

Analysis and Quantification of Transition Metal Borides with WDS and EDS

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Quantitative X-ray microanalysis using wavelength-dispersive (WDS) or energy-dispersive (EDS) spectrometers of light elements or using lines in the low energy range (<1 keV) is a challenge due to low X-ray yields and overlapping lines, especially for EDS. Furthermore, intensities can be affected by considerable absorption effects [1,2,3] and uncertainties in mass absorption coefficients (MACs).

In former work WDS and EDS analysis of steel was compared at an accelerating voltage of 6 kV and L lines of the 3d transition elements were used for quantification. Since EDS spectra present a significant overlap of L lines, spectral deconvolution was necessary [3,4] to get reasonable results. In addition, SDD-EDS quantification of chromium and lanthanum borides have been performed in [5].

In this work spectrum acquisition and quantitative analysis of different transition metal borides have been performed using EDS and WDS. Besides the challenges already mentioned peak shifts of a few eV and different peak shapes have to be considered as well. For WDS, integral measurements rather than peak-to-background measurements of the boron peak are recommended [1]. For EDS, peak shape variation is less important due to the lower energy resolution, Figure 1. However, the modelling of a correct bremsstrahlung background is an unavoidable issue for an accurate analysis. For our study the physical background model based on [6] was used.

WDS and EDS were measured under similar excitation conditions: a beam energy of 10 keV and a measuring time of 300 sec. A beam current of 10nA was chosen for EDS and 100 nA for WDS, respectively. For WDS two different multilayered crystals were selected and intensities of both were averaged. For EDS an XFlash Silicon Drift Detector with a Mn-K α resolution of 127 eV was used.

In order to evaluate the performance of the WD and ED spectrometers k-ratios are compared and related to the k-ratios given by Bastin [1]. Additionally, quantitative concentrations were determined from WDS and EDS k-ratios and compared to published data of other authors, i.e. Bastin using the same quantification algorithm. Depending on the $\Phi(\rho z)$ -methods and mass absorption coefficients (MACs) used considerable deviations are calculated.

The MAC of analyzed transition metals at the B K α energy varies by up to $\pm 20\%$ which can lead to variations in the resulting concentrations in the same range.

First results of a comparison for chromium boride is presented in Table 1.

References:

- [1] GF Bastin and JM Heijligers in “Quantitative Electron Probe Microanalysis of Boron in Binary Borides”, Technische Universiteit Eindhoven (1997) p. 1.
 [2] GF Bastin and JM Heijligers, *Scanning* **12** (1990), p. 225
 [3] X Llovet et al., *IOP Conf. Series: Materials Science and Engineering* **32** (2012), p. 012014.
 [4] R Terborg et al., *Microsc. Microanal.* **22** (S3) (2016), p. 404.
 [5] DE Newbury, WM Ritchie, *Microsc. Microanal.* **21** (2015), p. 1327.
 [6] G Castellano et al., *Spectrochim Acta B* **59** (2003), p. 31.

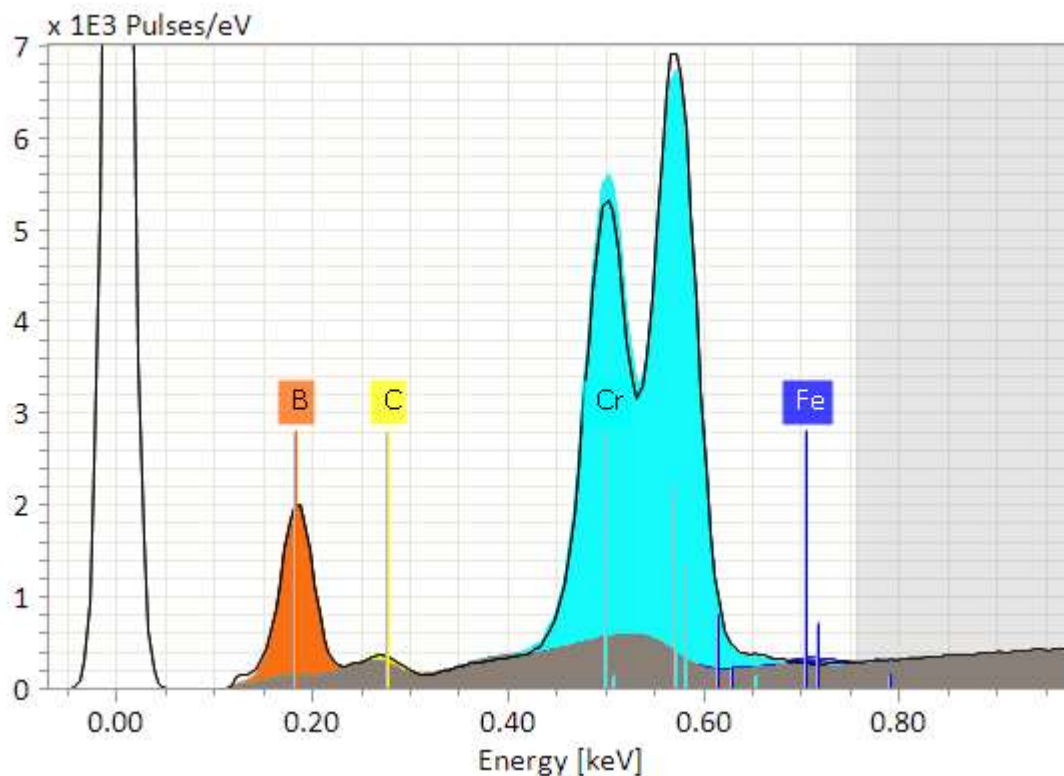


Figure 1. CrB spectrum (black) with calculated Bremsstrahlung background (grey) and peak deconvolution

CrB	B wt%	Cr wt%	Fe wt%	totals	B at%	Cr at%	Fe at%
WDS	17.7	83.3	0.1	101.1	50.6	49.4	0.1
EDS	17.7	83.4	0.2	101.3	50.5	49.4	0.1

Table 1. Quantitative analysis of chromium-boride CrB (given concentrations: B: 17.2 wt%, Cr: 82.8 wt%)