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Microbeam analysis — Analytical electron microscopy — Method for the determination of energy resolution for electron energy loss spectrum analysis

Analyse par microfaisceaux — Microscopie électronique analytique — Méthode de détermination de la résolution énergétique pour l'analyse spectrale de la perte d'énergie des électrons

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Foreword

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This document was prepared by Technical Committee ISO/TC 202, *Microbeam analysis*, Subcommittee SC 3, *Analytical electron microscopy*.

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Introduction

In order to understand the chemical composition, the atomic bonding and the electronic structure, electron energy loss analysis is often performed with the scanning transmission electron microscope or the transmission electron microscope (S/TEM) equipped with the electron energy loss (EEL) spectrometer.

In the analysis using EEL spectrometer system, the energy loss of incident electrons by the inelastic interaction via phonon and plasmon excitations, intra- and inter-band transitions and the inner shell ionization can be measured. The inner shell ionization is particularly useful and important as it gives the information on chemical composition of materials. For the precise analysis based on the energy loss peak decomposition and its energy shifts, it is vitally important to understand the energy resolution of the EEL spectrometer system. However, the determination method of the energy resolution is not standardized yet.

This document provides the procedures for energy step calibration and energy resolution determination useful for the electron energy loss spectrum analysis in the S/TEM equipped with the EEL spectrometer.

Microbeam analysis — Analytical electron microscopy — Method for the determination of energy resolution for electron energy loss spectrum analysis

1 Scope

This document specifies a determination procedure of energy resolution in the scanning transmission electron microscope or the transmission electron microscope equipped with the electron energy loss (EEL) spectrometer.

This document is applicable to both in-column type EEL spectrometer and post-column type EEL spectrometer. These EEL signal detecting systems are applicable to a parallel detecting system and a serial detecting system.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at https://www.iso.org/obp

3.1
beam diameter
full width at half maximum (FWHM) of the electron beam intensity profile for the STEM observation

3.2
Boersch effect
energy spread of electron beam due to *Coulomb interaction* (3.5) between electrons in the beam

3.3
channel
range of one pixel of the detector in the *parallel detection* (3.17) EELS

3.4
collection angle
EELS entrance aperture diameter divided by a camera length and a *geometric factor* (3.13) for the STEM or the TEM diffraction mode, or EELS entrance aperture diameter divided by the distance from crossover of the lens in front of the EEL spectrometer to the EELS entrance aperture for imaging mode of the energy-filtering TEM

3.5
Coulomb interaction
repulsion of electrons by electric charge

3.6
detection plane
plane where energy dispersed electron focus

3.7
electron energy loss
energy shift of the electron kinetic energy due to the inelastic scattering in solids

3.8
energy dispersion
degree of change in position of the dispersed electrons at the *detection plane* (3.6) per unit energy change

3.9
energy resolution
FWHM of the *zero-loss* (3.21) peak

3.10
energy step
energy selecting window (3.11) per *channel* (3.3) in the *parallel detection* (3.17) EELS, or energy range limited by the width of energy selecting slit in the *serial detection* (3.20) EELS

3.11
energy selecting window
energy range for selection of a specific energy loss value

3.12
entrance aperture
aperture for limiting the *collection angle* (3.4) of the EEL spectrometer

3.13
geometric factor
ratio of distance from a projector lens to an EELS entrance aperture to distance from the projector lens to an image detection device

3.14
in-column type EELS
EELS system with the EEL spectrometer located in the imaging system of the TEM

3.15
irradiation diameter
diameter of the electron beam irradiation region for the TEM observation

3.16
K edge
energy loss related to K shell electron transition to the lowest empty state

3.17
parallel detection
simultaneous EELS signal detection for all energy-dispersed electrons focused on the *detection plane* (3.6)

3.18
plasmon-loss
energy loss of electron due to excitation of the quantized plasma oscillations of electrons

3.19
post-column type EELS
EELS system with the EEL spectrometer located behind the imaging/detecting system of the TEM

3.20
serial detection
EEL spectrum detection by scanning the dispersed electrons across the energy selecting slit in front of the detector

3.21
zero-loss
unscattered and elastically scattered electrons (with only minimal loss of energy due to phonon excitation), giving rise to an intensity peak or the position of which defines zero in the electron energy loss spectrum

[SOURCE: ISO15932: 2013, 2.2.1.1]

4 Symbols and abbreviated terms

B	spatial width of energy selecting window in the serial detection of the EELS
CCD	charge coupled device
CFE	cold field emission
CH_1	sum of Ch_1(G, P) and the Ch_1(G, C-K). In the parallel detection system, CH_1 is the number of channels between the zero-loss peak and carbon K edge of graphite. In the serial detection system, CH_1 is distance between the zero-loss peak and carbon K edge of graphite.
CH_2	number of channels between the zero-loss peak [Figure 5, key 1] and the peak E_{CZLP} [Figure 5, key 2] on the calibrated energy step δE_{1C} in the parallel detection EELS. In the serial detection EELS, CH_2 is distance between the zero-loss peak [Figure 5, key 1] and the peak E_{CZLP} [Figure 5, key 2] on the calibrated energy step δE_{1C}.
CH_3	number of channels between the zero-loss peak and the peak E_{CZLP} on the energy step δE_2 in the parallel detection EELS. In the serial detection EELS, CH_3 is distance between the zero-loss peak and the peak E_{CZLP} on the energy step δE_2.
CH_4	number of channels corresponding to FWHM of the zero-loss peak on the calibrated energy step δE_{2C} in the parallel detection EELS. In the serial detection EELS, CH_4 is distance between the zero-loss peak and the peak E_{CZLP} on the calibrated energy step δE_{2C}.
Ch_1(G, C-K)	number of channels of the range from the graphite plasmon-loss (π + σ) peak [Figure 3, key 1] to carbon K edge $E_{C\text{-}K}$ [Figure 3, key 2] on the energy step δE_1 in the parallel detection system. In the serial detection system, Ch_1(G, C-K) is distance between the graphite plasmon-loss (π + σ) peak [Figure 3, key 1] and carbon K edge $E_{C\text{-}K}$ [Figure 3, key 2] on the energy step δE_1.
Ch_1(G, P)	number of channels of the range from the zero-loss peak [Figure 2, key 1] to the graphite plasmon-loss (π + σ) peak E_{CZLP} [Figure 2, key 2] on the energy step δE_1 in the parallel detection system. In the serial detection system, Ch_1(G, P) is distance between the zero-loss peak [Figure 2, key 1] and the graphite plasmon-loss (π + σ) peak E_{CZLP} [Figure 2, key 2] on the energy step δE_1.
CMOS	complementary metal oxide semiconductor
CRM	certified reference material
C1s	carbon K shell binding energy of graphite measured by the XPS
D	energy dispersion on the recording device of the EEL spectrometer
d	sample thickness of the electron beam irradiated area
E	value of electron energy loss such as plasmon-loss and ionization-loss
$E_{BN\text{-}P}$	measured plasmon-loss (π - π*) peak energy of boron-nitride under the condition of calibrated energy step δE_{1C}

E_{CZLP}	position of noticed low-loss peak close to the zero-loss peak
$E_{C\text{-}K}$	carbon K edge energy in the EELS
EEL	electron energy loss
EELS	electron energy loss spectroscope/spectroscopy
FWHM	full width at half maximum
GUM	guide to the expression of uncertainty in measurement
m	total number of available channels in the parallel detection of the EELS
n	iteration number in acquisition of electron energy loss spectrum
RM	reference material
STEM	scanning transmission electron microscope/microscopy
S/TEM	scanning transmission electron microscope/microscopy or transmission electron microscope/microscopy
s	detector spatial resolution for the parallel detection. For the serial detection, s is slit width of energy selecting window
TEM	transmission electron microscope/microscopy
t	acquisition time in acquisition of electron energy loss spectrum
XPS	X-ray photoelectron spectroscope/spectroscopy
ZLP	zero-loss peak
ΔE	energy resolution
ΔE_r	theoretical energy resolution
ΔE_{SO}	energy broadening
δE_1	selected energy step in the first energy calibration. In the parallel detection system, δE_1 is selected from the preset value. In the serial detection system, δE_1 is derived from the energy width and its spatial width in the energy selection window
δE_{1C}	calibrated value of energy step δE_1
δE_2	selected energy step in the second energy calibration. In the parallel detection system, δE_2 is selected from the preset value. In the serial detection system, δE_2 is derived from the energy width and its spatial width in the energy selection window.
δE_{2C}	calibrated energy step of energy step δE_2 by the second energy calibration step
δE_S	energy width of the energy-selecting window in the serial detection system of the EELS
λ	mean free path of electron inelastic scattering
π	π-bonding state
π*	π-antibonding state

σ	σ-bonding state
(π - π*)	resonant oscillation of the π-bonding state and the π-antibonding state
(π + σ)	resonant oscillation of the π-bonding state and the σ-bonding state

5 Definition of the energy resolution

The theoretical energy resolution ΔE_r is given from a convolution of an electron beam energy spread and a spectrometer resolution. The theoretical energy resolution is shown as Formula (1)[2].

$$(\Delta E_r)^2 \approx (\Delta E_0)^2 + (\Delta E_{SO})^2 + (s / D)^2 \quad (1)$$

where

ΔE_r is theoretical energy resolution

ΔE_0 is energy spread of the primary electron beam

NOTE ΔE_0 is affected by energy width of electron source and the Boersch effect.

ΔE_{SO} is broadening of energy

NOTE ΔE_{SO} is affected both the spectrometer focusing and the angular width of inelastic scattering.

s is a detector spatial resolution for the parallel detection. For the serial detection, s is a slit width of energy selecting window.

D is an energy dispersion of the spectrometer.

In addition, acquisition time t and acquisition iteration number n influence the energy resolution ΔE_r.

Measurement of energy resolution ΔE_r is not easy because of the complicated formation of the EELS system. It is well known that the full width at half maximum of the zero-loss peak is proportional to the energy resolution ΔE_r. Actually, FWHM of the zero-loss peak is very often used as the energy resolution[3]. The energy resolution ΔE is also defined as FWHM of the zero-loss peak in this document.

6 Reference materials and energy determination

6.1 General

In order to determine the energy resolution of the EELS equipped in the S/TEM, it is indispensable to calibrate the energy scale in advance. In this section, material selection for the energy scale calibration and the procedure for determining the energy scale are described.

6.2 Materials selection for energy scale calibration

For the energy resolution determination, calibration of the energy scale is necessary. As an EEL spectrometer cannot calibrate energy scale by itself, the reference material is necessary for the calibration. Since the energy calibrated certified reference materials (CRMs) and/or reference materials (RMs) are not available, it is necessary to select appropriate materials aiming to energy scale calibration, as (internal) reference materials. The following characteristics are required for the material.

— Easy to obtain

— Easy to handle,

— Homogeneous,

— Stable,

— Having loss peaks at a low-loss energy region,

NOTE 1 For measuring the energy resolution, energy scale calibration is needed to perform within loss energy region such as zero to several hundred electronvolt.

— Non-chargeable.

NOTE 2 In the first step of energy scale calibration on the EELS, loss energy known sample is needed. The loss energy value is obtained by the XPS analysis of the sample. Non-chargeable material is needed for XPS measurements.

In this document, graphite is recommended and used as a reference sample for the coarse energy scale calibration. The other reference sample for the following fine energy scale calibration should be selected from the materials, which has low-loss EELS peak, such as boron-nitride.

6.3 Binding energy measurement of graphite in the XPS

XPS C1s (carbon K shell binding energy) peak and EELS carbon K edge $E_{\text{C-K}}$ are equivalent. The correspondence of the energy values between XPS C1s and EELS carbon K-edge is described in Annex B. XPS measurement of C1s peak shall be done about graphite standard sample with calibrated XPS spectrometer.

The XPS shall be calibrated by ISO 15472:2010[4].

7 Measurement procedure and energy resolution determination

7.1 General

In this subclause, the energy scale calibration of EELS and the procedure for determining energy resolution are described. Annex A shows an example of actual measurement using this procedure.

A flowchart of measurement procedure is shown in Figure 1.

