# Optics and instrumentation upgrades for electronic and magnetic materials research at beamline 4-ID-C

This proposal is for an upgrade of the 4-ID-C beamline, which is dedicated to soft x-ray polarization dependent spectroscopy studies. The project consists of three major parts. First, the beamline optics (monochromator and mirrors) will be replaced to provide both greater beam stability and significantly increase incident flux (~1-2 orders of magnitude). This increased intensity will facilitate the study of much more dilute magnetic samples and enable time-resolved XMCD experiments currently at the limit of observation. In addition, two new experimental end-stations will be developed to provide enhanced and more flexible sample environments to the users, one for low-temperature high-field spectroscopy studies and another for the study of surface magnetism.

## Scientific Justification

The strong interaction of soft x-rays with matter offers a powerful tool to answer some of the most fundamental questions in condensed matter physics. Circularly polarized soft x-ray beamlines and associated spectroscopy, scattering and microscopy techniques, in particular, have enabled many experiments over the last 20 years which were previously unattainable. As a result, these techniques have become standard features in the study of magnetism, electronic ordering, and chiral systems, leading to a deeper understanding of the relation between electronic structure at the atomic level and macroscopic properties, as well as major technological advances. In order to address future challenges, however, will require enhanced beamline and sample environment capabilities. For example, three questions posed in a recent BES Workshop Report: "Directing Matter and Energy: Five Challenges for Science and the Imagination" directly relate to the science that will be enabled by the upgrades proposed here.

*"How do we control material processes at the level of electrons?"* This question is particularly relevant in the quest for new electronic devices that create and control spin-polarized carrier populations, with the promise of new functionality and greater efficiency in information processing and storage technology. To this end, understanding of the band structure in doped ferromagnetic semiconductors and hybrid structures has become critical. In these systems, very small magnetic moments induced on the host material or at interfaces are central to the magnetic ordering and spin transport properties. The element specificity of polarized soft x-rays offers a unique way to examine these states, but the magnetic moments are at or below the detection limits of current soft x-ray magnetic circular dichroism facilities, making systematic studies impossible. The beamline stability and flux enhancements in this proposal would enable such studies, thus providing key insight into the next generation of electronic devices.

*"How do remarkable properties of matter emerge from complex correlations of the atomic or electronic constituents and how can we control these properties?"* This question is directly related to the rich behavior that has been observed in strongly correlated electron systems such the transition metal oxides (manganites, cobaltites, cuprates, etc.). These systems contain a variety of competing interactions, creating a subtle balance to define the lowest energy state in the system (e.g. metal, insulator, superconductor). X-rays provide a direct probe of how the system's electronic and magnetic properties evolve, especially as it is driven through phase transitions between

different ground states via temperature dependence or the application of external pump fields (e.g. magnetic, electronic, or photonic). The behavior of these systems becomes especially interesting near the broken symmetry of a surface or interface. Through state-of-the-art oxide fabrication one can now create high-quality boundaries between systems with competing order. In this way, one can try to look for completely new states arising at the interface. Heterostructures then offer a direct route to development of novel devices, which provide a means to manipulate these ground-states as a pathway to new technologies. Element resolved x-ray probes are crucial to understand how the interface behaves, but require high intensity in the region of transition metal oxides given the small interface signals.

*"How do we characterize and control matter away—especially very far away—from equilibrium?"* The time scales for such processes range from nanoseconds to millennia. Perhaps the most intriguing examples of the latter are the pre-biotic chemical reactions that led to the genesis of life. It is widely believed that external delivery of pre-biotic material formed in interstellar molecular clouds may have been an important step in this process. If an interstellar chemical process is viable it must be demonstrated that the requisite molecules can be formed and, perhaps, more importantly, chirality can be achieved. Chirality of the products is essential for the origin of life, as we know it. To perform such measurements requires a means to determine the chirality of the product, *in-situ*. Natural circular dichroism at the N(K) and O(K) edges has the potential to achieve this, but only if the flux in 400-600 eV range can be significantly enhanced. This example emphasizes how enhancing the polarization capabilities on 4-ID-C could also be applied to discoveries in fields well beyond magnetism.

**Added Value of the Mid-term Upgrade**

We propose to upgrade the beamline optics and end-stations at 4-ID-C to provide 1–2 orders of magnitude greater photon flux over the present configuration, and to provide enhanced sample environments to users. The proposed upgrades are three-fold: new optics and detectors to improve flux and count rates, new optics to improve stability, and new end-stations to provide enhanced and more flexible sample environments to the users.

- *Optics upgrades to improve flux*

The present optical design of 4-ID-C is a carryover from the beamline's original installation in Sector 2. This design used a spherical grating monochromator and Rh-coated mirrors to achieve a useful energy range of 500-3000 eV. Since the move of this beamline to Sector 4 and installation of the circularly polarizing undulator (CPU), user demand has centered almost exclusively upon the lower part of this energy range (500-2000 eV), which encompasses resonances pertinent to transition metal oxides, rare earths intermetallics, and semiconductors. The original monochromator design, however, compromised efficiency at lower energies (<1000eV), where most of the users work, in order to be able to access higher energies (>2000eV). More efficient optical designs based on varied-line-spacing plane gratings have been built and tested at other synchrotron facilities, since the original commissioning of this beamline. A design analysis of the current optics has demonstrated a clear opportunity to re-task the 4-ID-C beamline to serve the energy range of 500-2000 eV with improvements in flux of 1–2 orders of magnitude at the lower end of this range where the oxygen K and transition metal L edges lie. It may also be possible, with separately planned upgrades to the CPU, to extend the useful energy range of the beamline down to 400 eV, allowing the N K edge to be accessed.

- *Optics upgrades to improve stability*

The canted undulator geometry in Sector 4 uses a permanent magnet dipole to separate the CPU (soft x-ray) and a linear 3.5cm undulator (hard x-ray) beams by 270 microradians. The 4-ID-C beamline then has two optics, a horizontal plane mirror and a horizontally focusing mirror to provide an additional separation of 4.4° between the canted x-ray beams. When the hard x-ray undulator is tuned for low energies (2.8-3.5 keV), however, a significant portion of its high-energy x-ray emission intercepts the first 4-ID-C mirror, resulting in heating, instabilities, and loss of flux on 4-ID-C. These mirrors are also the oldest optics on the beamline, and since they were installed improvements have been made in mirror fabrication such that significantly lower slope errors are now available. We propose to replace these optics with newer, higher quality mirrors, and to use internal water cooling on the first mirror to provide significantly better thermal stability. Furthermore, modifying the front end of 4-ID to permit a full 1 milliradian canting between the two beams, would aide in mitigating cross-talk issues between the two beamlines.

- *Instrumentation upgrades to improve sample environments*

The present instrumentation on 4-ID-C consists of 4 experimental end-stations arranged in series. A low-field (0.1 T) XMCD/scattering chamber with 20K base temperature and a high-field (7 T) XMCD/scattering chamber with 2.6K base temperature are permanently installed on the beamline. Two additional chambers are alternately placed behind these chambers: a photoemission microscope (PEEM) and a surface analysis chamber with x-ray photoemission spectroscopy (XPS). In a separate upgrade (not included here) the low-field chamber will be replaced with an intermediate-field (0.8 T) octupole magnet chamber with integrated scattering detector. Here, we propose to add a new high-field magnet chamber to achieve fields above 10 T, and temperatures below 1 K. In addition, multi-element Si drift diode detectors are now available which promise improvements in fluorescence count rates of a factor of 5–10 and significant increases in the x-ray resolution at low energies. Such a magnet/detector would open up entirely new types of physics, such as quantum phase transitions and heavy-Fermion physics, to soft x-ray spectroscopy studies. Finally, we propose to add a new instrument combining higher-resolution XPS, X-PEEM, and scanning probe microscopies (X-STM, AFM) to be permanently installed on 4-ID-C. Such a chamber would provide unprecedented opportunities for the study of magnetism at surfaces on length scales from 1Å to 1µm.

**Expected user communities**

The existing users of 4-ID-C form a broad international community of researchers primarily from academic, national laboratory, and industrial backgrounds. A partial list is given in "Partnerships and user interest". The majority are not traditional synchrotron users, but instead are typically experts in sample preparation and fabrication, and are specialists in one particular area of materials physics. All of these users would benefit from the proposed upgrades through the ability to take data faster, thus the beamline would be able to attract and accommodate more such users. Furthermore, the increased intensity would enable or enhance studies of more dilute magnetic systems such a doped semi-conductors, and time-resolved of structure materials such a spin-valve systems.

**Enabling technologies and infrastructure**

- Varied line spacing plane monochromator grating.
- Low figure-error and internally cooled mirror optics.
- 10T magnet with sub-1K cryostat.
- Multi-element Si drift diode detector.
- XPS/PEEM/STM end-station (including new energy analyzer, PEEM optics, and scanning probe microscope).

**Partnerships and user interest**

Prof. William E. Bailey, Applied Physics Dept., Columbia University
Dr. Dario Arena, NSLS, Brookhaven National Laboratory
Dr. Matthias Bode, Center for Nanoscale Materials, Argonne National Laboratory
Prof. Jacques Chakalian, Dept. of Physics, University of Arkansas
Prof. Bruce Wessels, Materials Science Dept., Northwestern University
Prof. Vitali Methlusko, Electrical Engineering, University of Illinois-Chicago
Dr. Scott Chambers, Pacific Northwest National Laboratory
Dr. Darrell Schlom, Dept. of Materials Science, Pennsylvania State University
Dr. John Mitchell, Materials Science Division, Argonne National Laboratory
Dr. Ken Gray, Materials Science Division, Argonne National Laboratory
Prof. Dr. Bernhard Keimer, Max Planck Institute – Stuttgart
Prof. Jim Eckstein, University of Illinois at Urbana-Champaign
Dr. Anand Bhattacharya, Center for Nanoscale Materials, Argonne National Laboratory
Prof. Sunhil Sinha, Dept. of Physics, University of California, San Deigo
Prof. Tom Callcott, Dept. of Physics, University of Tennessee, Knoxsville
Prof. Omar Chmaissem, Dept. of Physics, Northern Illinois University
Prof. Bogdan Dabrowski, Dept. of Physics, Northern Illinois University
Prof. Ying Liu, Dept. of Physics, Pennsylvania State University
Dr. Maria Laguna-Marco, University of Zaragoza, Spain
Prof. Song Jin, Dept. of Chemistry, University of Wisconsin at Madison
Dr. Annick Froideval, Paul Scherrer Institute
Dr. Xiao-Min Lin, Center for Nanoscale Materials, Argonne National Laboratory

**Industry and technology transfer**

None is foreseen at this time.

**Budgetary profile**

Beamline Optics Upgrades:

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| Internally cooled first mirror | \$200k |
| Low figure error mirror | \$ 60k |
| VLS monochromator and M3 mirror | \$600k |

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| | | $860k |
| High-magnetic field end-station | | |
| 10 T superconducting magnet | $350k | |
| $^{3}He$ low temperature cryostat | $100k | |
| Multi-element Si drift diode detector | $200k | |
| | | $650k |
| XPS/PEEM/STM endstation | | |
| Chamber with electron spectrometer | $200k | |
| Elmitec high-resolution PEEM | $200k | |
| Omicron UHV Variable Temperature STM | $300k | |
| | | $700k |
| **Total:** | | **$2,210k** |