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XAFS and micro-XAFS at the PNC-CAT beamlines

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The Pacific Northwest Consortium Collaborative Access Team (PNC-CAT) is constructing bending magnet and insertion device (ID) beamlines at the Advanced Photon Source. Both beamlines will be heavily used for XAFS and micro-XAFS experiments. This paper summarises their capabilities, and initial operational experience with the ID line. The ID beamline is based on APS undulator A, and has been running in an initial commissioning configuration since May 1997. Currently the undulator and monochromator energies can be scanned in synchrony for EXAFS measurements, and we have obtained focused beams as small as 0.7 micron using tapered capillaries. Initial testing has also begun on a MBE/UHV system for in-situ surface XAFS.

Keywords: beamline, Advanced Photon Source, micro-XAFS

1. Introduction

The PNC-CAT was formed to develop insertion device (ID) and bending magnet (BM) beamlines on a sector of the Advanced Photon Source. These lines are designed to be multipurpose, with an emphasis on spectroscopic measurements and microbeams. XAFS and micro-XAFS are often the techniques of choice for environmental studies, a major component of our research. The insertion device is the APS undulator A covering the range of 3-12 keV for the first harmonic, and higher energies with the third and higher harmonics. It is expected that the lower energy range will decrease to 2 keV when a smaller gap vacuum chamber is installed. For the ID line we are currently in the late stages of commissioning, while the BM line is in procurement with about 80% of the initial components ordered or in hand. This paper will concentrate on the ID line while briefly outlining the planned BM line design.

2. Beamline Hardware

Figure 1 shows a sector layout along with the configuration of the optical components in the first optical enclosure (FOE) for the insertion device (ID) beamline. The monochromator is based on a fixed output design from the BESSRC CAT (Ramanathan et. al. 1995). It employs a differentially pumped system with the main rotation stage (Huber 430) in a vacuum of about 10^{-7} torr, and the crystal mounts in a UHV compatible chamber which typically operates at $5x10^{-9}$ torr. We have added a Heidenhain ROD 800 encoder mounted directly on the Huber 430 for precise angle calibration. The first crystal is directly cooled with flowing liquid nitrogen, and the second crystal is indirectly cooled by copper braids to about –80° C. This removes most of the lattice parameter mismatch, and helps maintain a fixed output beam. The monochromator uses Si (111) crystals, which provide an energy range from 3-27 keV. Planned is a new version of the monochromator containing both Si (111) and Si (311) crystals. Crystal change will then be a simple translation of the monochromator and the upper energy limit will be approximately doubled. The new version will also provide more strain relief to the liquid nitrogen lines connecting to the crystals. Currently we see an approximately 50 μm vertical vibration of the beam position at 20 m from the monochromator (i.e. about 1 μrad vibration of the crystal) that seems to be from vibrations in the flexible liquid nitrogen lines. For our unfocused beam this is of only small consequence, but will need to be improved when the beam is focused.

The variation in temperature of the second crystal is approximately 10 °C, as the heat load on the first crystal varies with undulator gap changes. We observe a drift in crystal tuning larger than can be explained by the temperature change. This is likely due to Compton scattering from the first crystal heating components of the second crystal mount. We plan to increase the Compton scattering shields to improve this. In the meantime, we find that a simple feedback system adequately removes the long-term drifts in tuning. This consists of a split-ion-chamber beam-position-monitor immediately preceding the experiment. A PID type controller is used to vary a piezo adjustment of the second crystal tuning to maintain a fixed beam position in the hutch. In principle, the same system could be used to counteract the affects of the crystal vibration, but the noise and bandwidth of the current system need to be improved before this level of control can be achieved.

A focusing mirror based on a bent toroid will be added in the near future. It can be adjusted to focus the beam anywhere in the two end stations. The expected focus size is about 200x500 μm with a high-energy cutoff of about 30 keV. Currently we are running with an unfocused spot of about 1x3 mm. Typical fluxes are around 10^{13} ph/sec in a bandwidth $\delta E/E=1.5x10^{-4}$.

The layout of the BM line is similar to the ID line except that the focusing mirror is replaced by a sagitally bent second crystal. This will focus about 4 mrad of the horizontal beam. In addition, the line will have a vertically focusing/collimating white beam mirror in front of the monochromator. It is a singly bent flat mirror with a variable radius allowing two operating modes. In the collimating mode, it will allow us to collect the full vertical divergence of the beam without sacrificing any energy resolution. Alternatively, for experiments not as sensitive to energy resolution, the beam can be focused to a vertical focus of less than 200 μm.

3. Beamline controls and software

Beamline controls are based largely on EPICS, used extensively at the Advanced Photon Source.(Mooney et. al., 1995) It is used on our beamline to interface VME-based motor controllers and scalers, and will also be used to interface a Ge 13-element detector as well as other hardware. Control functions outside the EPICS environment are currently performed by software developed with LabVIEW, which is also used to develop some of the EPICS clients used at the beamline.

XAFS scans on the ID line are performed by stepping the monochromator energy and undulator gap together. The EPICS scan software executes on the beamline VME crate, with workstations monitoring progress of the scan over the network. Coordination between the beamline crate and the undulator is handled by EPICS without intervention by the workstation client. The net result is low overhead due to software, which we have

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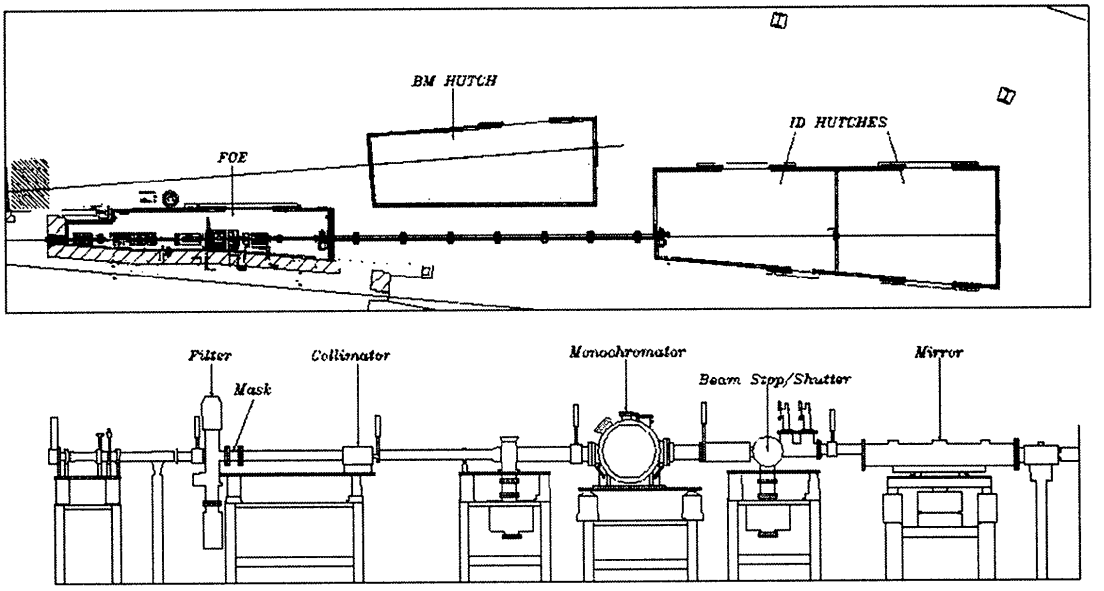

Figure 1

Sector layout showing the ID and BM hutches along with the ID optics enclosure. At the bottom is the optics layout for the ID line.

estimated as ~ 20msec per data point. The scan software is also capable of Quick ("on-the-fly") XAFS and this is planned for the future. This will require synchronisation between the motions of the monochromator and undulator, an issue currently being addressed by the APS

Micro-imaging has been performed with software utilising EPICS for motor control and a PC-based analog input board for data acquisition. Scans are conducted on-the-fly, with acquisition of pixel data triggered by motor control pulses. Some degree of signal integration is achieved by matching amplifier time constants to the sample scanning speed, and multiple images are averaged and viewed as they are acquired. Future plans include extending the capabilities to simultaneous multi-element imaging using a 13-element Ge detector.

4. Experimental equipment

4.1 XAFS equipment

The beamlines will have the full complement of experimental equipment, including ion chambers for transmission and fluorescence, filters and slits, low temperature refrigerators, and a 13-element solid state detector. It is expected that for bulk samples the beamlines will often provide more fluorescence signal than can be handled by the detector. We are therefore beginning to develop some crystal-based analysers. The 13-element detector should still be useful for micro-XAFS experiments where the incoming flux is smaller.

A variety of EXAFS experiments has been run to assess the monochromator operation. During this process a number of minor instabilities have been identified and eliminated. This process is ongoing, and as mentioned we still have a residual vibration from the liquid nitrogen flow. However, the instabilities are now at a level where good EXAFS can be obtained for a variety of samples. Fig. 2 shows some data that illustrates this. It is fluorescence data from a 1 mMol $FeSO_4$ solution obtained using the filter-slit combination with an ion chamber detector. This measurement also pointed up a problem with sample damage from the intense undulator radiation. The beam caused bubble formation in the sample presumably from the breakdown of the water. The bubbles limited the data collection time to about 20 min. before the sample had to be moved.

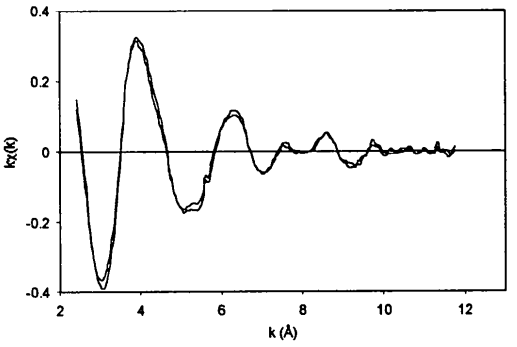

Figure 2
EXAFS from a 1mMol. solution of $FeSO_4$. Two scans taken at 2 sec./point.

4.2 Microfocusing optics

Several types of microfocusing optics are under development. Tapered capillary concentrators have been used to obtain sub-micron resolution images as shown in Fig. 3. For this image the sample was mounted on a high precision stepping motor scanning stage with 50 nm resolution. We have also begun commissioning a Kirkpatrick-Baez (K-B) mirror assembly based on a design by

the CARS CAT (Eng et. al., 1998). This will provide focal spots in the 1-10 μm range. The smaller focal spots require the use of shorter mirrors that are less efficient at collecting the beam. To increase the efficiency we plan to use short capillaries to condense a 10 μm focus down to submicron sizes. In this case the capillaries act as enhanced pinholes with gain of 5-10x in the flux density over a simple pinhole placed at the mirror focus. An ordinary pinhole of these dimensions would also be nearly impossible to fabricate. Testing of this configuration awaits final commissioning of the K-B mirror.

Figure 3
Image of a copper grid obtained with a capillary microbeam. The capillary input size is 150 μm, output size is 0.7μm, and the capillary is 0.5 m long. The x-ray energy is 10 keV, and the sample to grid distance is about 150 μm. At this energy and spacing the resolution of the image should be less than 1 μm. The image size is ~50X50 μm.

4.3 Molecular Beam Epitaxy and Other experimental equipment

A UHV system has been constructed to permit the *in situ* study of surfaces, interfaces and epitaxial growth relevant to environmental science or material science. It is configured for SEXAFS, polarization-dependent glancing incidence XAFS, reflectivity and x-ray standing wave measurements. The *in situ* growth capability includes three OMICRON EFM evaporators, quartz thickness monitor, RHEED optics and a cylindrical mirror analyzer. The sample manipulation is through a 6-axis goniometer with sample temperature control between -150 °C to 1200 °C.

The beamlines will also be equipped for diffraction and DAFS experiments. We are building a small 4-circle instrument that can be shared between the two beamlines. In addition, a dedicated large surface diffractometer is planned for the last station on the ID line. This will have a detachable UHV/MBE chamber to allow in-situ sample treatment or growth. These systems are still under design and will be discussed in detail in future publications.

An X-ray waveplate system will be available for use with or separate from the MBE system. Using a diamond (220) single crystal, the waveplate system will permit, for example, quarter or half wave operation over the energy range 5 keV to 12 keV. This will allow dichroism studies from the vanadium K-edge to the gold L_{III} edge.

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