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Use of the spindle stage for orientation of single crystals for microXAS: Isotropy and anisotropy in Fe-XANES spectra

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ABSTRACT

A new method has been developed to orient 50–100 μm size single crystals to obtain XANES or EXAFS spectra along any crystallographic orientation relative to a polarized synchrotron X-ray beam. The method uses a modified spindle stage and an X-ray goniometer head to hold a crystal and the glass fiber on which it is mounted. The orientation of the crystallographic axes within the crystal can be determined by X-ray diffraction in all cases or with the use of the polarizing light microscope if the minerals are biaxial. Once an optical orientation direction has been located, it can be oriented parallel to the polarization direction of the synchrotron source for spectral acquisition.

Mineral samples representing the three optical classes (i.e., isotropic, uniaxial, and biaxial) were oriented along their *a*, *b*, and *c* crystallographic axes, and Fe-XANES spectra were obtained. In all cases the interactions of polarized X-rays with the crystal structures are analogous to the interactions of visible light (e.g., as represented by refractive index). For the isotropic almandine sample the XANES spectra were similar at all orientations. For the uniaxial buergerite and scapolite samples the spectra were similar along the *a* and *b* axes and different along *c*. A detailed set of spectra acquired at multiple (known) optical orientations in the scapolite crystal demonstrates that spectral features vary in intensity as a function of orientation, with maxima and minima when the X-rays are polarized along optical orientations. For the biaxial fayalite sample, the XANES spectra differed along all three crystallographic axes, as expected by analogy to optical properties.