Comparing WDS and EDS quantitative analysis of light element alloys and standards

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The last decades have seen an astonishing evolution of the electron microscopes. At the same time they are much more versatile, more stable, easier to use, with higher resolution and magnification, more sensitive detectors and a much better signal to noise relation.

Historically scanning electron microscopes were always more a visualization tool where low current, of the order of pA are key for high resolution imaging. On the analytical side were the microprobes with their specific stable optics which allows currents of the order of nA and stability throughout the whole magnification range combining EDS and WDS detectors allowing reliable chemical quantification.

The stability of the current SEM's plus the Silicon Drift Detectors (SDD-EDS) is slowly turning them into both visualization and real analytical tools as there is a growing need for better, faster and higher magnification quantitative analytical data combined with imaging, on heterogeneous samples. Mineralogical standards and metallic alloys were measured on a SEM-FEG with SDD-EDS and compared to data obtained with a Microprobe with EDS/WDS, using approximately the same conditions: 15 to 25kV, spot-mode, between 30 and 50 measuring points for statistical relevance and ZAF matrix correction.
Table 1 shows the results for a metallic alloy sample: a thin wire, cross-section resin embedded, polished and carbon coated. Measurements were performed on a Quanta 3D - dual FIB from FEI with a Bruker Quantax 800 SDD Energy Dispersive Spectrometer (EDS) and on a Jeol 8900 Microprobe with a Thermo-Noran EDS and four Wavelength Dispersive Spectrometers (WDS), using, LiF, PETJ and TAP crystals. Trace elements: silicon and vanadium were identified only when counts were of the order of a million.

Standardless analysis with the software curve deconvolution methods were used for the SDD-EDS. Pure element standards, except for silicon where a SiFe standard was used, were used for the microprobe analysis.

Element	Iron	Chromium	Nickel	Manganese	Molybdenum	Silicon	Vanadium
SEM	52 (1)	20,2 (0,5)	16,3 (0,4)	7,5 (0,2)	2,8 (0,1)	0,39 (0,04)	0,17 (0,03)
Microprobe	52,96 (0,01)	20,85 (0,64)	15,90 (0,10)	7,87 (0,10)	2,66 (0,30)	0,38 (0,01)	0,17 (0,02)

Table 1 - comparison of EDS and WDS data for a metallic alloy.

Measurements on mineralogical standards with low Z elements are being performed and preliminary results are quite encouraging. A complete comparison and analysis of the data will be presented.
The preliminary agreement between the microprobe and SDD-EDS results might enable us to use a SDD-EDS SEM with almost the same reliability of a microprobe, especially with low Z mineralogical samples where the peak overlap is quite significant.

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