doi:10.1017/S1431927616003044
Microsc. Microanal. 22 (Suppl 3), 2016

Combined EDX and Micro XRF Analysis on SEMs

Ralf Terborg[1], Birgit Hansen[1], Stephan Böhm[1]

[1] Bruker Nano GmbH, Am Studio 2D, Berlin, Germany

Quantitative and qualitative micro X-ray fluorescence (XRF) analysis offers a wide range of applications. However, it is difficult to analyze light elements (Z<11) and elements having L or M lines in the low energy range (E<1keV) due to a small ionization cross section for excitation spectra of typical X-ray tubes. Additionally, significant absorption can occur if the measurements are not performed in a vacuum chamber. If an X-ray optics is used the transmission in the low energy range can also prevent an efficient excitation of low energy lines.

These limitations can be overcome at scanning electron microscopes (SEM)s equipped with energy dispersive x-ray spectrometry (SEM-EDX or ED-EPMA) where electrons are used as primary particles. These can excite low energy lines and light elements down to beryllium, which are detected with an energy dispersive spectrometer (EDS) which is also used for the acquisition of the XRF spectra. On the other hand, electron excitation also produces a significant bremsstrahlung background which reduces the detection limit for trace elements.

With an X-ray tube attached to an SEM samples can be analyzed with both, X-ray and electron excitation, fig 1. The electron excitation can be used for scanning and as an imaging technique which is useful for the orientation on the sample as well as for microanalysis of the major and minor elements. XRF spectra can additionally be used for trace element analysis using the same EDS. The combination of XRF analysis and EPMA can improve the results of a quantitative analysis because of simultaneous light element and trace element analysis.

However, there are also restrictions. While the electron beam can probe sizes <100nm the focused spot of the micro X-ray tube is in the range of 50µm. Also the depth information is 10 to 100 times larger for the XRF analysis. Samples need to be homogeneous in depth and lateral dimension in order to enable combined XRF and SEM-EDX analysis.

As SEMs are normally equipped with an x-y table in order to move the sample to different positions, it is also possible to perform one dimensional (line scan) or two dimensional (mapping) qualitative or quantitative analysis using XRF analysis, SEM-EDX or even both for qualitative applications.

Typical application for quantitative analysis could be minerals or glasses, which can contain trace elements but also – besides oxygen - boron or fluorine which cannot be analyzed by stoichiometry. In this case trace elements as well as the boron, oxygen and fluorine can be quantified in combination, table 1.

Other application fields could be the analysis of steels, semiconductors, ceramics or fly ash.

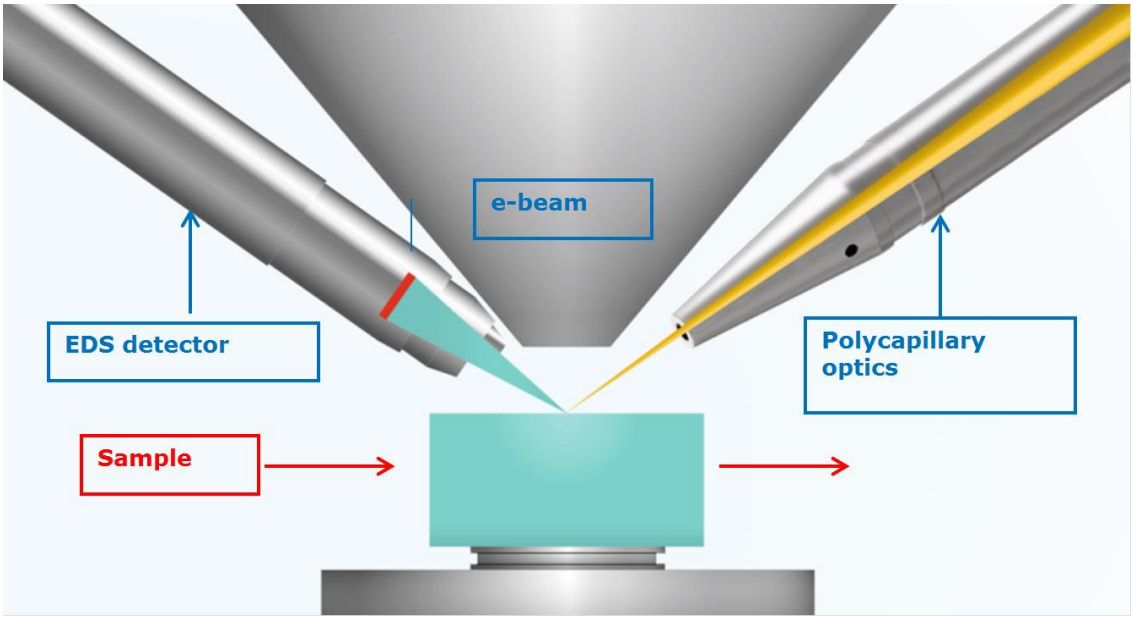

Figure 1. Experimental setup of the Xtrace XRF tube and an ED spectrometer on an SEM. The polycapillary optics and the EDS need to be aligned to the same spot.

Element	Certificate	EDS / wt% Norm.	XRF / wt% Norm.	EDS+XRF
O	46.8	45.7	45.6	46.0
Na	10.7	10.5	10.3	10.6
Mg	2.2	2.3	2.3	2.3
Al	0.95	1.34	0.89	0.89
Si	33.7	33.9	35.00	34.2
S	0.11	0.16	0.12	0.12
K	0.34	0.37	0.35	0.35
Ca	5.1	5.3	5.3	5.3
Ti	0.01	<LoD	0.01	0.01
Fe	0.03	0.28	0.03	0.03
As	0.04	0.04	0.06	0.06
Sr	-	0.00	0.04	0.04

Table 1. Quantitative analysis of the NIST 620 glass standard and comparison to the certificate values. XRF results and oxygen content were calculated assuming a fixed stoichiometry for all elements. EDX results were calculated by evaluation of peak intensities. For the EDS+XRF quantification the results of O, Na, Si, Ca results of both techniques were combined.

https://doi.org/10.1017/S1431927616003044 Published online by Cambridge University Press