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Performance Check of a Wavelength Dispersive X-Ray Spectrometer (WDS) attached to the SEM

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Besides energy dispersive spectrometers (EDS) that complete most of the scanning electron microscopes (SEM) there is an increasing number of wavelength dispersive spectrometers (WDS) that are attached to a SEM [1,2]. Hence, the analytical facilities available with a SEM are extended towards better element sensitivities and peak separation.

Procedures for a periodical check of the performance of the WDS attached to a SEM are less established than in the case of EDS. For accredited laboratories such procedures are even mandatory. Of primary interest is the stability of the peak count rate as a measure of the accurate operation of the gas flow proportional counter (stability of the composition/flow rate of the carrier gas), accurate mechanical adjustment of the whole "WDS geometry" (diffraction crystals, sample and whole spectrometer positioning) and the long-term stability of the transmission of the hybrid X-ray optics used. Spectrometer resolution (FWHM) and calibration of the energy scale are also considered.

In this paper the results of the periodical performance check of a parallel beam spectrometer (PBS) at a SEM over a longer period of time are presented. The check includes the measurement of FWHM, the intensity in counts per nA and the peak-to-background ratio. In opposite to the performance check of an EDS [3] this has been proven to be more laborious, not only more time-consuming, but also "tricky". Especially the exact mechanical alignment of the whole "WDS geometry" and its implications for the WDS quantification will be highlighted in this work.

Conventionally, a set of several specimens is used to perform the measurements with the diffraction crystals. We currently use a single test material specially developed at BAM (EDS-TM001) for these purposes [4]. It consists of five elements carbon, aluminum, manganese, copper and zirconium homogeneously distributed in a thick layer (ca. 10 μm) on a steel substrate, see Figure 1. X-ray lines of energies covering representatively the whole energy rangy of the five or six diffractors of the WDS are hence available in one specimen only (e.g. from Zr-Mζ at 0.152 keV (!) up to Cu-Kβ at 8.904 keV), see Table 1. Therefore, the whole check procedure becomes less time-consuming and delivers more reliable results. Some representative X-ray line maps are presented in Figure 2.

References

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[3] M. Procop, V.-D. Hodoroaba, M. Griepentrog, *Microsc. Microanal.* 12 (Supp 2) (2006) 870.
[4] www.webshop.bam.de ("Reference materials" / "Test materials" / "EDS-TM001").

TABLE 1. Energy ranges "covered" by the diffractors existing in the commercial PBS *MAXray* (Thermo Fisher Scientific) and the X-ray lines "offered" by the EDS-TM001 test material for the spectrometer performance check.

Diffractor	Energy range (keV)	X-ray line	Energy of the X-ray line (keV)
MoB4C200	0.066 to 0.257	Zr Mζ	0.152
NiC80	0.167 to 0.647	C K	0.286
WSi60	0.221 to 0.857	Mn Lα	0.645
TAP	0.509 to 1.979	Cu Lα, Al Kα	0.930, 1.489
PET	1.509 to 5.862	Zr Lα	2.046
LiF	4.633 to 17.994	Mn Kα, Cu Kα	5.887, 8.054

FIG. 1. Test material EDS-TM001 [4].

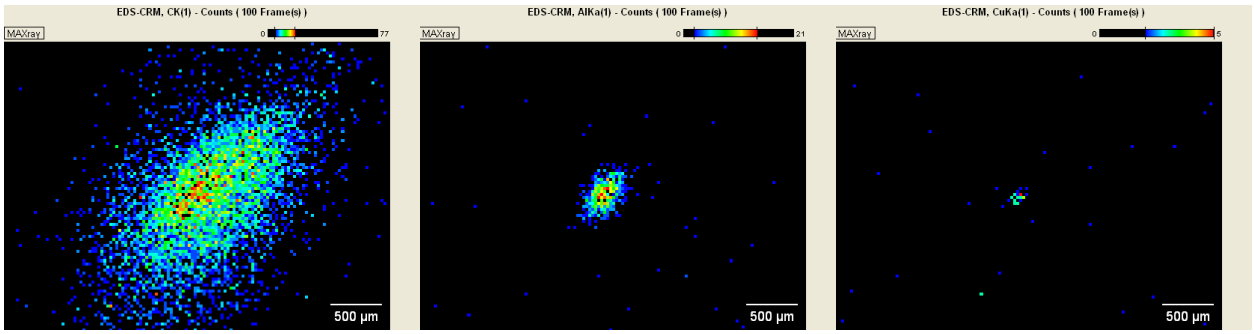

FIG. 2. Maps of some selected X-ray lines emitted by the test material EDS-TM001: C K (0.286 keV), Al Kα (1.489 keV) and Cu Kα (8.054 keV).

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