SDD-EDX: element analysis of nanostructures in STEM

M. Falke1, A. Kaeppel1, S. Scheller1, W. Hahn1, R. Terborg1, M. Rohde1, M. L. Trudeau2, I. MacLaren3, D.A. MacLaren3, Q. M. Ramasse4

1. Bruker Nano GmbH, Schwarzschildstr. 12, Berlin, Germany
2. Materials Science, IREQ, Hydro-Québec Research Institute, Varennes, Canada
3. SUPA, School of Physics and Astronomy, University of Glasgow, Glasgow G12 8QQ, UK
4. SuperSTEM, Daresbury, Science and Innovation Campus, Keckwick Lane, Warrington WA4 4AD, UK

Meiken.falke@bruker-nano.de
Keywords: EDXS, atomic resolution, STEM

Energy dispersive X-ray spectroscopy (EDX(S)) in the electron microscope uses characteristic X-rays generated during the interaction of electrons with a sample for element identification and quantitative composition analysis. EDX is ideal for cases where many elements have to be identified at once and other complementary techniques suffer from ambiguities such as peak overlaps or intensity reversals in case of shallow edges in EELS, low z-axis resolution in atom probe tomography or preparation artefacts and other, different from the EDX-typical, complications.

The smaller the structure to be investigated, the higher the spatial resolution of the microscope has to be. It is not straightforward though, to form an appropriate electron probe ensuring the necessary spatial resolution while providing a high beam current for efficient spectroscopy. Measures taken to reduce sample damage, such as low accelerating voltages and low dose techniques, make this problem even more complicated. Collecting a sufficient part of the sparsely generated X-ray photons in case of a limited electron probe current incident on a minor amount of matter is a challenge though. Since characteristic X-rays are emitted roughly into all directions, an ideal detector would cover a sphere around the sample. Various approaches have been implemented to tackle this engineering problem, ranging from single large detector areas, via multi detector systems to ring-shaped and a Pi-steradian (sr) detector [1].

The ideal solution for chemical analysis on the nanoscale though is to combine an improved EDX system with adjustments in pole piece and port geometry as well as with a high brightness electron source [2] and aberration correction [3]. This approach enables single atom X-ray spectroscopy in STEM even at 60keV accelerating voltage and at a solid angle of just 0.1sr [4].

It is difficult though to make changes to the pole piece geometry or source performance of existing microscopes, either standard or aberration corrected. This paper will demonstrate element analysis on the nanoscale using of the shelf stand-alone silicon drift detector (SDD) solutions for such microscopes. So-called slim line technology was implemented to improve the solid angle in TEM without having to tilt the sample and without significant sacrifice in take-off angle (TOA). This approach enables the fit of a larger detector area into an unchanged detector tube size. With the new design, element identification and quantification remains of high quality. Figure 1 shows BN spectra acquired using a 60mm² detector at 0.4sr solid angle and 0° sample tilt. The standard less quantification with theoretical (calculated) Cliff-Lorimer factors results in 50.5at% B and 49.5at% N. The spectra in Figure 2 were taken with a 30mm² and a 60 mm² detector under otherwise equal conditions. To obtain the same number of pulses in the main sample relevant peaks, the spectrum obtained by the 30mm² detector had to be multiplied by a factor of 2, which proves that the solid angle was doubled.

Figure 3 demonstrates the quantification of the Cr and Si composition in a line scan over nm-sized embedded CrSi₂ precipitates using a 30mm² detector at 0.12sr. The Cliff-Lorimer factors were calibrated on a continuous CrSi₂ layer part present in the same sample, where the beam passes through CrSi₂ only. The data acquisition took 30s. Nanometre-sized precipitates are used to set the texture and epitaxial orientation of thermoelectric silicide films. The function of precipitates and other seed layers is still not fully understood. Thus, revealing the chemical composition of these nanocrystals is the first step to analyse their crystallographic phase and clarify their role during epitaxial growth.

In summary, we demonstrate, that element identification and quantification of nanostructures under challenging conditions is possible using commercial high-end SDD-EDX detectors.
References


Figure 1. Spectra of BN acquired at different energy dispersions in a C$_s$-corrected cold-FEG STEM at 200keV, 0.4sr solid angle and no sample tilt using a detector of 60mm$^2$ area.

Figure 2. Part of a spectrum obtained using 60mm$^2$ detector area achieving 0.4sr solid angle and a spectrum obtained under the same conditions using 30mm$^2$ detector area with 0.2sr solid angle. The latter spectrum had to be multiplied by a factor of 2 to normalize to the same number of counts in the energy channels marked. The slightly better peak to background ratio in case of the smaller detector size may be due to less background from the bulk support, e.g. Cu.

Figure 3. Left: net count element profile across a CrSi$_2$ precipitate using a 30mm$^2$ EDS detector in a standard 200kV STEM and calibrated Cliff-Lorimer factors. Very thin nm-sized features of Cu and Si contamination caused by ion milling and surface oxidation are clearly visible in 30s. Right: HAADF, Si-, Cr-, Cu-map, the arrow shows the line scan region.