

Solving the iron quantification problem in low-kV EPMA: An essential step toward improved analytical spatial resolution in electron probe microanalysis—Olivines

AURÉLIEN MOY^{1,*}, JOHN H. FOURNELLE¹, AND ANETTE VON DER HANDT²

¹Department of Geoscience, University of Wisconsin, Madison, Wisconsin 53706, U.S.A.

²Department of Earth Sciences, University of Minnesota, Minneapolis, Minnesota 55455, U.S.A.

ABSTRACT

The relatively recent entry of field emission electron microprobes into the field of microanalysis provides another tool for the study of small features of interest (e.g., mineral and melt inclusions, exsolution lamellae, grain boundary phases, high-pressure experimental charges). However, the critical limitation for accurate quantitative analysis of these submicrometer- to micrometer-sized features is the relationship between electron beam potential and electron scattering within the sample. To achieve submicrometer analytical volumes from which X-rays are generated, the beam accelerating voltage must be reduced from 15–20 to ≤ 10 kV (often 5 to 7 kV) to reduce the electron interaction volume from ~ 3 to ~ 0.5 μm in common geological materials. At these low voltages, critical $K\alpha$ X-ray lines of transition elements such as Fe are no longer generated, so L X-ray lines must be used. However, applying the necessary matrix corrections to these L lines is complicated by bonding and chemical peak shifts for soft X-ray transitions such as those producing the $FeL\alpha$ X-ray line. It is therefore extremely challenging to produce accurate values for Fe concentration with this approach. Two solutions have been suggested, both with limitations. We introduce here a new, simple, and accurate solution to this problem, using the common mineral olivine as an example. We also introduce, for the first time, olivine results from a new analytical device, the Extended Range Soft X-ray Emission Spectrometer.

Keywords: EPMA, olivine, low-kV, iron, microprobe, SXES, EMPA