

## Quantification and Precision in Particle Analysis Using SEM and EDS

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Microscopes in general and scanning electron microscopes (SEM) in particular are widely used for particle analysis where numerous features over a large area can be analyzed in an automated fashion [1] [2]. The SEM on its own will give information about particle size, density, shape, distribution etc., but combining the SEM with energy dispersive spectroscopy (EDS) will couple the morphology measurements with chemical classification. This classification typically relies on comparison of net intensities, least squares fit of spectra or more commonly the calculated composition of the particle represented in weight or atomic percent.

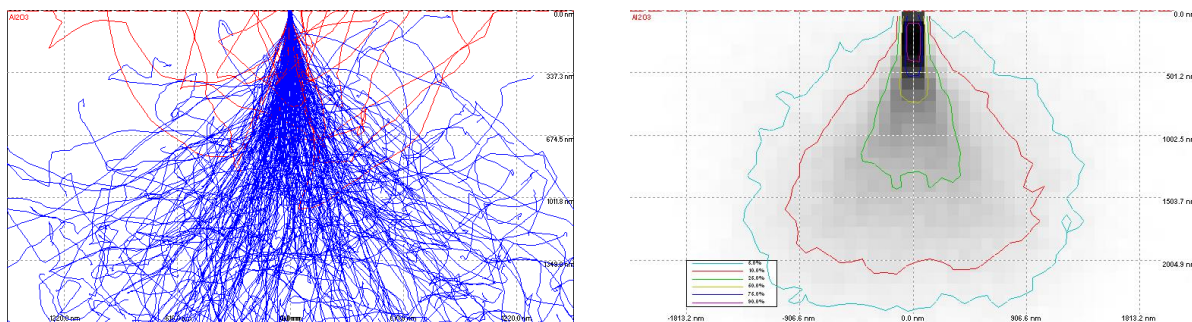
The models used for quantification are typically developed for bulk samples that are assumed to be flat and homogenous within the interaction volume [3]. As illustrated in Figure 1, electron penetration into a sample and the volume from which X-rays are generated can be quite significant and is often on a scale comparable to or larger than the volumes of interest in particle analysis. Since the model assumptions are not valid for particles of random shapes and sizes, significant variations from the true particle composition can be expected when the models are applied.

To qualify the amount and identify the cause of the variation, we are using pure standards (99.99% or higher purity) that have been ground into particles. Multiple measurements on a large flat grain is used as a base line for quantification. Spectra were collected at 15 kV for 2 live seconds with an input count rate of approximately 80k and 16% dead time for large flat particles. For automated spectrum collection the spectra were either collected from a single point in the geometric center of the particle or from a raster covering 80% of the particle core. For a stoichiometric Al<sub>2</sub>O<sub>3</sub> the expected weight percent of Al and O is 52.925 and 47.075 respectively and as can be seen in Figure 2, the mean composition of the measured particles is within 1 weight % of the large grain. However, a significant spread in the calculated composition is seen with the single point acquisition being significantly worse than the raster acquisition.

By looking at individual particles that deviate from the expected composition, the root cause can often be identified. Figure 3 is an example where the quantification model fails due to absorption outside of the particle. The particle under investigation is surrounded by larger particles that absorb the emitted X-rays leading to relative higher attenuation in the low energy range, which leads to an underestimation of the O content. In this case, both center spectrum collection and raster scan will result in large deviations in the quantification results. Figure 4 shows a particle with a high degree of topography with a face of the particle oriented towards the detector (top in image). As the model assumes the particle to be flat, this corresponds to a change in take-off angle, which again leads to deviations in the quantification results. Changes as small as 10 degrees can easily lead to deviation on the order of 2-3 weight percent.

References:

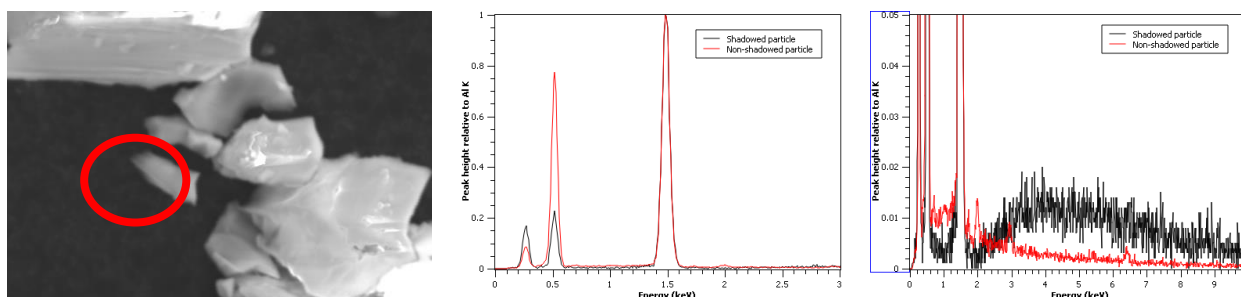
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- [2] A Laskin et al., 2006, Journal of Electron Spectroscopy and Related Phenomena, **2-3** (2006), p. 260.
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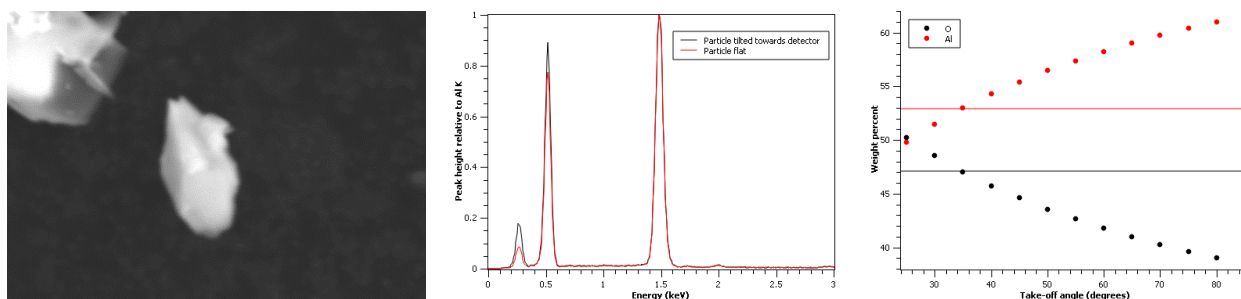
**Figure 1.** Monte Carlo simulation of electron scattering (left) and energy deposition (right) in a bulk Al<sub>2</sub>O<sub>3</sub> sample with 15 kV of acceleration voltage.

|                      | O wt% mean | Al wt% mean | Standard Deviation |
|----------------------|------------|-------------|--------------------|
| Single grain         | 47.1       | 52.9        | 0.2                |
| Particle raster scan | 47.0       | 53.0        | 3.8                |
| Particle center      | 46.3       | 53.7        | 8.5                |

**Figure 2.** Quantification results and standard deviation for the different spectrum collection modes.



**Figure 3.** SEM image (left) of particle surrounded by larger particles causing shadowing. Spectra (center and right) comparing the shadowed particle to a non-shadowed particle shows abnormal bremsstrahlung background and attenuation of the O peak.



**Figure 4.** SEM image (left) of particle with top part facing the detector. Spectra (center) comparing the particle to a flat particle shows increase of the O peak due to the change in escape path. The graph on the right shows the variation in quantification results as the take-off angle is changed for a spectrum collected from a flat particle.