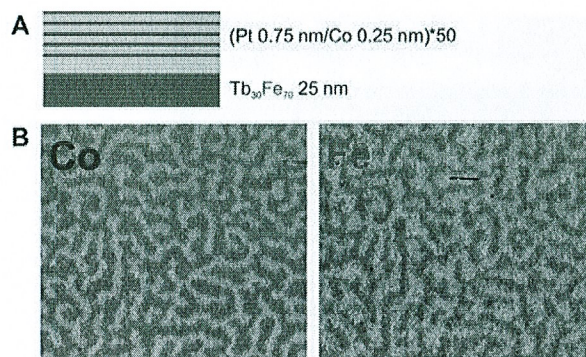
Figure 4 (A) Scheme of the (Pt 0.75 nm/Co 0.25 nm) 50/$Tb_{30}Fe_{70}$ 25 nm/Pt 5 nm multilayered film. (B) Magnetic X-ray microscopy images recorded at the Co L_3 (left) and Fe L_3 (right) edge at a magnetic field of approximately 3 kOe. The two images show the same domain pattern with reversed contrast demonstrating the antiparallel alignment of the Fe and Co moments and their direct coupling. From Ref. [44].

Figure 5 Schematic diagram of the experimental setup including the sample and the Elmitec PEEM endstation at the elliptically polarized soft X-ray undulator beamline UE49-PGM-a at the storage ring BESSY-II and cross-sectional as-grown schematic of the Co microdot structure, with the photoemission core peaks used in the element-specific study of the constituents of the structure indicated by arrows. From Ref. [47].

layer, sensitivity by either the Fe L_3 edge at 707 eV or the Co L_3 edge at 778 eV, respectively. A strong magnetic contrast is observed at both edges, however, with the sign at corresponding edges pointing in opposite directions. This immediately confirms the antiparallel alignment of the Fe and the Co moments on a microscopic length scale.

Another approach to obtain depth resolved information is based on excitation with soft X-ray standing waves (SWs) generated by Bragg reflection from a multilayer mirror substrate. The SW can then be moved vertically through the sample by varying the photon energy around the Bragg condition [45], or by moving the X-ray spot across a sample in which one layer has a wedge profile [46]. Photoemission intensities which are recorded as a function of the incoming photon energy or spot position can then be used quantitatively to derive the depth-resolved film structure of the sample by comparing the intensities to X-ray optical theory calculations. Again, if one combines this method of depth resolution, e.g., with lateral information from PEEM, a three-dimensional representation of the magnetic structures inside the specimen can be obtained. Figure 5 shows an example from a nanostructured system consisting of square arrays of circular magnetic Co nanodots, nominally 4 nm in thickness and 1 μm in diameter, which were grown on a multilayer substrate of configuration (23.6 Å-Si/15.8 Å-Mo)×40 to act as the SW generator. The experiments were conducted at the elliptically polarized soft X-ray undulator beamline UE49-PGM-a at the storage ring BESSY-II in Berlin, Germany. This microfocus beamline is equipped with an Elmitec PEEM-III endstation with an integrated photoelectron energy analyzer (Figure 6). The experimental geometry allowed for photon incidence angles between 13.8° and 17.8°, as measured from the sample surface plane. For the particular multilayer substrate and for the incidence angle range of 13.8°–17.8°, the Bragg condition was achieved by using a photon energy range of 663 eV and 516 eV, respectively. As confirmed from simulations for

electron spectra, the combination of a photon energy of 663 eV and a grazing angle of 13.8° yields the best resolved Co 3p, Al 2p, Si 2p and C 1s photoemission spectra, unobstructed by any Auger electron features. In conjunction with X-ray optical theoretical modeling, quantitative information about the depth-dependent chemical and magnetic composition of the sample can be extracted from the photoemission data [45].

Magnetic depth profiles have also been studied with magnetic resonant X-ray reflectivity (MRXR) [48], which is similar to neutron reflectivity measurements; however, it has the
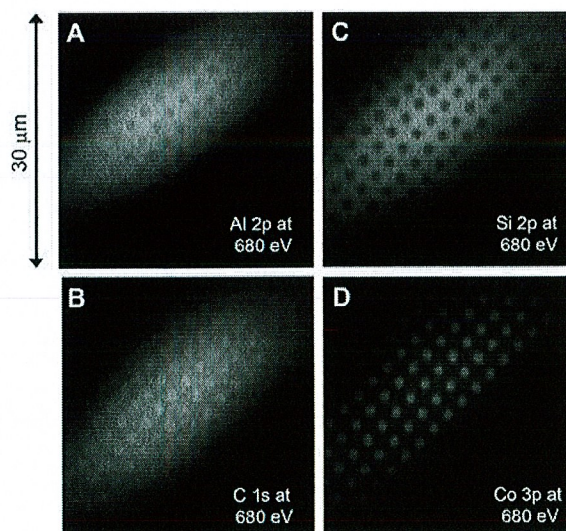
Figure 6 Standing-wave PEEM images captured at hν=680 eV, for (A) Al 2p, (B) C 1s, (C) Si 2p and (D) Co 3p core levels; 680 eV is the energy for which the Al image is a maximum off the microdots. From Ref. [47].

interesting and technologically relevant systems. It combines inherent elemental specificity with the capability to image spin structures and their dynamics down to fundamental magnetic length and time scales. These are the in nm length and the fs temporal range. The spatial resolution, which is inherently limited by the wavelength of soft X-rays has been demonstrated with state-of-the-art zone plate optics to achieve <10 nm. Novel approaches are currently exploring the feasibility of obtaining three-dimensional images, e.g., by combining depth-resolved techniques with two-dimensional imaging microscopies. In terms of sample environment, soft X-rays can operate at low and high temperatures, in infinite magnetic and electric fields, at low and high pressures, which are all relevant parameters for magnetic behavior. Only a fraction of these parameters is currently available at existing X-ray microscopes, but it can be foreseen, that in the near term future these parameters will become available. The obtainable time resolution is limited by the time structure, i.e., the X-ray pulse length of current soft X-ray sources at third generation synchrotrons. However, next generation light sources, such as X-ray free electron lasers (XFEL) are showing up on the horizon and they will be capable of delivering fs X-ray pulses with a pulse intensity, which is sufficient for a single shot magnetic image. It can thus be foreseen that these facilities will become more widely available as more routine user tools at numerous synchrotron facilities and next generation free-electron lasers around the world.

Current scientific activities of the authors

Dr. Fischer and Prof. Fadley are both principal investigators in the Magnetic Materials Program at the Lawrence Berkeley National Laboratory (LBNL). This program aims to explore and create a basic understanding of novel magnetic materials for spintronics applications, where the spin of the electrons is the dominant physical quantity. Particular emphasis in their research is to look for magnetic effects occurring on fundamental magnetic length and time scales as well as ways to minimize the energy consumption when used in magnetic devices. As progress in this research requires enhanced and new capabilities of advanced characterization, a major component of their scientific activities is to develop instrumentations which are largely based on utilizing the unique properties of X-rays.

Dr. Fischer is in charge of the full-field soft X-ray microscope XM-1 (see Figure 7) which is jointly operated between the Materials Science Division (MSD) at LBNL and the Advanced Light Source (ALS) at beamline 6.1.2 at the ALS. Worldwide, this instrument is still the only full-field soft X-ray transmission microscope used for magnetic imaging with XMCD as the magnetic contrast mechanism. The key components to run this instrument are the Fresnel zone plate (FZP) optics, which are developed and fabricated at the Center for X-ray Optics (CXRO), a unique research facility within MSD at LBNL. Highest quality FZPs have demonstrated at XM-1 a world record 10 nm spatial resolution with soft X-rays and the current standard spatial resolution, enjoyed by a large general and worldwide user community of XM-1, is approximately 20–25 nm. The most attractive feature of XM-1 is the combination of high spatiotemporal resolution and chemical and magnetic sensitivity, which is of utmost importance for nanomagnetism research.

Dr. Fischer's current scientific interest is to investigate ways to control spins on the nanoscale by imaging with MTXM, e.g., spin current induced domain wall and vortex core dynamics, as well as to study the relatively simple, but yet unanswered question as to whether magnetic processes on a nanometer length scale exhibit a deterministic behavior or show stochastic character. This question is of both fundamental interest but moreover of high technological relevance.

One out of many of Prof. Fadley's scientific research interests is the question how buried interfaces determine the functional behavior of complex and multicomponent nanoscale magnetic devices. Also here, new instrumentation utilizing X-rays can provide very valuable and new insight (see Figure 8). Using the standing wave approach, which Prof. Fadley and his team has pioneered in the recent past with experiments at various worldwide synchrotron radiation facilities used a high precision and tailored multilayer substrate fabricated also at CXRO. With the advanced instrumentation and the sophisticated X-ray optics analysis calculations, his team was able to demonstrate the feasibility to fully gain three-dimensional information of the magnetism at deep-buried interfaces in technologically relevant systems. Another important scientific direction of Fadley's team is to enhance the capabilities of photoemission spectroscopy, which owing to the limited escape depth of soft X-rays has so far remained a highly surface sensitive technique, into the hard X-ray regime, which allows to probe deep into the bulk of interesting materials. Fadley is also closely collaborating with theory groups to achieve a solid understanding of the experimental data.

Both Dr. Fischer and Prof. Fadley are looking forward to next generation of soft X-ray instrumentation such as a next generation full-field soft X-ray microscope for materials, environmental and energy related research and a new hard X-ray photoemission instrument, as well as to the next generation of light sources, such as the upcoming X-ray Free Electron Laser (X-FEL) facilities, which will completely provide new capabilities for X-ray sciences in terms of peak brilliance, coherence and time scales which will become accessible to the community.
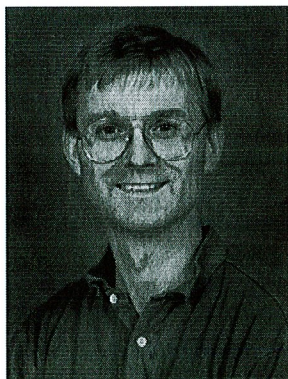
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Dr. Peter Fischer received his PhD in Physics (Dr.rer.nat.) from the Technical University in Munich, Germany in 1993 and his habilitation from the University in Würzburg in 2000 based on his pioneering work on magnetic soft X-ray microscopy.

Since 2004, he has been staff scientist and principal investigator at the Center for X-ray Optics within the Materials Sciences Division at Lawrence Berkeley National Laboratory (LBNL) in Berkeley, CA. His current research program is focused on the use of polarized synchrotron radiation for the study of fundamental problems in nanomagnetism. He has pioneered magnetic soft X-ray transmission microscopy for the imaging of magnetic domain structures and their fast dynamics. He is involved in developing the scientific case for a next generation soft X-ray free electron laser at LBNL. Dr. Fischer has published more than 130 peer-reviewed papers and has given more than 180 invited presentations at national and international conferences. For his achievements of "hitting the 10 nm resolution milestone with soft X-ray microscopy" he was co-awarded with the Klaus Halbach Award at Advanced Light Source in 2010. He was appointed as Distinguished Lecturer of the IEEE Magnetics Society in 2011.

Dr. Fischer serves the magnetism and X-ray community as a member of various program and review committees worldwide. He was conference chair of the 7th MML in Berkeley in 2010. He is a member of the APS, AVS and senior member of the IEEE Magnetics Society.

Professor Charles S. Fadley received his BS from MIT and his MS and PhD from UC Berkeley, CA. He is Distinguished Professor of Physics at UC Davis, CA and Faculty Scientist at the Materials Sciences Division at Lawrence Berkeley National Laboratory in Berkeley, CA. His general research areas include surfaces, buried solid-solid interfaces, multilayer nanostructures and complex multicomponent materials that form the active elements in many devices for information storage and processing, as well as energy conversion, storage and utilization. Research in the Fadley Group is directed towards developing new X-ray based methods for studying such surfaces, interfaces, nanostructures and complex materials. The principal technique used by Fadley is photoelectron spectroscopy (photoemission) as excited by synchrotron radiation, and in the future, free-electron lasers. One new direction of interest is the use of X-ray standing waves created by Bragg reflection from multilayer mirrors to probe the depth dependence of composition, electronic structure and magnetism in spintronic-, complex oxide- and semiconductor-multilayer nanostructures. A second is the use of harder X-rays at 3–6 keV or more to excite photoelectrons from deep within nanostructures and bulk materials. The systems studied are of relevance to next-generation magnetic information storage and logic, and other emerging ideas for logic, memory and energy conversion. Fadley and his team carry out experiments with unique facilities at the Advanced Light Source in Berkeley, as well as other laboratories in Germany, Japan and Switzerland.

Prof. Fadley has received numerous awards and honors, including the Medard W. Welch Award of the AVS (2005), the Helmholtz-Humboldt Research Award (2006), the Japanese Society for the Promotion of Science, Microbeam Analysis Committee Award (2007) and an Alfred P. Sloan Foundation Fellowship, and has been elected fellow of the APS, AVS and IOP, and a foreign member of the Russian Academy of Sciences and the Royal Society of Sciences in Uppsala, Sweden. He has published close to 300 peer-reviewed papers and given close to 200 invited talks at international venues.

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