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Microbeam analysis — Scanning electron microscopy — Guidelines for calibrating image magnification

Analyse par microfaisceaux — Microscopie électronique à balayage — Lignes directrices pour l'étalonnage du grandissement d'image

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

The committee responsible for this document is ISO/TC 202, *Microbeam analysis*, Subcommittee SC 4, *Scanning electron microscopy (SEM)*.

This second edition cancels and replaces the first edition (ISO 16700:2004), which has been technically revised.

Introduction

The scanning electron microscope is widely used to investigate the surface structure of a range of important materials such as semiconductors, metals, polymers, glass, food and biological materials, and this International Standard is relevant to the need for magnification calibration of the images. It describes the requirements for calibration of the image magnification in the scanning electron microscope using a reference material or a certified reference material.

Microbeam analysis — Scanning electron microscopy — Guidelines for calibrating image magnification

1 Scope

This International Standard specifies a method for calibrating the magnification of images generated by a scanning electron microscope (SEM) using an appropriate reference material. This method is limited to magnifications determined by the available size range of structures in the calibrating reference material. This International Standard does not apply to the dedicated critical dimension measurement SEM.

2 Normative references

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

ISO/IEC 17025:2005, *General requirements for the competence of testing and calibration laboratories*

ISO Guide 30, *Reference materials — Selected terms and definitions*

ISO Guide 34, *General requirements for the competence of reference material producers*

ISO Guide 35, *Reference materials — General and statistical principles for certification*

3 Terms and definitions

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

3.1
scanning electron microscope
SEM
instrument that produces magnified images of a specimen by scanning its surface with an electron beam

3.2
image
two-dimensional representation of the specimen surface generated by *SEM* (3.1)

Note 1 to entry: A photograph of a specimen taken using an SEM is a good example of an image.

3.3
image magnification
ratio of the linear dimension of the scan display to the corresponding linear dimension of the specimen scan field

3.4
scale marker
line/generated line (intervals) on the *image* (3.2) representing a designated actual length in the specimen

3.5
reference material
RM
material, sufficiently homogeneous and stable with respect to one or more specified properties, which has been established to be fit for its intended use in a measurement process

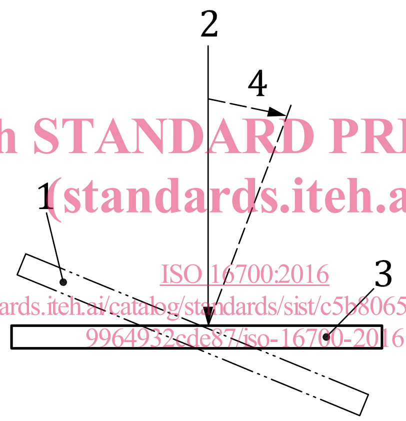
3.6
certified reference material
CRM
reference material (3.5) characterized by a metrologically valid procedure for one or more specified properties, accompanied by a certificate that provides the value of the specified property, its associated uncertainty, and a statement of metrological traceability

Note 1 to entry: For the purposes of this International Standard, an RM/CRM possesses pitch pattern(s) with the desired range of pitch size(s) and accuracy, to be used for the calibration of the *image magnification* (3.3).

3.7
calibration
set of operations which establish, under specified conditions, the relationship between the magnification indicated by the *SEM* (3.1) and the corresponding magnification determined by examination of an *RM* (3.5) or a *CRM* (3.6)

3.8
tilt angle
angle of the inclined specimen surface from the plane perpendicular to the electron beam axis

Note 1 to entry: See Figure 1.



Key

1 tilted specimen

2 electron beam

3 specimen

4 tilt angle

Figure 1 — Tilt angle

3.9
display
analog or digital device used for visualization of *images* (3.2)

Note 1 to entry: Examples of display are a cathode ray tube, plasma display panel, liquid crystal display, etc.

3.10
working distance
distance between the specimen surface and the bottom plane of the objective lens of the *SEM* (3.1)

3.11
pitch
closest separation of two similar features on a specimen which are equivalent points on a repeat pattern

3.12
accuracy
closeness of agreement between a test result and the accepted reference value

Note 1 to entry: A "test result" constitutes the observed values of a *pitch* (3.11) of a *CRM* (3.6) obtained by the procedure outlined in this International Standard.

Note 2 to entry: The term "accepted reference value" is a value certified by a national or an international calibrating laboratory. There will be an uncertainty associated with this value which should also appear on the certificate.

Note 3 to entry: Accuracy and precision are different. Precision is defined as the closeness of agreement between independent test results obtained under stipulated conditions. See ISO 5725-1.

4 Image magnification

4.1 Scale marker

To indicate magnification, superimpose on the image a scale marker and the corresponding length, in SI units, that it actually represents on the specimen. An example is shown in Figure 2.

NOTE In Figure 2, the length indicated by the arrows corresponds to 500 nm after the calibration.

Figure 2 — Scale marker and its length

4.2 Expressing magnification

Magnification of an image is given by a number representing the number of times the object has been magnified and it is accompanied by the symbol "×" (e.g. 100 ×, 10 000 ×, 10k × or × 100, × 10 000, × 10k, where 100, 10 000 and 10k are magnitude numbers).

NOTE 1 It is not always necessary to show the magnification when the scale marker is shown on the image.

NOTE 2 The magnification shown on the image corresponds to a chosen output device, which can be a display monitor or a printer or a photographing device. The scale marker shown on the image is independent from the output device chosen by the operator of the SEM. The magnification shown corresponds to the scale marker only when the image is displayed or printed on the operator-chosen output device.

5 Reference material

5.1 General

See ISO Guide 30.

For calibrating the magnification of an image, wherever possible, choose a CRM that is produced in accordance with ISO Guide 34 and certified in accordance with ISO Guide 35.

When a suitable CRM is not available, an RM produced in accordance with ISO Guide 34 may be used.

5.2 Requirements for CRM

Ensure that the chosen CRM

— is stable with respect to vacuum and repeated electron beam exposure,

— provides good contrast in the SEM image,

— is electrically conductive,

— can be cleaned to remove contamination occurring during normal use without causing mechanical damage or distortion, and

— has an associated valid calibration certificate.

5.3 Pitch patterns on CRM

Pitch patterns on the CRM may be in any one or more of the following forms:

— an orthogonal cross grid;

— a line array;

— a dot array;

— an orthogonal dot array.

Ensure that the chosen CRM contains pitch patterns that allow for calibration in at least one direction, and that the uncertainty in the pitch patterns is consistent with the targeted accuracy.

NOTE 1 There are cases where the CRM contains pitch patterns both in X and Y directions so that the measurements can be performed in orthogonal directions without the necessity of mechanically rotating the CRM. In some cases, the CRM additionally contains other structures for testing image distortion and/or resolution.

NOTE 2 There are instances where the chosen CRM has different-sized pitch patterns to cover the whole range of magnifications for which calibration is needed. It can also be necessary to have more than one CRM to cover the desired range of magnifications.

5.4 Storage and handling

Store the CRM in a desiccating cabinet or in a vacuum container.

NOTE To ensure minimal handling of the actual CRM, it can be permanently mounted on a stub.

Handle the CRM using fingerstalls, clean room gloves or tweezers.

Visually inspect the CRM surface for contamination and deterioration, as this may affect calibration. Do not use the CRM if it is damaged or grossly contaminated.

Remove any dust, loose debris or other contamination from the CRM using clean dry air or nitrogen gas, taking care not to damage the CRM.

Check the calibration of the CRM at intervals by comparison with other CRMs; record the results. The frequency of verification may depend on the nature and usage of the CRM.

Use the CRM for calibration purposes only.

6 Calibration procedures

6.1 General

Parameters that influence the resultant magnification of an SEM may cause systematic errors. These are listed in Annex B.

The stability of the SEM will be a major factor in determining the calibration interval. Initially, it will be necessary to perform calibration at frequent intervals in order to verify that the SEM is stable.

The results obtained will provide an estimate of the reproducibility within the laboratory and the bias inherent in both the display and the data automatically superimposed on any output.

The selection of the CRM depends on the magnification being used and accuracy required. For the purposes of this International Standard, ensure that the accuracy of calibration is better than 10 %.

6.2 Mounting CRM

At the time of mounting the specimen, ensure that handling of the CRM is carried out in accordance with 5.4.

Mount the CRM in accordance with the SEM and the CRM manufacturer's instructions.

Ascertain that there is a good electrical contact between the CRM and the specimen stage of the SEM.

Check that the CRM is securely fixed on the specimen stage so that it does not move from its mounting. This enables one to minimize any image degradation caused by vibration.

6.3 Setting SEM operation conditions for calibration

Evacuate the specimen chamber to the working vacuum in accordance with the SEM manufacturer's instructions.

Optimize the electron beam brightness and alignment in accordance with the SEM manufacturer's instructions.

Set tilt angle to 0°, following the SEM manufacturer's instructions so that CRM surface is perpendicular to the electron beam axis during operation.

Check the tilt of the CRM by the following procedures.

a) Turn off the tilt angle correction, the scan rotation and the zoom control of the magnification.

b) Select the imaging mode (secondary electron and/or back scattered electron).

c) Bring the image into focus without visible stigmatic distortions in the image.

d) Select the magnification at which the entire area of measurement is visible.

e) Determine the tilt position where the measured value of pitch is maximum. If the difference of measured values is not found, assume that the tilt angle is 0°. Carry out subsequent recording of the image in this position.

   NOTE 1 If the image of the whole area cannot be brought into focus, then it is necessary to remount the CRM or readjust the mechanical alignment of the SEM.

f) Select the accelerating voltage and the working distance for which the calibration is to be performed and bring the CRM into the correct position using the specimen stage controls.

g) Wait until the instrument is fully stabilized at the desired operating conditions in accordance with the SEM manufacturer's instructions.