X-ray Microprobe (XMP)

Beamline Description

The XMP beamline is currently used both as an X-ray absorption spectroscopy beamline and an X-ray microprobe beamline. The beamline was reconfigured in 2002 to be very similar to the DCM beamline on port 5b (accepting 2 mrad), except for the added microprobe capability.

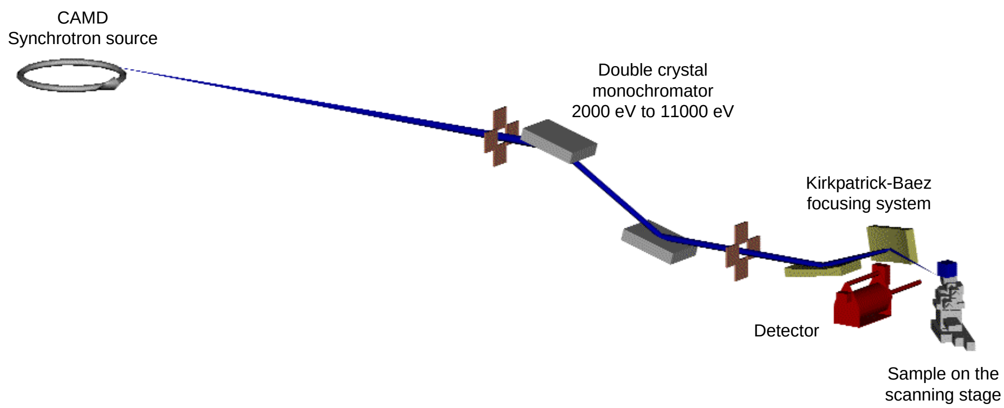

Figure 1
Schematic Drawing of the XMP beamline

Historically, the beamline was built as a collaborative effort between the Argonne National Laboratory and CAMD. The micro-focusing optics was built with the help from Mark Rivers and Peter Eng from the GSE-CARS group at the University of Chicago. A schematic drawing of the beamline and detailed one of the microprobe are shown in Figures 1.
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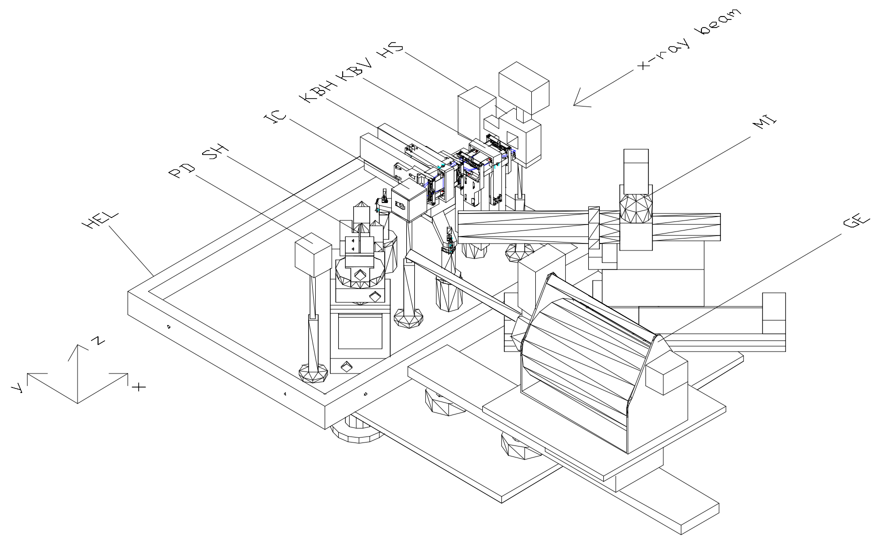

Figure 2
XMP Microfocusing Optics

The incident white or monochromatic x-ray beam (indicated by an arrow, Figure 2) passes through a motorized 4-jaw entrance slit, defining the beam size, which illuminates the vertical and horizontal focusing mirror of the Kirkpatrick-Baez system (KBV, KBH) . After being reflected off the KB system, the beam travels through a 1" long ion chamber (IC) and impinges as a small spot on the sample. The sample, with a typical size of 45 mm x 15 mm, is mounted to the sample holder (SH), which is located on the motorized goniometric sample stage assembly. The sample stage assembly, consisting of 2 translation stages (x, z) and 2 rotation stages (phi, chi), is positioned under 45˚ to the incident beam. To collect data in transmission a photo-diode is positioned behind the sample in the direct beam. Fluorescence data, on the other hand, is acquired with an energy-dispersive germanium detector, which is also coupled in to the helium atmosphere. To obtain an optical microscope image of the sample, a long-working distance microscope (MI) is pointed through a view port under 90˚ to the sample.

The initial design consisted of a plexiglass chamber around the micro-focusing system which allowed low-Z measurements when it was filled with helium. In 2005 a large metal chamber replaced the original plexiglass chamber which allowed operation at moderately low vacuum in addition to helium atmosphere. Measurements have been made at phosphorus and sulfur K edges in 2006.

Table 1 shows the crystals available for the monochromator at the XMP beamline. The crystals can be changed in a matter of hours.

Table 1
Monochromator Crystals

Crystal	2*d (Å)*	E (eV)
InSb (111)	7.481	1830 - 6400

Si (111)	6.271	2180 - 7640
Ge (220)	4.00	3420 - 11980
Si (220)	3.840	3560 - 12470
Ge (422)	2.306	5930 - 20770

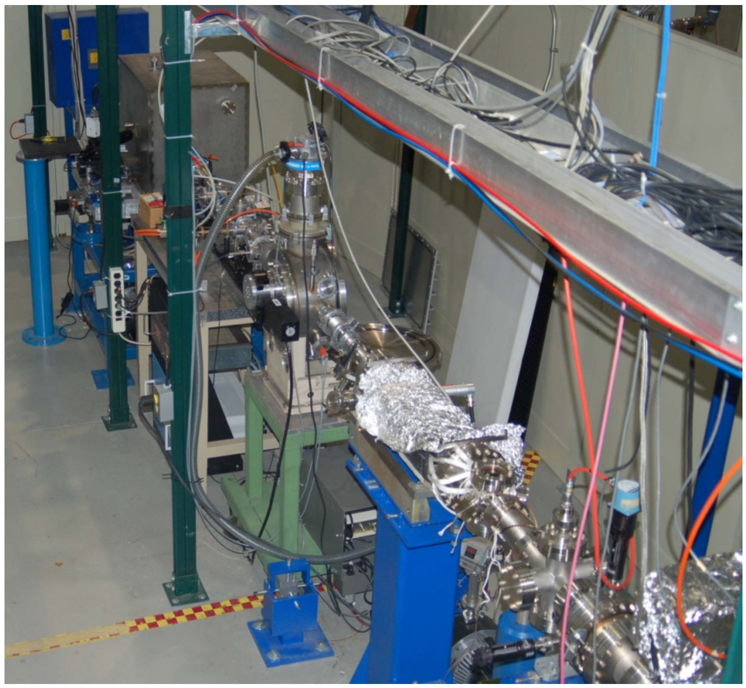

Figure 3
XMP Experimental Hutch

Figure 3 shows a photograph of the X-ray microprobe beamline. The double crystal monochromator is in the middle. At the top middle is the experimental chamber.

Figure 3
XMP Experimental Chamber

A new PC based beamline control and data acquisition system has been installed to replace the original VMS/VAX Alpha system.

Current Status

The beamline is operational for standard XAS experiments and as an X-ray microprobe. There has been a surge of interest in using the X-ray microprobe in 2007. In early 2000, a spot size of 20 μm (vertical) x 40 μm (horizontal) could be routinely achieved. At present (June 2007) that size is ca. 80μm x 50 μm. The mirrors are now almost ten years old and need replacement. This should allow attainment of previous spot size. In addition, the brightness of the ring has improved over the past ten years. This should result in better flux at the XMP beam line.

Selected Publications

de Silva, R.M., Palshin, V., Hormes, J., Fronczek, F.R., and Kumar, C.S.S.R. (2007) Investigation of the Influence of Organometallic Precursors on the Formation of Cobalt Nanoparticles. Journal of Physical Chemistry C.

Modrow, H., Palina, N., Kumar, C.S.S.R., Doomes, E.E., Aghasyan, M., Palshin, V., Tittsworth, R., Jiang, J.C., and Hormes, J. (2005) Characterization of Size Dependent Structural and Electronic Properties of CTAB-Stabilized Cobalt Nanoparticles by X-ray Absorption Spectroscopy. Physica Scripta, T115, p. 790-793.

Molders, N., Schilling, P.J., Wong, J., Roos, J.W., and Smith, I.L. (2001) X-ray Fluorescence Mapping and micro-XANES Spectroscopic Characterization of Exhaust particulates Emitted From auto engines Burning MMT-Added Gasoline. Environmental Science and Technology, 35(15), 3122-3129.

Palshin, V., de Silva , R.M., Hormes, J., and Kumar, C.S.S.R. (2007) Effect of Precursor on the Electronic and Geometric Properties of Cobalt Nanoparticles Investigated by Co-K XANES and EXAFS. In B. Hedman, and P. Pianetta, Eds. X-Ray

Absorption Fine Structure - XAFS13, 882, p. 758-760. American Institute of Physics, Palo Alto, California.
Roy, A., Moelders, N., Schilling, P.J., and Seals, R.K. (2006) Role of an amorphous silica in portland cement concrete. Journal of Materials in Civil Engineering, 18(6), 747-753.
Song, Y.J., Modrow, H., Henry, L.L., Saw, C.K., Doomes, E.E., Palshin, V., Hormes, J., and Kumar, C.S.S.R. (2006) Microfluidic synthesis of cobalt nanoparticles. Chemistry of Materials, 18(12), 2817-2827.
Zinoveva, S., Datta, P., deSilva, R.M., Louis, R., Kumar, C.S.S.R., and Hormes, J. (2007) Time resolved observation of the wet-chemical synthesis of nanoparticles by spatially-resolved X-ray spectroscopy. In E. Morikawa, J. Scott, and A. Roy, Eds. Synchrotron Radiation Instrumentation 2007. Elsevier, Baton Rouge, Louisiana.