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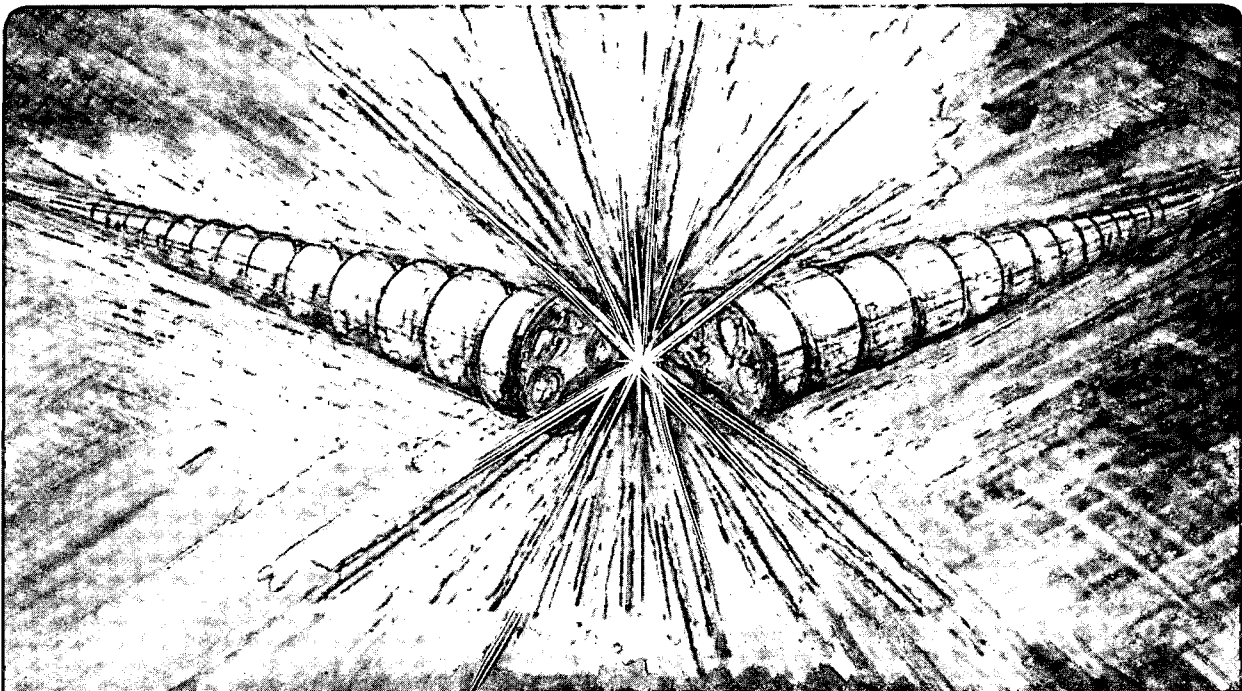
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**X-RAY MAGNETIC MICROSCOPY AND SPECTROSCOPY USING A THIRD
GENERATION SYNCHROTRON RADIATION SOURCE***

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X-Ray Magnetic Microscopy and Spectroscopy Using a Third Generation Synchrotron Radiation Source

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Abstract

Applications of x-ray magnetic circular dichroism (XMCD) to the study of magnetic materials are described. XMCD spectra can be used to quantitatively determine magnetic properties on an element-specific basis. These spectra are also sensitive to the chemical state and environment of the element being probed. The dichroism effect can also be used to produce images of microscopic magnetic structures and domains. Third generation synchrotron light sources are well suited to these experiments. Current and planned facilities at the Advanced Light Source, the first of the new light sources in the U.S., are described, focusing on a new facility with specialized undulators which will directly produce high flux, high brightness beams of circularly polarized x-rays. With new beamlines which have been optimized for either spectroscopy or microscopy, this facility will provide the capability to provide detailed information about magnetic materials.

I. INTRODUCTION

The application of circularly polarized x-rays to studies in chemistry and physics has recently become a field of intense interest. Studies of systems as diverse as biological molecules¹ and monolayer/thin film structures² have been performed using circularly polarized x-rays. In particular, studies of magnetic materials have been of special interest. Starting with the theoretical study by Erskine and Stern³ to the first experimental studies by Schutz *et al* ⁴ and Chen and co-workers⁵, XMCD has been used to study a variety of magnet phenomena such as the measurement of magnetic moments and the determination of magnetic coupling. XMCD has also been used to produce images. "Magnetic microscopy" has been used to create element specific images of the magnetic domains and structures of interest to the semiconductor/magnetic recording industry.⁶

The Advanced Light Source (ALS) at Lawrence Berkeley National Laboratory is ideally suited for both spectroscopy and microscopy using circularly polarized x-rays. In addition to existing beamlines which have capabilities for circularly polarized x rays, ALS is designing and building a complement of new insertion devices and beamlines to facilitate this research. Using "insertion devices" which directly produce circularly polarized light makes available high flux, high brightness beams of x-rays at "application-specific" beamlines.

This paper first reviews a few general properties of XMCD spectra. This review will be brief and will treat the physics only qualitatively. More detailed reviews of the theory of XMCD are available.^{7,8} It then describes some of the applications of XMCD to magnetic materials research. Again, only a few examples of interest to magnetic measurements are presented. The present capabilities of the ALS with circularly polarized x-rays is described. New facilities being built at the ALS are discussed; in particular, a new facility with circularly polarized undulators and optimized beamlines is described. Finally, the end stations will be described.

II. GENERAL PROPERTIES OF X-RAY MAGNETIC CIRCULAR DICHROISM

XMCD uses the property that some materials, when magnetized, exhibit a different absorption coefficient for left circularly polarized x-rays than for right circularly polarized x-rays. The effect is dependent on the direction of both the magnetization vector and the polarization vector of the x-rays, which for circular polarization is either parallel or anti-parallel to the direction of photon propagation. Two spectra are possible, one where the magnetization and the photon polarization vectors are parallel, and the other with the magnetization and the polarization anti parallel. Switching between these two spectra can be done by changing either the magnetization direction or the photon polarization.

Excitation by x-rays produces atoms in which one of the inner shell, or core level, electrons is promoted to an empty valence shell energy level. X-ray absorption probes the population and state density of both the lower state and the upper state. For transition metal elements, the upper levels are the 3d energy levels, which are the states responsible for magnetic properties. The strongest absorption to these levels are the L_{2,3} lines (edges), which have the 2p levels as their lower state. The transition energies lie between 574 eV for vanadium to 952 eV for copper. Rare earth elements have 4f levels as the upper states. The strongest lines for these levels are the M_{4,5} lines, which have the 3d levels as their lower states. These transition energies 880 eV to 1600 eV.

Figure 1 shows typical absorption spectra for the L_{2,3} lines of nickel. In the upper panel, the solid and dashed lines are the spectra obtained with the magnetization vector and the photon helicity parallel and anti-parallel. The lower panel shows the resulting XMCD spectra, that is, the difference between the two absorption spectra. The intensity ratios of both the absorption spectra and the XMCD spectra are informative. Sum rules which relate the integral of the XMCD spectra to the orbital and spin magnetic moments have been developed.^{9,10}

To use XMCD to produce images, the energy of the exciting x-ray beam is held

fixed at the center of the XMCD peak. With a given photon helicity, the intensity of the response of the material will depend upon the magnetization of the material. If the response from the sample can be spatially resolved, a map of the magnetization of the material can be produced.

There are two primary schemes to image the surface. The first technique uses the x-ray beam to illuminate a small but finite area. An electron microscope is then used to create an image from the photoelectrons that have been liberated. This technique has the advantage of acquiring the entire image at once. Ultimate resolution is determined by the resolution of the electron microscope. The second technique focuses the x-ray beam to as small a focal spot as possible and recording the signal. Spatial resolution is set by the size of the focal plot. The spot is then scanned over the entire sample, either by translating the focusing element. or by moving the sample. This technique has the advantage that a variety of detection techniques can be used, including transmission, fluorescence, and photoelectron detection.

III. APPLICATIONS OF XMCD

There have been a large number of experiments using XMCD. These can be divided into two major categories, spectroscopy and microscopy. A variety of systems have been studied with spectroscopy. Magnetic multilayer thin films have been a frequent topic of interest. Monolayer films of iron on single crystal copper have been studied¹¹, with large dichroism effects detected. Nickel ultrathin films have been studied¹², and it was determined that the easy axis of magnetization varied with the thickness of the layer. It was determined that the axis changed from parallel to the surface for 5-9 monolayers to perpendicular for 10-75 monolayers. In another experiment, the exchange coupling between two layers has been studied as a function of the width of an intervening layer.¹³ In this experiment, the coupling between a cobalt layer and a nickel-iron layer with an intervening wedge of ruthenium was determined using XMCD. It was found that the direction of magnetization oscillates with the

thickness of the ruthenium.

Quantitative measurements of magnetic properties have also been made. For example, element specific magnetization curves have been made.¹⁴

Iron/copper/cobalt trilayers were measured using XMCD. Figure 2 shows the hysteresis curves obtained with XMCD and a conventional method. As can be seen, the iron and cobalt hysteresis curves are very different, with different saturation and coercive fields. This is in contrast to measurements made on bulk alloys, in which the hysteresis curves for the two elements are identical. The sum of the two curves yields the curve obtained conventionally. This experiment also illustrates the fact that XMCD measurements are element specific. Another experiment illustrates that the magnetic moment can be decomposed into its orbital and spin contributions. Using the sum rules mentioned above, Chen and co-workers¹⁵ determined that the orbital moment for a cobalt thin film is $0.14 \mu_B$, and the spin moment is $1.52 \mu_B$.

Much less work has been done on magnetic microscopy. Stohr *et al* have imaged the magnetic bit structure of a magnetic recording disc.⁶ In this case, the material was a cobalt/platinum/chromium alloy, and the XMCD spectrum was taken at the cobalt edge. Spatial resolution of $1 \mu m$ was demonstrated. Another example of imaging is the work by Tonner *et al*, who measured iron/terbium/cobalt thin films.¹⁶ In this experiment, the images were obtained at the iron and terbium edges.

IV. CIRCULAR POLARIZATION CAPABILITIES AT THE ADVANCED LIGHT SOURCE

X-ray science has enjoyed a renaissance in recent years with the introduction of synchrotron light sources. These light sources use the fact that charged particles emit light when bent by a magnetic field. The spectrum of light that is emitted is a function of the mass and energy of the particle and the strength of the magnetic field. To produce high energy photons such as x-rays, electrons accelerated to the GeV energy range are used in combination with magnetic fields of about 1-5 Tesla. This produces

light from the far infrared to x-rays beyond 10 keV (a wavelength of 1.2 angstroms.) In addition, by using these high energy electrons, which are relativistic at this energy, the emitted photons are directed in a very narrow, well directed beam, propagating in the same direction as the electron. Pictorially, this is analogous to the searchlight (the x ray beam) sweeping around an arc.

Third generation light sources, of which the ALS is the first in the world designed for soft x-rays, have optimized the design of the storage ring to produce high quality photon beams. Third generation sources use an electron beam which has a small cross sectional area and small divergence, giving "low emittance." This makes it possible to focus the emitted photon beam to small sizes. In addition, the design of the electron storage ring is in the shape of polygon with many sides or straight sections, rather than a circle or oval. In the case of the ALS, there are twelve straight sections. At the vertices of the polygon, bend magnets are placed which produce synchrotron radiation as described above. In the straight sections, special periodic arrays of magnets can be placed. These periodic arrays, or insertion devices, produce a much higher photon flux than a single bend magnet.

There are two types of insertion devices, wigglers and undulators. Wigglers use magnets with a high magnetic field, and produce a high flux photon beam with a broad energy spectrum. Undulators use periodic magnet arrays of lower field; however, because of constructive interference between the photons in the undulator, the beam is much narrower than the bend magnet beams, and is quasi-monochromatic. The beams produced thus have high "spectral brightness", a measure of the number of photons per cross sectional area, angular degree, and photon energy spread per second. The spectral brightness of a typical ALS undulators is 4-5 orders of magnitude higher than that from bend magnets sources.

Synchrotron radiation is emitted with specific polarization properties. From bend magnets, the light emitted in the plane of the electron orbit is polarized horizontally. As

one moves out of the orbit plane, though, the polarization of the beam becomes elliptical, becoming more and more circular the further out-of-plane one goes. The flux of photons drops rapidly, however. The variation of flux and polarization with angle is a function of the photon energy. Using apertures, the degree of circular polarization can be selected.

Undulators and wigglers also produce horizontally polarized light. However, conventional wigglers and undulators do not produce circularly polarized radiation. In order to produce circularly polarized radiation, a variety of special insertion devices have been proposed, although not all designs allow for complete control of the energy or helicity of the radiation. The ALS is designing and building a set of these special undulators which allows for user-controlled energy and polarization.

A. Presently Operating Facilities

Beamline 9.3.2 is a bend magnet beamline which has been optimized for high resolution spectroscopy.¹⁷ With an energy range from 30-1500 eV, it enables research with the most interesting 3d transition metals and 4f rare earths, including nickel, iron, cobalt, and neodymium. Equipped with a monochromator which has a resolving power greater than 7000, it is capable of determining shifts in the absorption spectra which occur due to differences in the chemical environment of the element probed.

To use the circularly polarized x rays, an aperture is used to select a portion of the out of plane radiation. At a modest reduction in photon flux, the beam can have a high degree of circular polarization. Typically, a degree of circular polarization of about 0.8 is used, corresponding to collecting radiation from about 0.2 to 0.5 mrad. Measurements of the polarization using a reflection polarimeter have verified the performance of this system.

Multiple end stations can be accommodated at this beamline. These include: the Advanced Photoelectron Spectrometer/Diffractometer (APES), which is capable of a variety of surface science experiments, such as high resolution photoelectron diffraction;

the Angle-Resolved Photoemission Spectrometer (ARPES), with a rotatable electrostatic electron-energy analyzer; and the Applied Materials Chamber (AMC). Using the AMC, XMCD measurements can rapidly be made on a variety of samples.

B. Facilities Under Construction

1. Beamline 7.3.1 Bend magnet endstation optimized for microscopy

Beamline 7.3.1 is also a bend magnet beamline. It is optimized for full field photoelectron microscopy applications.¹⁸ Because the electron beam emittance from third generation synchrotrons is so low, with careful optical design, bend magnet radiation can be focused to produce very small illuminated areas, typically 50 micron diameter. Microscopy experiments in general require a high photon flux. Because of this, the optical design of the beamline has been optimized to maximize the photon flux through the beamline to the endstation. The microscopy applications generally do not require high spectral resolution. This leads to a simplified monochromator design which improves the photon throughput. Spectral resolution of at least 1000 will be achieved throughout the energy range of the system. This range, 275-1500 eV, covers the carbon 1s absorption edge at the low end, to the M-edges of the rare earths at the high end. The performance is optimized at approximately 800 eV, the energy of the L-edges of iron, cobalt, and nickel.

Selection of the helicity of the circular polarization will be performed using a chopper/aperture to collect out of plane radiation. To improve the XMCD performance, the chopper can be used to switch the helicity at rates up to 0.5 Hz. This capability will allow for reductions in noise, as well as a modest capability to measure time dependent processes.

The endstation for beamline 7.3.1 will be photoemission electron microscope (PEEM). Details of this type of microscope have been detailed elsewhere.¹⁶ With this type of microscope, the photoelectrons ejected from the sample by the x-rays will be used by an electron microscope to image the surface. Sub-micron spatial resolution has

been demonstrated with this instrument, and with planned improvements, resolution of < 100 nm structures should be achievable. This experimental facility is currently under construction and will be ready for use by June of 1996.

2. Beamline 4.0 Undulator Facility for Spectroscopy and Microscopy

Beamline 4.0 is an undulator-based facility designed specifically for circular polarization applications. The insertion devices will be located in a single insertion device straight section, where two "undulator stations" will be placed end-to-end. Small bending magnets will produce a "chicane" in the straight section, directing the electron beam first through one station, then through the other, producing a 1.65 mrad angle between the two optical axes. Undulators can be positioned and operated at both stations simultaneously. In addition, at each station, a translation mechanism will allow either of two undulators to be placed in the beam. With this arrangement, a total of four different undulators will be available for use at the two beamlines, allowing for great flexibility in the energy range or helicity switching. The output from either station can be directed to either of the two branchlines, or the output from both stations can be directed to the same branchline.

To directly produce circularly polarized radiation, an undulator design similar to that of Sasaki¹⁷ and Carr¹⁸ has been adopted. The magnetic design of this insertion device has been modified to produce higher magnetic fields on the axis of the undulator, leading to an expanded photon energy range. The device is capable of producing polarized light of any ellipticity: horizontal, vertical, circular, or elliptical. Users will be able to select the mode used. In addition they will be able to change the helicity of the circularly or elliptically polarized light every few seconds.

The figure of merit considered for the evaluation of performance is defined as:

$$M = P_C^2 \cdot F$$

where P_C is the degree of circular polarization, and F is the flux. The merit function brightness is also considered (where brightness is substituted for flux in the above

equation). Flux, brightness, and degree of circular polarization have been calculated using the formalism described by Kim¹⁹ for the planar and helical cases and generalized by Marks²⁰ for the elliptical case.

The first undulator to be installed in the ALS circular polarization facility will be a 5 cm period, 1.95 m long device with 37 full strength periods. The energy ranges of the various modes of polarization are summarized in Table 1. These correspond to an electron energy of 1.9 GeV. Figure 3 shows the calculated performance for the merit function flux and brightness for this device for pure helical mode and for the 1st, 3rd, and 5th harmonics when the undulator is operated in an elliptical mode. As can be seen, operation in pure helical mode gives slightly higher merit function performance, but with a very restricted energy range, as pure helical motion produces no higher harmonics. In elliptical mode, this undulator produces usable output from 100 to 1800 eV. This energy range covers the important core levels for magnetic materials -- the L(2,3) edges of transition metals and the M(4,5) edges of rare earths. Performance has been optimized for coverage of the iron, nickel, and cobalt absorption edges, near 800 eV. These calculations do not include electron beam energy spread, which will decrease the performance of the higher harmonics. For this reason, in determining the high energy performance, only the first and third harmonics have been considered. To reach even lower energies, a 7.5 cm period device has also been designed. With this device, energies as low as 20 eV will be available.

Emerging from the undulators, the two photon beams are separated by 1.65 mrad. After passing through the shield wall, the beams will enter a "mirror switchyard." Using this set of mirrors, the output from either (or both) undulator station(s) can be directed to either of the branchlines. In the "standard" mode, each undulator beam would be directed to one of the beamlines by a translating mirror. To send both beams to the microscopy branchline, the mirror for the spectroscopy line would be retracted and the beam allowed to propagate to a second mirror which would then direct the

beam down the microscopy line. A similar procedure is used to direct both beams to the spectroscopy line. Sending the output from both undulator stations down the same beamline will allow for experiments which require rapid changes in polarization. For these experiments, undulators in the two stations will be set to the same photon energy and equal but opposite helicity. Although both beams propagate down the same beamline, in some places they are physically separated. At one of these points, a mechanical chopper is placed which will alternately block one polarization or the other.

The conceptual design of the circular polarization beamlines is shown in figure 4. Two main beamlines will be built for this facility. The optical design of each branchline is tailored to suit one of the two major research areas, microscopy or high resolution spectroscopy. For spectroscopy studies, a wide energy range will be covered, from 20 to 1800 eV, with a resolution of up to 10,000 at 100 eV. For the microscopy beamline, a higher-throughput, lower resolution monochromator has been designed. It is entrance-slitless, with an energy range of 100-1800 eV and a resolution of about 1500.

The microscopy beamline will have provisions for two end stations, each with its own monochromator exit aperture. One end station will be a PEEM, a similar but improved version of the instrument used on beamline 7.3.1. The current generation of PEEM's suffer from resolution degradation caused by chromatic aberration of the lens, caused by the spread in the kinetic energy of the photoelectrons. The new PEEM will focus all electrons to the image plane, independent of their kinetic energy. Ultimate resolution of an advanced PEEM should be better than 20 nm.

The other microscope end station will be equipped for scanning Fresnel zone plate microscopy. Zone plates are focusing elements which rely on diffraction rather than refraction, like lenses, or reflection, like mirrors. They consist of a series of concentric rings, with a decreasing spacing as the radius increases. The radii of the rings is chosen so that the light passing through all the rings is diffracted to a single point. Zone plates are excellent optical elements for x-ray applications as, because of absorption, there are

no materials which can be used as lenses. Mirrors for x-ray applications also suffer from absorption as well as the requirement that the mirrors need to be used at grazing incidence. A typical zone plate in use at the ALS has 100 zones (rings), a diameter of 100 μm , and an outer zone width of 30 nm.

The resolution of a zone plate microscope is derived from the size of the focused spot. This, in turn, is approximately equal to the width of the outer zone. This is currently limited by microfabrication technology to about 30 nm.

Detection in a zone plate microscope can be via a variety of techniques, depending upon the sample. Using x-ray detectors, transmission through the sample can be measured, as can fluorescence excited by the synchrotron beam. Alternatively, photoelectrons can be collected. Images are collected by scanning the x-ray spot across the sample, either by translating the zone plate or by moving the sample.

The spectroscopy beamline will also have provisions for two end stations. For this beamline, a rotating experimental platform will allow two end stations to be attached and aligned to the beamline at a time. Many of the types of experiments to be run on this beamline are similar to the experiments performed on beamline 9.3.2, thus, many of the endstations will be similar. However, the much greater photon flux available from the undulators will enable experiments which are not currently possible. For example, experiments with samples which are very dilute in the species of interest or which have a finite lifetime will benefit from the increased photon flux. Typical end stations which will be used on this beamline will include UHV angle-resolved photoelectron spectroscopy chambers and general purpose chambers equipped for surface science and materials research. For XMCD measurements, chambers with magnets which can alternate their polarity will be used. Systems with permanent magnets or electromagnets can be used. In particular, superconducting magnet chambers for measurements at fields above 1 Tesla will be available.

SUMMARY

X ray magnetic circular dichroism has shown, in a short time, to be a valuable technique in characterizing magnetic materials. Third generation synchrotron light sources like the ALS can provide high flux, high brightness beams of x-rays for this research. At the ALS, in addition to presently available bend magnet beamlines, two new facilities are being constructed. A new microscopy beamline is being built, and a facility with circularly polarizing undulators and beamlines optimized for high resolution spectroscopy and microscopy are being constructed. These new facilities will provide a new resource for probing magnetic materials.

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Table 1 Energy Range of a 5 cm period elliptically polarizing undulator

Mode	Peak Bx T	Peak By T	Min. Energy eV	Max. Energy eV
Elliptical	0.228	0.502	100	1500
Helical	0.424	0.424	140	680
Horizontal	0	0.793	85	1500
Vertical	0.502	0	180	1500

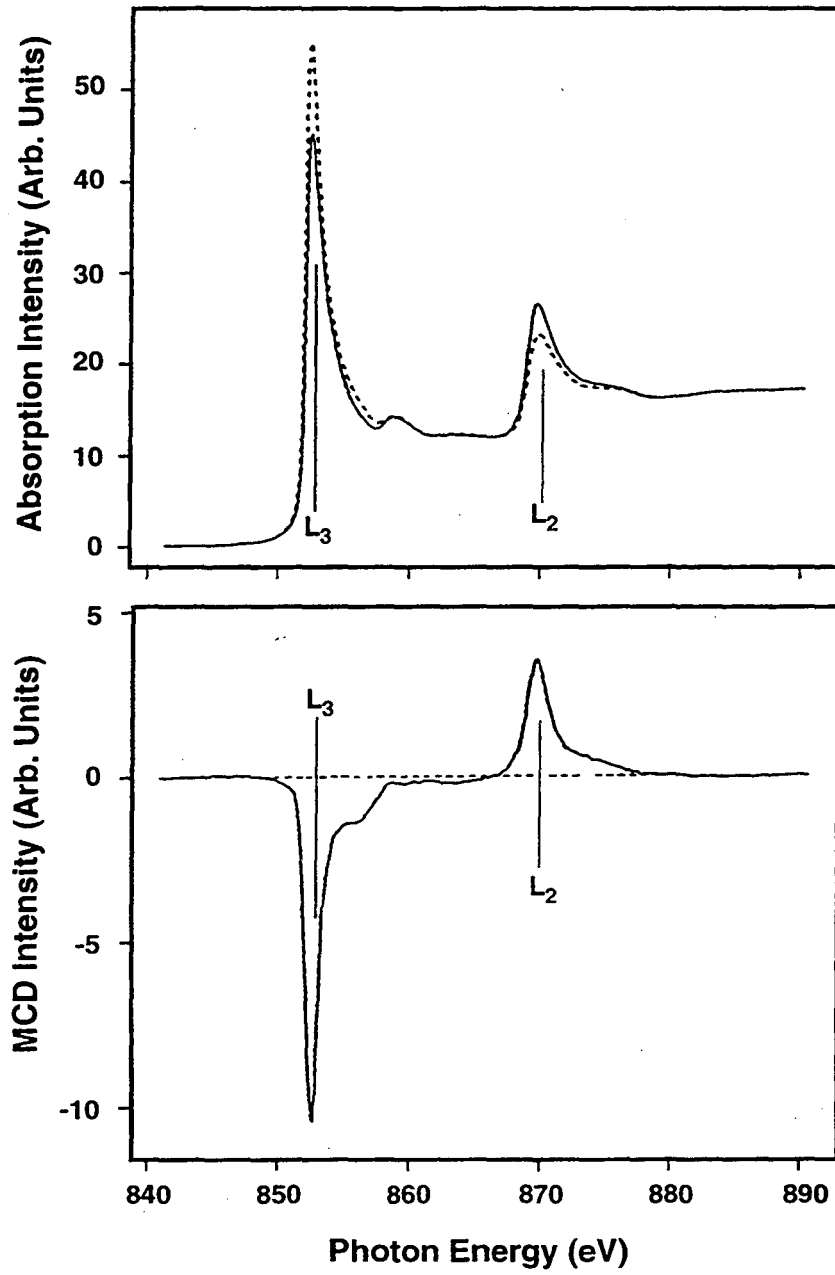
Figure Captions

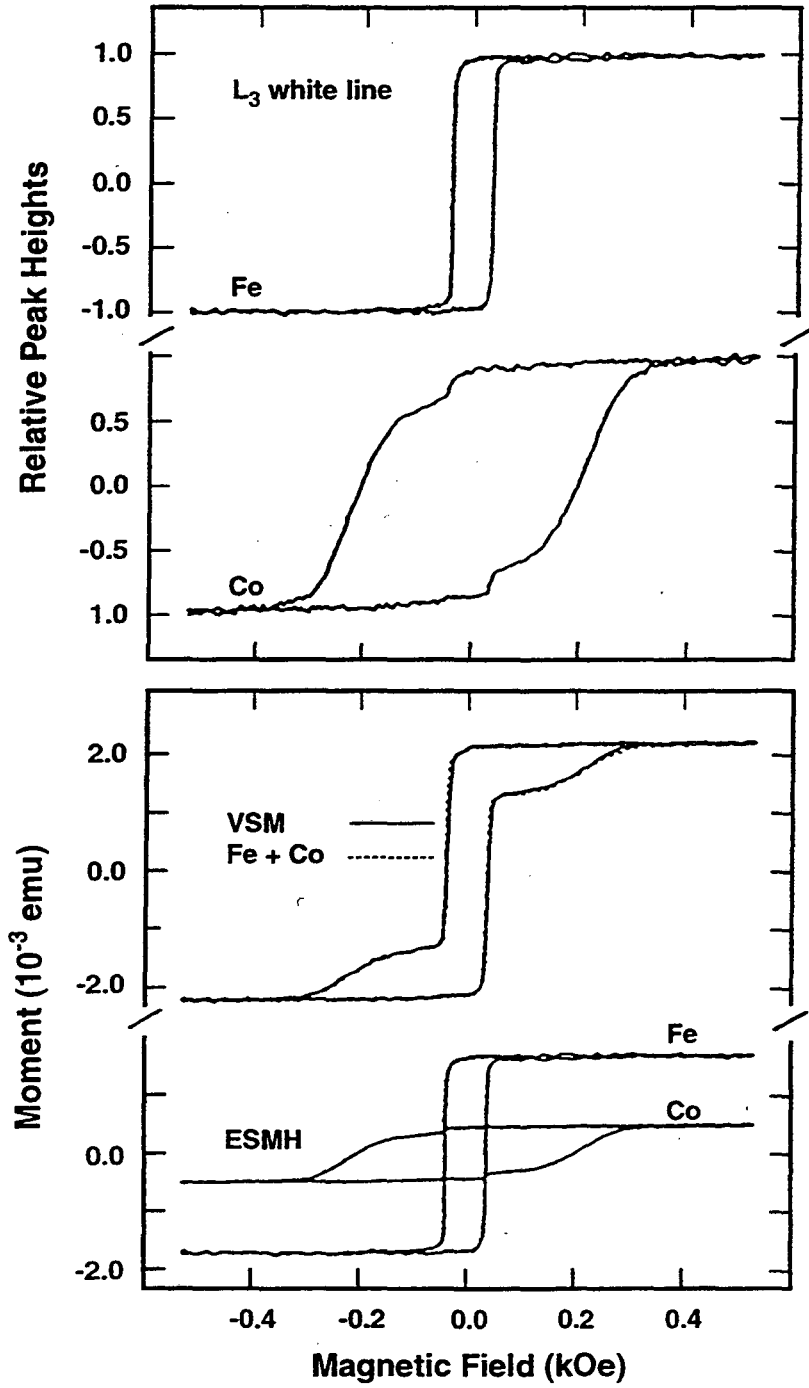
Figure 1 X-ray absorption and XMCD spectra of nickel at the L_{2,3} core level edges. Upper panel: absorption spectra with photon helicity parallel (solid) and anti-parallel (dashed) Lower panel: XMCD spectrum

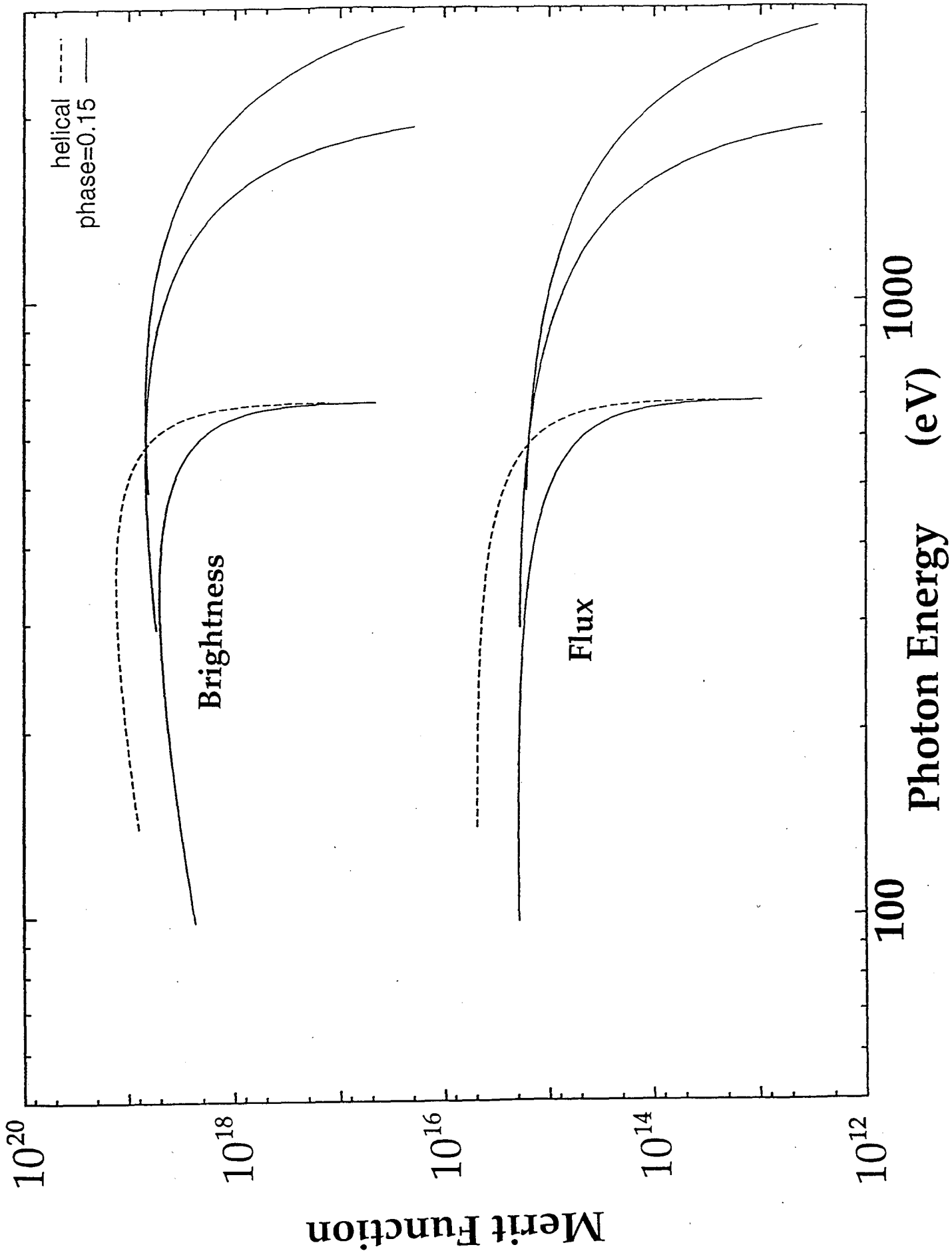
Figure 2 Element specific magnetometry using XMCD. The top panel shows the Fe and Co L₃ line intensities as a function of applied magnetic field for a Fe/Cu/Co trilayer. The bottom panel shows the comparison between an overall hysteresis curve obtained using conventional methods (solid) and a linear combination fit of the two XMCD hysteresis curves (dotted.)

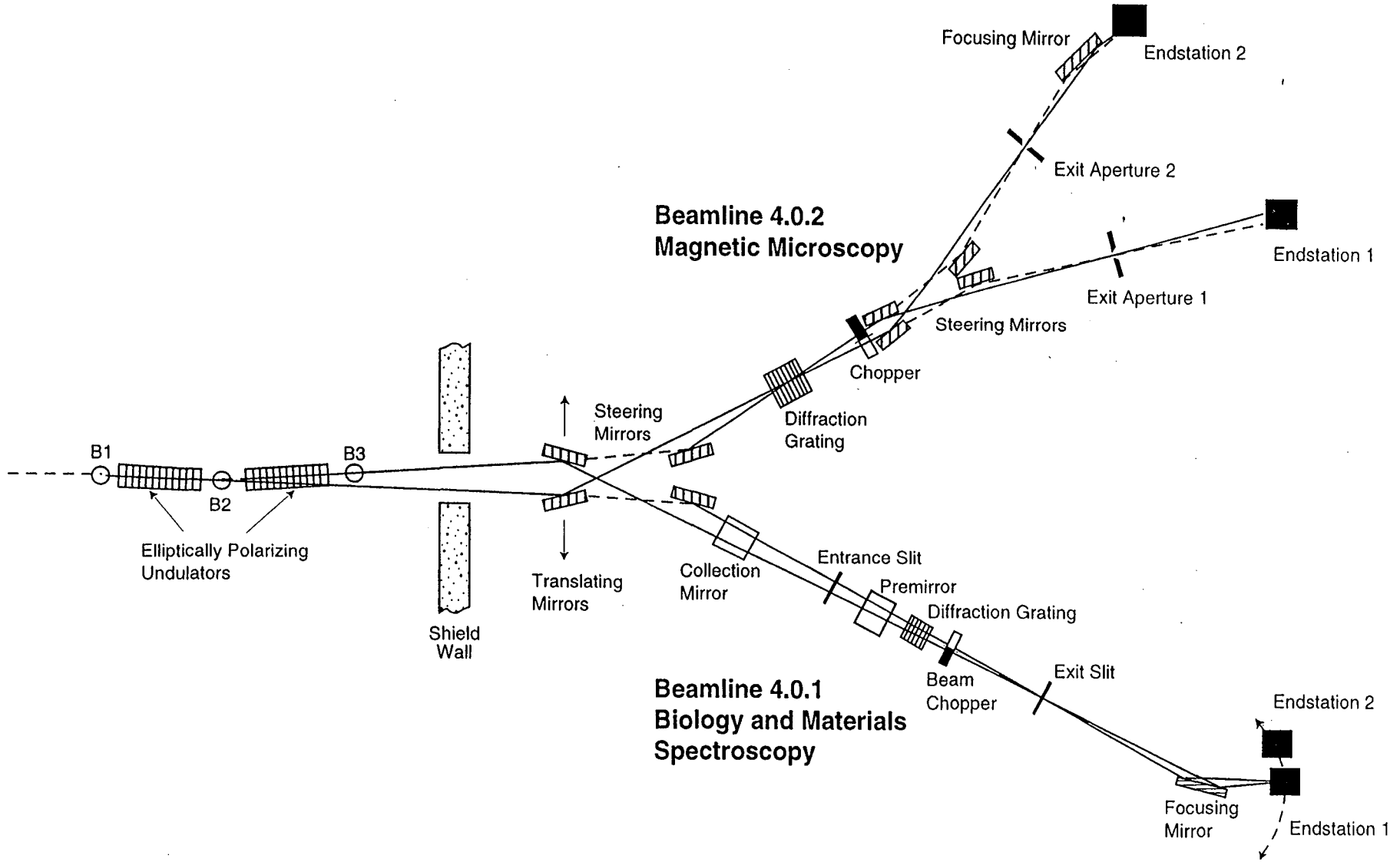
Figure 3 Calculated performance curves of a 5.0 cm period elliptical polarized undulator. The flux (brightness) merit function is plotted, which is the flux (brightness) x degree-of-polarization². Electron beam energy is 1.9 GeV.

Figure 4 Conceptual design of the Advanced Light Source beamline 4.0, a new facility with elliptically polarizing undulators and beamlines optimized for spectroscopy and microscopy









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