Canted Undulator upgrade at Sector 20 providing a high throughput dedicated Micro-XAFS station

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Introduction

A canted undulator expansion will double the available beamtime and allow the operations at Sector 20 to be optimized. Sector 20 began as a CAT with a specialization in spectroscopy while supplying a wide variety of other capabilities. With the transfer to XOR we have been working to concentrate on our strength in spectroscopy. **Our long term goal is to be the premier sector for x-ray spectroscopy at the APS.** Our capabilities for micro-spectroscopy using KB mirrors are an important component of the current program, and we have highly utilized stations on both 20-ID and 20-BM. We also have some unique capabilities for time-resolved spectroscopy, low energy spectroscopy using x-ray Raman (LERIX), and spectroscopy combined with UHV surface science. In addition to doubling the beamtime available to these instruments, the proposed canted undulator expansion allows us to enhance the capabilities and operational efficiency of all of these stations.

We have chosen to create a dedicated microprobe station that will exclusively utilize one of the undulator beams. A microprobe is a good candidate for a side station, and it will serve a large and growing user base. On the original beam line, an additional hutch will allow reconfiguration of the stations into a more convenient and efficient arrangement for our other capabilities. Also, some of the same KB mirror technology planned for the new microprobe will be provided for them to enhance their performance and range of application.

Science

Worldwide there is high demand for x-ray microprobes capable of high quality micro-XAFS and imaging. This comes mainly from the Environmental and Earth Sciences communities, but important applications also exist in areas such as materials science and catalysis. Whenever samples are inhomogeneous, the microprobe can be used to locate interesting regions for detailed analysis by micro-XANES, micro-EXAFS and micro-diffraction. The microprobe can also be used to study micron scale samples as found, for example, in diamond anvil high pressure cells.

The existing part-time x-ray microprobes have been invaluable in a variety of areas related to the basic research needs identified by DOE. Already the microprobe has contributed to major advances in geosciences related to cleanup of DOE sites. Both laboratory and field samples have been used to determine the transport, binding, and reactivity of actinides and other hazardous elements in a variety of sediment systems. Toxic and radioactive elements released into complicated environments can be located and studied in detail. Cleanup efforts can be monitored and evaluated. Highly radioactive materials such as Hanford tank waste have been studied. The microprobe can separately study different components of these complex materials, and allows the use of small quantities that are safer to handle.

Scientists from the EPA and equivalent agencies in other countries have also used the microprobe to monitor pollution problems at their sites. A good example is arsenic in the environment, which has been the focus of many national and international studies. The microprobe has been used to look at As in the soil, in plants, in animal tissues and even in human foodstuffs. In all of these cases it is necessary to image the samples to determine where the As is located, and to run XANES and/or EXAFS to determine its chemical state. This information is often needed for concentrations at the ppm level. Micro-diffraction can then be used to determine the minerals to which the As is binding. Only an x-ray microprobe can provide the needed information to make progress in these types of important societal problems.

In materials and catalysis science, the microprobe has been applied to energy related problems such as solar cell materials and fuel cell electrodes. For polycrystalline Si solar cell material, the goal was to locate and identify small impurity particles. These measurements demonstrated that isolated nanoscale (<20nm) particles could be detected and XANES obtained for somewhat larger particles. The new microprobe will push down these size limits, and will allow much larger areas to be surveyed to locate interesting regions. The fuel cell electrodes were composed of several thin (20 micron) layers of complex ceramics in contact with stainless steel. The performance of these electrodes can be degraded by Cr diffusion from the stainless steel contacts. An x-ray microprobe can image the Cr distribution within the electrodes, and use micro-EXAFS and micro-diffraction to identify the Cr phases formed.

The utility of x-ray microprobes is highlighted by the number of beamlines at the APS that have microbeam capabilities. These include the new nanoprobe beamline. In this case the smallest beams are achieved using zone plate optics. For spectroscopic applications, achromatic focusing mirrors (KB mirrors) are more useful by allowing large and rapid energy changes. Currently there are several KB mirror based microprobes at the APS operated on a part time basis, but no full time KB mirror based facility. This is in contrast to most other third generation sources that have or are planning dedicated micro-spectroscopy beamlines.

While the primary goal of this project is the construction of the new x-ray microprobe, it will also enhance the science at the existing sector 20 instruments by making more beamtime available, and through enhanced focusing. We plan to implement the same KB mirror focusing technology for the x-ray Raman (LERIX) instrument and the time-resolved EXAFS station. In the case of LERIX a smaller spot will allow use of diamond anvil cells, which will allow the measurement of low-Z edges at high pressures. Smaller spots also enhance the performance of the backscattering analyzers, allowing faster data collection and higher resolution.

The time-resolved EXAFS station currently depends on x-ray microfocusing to match the x-ray beam size to the small laser beams required for operation at high repetition rates (272 kHz to match the single bunch repetition rate). New focusing mirrors should enhance the flux by at least 5x. When combined with multi-element detectors, they should enhance count rates by as much as an order of magnitude. This will speed up the measurements on presently accessible samples, and allow the measurement of much more difficult samples. A good example is individual micron sized gates in phase change memory devices. Phase change devices use electrically driven heat pulses to cause a phase change in the memory element, and could form the basis for the next generation of static memory. Since the thermal properties are dependent on the device size, it is important to make measurements at the same size scale as for actual devices.

Added Value of the Mid-term Upgrade

In addition to improved performance, a dedicated microprobe station allows for better operational efficiency by reducing setup and alignment effort. It will more then double the beamtime available to general users over the current station. It also makes it simpler to develop a large suite of specialized sample environments and holders.

The proposed microprobe will provide an order of magnitude improvement in overall efficiency by taking advantage of better detectors, optics and software. This can provide not only much more efficient data collection, but a qualitative improvement in the experimental results. Currently long acquisition times mean that a typical experiment can only measure a few locations in a few samples. There is always the issue of whether these limited experiments are truly representative of the whole range of possibilities in complicated environmental samples. It is planned to allow a user selectable beam size from 0.5 to 10 microns by adjusting an intermediate slit. By providing an easily selectable beam size, the new microprobe will allow the rapid survey of large sample areas, followed by high resolution studies of interesting regions.

The resolution limit of 0.5 microns is an improvement over our current limit of 1-2 microns. Even better resolution is possible from KB mirrors. However, the performance of the microprobe is ultimately limited by the brilliance of the source, and smaller beams imply less flux. This design value is a good compromise of resolution and flux, and also matches well the needs of our user community. To take full advantage of small beams requires samples of similar thickness, something that can be difficult to achieve for many environmental samples. The 0.5-10 micron range also provides a good complement to nanoprobe beamlines where the ultimate resolution can be provided when needed.

The improved beamlines will also be well positioned to take advantage of a potential ERL upgrade. The high brilliance of the ERL will make possible smaller microprobe beams without a corresponding flux loss. With appropriate fill patterns the time-resolved station would be capable of sub-ps XAFS with relatively minor modifications.

Expected user communities

The existing microprobe has already developed an active user community. As mentioned, it is expected to be a major resource for the Environmental and Earth Sciences communities. During the previous 6 cycles (2006-2 through 2008-1) of part time use, there have been 51 separate microprobe experiments carried out by 24 different user groups. Typically there have been at least 2x more requests for the microprobe then the total available beam time. The improved performance of the new beamline should attract even more users to apply.

Similarly the user communities for the time-resolved XAFS station and LERIX spectrometer have shown strong growth. In 2008-1 the requests for these two instruments was about equal to the total time available at 20-ID. This does not include time needed for the two UHV stations also supported at 20-ID. As both of these stations have only recently opened to general users, we expect the user demand to continue to grow.

Enabling technology and infrastructure

The improved performance is made possible by several recent developments. Mirror fabrication has shown steady improvement, and it now appears possible to get reasonably large (300 mm) mirrors with sub-microradian slope errors. To collect the full beam with a 0.5 micron focus, we will need about 0.5 microradian slope errors.

Improved detectors will also be important. Large arrays of Si drift detectors are currently the preferred option. They have higher count rates, reduced escape peaks, and better resolution then the current Ge detectors. These are now becoming available from several sources. The goal is to collect up to 20% of the available solid angle, approximately 5x more then is typically used now. It has also been recently demonstrated that the detector output can be processed in real time, providing the user with a live display of the elemental concentrations even for complex samples with many overlapping fluorescence lines. This is a critical capability that should be provided at the new microprobe. Ultimately, to take full advantage of the available signal, it will be necessary to implement very many small detectors covering a large solid. The most cost effective and efficient approach would be a pixel array detector for spectroscopy. With recent technology advances this seems an achievable goal for an APS detector development program and would benefit many beamlines.

The canted undulator option is now in standard operation at several beamlines. For best performance we would also like to see accelerator improvements to allow higher currents and longer undulators. That way the new canted undulators could each have higher performance then our current undulator A.

Partnerships

The goal of this project is to provide a state of the art micro-spectroscopy beamline to be used by the general user community of the APS. We have not sought out partners for the beamline development effort. We have, however, discussed possible partnerships for detector development with Chris Ryan (CSIRO) and Peter Siddons (BNL) in developing a version of their multi-element detector and processing electronics for our beamline.

Industry and technology transfer

We do not anticipate any technology transfer.

Estimated budget

A detailed budget can be provided. About $4800K in capital equipment will be needed spread out over approximately 3 years. It can be broken down into 5 main categories:

1. New hutches and shielded beam pipe – 900K
2. New beamline optics (mirrors and monochromator) – 1200K
3. New detectors and electronics – 800K
4. Misc. (utilities, vacuum components, motors, LN2 pump) – 700K
5. Canted undulator and front end modifications – ~1200K for standard system, but we would prefer a more advanced system (longer straight section and undulators) with higher performance.

To operate the beamline we will need at least two new staff members. We would like at least one of the new staff to be hired at the start of the project to help oversee construction and to become familiar with the beamline equipment. We will also need of order 1.5 FTE (0.5 FTE/yr) of engineering and technician help during the project.