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Fe XANES Spectra of Iron-Rich Micas

M.D. Dyar (Mt. Holyoke Col.), J.S. Delaney (Rutgers U), S.R. Sutton (U Chicago)

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Introduction: Mica group minerals offer a challenge to spectroscopic analysis by any technique because they commonly contain significant amounts of Fe in two different valence states and three structural sites. Micas are commonly zoned at thin section scales, making bulk spectroscopic measurements of their Fe valence and site distribution by wet chemistry of Mössbauer spectroscopy potentially too simplistic. Precise measurements of the oxidation states and site occupancies of Fe in micas, particularly at micrometer scales, have long been a source of frustration for mineralogists, petrologists, and geochemists.

Methods and Materials: Fe-rich mica crystals have been studied using microbeam XANES spectroscopy of the Fe K edges in order to evaluate the dependence of XANES features and resultant $Fe^{3+}/\Sigma Fe$ values on the orientation of the crystals with respect to the polarization of the synchrotron beam. Polished, thinned samples were prepared with cleavages perpendicular to the plane of their thin sections, and these sections were then rotated relative to the horizontal plane of the ring and the 10 x 15 μm beam.

Results: Because the samples and their orientations are exactly the same as those used in a previous optical study of Fe-rich micas, correlations can be drawn between optical and pre-edge features for which the quantum mechanical selection rules that control the transition probabilities are the same. Data show that both pre-edge and main-edge features in these micas exhibit orientation-dependent changes in both peak intensity and energy. These shifts constitute the majority of the error (~±10%) in determinations of $Fe^{3+}/\Sigma Fe$ by pre-edge energy, a problem that will be overcome in future work through techniques for quantification of grain orientations. Results show distinctive orientation-dependent features in the main edge region, from which it will soon be possible to deconvolute contributions from multiple scattering interactions to obtain information about the electronic properties of micas. Data also demonstrate the exciting potential of XANES pre-edge spectroscopy for routine, *in situ* analyses of $Fe^{3+}/\Sigma Fe$ on comparable scales to electron microprobe analyses.

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