

## Micro-EXAFS at X-26A

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Beamline: **X26A**

**Introduction:** Acquisition of EXAFS spectra of bulk samples has become relatively routine in recent years and there is increasing interest of collecting EXAFS spectra at micro-probe beamlines. Micro-XANES is routine at X-26A using Kirkpatrick-Baez (KB) mirrors to achieve focal spot sizes of approximately 10  $\mu\text{m}$ . KB mirrors also reject higher order harmonics as well as fix the focal spot position when using a variable exit channel cut monochromator. In collecting EXAFS spectra with a micro-beam, there are many issues to be considered. Mechanically ground particles are often 1 to 10  $\mu\text{m}$  in diameter, which adds to the challenge of achieving a uniform sample at the size of the X-ray beam. EXAFS oscillations are typically less than 1% of the total signal and a sample stage drift of less than 100 nm can negatively affect the EXAFS signal from a particle of similar size to the X-ray beam. Also, small particles may be oriented and thus give rise to polarized-EXAFS signals.

**Methods and Materials:** A number of reference minerals for which bulk EXAFS have been previously collected were investigated by micro-EXAFS using transmission and fluorescence detection. The sample stage, which is usually oriented at 45 degrees to the incident beam, was rotated to 35 degrees to achieve the magic angle between the surface normal and the plane of polarization of the incoming beam and the sample to eliminate orientation effects of micro-crystallites.

**Results:** High quality transmission micro-measurements that match bulk EXAFS measurements have been achieved proving the potential for micro-EXAFS. However, particle size was related to spectral quality in that the best spectra were achieved when particle sizes were much smaller than the size of the probe beam. Under these conditions, acquisition was similar to that of statistical averaging of bulk EXAFS spectra. Spectral quality was extremely erratic when particle sizes approached the size of the beam. Additionally, high quality spectra were achieved on samples that were homogeneous on a scale much larger than the size of the beam (i.e., reference foils). Fluorescence spectra were not highly dependent on sample homogeneity. Also, self-absorption is less of a problem on samples with a less than 10  $\mu\text{m}$  in thickness. A simple methodology of imaging a small X-Y array to ensure sample homogeneity within particles that are slightly larger than the size of the X-ray beam was sufficient to achieve satisfactory fluorescence data.

**Conclusions:** In general, micro-EXAFS can be conducted in transmission and fluorescence at X-26A, but the experimental methodologies must be carefully defined to get satisfactory results.