

Strategies for Low Accelerating Voltage X-ray Microanalysis of Sub-Micrometer Features with the FE-EPMA

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X-ray microanalyses of sub-micrometer features require that a lower accelerating voltage be used. This reduction in the accelerating voltage reduces the penetrating distance of the beam electrons and thereby reduces the analytical volume. Two general strategies have been proposed for achieving the smallest analytical volume. The first approach involves significantly reducing the accelerating voltage to minimize the electron interaction volume. For many element systems, the optimum accelerating voltage is in the 5-8kV range, assuming a field emission electron gun is used for the analysis. A lower kV will reduce the penetrating depth of the electrons further. However with lower kV, the diameter of the electron beam becomes a critical factor in determining the analytical area, and with a lower kV, the beam diameter becomes larger. Mostly this comes from the higher required beam current used due to the lower X-ray production rate at the lower kV. This low-kV strategy also typically requires a different set of X-ray lines be used, since some of the more commonly used X-ray lines are not generated at these lower accelerating voltages. However, it can produce a very small analytical volume (Fig. 1a).

The second approach involves using a low-overvoltage. First, the X-ray lines of interest are selected, and then an accelerating voltage is used that is only 1-3kV greater than the critical ionization energy of the highest energy X-ray line. This low-overvoltage strategy can produce a larger electron interaction volume, but the electrons quickly decelerate on entering the sample, dropping below the critical ionization energy of the element in question. Therefore they can only generate X-rays very near the surface, even though the electrons continue deeper into the sample (Fig. 1b).

Using the example of the Fe-Ni binary, both the low-kV method and the low-overvoltage method produce similar analytical volumes (Fig. 1). However when it comes to analyzing sub-micron features, there are a number of other factors that need to be evaluated. These would include: (1) minimum detection limits, (2) quality of the matrix corrections, (3) secondary fluorescence, and (4) effects of surface contamination, oxide layers, and carbon coatings. These all play into the overall quality of the analysis. For some of these issues, the low-overvoltage method performs better, in other the low-kV method does. For example, switching to the L-lines for the transition elements can be very problematic. Therefore the low-overvoltage method can have some significant advantages in that it allows the user to pick the better-behaved, X-rays lines. However the low-kV method has real advantages when considering secondary fluorescence and how close one can get to a phase boundary without measuring a fluoresced element from the neighboring phase. Again using the Fe-Ni binary as the example, significant Fe K α X-rays (0.5%) can be measured in a 100% Ni phase, even 20 microns from the Fe-Ni interface. However, if the low-kV method is used, along with the L-lines, the maximum distance from the interface that the Fe L α X-rays can be measured is less than 200nm.

A third approach that could be used combines the advantages of the two preceding approaches. This method uses multiple accelerating voltages. It allows the analyst to craft the analytical setup to take

advantage of the best of both the low-kV and low-ovoltage methods. Using a SiO₂-FeO-MgO-CaO glass as an example, the low-kV method would produce an analytical volume of about 500nm using an accelerating voltage of 7kV (Fig. 2). However this would require using the Fe L α line for the analysis. This resulted in a measured FeO content higher than the expected value (18.5 wt.%, versus 14.4 wt.% expected). Using the low-ovoltage method and 10kV, better results for FeO (14.8 wt.% versus 14.4 wt.% expected) can be obtained. However, at 10kV the analytical volume is about 1000nm for SiO₂, MgO and CaO (Fig. 2). One solution is to use a multiple kV approach and combine the 10kV k-ratios for FeO with the 7kV k-ratios for SiO₂, MgO and CaO. This produces a 500nm analytical volume for all the elements, along with good analytical results for all the elements, including FeO (Table 1).

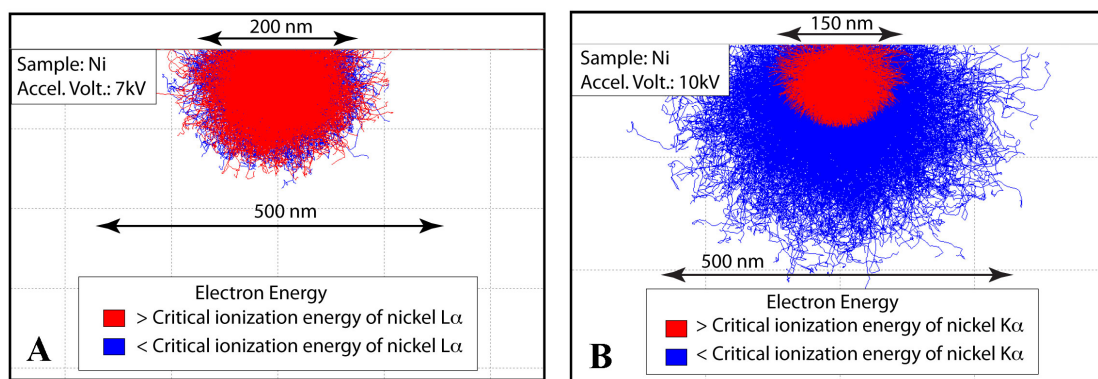


Figure 1. Monte Carlo simulation of the analytical volume for a Ni sample using the low-kV method at 7kV using Ni L α (a), and the low-ovoltage method at 10kV using Ni K α .

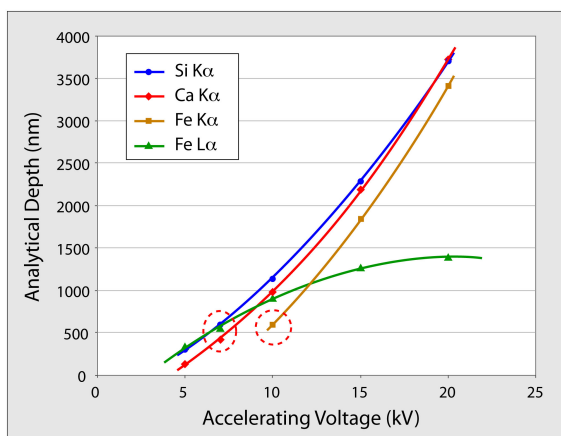


Figure 2. Plot of the analytical depths for the X-rays line generated from a SiO₂-MgO-CaO-FeO glass. Using the Fe L α and 7kV, results in an analytical volume for each elements of about 500nm. This same analytical volume can be obtained for Fe K α , if 10kV is used for it.

Table 1. Three strategies were used to measure the NIST glass K-411. Fe analyzed using its K α line are denoted (^), and the L α line as (*). In the multiple-kV analysis, elements in green were analyzed at 7kV, those in yellow at 10kV.

	Acc. Voltage	SiO ₂	FeO	MgO	CaO
Expected		54.3	14.4	14.7	15.5
Low-U	10kV	54.0	14.8 ^	14.4	15.4
Low-kV	7kV	53.7	18.5 *	14.6	15.3
Multi-kV	7 + 10 kV	53.8	14.8 ^	14.5	15.4