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RESEARCH PAPER

New reference and test materials for the characterization of energy dispersive X-ray spectrometers at scanning electron microscopes

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Abstract Checking the performance of energy dispersive X-ray spectrometers as well as validation of the results obtained with energy dispersive X-ray spectrometry (EDX) at a scanning electron microscope (SEM) involve the use of (certified) reference and dedicated test materials. This paper gives an overview on the test materials mostly employed by SEM/EDX users and accredited laboratories as well as on those recommended in international standards. The new BAM reference material EDS-CRM, which is currently in the process of certification, is specifically designed for the characterization of EDS systems at a SEM through calibration of the spectrometer efficiency in analytical laboratories in a simple manner. The certification of the spectra by means of a reference EDS is described. The focus is on the traceability of EDS efficiency which is ensured by measurements of the absolute detection efficiency of silicon drift detectors (SDD) and Si(Li) detectors at the laboratory of the PTB using the electron storage ring BESSY II as a primary X-ray source standard. A new test material in development at BAM for testing the performance of an EDS in the energy range below 1 keV is also briefly presented.

Keywords EDX · EDS · Performance check · SEM · Test materials · Spectrometer efficiency

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Introduction

For more than four decades, one of the most widely employed analytical methods to quickly determine the elemental composition in solid materials at the microscale is the energy dispersive X-ray spectrometry (EDX) with electron excitation at a scanning electron microscope (SEM). The method is also called ED-EPMA (electron probe microanalysis with energy dispersive X-ray spectrometry). The quantification works "classically," by using certified reference materials (CRMs) with an elemental concentration similar to the unknown sample, as well as "reference-free," by relating the measured intensities in the unknown sample to intensities of CRMs either calculated from first principles or premeasured under well-known conditions and stored in the quantification software package. After proper correction of matrix effects (atomic number, absorption, and fluorescence excitation) by using empirical models based on physical approaches, the elemental concentrations are obtained. The establishment of the quantitative ED-EPMA as a true metrological tool for traceable results in industrial laboratories requires detailed knowledge of the uncertainties introduced by the spectrometer performance parameters and matrix corrections. For well-known approaches, experimental conditions, and samples, this type of measurement uncertainties can be calculated [1]. Nevertheless, the ED-EPMA quantification on a traceable basis remains challenging. For the case of reference-free quantification, the detailed knowledge of the instrumental performance parameters of the energy dispersive X-ray spectrometer (EDS) is of crucial importance.

Both energy and intensity scales of the X-ray spectra must be well controlled. While the energy scale can easily be calibrated and the EDS energy resolution is determined as FWHM (full-width-at-half-maximum) of well-defined peaks, the calibration of the intensity scale over the whole energy range of interest for EDX at a SEM, i.e., up to about 15 keV,

is challenging. Using synchrotron radiation at a national metrology laboratory like the PTB at the electron storage ring BESSY II in Berlin is a highly accurate but rather expensive way to measure the absolute spectrometer detection efficiency [2, 3]. It is based on relating the measured X-ray spectrum to the well-known input spectrum from the storage ring as a primary X-ray source and yields very low measurement uncertainties of the spectrometer efficiency. For "common" SEM/EDX user laboratories, simple practical procedures based on reference materials for the determination of the spectrometer efficiency have also been developed. A brief overview is given in the following section.

Generally, the validation of the quantitative EDX results requires CRMs with certified elemental composition. This category of materials is rich enough in order to select the most suitable ones for the specific application. Furthermore, the routine check of the performance of an EDS necessitates well-selected test specimens available as simple/common materials. Test materials for checking the EDS performance parameters, in particular below 1 keV, are limited and need special attention for their proper application.

Available test materials for EDS check and specification

In the following, an overview is given for test materials employed mostly to check and specify the main instrumental parameters of an EDS. Table 1 is a compilation of recommendations in the standard ISO 15632 [7] as well as given by EDS manufacturers. In principle, all the relevant instrumental parameters can be checked or specified by means of properly selected test materials, which shall be easily available in an analytical laboratory. Furthermore, the procedures to be applied to determine the corresponding EDS parameters are simple and can be run quickly and deliver unambiguous results.

EDS-TM test material as precursor of the reference material EDS-CRM

Recently, it was demonstrated that a test material, EDS-TM, can be employed to easily check all the relevant performance parameters of an EDS by means of a single 10-kV measurement at a SEM/EDS system [8]. The synthetic test material consists of an optimized elemental combination of C, Al, Mn, Cu, and Zr, such that a set of well-separated X-ray lines, C K, Mn Lα, Cu Lα, Al K, Zr Lα, and Mn Kα, see Fig. 1, serves to the overall characterization of an EDS. The material is a good practical example of test materials as recommended by ISO 15632 to be used for the periodical control of the EDS critical performance parameters in end user laboratories and required for those test laboratories operating under accreditation schemes [9].

Optional to the EDS-TM test material, a dedicated software package, "EDX Performance Test," is offered by BAM for the automatic processing of the measured 10-kV spectrum [10]. A screenshot with the diagrams produced by this software is shown representatively in Fig. 2 for a silicon drift detector (SDD) EDS system used for metrological measurements [11]. The result of the determination of the FWHM of C K and Mn Kα lines and of the Mn Lα/Mn Kα intensity ratio as a measure of the EDS efficiency with respect to the detector contamination is displayed in the upper two diagrams. The

Table 1 EDS instrumental parameters to be checked or specified with various test materials as recommended in the standard ISO 15632, by EDS manufacturers or other sources in the literature

Instrumental parameter	Test material	X-ray line	Scope (check/specification)	Source of recommendation
Energy scale	Pure copper	Cu Kα and zero peak	(Re-)calibration	EDS manufacturers
Energy resolution	^{55}Fe source or polished pure Mn	Mn Kα	Specification and check	ISO 15632, EDS manufacturers
	PTFE (Teflon®) or glassy carbon	C K	Check and specification	
	PTFE (Teflon®) or F containing mineral, e.g., CaF_2	F K	Check and specification	
Peak-to-background	^{55}Fe source	Mn Kα/"background at 1 keV" intensity ratio	Specification	ISO 15632, EDS manufacturers
Dead time	Various	Various	Check and specification	ISO 15632
Energy dependence of the efficiency	Pure copper or nickel	L/K intensity ratio	Check and specification	ISO 15632
Detector/window contamination	Pure chromium	(Cr Lι+Lη)/(Cr Lα1+Lβ1) intensity ratio	Check	EDS manufacturer [4]
	Freshly prepared cleaved surface of Si(111) wafers	Spectral background above O K_{ab}	Check	[5, 6]
	Boron nitride	B K/N K intensity ratio	Check	[5]
Pile-up effects	Various	Various	Check	ISO 15632

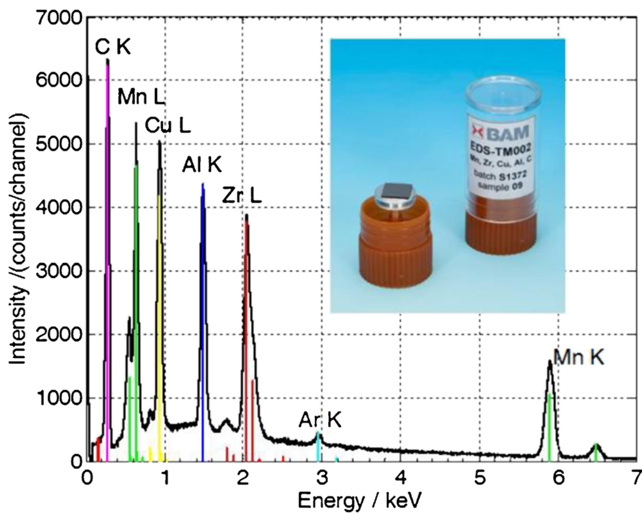

Fig. 1 A 10-kV X-ray spectrum of the EDS-TM002 test material

energy dependence of the measured EDS resolution is plotted in a separate diagram (bottom left in Fig. 2). Also, based on the set of X-ray lines offered by EDS-TM, the state of calibration of the EDS energy scale is represented in the bottom right diagram in Fig. 2. A more detailed explanation with representative practical examples of implementation of the EDS-TM and related software are given in [8]. As stated above, control charts documenting the calibration state of the EDS are required in accredited laboratories. The result of the periodical check of a Si(Li) EDS at BAM over a time span of more than 13 years, employing the same standard operation procedure, is illustrated in Fig. 3. Note that the manufacturer specifications of the EDS energy resolution, of 129 eV at Mn Kα and 58 eV at C K, respectively, were met practically over the whole period. Just during its last 3 years of operation, the specified values have been occasionally slightly exceeded.

Fig. 2 Results of processing the spectrum from Fig. 1 by the software package “EDX Performance Test”

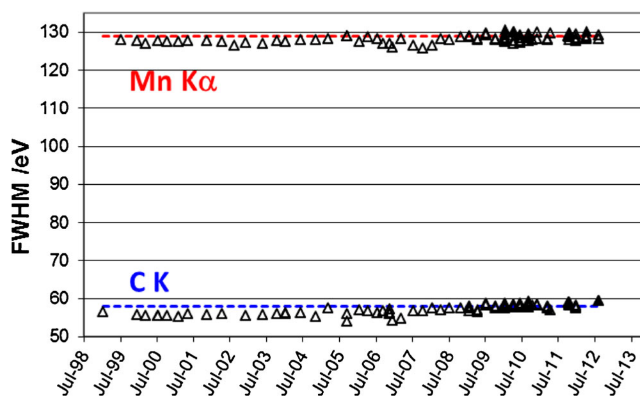

Fig. 3 Result of the periodic check of a Si(Li) EDS with respect to the energy resolution at Mn Kα and C K over a period of more than 13 years. The specifications of the EDS manufacturer were 129 eV at Mn Kα and 58 eV at C K, respectively (see the *dashed lines*). Note the slight alteration of the EDS energy resolution in its last 3 years of operation (of 1 to 2 eV)

Certification process

The EDS-TM test material is being successfully employed to test the performance of EDS systems in more than 100 laboratories. For the first generation of test materials, EDS-TM001, the manufacturing procedure of the 10-μm thick C-Al-Mn-Cu-Zr layer was reactive (magnetron) co-sputtering [12]. Due to the insufficient homogeneity and repeatability of the batch manufacturing, each individual EDS-TM001 sample should have been certified with respect to its elemental composition and of the emitted X-ray spectrum, respectively. This very tedious certification of the elemental composition has been waived. Nevertheless, EDS-TM001 has been kept as a valuable test material for checking almost all EDS performance parameters, apart from the challenging EDS efficiency. Also, the quick check of WDS spectrometers with EDS-TM001 has been demonstrated [13].

The need to manufacture new batches of EDS-TM has triggered a survey of deposition techniques applicable for the deposition of C-Al-Mn-Cu-Zr layers with excellent homogeneity and stability of the elemental concentration. Pulse laser deposition (PLD) was chosen to deposit the 6-μm layer onto silicon wafer instead of steel as a substrate. It should be noted that the “bulk” 6-μm C-Al-Mn-Cu-Zr layer consists of a multilayer stack of thin, individual layers of pure elements, C, Al and Cu, and Mn mixed with Zr, respectively. The thicknesses of the single layers in the range of a few nanometers have been optimized such in a way that the resulting EDX spectrum excited at 10 kV electron beam voltage in a SEM shows a similar spectral distribution as that of the EDS-TM001 material (see Fig. 1). The homogeneity and stability characterization of one EDS-CRM batch has been carried out as follows:

- From one 4″ wafer, 50 samples of 8.5 mm×8.5 mm size can be used for certification purposes; samples near to the margin region of the wafer are excluded.
- Five samples as sketched in Fig. 4 (left) have been selected as representative for one batch, i. e., one wafer, and have been investigated concerning homogeneity and stability.
- Each selected EDS-CRM sample has been measured with two EDS systems on five well-distributed positions—as indicated in Fig. 4 (right)—at two neighbored 100 μm× 100 μm areas. The overall relative standard deviation associated to the line intensities measured at all locations on all five samples is below 5 %.
- Two (of the five selected) samples have undergone accelerated climatic tests at 70 °C and a relative humidity of 30 and 50 %, respectively, for 3 weeks—this corresponds to at least 2 years of standard condition for temperature and pressure. EDX spectra before and after the climatic tests have shown no significant changes in the line intensities (well below 5 %). Moreover, surface-sensitive depth profiling with Auger electron spectroscopy has confirmed that no alteration, e.g., oxidation, of the surface has occurred.

Thus, the prerequisite is given for a CRM to be applied also for the task of calibrating EDS efficiency. The X-ray spectra emitted by EDS-TM002 can be certified (see Table 2) by means of measurements carried out with a calibrated, reference EDS. The particular procedure to calibrate absolutely the efficiency of the reference EDS is described in the next section, followed by a section on the transfer of the efficiency calibration to a laboratory EDS. An overview flowchart with the individual steps of the EDS calibration procedure proposed is given in Fig. 5.

The certified values will be intensity ratios and not absolute intensities or elemental concentrations. Furthermore, two excitations, 10 and 30 kV, have been considered, so that a number of eight well-excited X-ray characteristic lines of the EDS-CRM can be taken into account for the direct calculation of the efficiency at those energies. The reference X-ray line to which the other lines shall be normalized has been chosen as Mn Kα, a line relatively well excited at both acceleration voltages. The efficiency value of the EDS at the energy of Mn Kα can be calculated in good approximation by just taking the transmission of the detector window into account. Note that the measurement uncertainties resulting from the homogeneity and stability measurements as well as those generated at the EDS calibration are contained in the

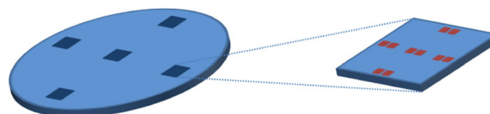

Fig. 4 Scheme with the 4″ coated silicon wafer showing the EDS-CRM samples selected for homogeneity and stability measurement of one batch (*left*) and detail with the areas selected for homogeneity measurements of one 8.5 mm×8.5 mm sample (*right*)

Table 2 Example of the certification of the EDS-CRM with the intensity ratio of eight X-ray characteristic lines as the certified parameter. The associated relative uncertainties are less than 5 % for all ratios apart from the normalized intensity ratios of C K and Zr Kβ which do not exceed 10 %

Normalized line intensity ratios	Reference value	
	10 kV	30 kV
C K/Mn Kα	4.2	–
Mn Lα/Mn Kα	2.7	–
Cu Lα/Mn Kα	2.2	–
Al K/Mn Kα	1.5	0.10
Zr Lα/Mn Kα	2.8	0.35
Cu Kα/Mn Kα	–	0.18
Zr Kα/Mn Kα	–	0.032
Zr Kβ/Mn Kα	–	0.0056

measurement uncertainties accompanying the intensity ratios given in the EDS-CRM certificate.

Absolute calibration of the efficiency of a reference EDS with synchrotron radiation

The calibration of X-ray detectors as used in EDS systems like SDD or lithium drifted Si detectors (Si(Li)) is performed routinely in the PTB laboratory at BESSY II by using the electron storage ring as primary X-ray source standard. It should be noted that this is the only metrology facility worldwide where such dedicated experiments may be performed. The number of stored electrons is exactly known and kept so low that a moderate flux of X-ray photons, in the range of a few thousands photons per second, can be adjusted and impinges on the EDS detector to be calibrated. To calculate the emitted spectrum with relative uncertainties of about 10^{-4}, not only the number of stored electrons but also the electron energy, the magnetic induction in the bending magnet, and the size of the flux-determining aperture and its distance from the source point have to be known with low uncertainty [3].

The calibration procedure using undispersed radiation requires a physical model for the calculation of the detection efficiency with absorbing layer thicknesses as variable

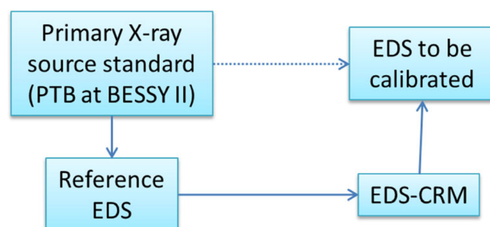

Fig. 5 Flowchart showing the sequence to carry out for calibrating the efficiency of an EDS by means of the EDS-CRM as an alternative to the "direct," absolute calibration at the PTB laboratory at BESSY II

parameters to be adjusted so that the measured spectrum can be reproduced [2, 3]. Figure 6a shows the X-ray spectra as measured with an SDD and the incoming spectrum, multiplied with the resulting efficiency and convoluted with a calculated response function. The latter step is necessary in order to take into account the considerable amount of counts registered in channels other than the excitation channel, i. e., in the main peak (around the excitation channel) which is provided with a low-energy tail (due to incomplete charge collection), in escape peak, sum peaks, fluorescence peaks, and a so-called shelf below the main peak down to zero, caused by artifacts in the front window materials [14]. The parameters of the SDD physical model, i.e., the thickness of the absorbing layers in the detector window front contact, are varied until the measured and calculated spectra coincide. The resulting model parameters and efficiency are shown in Fig. 6b together with the associated measurement uncertainties. These measurement uncertainties are of decisive relevance when the EDS-CRM spectra are certified based on measurements with such a reference detector. In order to validate the detector efficiency values as determined with the undispersed radiation approach described above, the sensitive low energy range up to 1.8 keV is counterchecked by means of measurements with monochromatic radiation at another beamline in the same laboratory of PTB [2]. At all energies close to absorption edges of B, C, N, O, Al, and Si, the results agree very well with the data from undispersed radiation. Similarly robust results have been obtained for the detector of the second EDS system, a Si(Li) EDS, calibrated for the same purpose of certification of the EDS-CRM.

Obtaining the absolute efficiency of the detector of an EDS system yields a calibrated system when the detector efficiency data is used in the data evaluation of the EDS system.

Calibration of the EDS efficiency in a SEM/EDX laboratory

Once having the EDS-CRM material accompanied with the certified emitted spectra at specified conditions, the absolute efficiency of an unknown EDS can be now determined on site, in common service SEM/EDX laboratories. Of course, the measurement uncertainties accompanying the determined efficiency must be larger than those attained with synchrotron radiation as described in the previous section. The EDS-CRM material must be measured at two accelerating voltages, 10 and 30 kV, and the eight intensity ratios as in Table 2 must be calculated from the measured spectra. By relating the measured intensity ratios to certified values, the efficiency of the EDS to be calibrated at nine energies is obtained, see Fig. 7 for the example of a Si(Li) EDS system successfully used for metrological measurements [15, 16] and for validating various models [17–19]. As mentioned above, the efficiency at the Mn Kα line energy is considered as the additional ninth point and can be calculated using the transmission of the detector

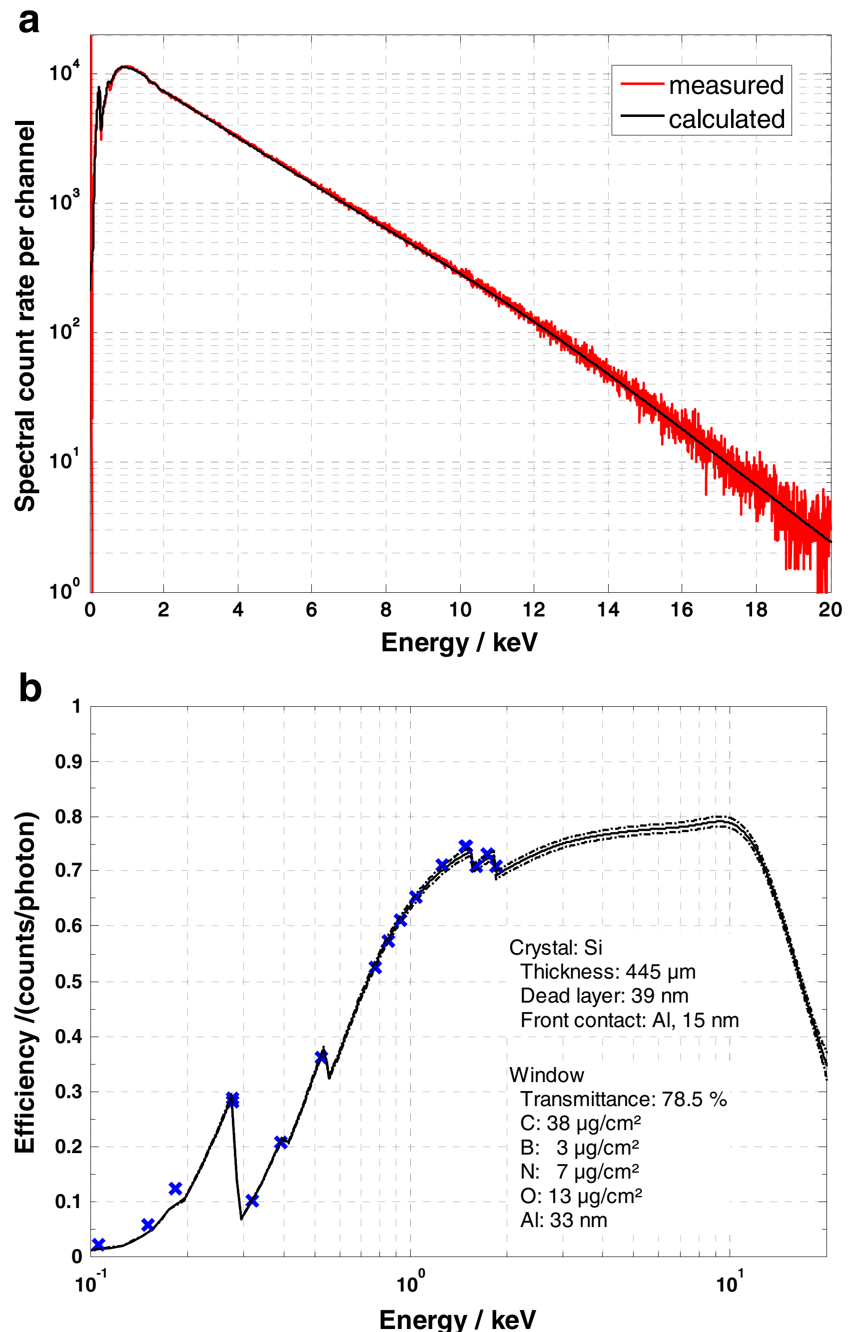

Fig. 6 **a** X-ray spectrum as measured with an SDD EDS (*red*) with undispersed synchrotron radiation at the PTB beamline @ BESSY II and corresponding calculated X-ray spectrum (*black*), multiplied with the detection efficiency and convoluted with the response functions. **b** Absolute detector efficiency of an SDD obtained by optimization of the SDD physical model parameters (see legend) to match the measured spectrum from Fig. 6a, together with the associated measurement uncertainties (*dashed lines*). The *dashed lines* indicate the confidence interval 2σ, i.e., 95 %. The *blue crosses* in the energy range below 1.8 keV represent the absolute efficiency calibration as determined using monochromatic radiation

window. The procedure was initially proposed by Alvisi et al. [20], however, at that time just as proof-of-principle, without considering consistently the traceability of the determined efficiency. Measurement uncertainties resulting mainly from the homogeneity and stability of the CRM, efficiency of the reference EDS, measurement parameters (counting statistics, background subtraction, etc.), and model fitting of the point efficiency are rigorously calculated as part of the ongoing certification.

To obtain the efficiency in the entire energy range up to 20 keV, a physical model as mentioned above must be applied. Thus, decisive EDS parameters such as layer thicknesses responsible for absorption in the detector (dead layer) and window and front contact can be extracted. Finally, these true parameters characterizing the EDS to be calibrated should be compared with the manufacturers' values. Ideally, the true efficiency, respectively the absorbing layer thicknesses determined, should be updated in the available quantification software.

Another practical approach to transfer the reference efficiency to other EDS systems on site has also been studied for the case of excitation with an XRF source at a SEM [21].

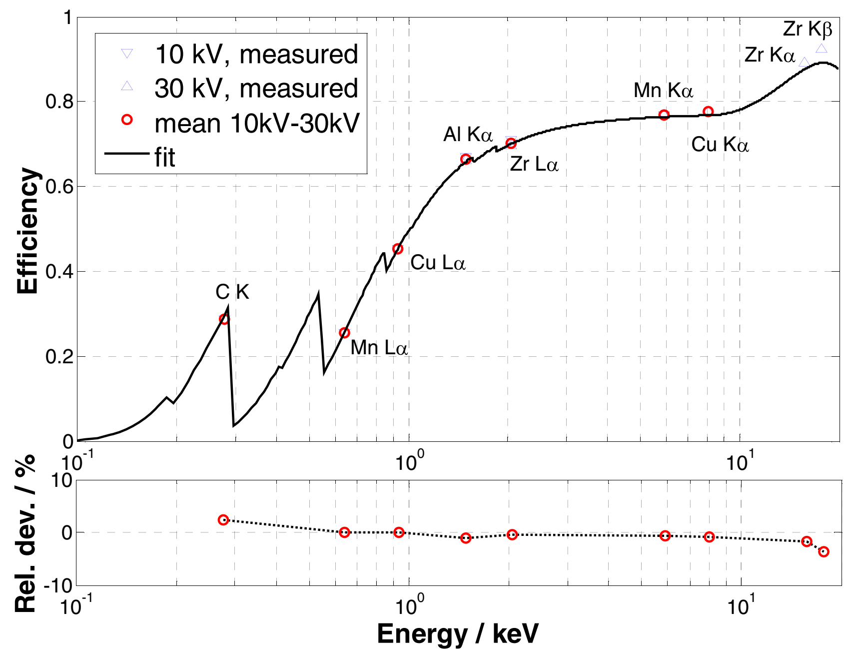

Fig. 7 An example of a measured efficiency of a Si(Li) EDS as determined by means of the EDS-CRM on site, in a SEM/EDX laboratory. The following parameters were used: Si crystal thickness, 3 mm; dead layer thickness, 0 nm; Ni front contact thickness, 11.8 nm, window transmittance, 77 % at Mn Kα energy; Al layer thickness, 23 nm; mass thicknesses of C, 48.6 μg/cm^2; B, 4.5 μg/cm^2; N, 3.6 μg/cm^2; and O, 43.4 μg/cm^2

However, the procedure has not been proven to be robust enough, mainly due to the rather large and undefined excitation volume in a light sample matrix as "reference" X-ray source for an EDS calibration.

EDS-TM003 for the low energy range

Modern EDS systems provide increasing performance in the low energy range below 1 keV. The capability to detect X-ray lines situated below 100 eV is reported by EDS manufacturers as state-of-the-art. Accordingly, the characterization of EDS performance below 1 keV is not only necessary but also more challenging as for the high energy range. The C K line is taken into account in the ISO 15632 standard for the EDS characterization; nevertheless, consideration of more K X-ray lines of light elements such as Be, B, N, and O can supply a more complete picture of EDS behavior in the range below 1 keV. Mostly, compounds like boron nitride are used as EDS test materials in the low energy range, either for quantification, if stoichiometry can be guaranteed [5], or simply as B K, C K, N K, and O K X-ray lines "provider" if oxidized and after carbon deposition. Pure materials like beryllium (Be K line at 108 eV), silicon (Si L lines at 92 eV), aluminum (Al L lines at 79 eV), lithium (Li K line at 54 eV), or magnesium (Mg L lines at 49 eV) are used by EDS manufacturers to demonstrate detection capabilities of the newest generation SDD EDS in the very low energy range, e.g., as reported in [22]. Moreover, stoichiometric compounds such as boron nitride or calcium carbonate are used to check the quantification accuracy in this delicate range.

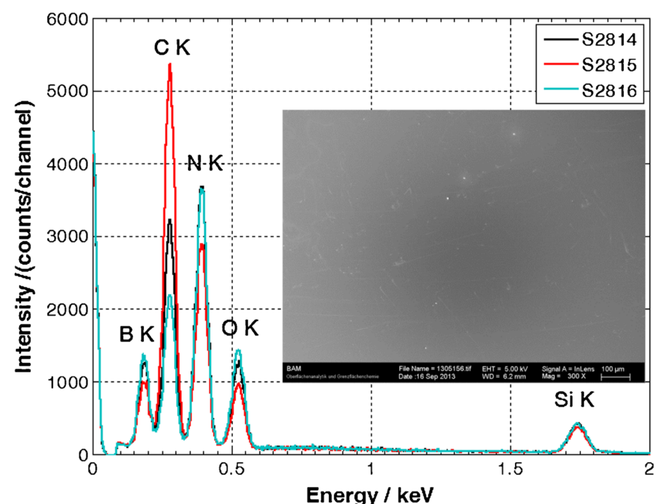

Fig. 8 The 3-kV X-ray spectra of three candidates for EDS-TM003 as a test material to characterize EDS performance below 1 keV. A representative SEM micrograph illustrating the very smooth surface morphology is inserted

Impelled by the success of the EDS-TM test material, we have tried to apply the same PLD deposition technology for a set of light elements in the form of a thick synthetic layer on a silicon wafer. Of course, by having elements like B, C, N, O, or F in one sample, it is expected that the stability of the new layer may be a critical parameter. Many optimization deposition trials have been undertaken in order to ensure both the layer stability and a reasonable elemental concentration for sufficiently intense K line peaks. One initial idea was to take Teflon® as a substrate in order to have F K in the emitted spectrum—and come hence in agreement with ISO 15632 where F K is an X-ray line at which the EDS energy shall be specified. However, we had to abandon the inclusion of F due high instability of the manufactured material.

The optimized elemental combinations in the new test material, EDS-TM003, foreseen for EDS characterization in the energy range below 1 keV, by only one measurement at the SEM/EDS, generate X-ray spectra as in Fig. 8. The B-C-N-O layer of total thickness of about 110 nm and being constituted from individual alternating B-C and C-N-O ultrathin layers is deposited on Si(100) wafer as a substrate. The mass fraction of carbon has been varied so that three samples of different compositions could be produced. According to Fig. 8, the optimal elemental composition corresponds to the X-ray spectrum in blue (sample S2816).

The stability and homogeneity measurements have been finished successfully, so that the EDS-TM003 can be scheduled to be commercially offered by BAM. A software package accompanying the new material is not planned. The periodical measurement of the EDS-TM003 under the same conditions is sufficient to alert distortions in the operation of an EDS in the low energy range, by simply comparing (overlapping) the measured spectrum with spectra taken earlier (preferably at the installation by the EDS manufacturer). Also, the direct comparison of EDS-TM003 spectra taken using different spectrometers under the same geometrical (take-off-angle) and excitation conditions at SEM directly shows differences in the performance of the spectrometers in the sensible energy range below 1 keV.

Conclusions

Many test and reference materials are employed by users and recommended by EDS manufacturers and by ISO standards to test different parameters characterizing the performance of an EDS. The new BAM test material whose certification is in progress will allow the rapid and simultaneous determination of all parameters including the EDS efficiency. For the emitted X-ray spectra of the EDS-CRM at 10 and 30 kV accelerating voltage in a SEM, the intensity ratios of the eight X-ray lines C K, Mn Lα, Cu Lα, Al K, Zr Lα, Cu Kα, Zr Kα, and Zr Kβ will be certified with respect to the intensity of the Mn Kα line. In order to ensure the traceability of these intensity ratios, measurements with a primary X-ray source standard at the PTB laboratory at BESSY II were carried out to calibrate the efficiency of the reference EDS which was used to certify the EDS-CRM spectra. The ongoing certification indicates relative measurement uncertainties associated to the certified intensity ratios below 5 %. For the ratios containing C K and Zr Kβ, the corresponding value is below 10 %. The practical procedure of determining the true efficiency of an EDS on site in a SEM/EDX laboratory by means of the new EDS-CRM is also presented.

Developments at BAM regarding other potential candidates for test materials in the increasingly interesting energy range below 1 keV are briefly discussed. The EDS-TM003 containing B, C, N, and O in such proportions that all their K lines are sufficiently intense at 3 keV excitation energy is the most suitable candidate planned to be commercialized by BAM in the near future.

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