TEM-EDS: Energy-dispersive X-ray Spectroscopy of Electron Transparent samples; Possibilities and Challenges

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Quantitative chemical characterization of mixtures of light and heavier elements using EDS at high spatial resolution in the electron microscope is a challenge. While quantitative bulk analysis by EDS in SEM has been developed extensively, quantitative EDS of nanostructures and in transmission is still being explored. Complicating factors are for instance absorption effects, radiation damage and preparation artefacts.

Various single and multiple detector arrangements providing high solid angles of up to 1.1sr and high take-off angles between 20° and 70° are available now for electron microscopes. While single Bruker XFlash® standard detectors of 0.1sr allow quantification on the atomic level within minutes, multiple detector systems provide fastest data acquisition, which is suitable to analyze large or 3D samples in a reasonable amount of time and is furthermore important to avoid extended exposure of electron beam sensitive samples. Additionally, in SEM, the analysis of complex bulk topography becomes possible, limiting the need for sample preparation. Our webinar reports on ways to exploit the available Bruker XFlash® technology for quantitative EDS on the nanoscale using electron transparent samples in STEM and SEM.

In STEM small solid angles of 0.1sr of the 30mm^2 Bruker XFlash® detector in combination with high beam current, high brightness and the correction of spherical aberration allow the identification of single atoms in graphene by EDS [1]. The Cliff-Lorimer method, widely used for the quantification of electron transparent objects larger than that, can provide data on the accuracy level of a few at%. Using high solid and take-off angles as mentioned above for multiple detector arrangements, even the ppm level (e.g. 0.02 at% for Rb in an Orthoclase mineral standard [2]) can be accessed. The at% level results of the Cliff-Lorimer quantification method are only valid relative to a suitable standard of similar thickness and composition though, which is often difficult to obtain. One alternative is the Zeta-factor method [3]. It additionally includes information on the electron dose (beam current) and accommodates the use of any standard(s) with known thickness, density and composition. The Zeta-factor method can deliver absolute quantification while accounting for absorption and fluorescence effects. The implementation, further development and options to combine EDS with other complementary STEM-analysis techniques, such as, electron energy loss (EELS) spectroscopy [4] will be discussed. To correctly interpret STEM-EDS element maps on the atomic scale, simulations of relevant scattering and radiation effects are necessary [5].

EDS of electron transparent samples in SEM (T-SEM) allows the analysis of large sample areas, e.g. using automated particle analysis and it can be combined with other emerging complementary SEM-based techniques: micro-XRF allows trace analysis for higher Z elements at moderate spatial resolution and higher penetration depth than EDS and Transmission Kikuchi Diffraction (TKD) offers crystallographic analysis on the nm-scale [6].

References

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