American Mineralogist, Volume 107, pages 1532–1544, 2022

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

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Abstract

The use of the field emission gun in scanning electron microscopy permits the imaging of sub-micrometer-size features. However, achieving sub-micrometer analytical spatial resolution in electron probe microanalysis (EPMA) requires both reducing the electron beam size and reducing the accelerating voltage to achieve the desired sub-micrometer interaction volume. The resulting quantification of the first-row transition metals at low accelerating voltage, i.e., below 7–8 kV, is problematic as the main characteristic X-ray lines ($K\alpha$) cannot be excited at these conditions. Furthermore, the use of the $L\alpha$ and $L\beta$ soft X-ray lines for quantification is complicated by bonding and self-absorption effects resulting in not-yet-determined mass absorption coefficients and hence in the failure of the traditional matrix correction procedure. We propose two methods to circumvent these low-kilovolt (low-kV) analysis limitations: using the non-traditional Fe$L\ell$ line and using universal calibration curves for the more traditional Fe$L\alpha$ and $L\beta$ lines. These methods were successfully applied to Fe-sulfide minerals showing accurate quantification results by EPMA at reduced kV, necessary for accurate quantification of sub-micrometer sulfide grains.

Keywords: EPMA, sulfides, low kV, iron, microprobe, SXES, EMPA, X-ray