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Systematic Error Determination of ζ-Factor Quantification in XEDS Analysis

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In (S)TEM the energy dispersive X-rays spectroscopy (EDS) is used for compositional analysis with high lateral resolution. The accuracy of the quantification result is crucial for the understanding of the materials properties. A key advantage of ζ-factor quantification method [1] discussed in this contribution compared to the more commonly used Cliff-Lorimer quantification is that the sample thickness does not need to be determined for the quantification, but that it can be derived from the easier accessible beam-current used in the experiment. On the other hand some systematic errors do not cancel out in the ζ-factor method whereas they do in Cliff-Lorimer quantification. The EDS live time, effective solid-angle and beam current have to be measured with high accuracy. Even though the theory is established [2], an overview of these systematic errors on the field of the ζ-factor is missing.

In this work, an investigation on the reproducibility of the ζ-factor among different experimental conditions is presented. EDS spectra are acquired under variation of several parameters like: beam current, size of the illuminated area, position on the sample, tilting angle, apertures settings, beam voltage, sample compound, and on different machines. For a proper ζ-factor calibration, an accurate measure of the beam current and a sample with known thickness is required. The beam current read out from the flu screen can be influenced by several effects: e.g. inhomogeneity, charging, non-linearity. In order to validate the flu-screen read out, a sample holder with integrated Faraday cup is used. The sample used is a Si3N4 amorphous film with 200nm thickness, which is produced using lithography techniques to have a well-defined thickness and homogeneous thickness. The Si3N4 film is deposited on a silicon waver and afterwards the waver is etched away on the backside of the film to make the sample electron transparent.

Figure 1 shows machine-to-machine reproducibility of the ζ-factor at 200 and 300kV. Measurements were performed in the factory, following a routine protocol on the machine before shipment. The 200kV results show a good reproducibility across the tools. For the 200nm thick Si3N4 sample an error in the thickness estimate of %10, results in a quantification error of 1.6 at% at zero tilt. So even with the 17% variation of zeta-factors at 300 kV, a quantification error of less than 3 at% is expected. Cliff-Lorimer quantification would give an error of 14 at%.

An estimation of the uncertainty of the ζ-factor value relative to different sources is reported. The sources of uncertainty using different tools are further investigated.. Nevertheless the stability of both the sample and the system were investigated by a 60-hour continuous EDS acquisition. Figure 2 shows that the system stability is excellent. Additionally model-based correction of the effect of holder shadowing on the effective solid-angle is discussed in the ζ-factor measurements of this contribution [3]. It is shown a single zeta-factor can be used across machines. In order to compensate for shadowing of the EDS signal by the holder, a model based approach can be used. Although the a built-in flu-screen has a higher measurement error than a dedicated faraday-cup, the resulting mass-density estimates are sufficient for improving the quantification results compared to Cliff-Lorimer quantification without absorption correction.

References:

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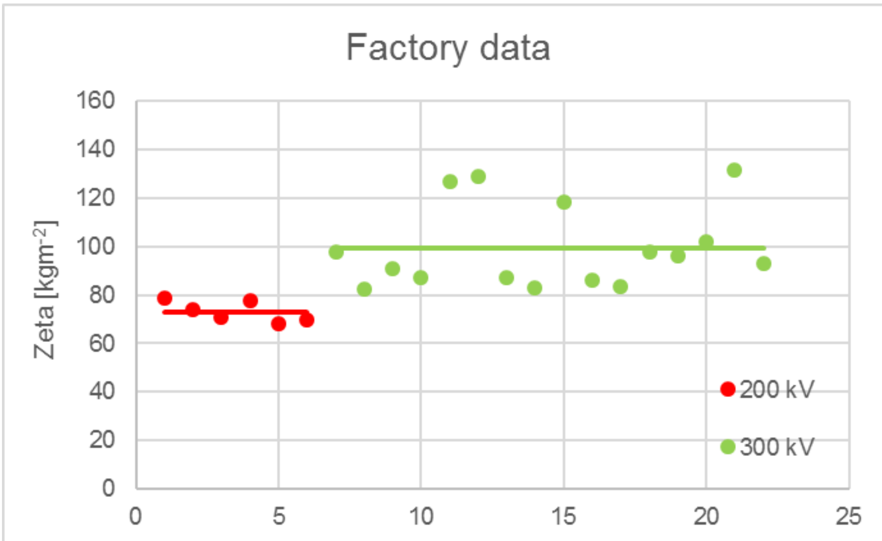

Figure 1. Machine-to-Machine variations across 22 Themis electron microscopes. The ζ-factors are calculated from EDS spectra of Si_3N_4. The ζ-factor has a value of 73±4 kg·m^{-2} and 100±17 kg·m^{-2} for beam voltages of 200 kV and 300 kV respectively.

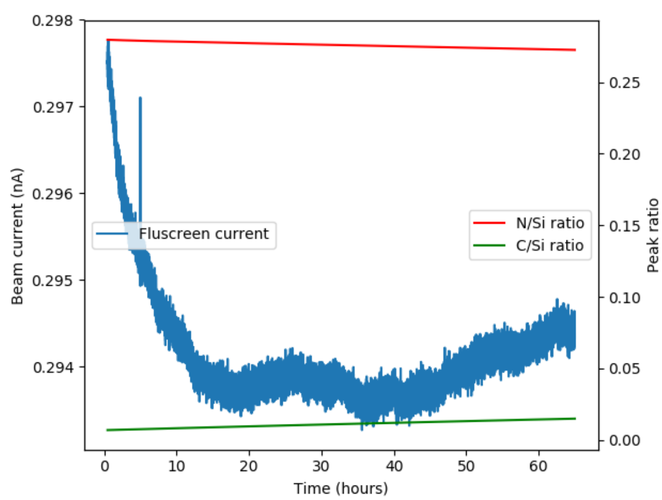

Figure 2. Stability measurements on a Si3N4 sample. Over a span of 60 hours, the variation in beam-current was 1.5%, the change in peak-ratio was 2.6% over 60 hours. The change in zeta-factor was below the measurement error.

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