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Microbeam analysis — Electron probe microanalysis — Guidelines for the determination of experimental parameters for wavelength dispersive spectroscopy

Analyse par microfaisceaux — Microanalyse par sonde à électrons — Lignes directrices pour la détermination des paramètres expérimentaux pour la spectroscopie à dispersion de longueurs d'onde

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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ISO 14594 was prepared by Technical Committee ISO/TC 202, *Microbeam analysis*, Subcommittee SC 2, *Electron probe microanalysis*.

Microbeam analysis — Electron probe microanalysis — Guidelines for the determination of experimental parameters for wavelength dispersive spectroscopy

1 Scope

This International Standard gives the general guidelines for the determination of experimental parameters relating to the primary beam, the wavelength spectrometer and the sample that need to be taken into account when carrying out electron probe microanalysis. It also defines procedures for the determination of beam current, current density, dead time, wavelength resolution, background, analysis area, analysis depth and analysis volume.

This International Standard is intended for the analysis of a well-polished sample using normal beam incidence, and the parameters obtained may only be indicative for other experimental conditions.

This international standard is not designed to be used for energy dispersive X-ray spectroscopy.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO Guide 25:1990, *General requirements for the competence of calibration and testing laboratories*

3 Terms and definitions

For the purposes of this International Standard the following terms and definitions apply.

3.1
analysis area
two-dimensional region of sample surface measured in the plane of that surface from which the full signal or a specified percentage of that signal is detected

3.2
analysis depth
distance from the sample surface to the bottom normal to the surface from which the full signal or a specified percentage of that signal is detected

3.3
analysis volume
three-dimensional region of a sample from which the full signal or a specified percentage of that signal is detected

3.4
background
non-characteristic component of an X-ray spectrum arising from the X-ray continuum

3.5
beam current
electron current contained within the focused beam

3.6
beam current density
beam current incident on the sample per unit area

3.7
dead time
time associated with the measurement of a signal photon in a detector and/or counting system, representing the time that the system is unavailable to process the next photon

3.7
wavelength resolution
full peak width at half maximum of a peak obtained from a single X-ray transition

4 Abbreviated terms

— EPMA: Electron Probe MicroAnalysis

— FWHM: Full Width at Half Maximum

— WD: Wavelength Dispersive

5 Experimental parameters

5.1 General

The parameters given in 5.2.1 to 5.2.4 should be known and recorded. Checking the calibration of beam energy, beam current and magnification together with counter dead time should be included in the maintenance schedule of the instrument.

5.2 Parameters related to the primary beam

5.2.1 Beam energy

The beam energy may typically range from 2 keV to 30 keV. In most cases the calibration of the beam energy is not critical for qualitative analysis.

NOTE Calibration is very critical in the case of use of low overvoltage ratio or during measurements relating to layer thickness or elemental depth distributions.

5.2.2 Beam current

Because X-ray peak intensity is directly proportional to beam current, the precision of the measurement of the beam current should be better than the precision required for quantitative analysis.

NOTE The beam current stability over long periods of time is absolutely essential for consistent quantitative analysis. The beam current stability should be tested periodically, especially prior to quantitative calibration and analysis. It is possible to compensate for small changes in beam current if this is recorded prior to or following each measurement. Then all X-ray peak and background measurements should be scaled appropriately by $I_\mathrm{i}/I_\mathrm{m}$ where I_i is the initial beam current and I_m is the beam current at the time of the measurement.

5.2.3 Beam current density

Beam current density is especially important when analysing beam sensitive materials. The current density in a focused probe may exceed 10^4 Am^{-2}. The effective current density may be reduced for a measurement by lowering the incident electron beam current or, where lateral resolution is not critical, by either defocusing or rastering the probe. If a rastered probe is used a similar scan should be used for comparative measurements on standards and other specimens because the effective spectrometer efficiency for the selected wavelength decreases as a function of the beam deflection. See NOTE 3 in 5.3.5.

5.2.4 Magnification

To properly define the dimensional scale for line-scans and images acquired by deflecting the primary electron beam, it is essential to calibrate the magnification scale while operating in the scanning electron mode.

5.3 Parameters related to wavelength dispersive (WD) spectrometers

5.3.1 General

An instrument may be fitted with one or more WD spectrometers, each with a number of diffracting crystals that may be selected to cover a particular range of X-ray wavelengths depending on the line of the analysed element. The following parameters are important for the proper operation of WD spectrometers.

5.3.2 Take-off angle

The take-off angle affects quantitative analysis. Any comparison of measurements from instruments with different take-off angles should be taken into account and the procedures used be noted in the analysis report.

NOTE The value of this angle, which is normally fixed, is provided by the instrument manufacturer.

5.3.3 Wavelength resolution

The spectral resolution depends on many parameters:

- crystal material (and Miller indices of the crystal planes);
- the radius of curvature of the diffracting crystal (fully focusing vs. semifocusing crystal);
- the presence of a crystal mask (if semifocusing crystal);
- the size and position of the counter entrance window or of the entrance slit if present.

All these settings determine the wavelength resolution of the measured X-ray spectrum and the observed line-width (FWHM) of the characteristic X-ray peaks.

Resolution can also influence the ability of the system to discriminate against overlapping peaks, background signals and the sensitivity of measurements to specimen height and beam position on the specimen.

5.3.4 X-ray detector and counting chain

Many spectrometers use a gas-filled proportional counter to detect X-rays. The magnitude of the output pulses from these detectors is determined by the incident X-ray energy and/or the voltage applied to the counters. Two discriminators are used to select the pulse of interest. A low discriminator setting is used to eliminate pulses due to noise, while a high discriminator setting excludes pulses from high order reflections of more energetic X-rays. Optimum settings depend on the X-ray lines of interest.

It is important to set the discriminator to ensure that any unintended shift in pulse amplitude, due for example to high count rates or changes in atmospheric temperature and pressure (flow counter), has no significant effect on the measured count rate.

NOTE Because X-ray counting efficiency decreases with increasing count rate, it is important to correct the measured count rate for the effect of the dead time. In an automated system the discriminator settings may be set automatically. These settings should be routinely checked to ensure proper automatic operation.

5.3.5 Peak location (wavelength)

Under normal circumstances the wavelength which has the maximum peak intensity is used to define the location of an X-ray peak. It is necessary, using suitable reference materials, to periodically check and correct for the difference in a peak's theoretical position and its actual measured position on a given spectrometer and diffraction crystal. The time between checks will depend on the stability of the instrument spectrometers.

The measured maximum intensities of peaks which have narrow FWHM values are strongly affected by the errors in peak location. The peak intensity may be changed due to the chemical state and polarization effects.

NOTE 1 If the element in the sample of interest is in a different chemical state than that of the reference material, then the shape of the characteristic X-ray peak may be different for sample and standard. In this case the peak maximum may not provide a reliable measure of the total peak intensity and an alternative approach, such as peak area measurements, may be required to obtain reliable results. These chemical state effects are particularly important for X-ray peaks with low energy values.

NOTE 2 If a crystalline sample causes the polarization effects in relation to the position between the sample and the analysis crystal, the peak shape and location may be changed. This can be checked by rotating the sample around an axis perpendicular to the electron beam and observing the effect on peak shape and location. The problem may occur in systems with symmetry lower than cubic and higher than triclinic and is worst when the Bragg angle is close to 45°. The phenomenon has been found in graphites [1] and certain borides [2]. These can be much reduced by using peak area measurements.

NOTE 3 The position of the peak maximum varies with deviation of the probe from the focal point of the spectrometer on the sample. Calibration measurements and quantitative analysis on the sample should normally be made with the probe in the same position relative to this focal point, and using the same beam defocus or raster setting if applied. For all quantitative and qualitative analyses carried out using a defocused and scanned beam, the area of the sample surface irradiated should not be so large as to cause a significant fall in X-ray counts from that obtained with the static focused electron beam.

5.3.6 Background

The characteristic X-ray peaks are superimposed on a background of continuum X-rays.

To properly calculate the intensities of characteristic X-rays, the magnitude of this background needs to be determined and corrected if it is statistically significant.

5.4 Parameters related to the sample

5.4.1 Sample stage

High precision X, Y, Z stages allow the sample and standards to be accurately positioned under the electron beam. By using an attached optical microscope, the user can set the height of the sample so the axis of the WD spectrometer and the primary beam position coincide at the surface of the sample. Orthogonality between the electron beam (the optical axis) and the sample stage is essential in order to perform a proper quantitative analysis. A check on the adjustment of the optical microscope should be included in the routine instrument maintenance schedule.

In an automatic mode of operation, where the measurements are to be made at pre-set points on the standards and the sample, it is important to know the reproducibility with which the stage retrieves pre-set points and to adopt appropriate strategies to overcome any obvious limitations.

5.4.2 Surface roughness

For best results the surface roughness of the specimen should be minimized.

5.4.3 Analysis volume

Analysis volume is determined by the incident beam area, the depth of beam penetration, the spread of the incident beam within the sample and the energy of the characteristic X-ray line. This analysis volume may be significantly increased by unwanted fluorescence effects which are caused by the characteristic and continuum X-rays.

6 Procedures and measurements

6.1 General

The following procedures should be adopted to determine a number of critical parameters.

6.2 Beam current

6.2.1 Measurement

Measure the beam current using a Faraday cup. It should be positioned after the final aperture. If the measurement is carried out at another position, the relationship between the above mentioned position and this position shall be shown.

6.2.2 Density

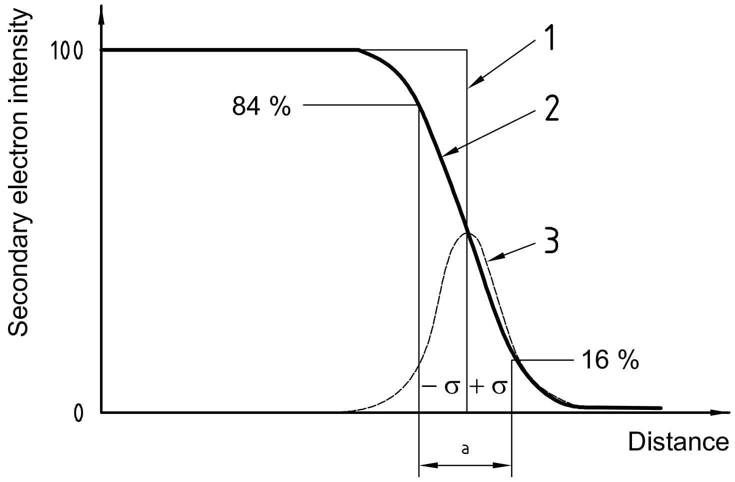
This procedure gives an average current density within the beam. The local current density may be calculated assuming a Gaussian beam profile and using the value measured in 6.2.2.1 a) or 6.2.2.1 b).

6.2.2.1 The diameter of the beam shall be defined by one of the following methods:

a) the diameter of electron beam shall be defined as the interval where the emitted secondary electron intensity drops from 84 % to 16 % of the maximum peak intensity, which is equivalent to two standard deviations (2σ) of the error curve (see Figure 1). This measurement should be done such that the primary electron beam crosses a knife edge at a right angle;

b) the diameter of area exhibiting optical fluorescence for a material such as aluminum oxide, zirconium oxide or thorium oxide whereby that diameter is determined by using an optical microscope. This measurement should be done when the beam diameter is more than 5 µm.

6.2.2.2 The beam current density shall be calculated by dividing the incident beam current (as defined in 6.2.1) by the area of the electron beam. For a round defocused beam the area would be $\pi d^2/4$, where d is the beam diameter.



Key

1	true boundary
2	measured curve
3	error function

a	Diameter.

Figure 1 — Method for measuring beam diameter

6.3 Parameters related to measured peaks

6.3.1 Dead time correction

Measure the beam current, i, in accordance with 6.2.1 and the count rate (N).

6.3.1.1 As shown in Figure 2, make a calibration curve by using the count rate, N, divided by the beam current, i, values as the ordinate values and the count rate, N, as the abscissa values.

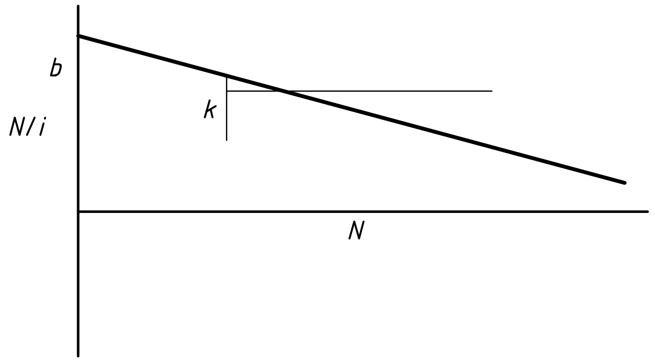


Figure 2 — Counting loss by dead time

In order to confirm the linearity of the current measuring device, it is advisable to monitor the count rate, n, of a low intensity line at the same time as the count rate, N, of a high intensity line; the ratio $n{:}i$ should be constant for the whole range of measurements.

Typically a Kα line may be used to determine N and the corresponding Kβ line for n.

NOTE If the probe current cannot be measured accurately, the dead time can be determined by measuring the ratio of two X-ray intensities as a function of count-rate on two X-ray spectrometers [2, 3].

6.3.1.2 Determine the gradient, k, and the intercept value, b, at the ordinate axis from the calibration curve.

Calculate the dead time, τ, by using equation (1).

$$\tau = \frac{-k}{b} \qquad (1)$$

6.3.1.3 Calculate the true count rate, N_0, by using equation (2).

$$N_0 = \frac{N}{1 - N \times \tau} \qquad (2)$$

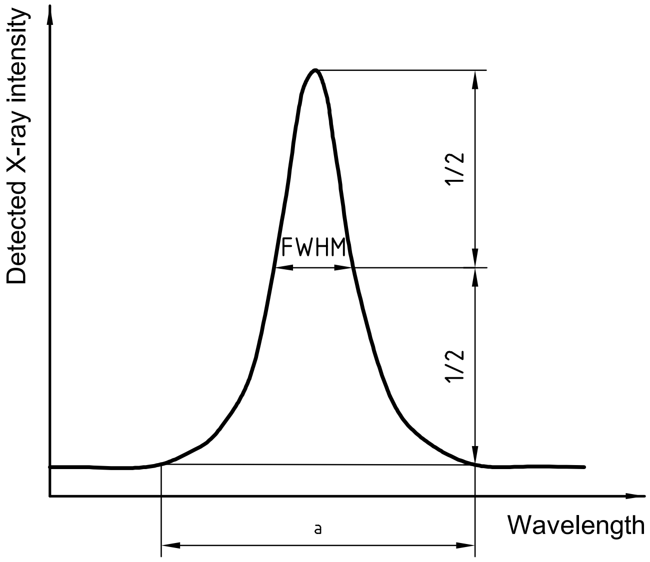
For accurate measurements the count rate should be restricted so that the correction for dead time does not exceed 5 %.

6.3.2 Wavelength resolution of detected characteristic X-ray peaks

6.3.2.1 Measure a characteristic X-ray intensity versus wavelength spectrum for the elements of interest by measuring the intensity of the X-ray signals while scanning over the wavelengths of interest.

6.3.2.2 Calculate the wavelength resolution of the detected characteristic X-rays by using the following definition.

After subtracting the background (see 6.3.3) wavelength resolution is equal to FWHM as shown in Figure 3.



[a] Background.

Figure 3 — Definition of FWHM