EDS Analysis of Light Elements with Silicon Drift Detectors: Quantification of Boron

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Introduction
The analysis of light elements (Be, B, C, N, O and F) represents a challenge for energy dispersive X-ray spectrometry (EDS). Some of the problems are technical in nature, relating to instrument design and measurement procedure, while others are due to inherent physical effects (e.g., low fluorescence yields). With recent advances in EDS technology, dramatic progress in the detection of light elements has been made. Ultra-thin polymer windows have made the detection of light elements possible (e.g., C-K\(_\alpha\)) and their high count rate makes them attractive for the detection of elements such as boron and discuss remaining challenges for light element analysis with EDS.

Figures and Tables

- Fig. 1. Efficiency curves for an SDD with a super light element window (SLEW) compared to an SDD with conventional Be window.
- Fig. 2. Energy resolution for different elements (e.g., C-K\(_\alpha\) at 46 eV, Mn-K\(_\alpha\) at 5,803 eV) measured with a Bruker XFlash® 5010 SDD on a standard sample (EDS-TM001), specifically designed by the German Federal Institute for Materials Research and Testing (BAM) for testing the resolution of EDS detectors [1].
- Table 1. Quantification results (standard-based, PhiRhoZ) for Ni\(_3\)B. Average of 5 measurements for each condition.

Results
Figure 3 shows spectra of B\(_4\)C (purple), LaB\(_6\) (green), BN (blue) and Ni\(_3\)B (red) compared to spectra of a pure boron standard (black) and a pure carbon standard (gray). Analytical conditions: 5 kV accelerating voltage, 0.4 nA beam current, 3.5-7 kcps input count rate, XFlash® 5030 silicon drift detector, 60 s measurement time.

When comparing the Ni\(_3\)B spectrum with the spectrum of the carbon standard (which does not contain any boron), the boron peak of Ni\(_3\)B (with 5.8 wt% B) is small but clearly visible. It is also possible to map such minor boron concentrations. Figure 4 shows an element map of boron for an area within a weld seam (base metal was a Cr-Ni stainless steel). The sample contains phases with 3 different boron contents as indicated by the color scale: red = highest (Cr-boride), green = intermediate (Cr-Ni-boride) and blue = lowest (Ni\(_3\)B).

Quantification was carried out standard-based (with pure element standards for B and Ni) using the XPP-optimized PhiRhoZ model. Figure 6 shows the quantification results in terms of intensity ratios as a function of accelerating voltage.

Discussion
Our results underline the importance of using low accelerating voltages (≤5 kV) to ensure a sufficient peak to background ratio for the quantification of light elements. This, however, complicates the analysis, because K lines of many other elements, which may be present in the sample, are not excited when optimum overvoltages for the light elements are attained. Uncertainties in mass absorption coefficients [2] and peak overlaps with L, M and N lines of heavier elements represent further challenges that need to be considered.

References:

Conclusions
Advances in detector technology have triggered dramatic progress in the analysis and quantification of light elements with EDS!