

Improving spatial resolution for quantitative microanalysis by SEM-EDS using lower accelerating voltage

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Abstract

It is challenging to measure the chemical compositions of very fine-grained minerals. A method with broad utility to measure with high spatial resolution for quantitative microanalysis has not yet been established for geological materials. We are developing such a method using energy-dispersive spectrometry (EDS) in a scanning electron microscope (SEM), evaluating the effects of two corrections to improve the spatial resolution and quantitative accuracy: (1) reducing the accelerating voltage and (2) reducing the probe diameter.

We impose these corrections to counteract the fact that the interaction volume of incident electrons within a mineral expands due to electron scattering, resulting in X-rays being generated from a wider region than the incident probe diameter. It has been demonstrated that the X-ray generation volume is smaller at lower accelerating voltage, so reduction of the accelerating voltage results in an improvement of spatial resolution (e.g., Barkshire et al., 2000; Burgress et al., 2013). Reducing probe current also reduces the size of the X-ray generation volume and improves spatial resolution. Both of our corrections also result in a significant decrease in the signal intensity of the X-ray spectra.

Analyses were performed by EDS (Oxford X-Max^N 80mm² with AZtec) in a HITACHI SU-3500 SEM at the GSJ-lab in the Geological Survey of Japan, AIST. We have evaluated the spatial resolution and the signal intensity of the X-ray spectra for a range of SEM optics and EDS detector settings. A minimum accelerating voltage of 9 kV is required to quantify Fe-oxides using the signal intensity of Fe K α X-rays. Count rates of X-rays and probe currents measured through a faraday cup can be decreased by reducing accelerating voltage, spot intensity or size of aperture. The count rates of X-rays at dead times of ~45% can be processed to yield acceptable quantitative analyses. A spot intensity (condenser lens) of 63 is required to obtain a dead time of ~45% when accelerating voltage is 9 kV and aperture is No. 3 (50 μ m) in the system at the GSJ lab, by setting the longest counting time (processing time of 6) possible in AZtec. A probe current of ~0.6 nA is generated by these SEM settings.

We have so far verified the method through measurement of anhydrous and hydrous natural mineral samples. The analyses reveal that the best possible operating conditions are 9 kV accelerating voltage, spot intensity of 63, aperture No. 3, and processing time of 6. Line scans across (potentially dipping) natural mineral boundaries suggest a maximum spatial resolution of the analyses of ~1 μ m, i.e., analysis of 2 μ m diameter grains is possible. The accuracy of weight percentages of oxides is estimated as a relative difference of < 5% for MgO, Al₂O₃, SiO₂, Na₂O and K₂O and < 10% for Fe-oxides compared to values obtained by WDS or at an accelerating voltage of 15 kV.

In future we will work to increase the accuracy of quantification of iron by obtaining proper standard

spectra, and to measure spatial resolution by a combination of Monte Carlo simulations, which allow theoretical predictions of spatial resolution, a 'knife edge' technique using a sharp edge which allows measurement of lateral analytical spatial resolution, and further line scan analyses across mineral boundaries of known orientation.

Reference

Barkshire, I., et al. (2000). *Microchimica Acta*, 132(2-4), 113-128.

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