

Low Overvoltage Quantification with SCC

Energy Dispersive Spectroscopy (EDS) analysis of small structures or surface contaminants can be exceedingly challenging due to the X-ray signal generation volume, which is typically orders of magnitude larger than the resolution seen in the electron image. One way to reduce the X-ray volume is to reduce the acceleration voltage, however, this limits the number of X-ray lines available and makes quantification very challenging due to the large absorption corrections. Another approach is to use low overvoltage. Overvoltage is the ratio of the electron energy to the critical ionization energy of a given absorption line. A simulation of the signal depth for Si and Fe can be seen in Figure 1.

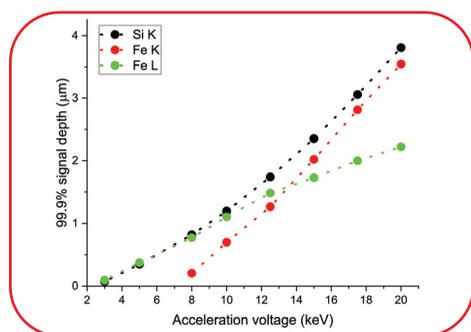


Figure 1: Simulation of signal depth as a function of acceleration voltage for Si K, Fe K, and Fe L lines using NIST DTSA-II¹. The figure shows that the X-ray depth resolution is better using the Fe K line relative to the low energy Fe L line from roughly 13 kV acceleration voltage down to the excitation energy of 7.113 keV.

While low overvoltage measurements limit the X-ray generation volume, quantification is exceedingly challenging since the ionization cross-section changes dramatically as the acceleration voltage approaches the critical ionization energy (Figure 2). Significant deviations from theoretical models are to be expected² and consequently, standard-less models frequently require an overvoltage of at least 1.5 before a given energy line can be used for quantification.

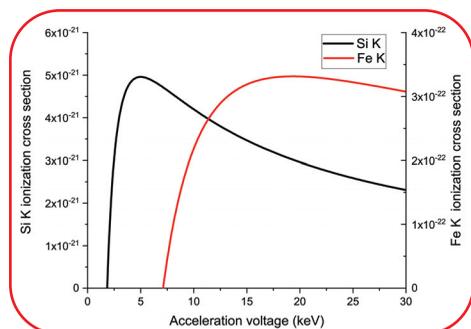


Figure 2: Calculated ionization cross-section of Si K with an ionization energy of 1.839 keV and Fe K with an ionization energy of 7.113 keV³.

However, by using the Standard Customized Coefficients (SCC) option available in the APEX™ 2.0 software, it is possible to measure and compensate for deviations between the implemented model and the actual ionization cross-section for a given element and voltage combination.

To illustrate the improvements with SCC, an iron silicide standard with two distinct phases was examined (Figure 3). A pure Si sample was used as a reference sample at different voltages, while a pure Fe sample was used to determine the Fe K SCC values. Spectra were collected in a range from 9 - 20 kV corresponding to an overvoltage range of 1.1 to 2.8.

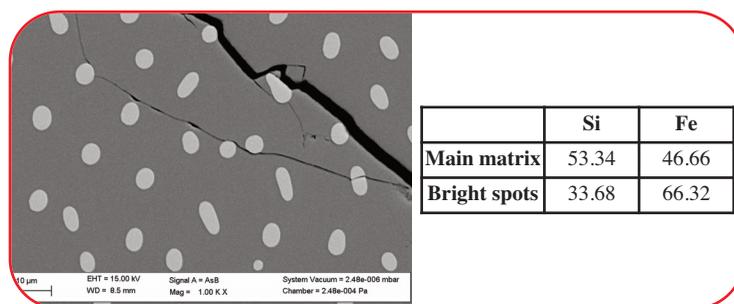


Figure 3: Backscatter image of an iron silicide standard with composition in weight %.

The quantification results in Figure 4 show a significant overestimation of Fe as the acceleration voltage approaches the Fe K ionization energy. By applying SCC measurements from a pure Fe sample, the deviations from implemented models can be compensated for and satisfactory quantification results can be achieved.

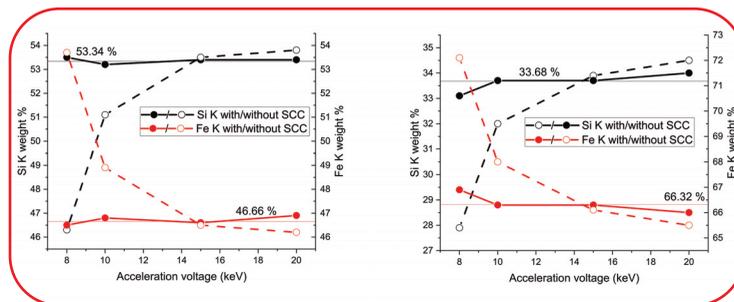


Figure 4: Quantification results with/without SCC for the main matrix (left) and bright spots (right) shown in Figure 3. The certified compositions are indicated by black/dotted lines for Si/Fe.

The SCC adjustments allow the user to optimize the quantification routine and compensate for model deviations in specific samples or uncommon conditions. The example presented here demonstrates optimization using simple, pure element samples. For effects with no significant inter-element influence, the SCC measurements for a given element can be applied irrespective of composition.

[1] Ritchie, N.W.M. (2019). NIST DTSA-II software, including tutorials. Available at <https://cstl.nist.gov/div837/837.02/epq/dtsa2/>

[2] Llovet et al., Journal of Physical and Chemical Reference Data 43, 013102 (2014)

[3] Goldstein et al., Scanning Electron Microscopy and X-Ray Microanalysis (2018). ISBN: 978-1-4939-6674-5. 10.1007/978-1-4939-6676-9.