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# Metallic coatings — Measurement of coating thickness — Scanning electron microscope method

*Revêtements métalliques — Mesurage de l'épaisseur de revêtement — Méthode au microscope électronique à balayage*



**ISO/CEN PARALLEL PROCESSING**



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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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This document was prepared by Technical Committee ISO/TC 107, *Metallic and other inorganic coatings*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 262, *Metallic and other inorganic coatings*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).



This second edition cancels and replaces the first edition (ISO 9220:1988), which has been technically revised.

The main changes are as follows:

— addition of two further calibration methods in 5.2, 8.2, and 8.3;

— deletion of technically outdated content concerning instability of SEMs and analog photos or concerning the operation of SEMs [removal of old Subclauses 6.11, 6.12, 6.13, 8.4, 9.2.1, 9.2.2, 9.3, A.2.3, A.3.2, A.3.3, A.3.4, and A.3.7; revision of item e) in Clause 12];

— discussion of influences of imaging parameters on measurement uncertainty (new 6.11);

— revision of Clause 10 and addition of Annex B with precision data from round robin tests;

— revision of Annex A to (re-) align it with ISO 1463:2021;

— adding a bibliography with informative references.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

# Metallic coatings — Measurement of coating thickness — Scanning electron microscope method

## 1 Scope

This document specifies a destructive method for the measurement of the local thickness of metallic and other inorganic coatings by examination of cross-sections with a scanning electron microscope (SEM). The method is applicable for thicknesses up to several millimetres, but for such thick coatings it is usually more practical to use a light microscope (see ISO 1463). The lower thickness limit depends on the achieved measurement uncertainty (see Clause 10).

NOTE The method can also be used for organic layers when they are neither damaged by the preparation of the cross-section nor by the electron beam during imaging.

## 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:



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

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

**3.1**
**local thickness**
mean of the thickness measurements, of which a specified number is made within a reference area

[SOURCE: ISO 2064:1996, 3.4]

## 4 Principle

A test specimen is cut, ground, and polished from a cross-section of the coating for materialographic examination by a scanning electron microscope. The measurement is made on the digital image generated by the SEM using either the tools of the SEM's operating software or by importing the image file together with its calibration data into an image processing software and using that software's tools.

## 5 Instrumentation

### 5.1 Scanning electron microscope

Suitable instruments are available commercially.

### 5.2 Tools to calibrate the length measurement function of the SEM software

Suitable tools are required for the calibration of the length measurement function of the SEM's software, e.g. a stage micrometre, or a graticule, or a piece from a silicon wafer with a regular pattern of (cylindrical) metallic bumps with a certified distance of the cylinder axes, or spherical polymer

particles of certified diameter in the range of a few tenths of a micrometre to a few micrometres can be used, all of which are commercially available. They should have an uncertainty of less than 5 %.

# 6 Factors influencing the measurement results

## 6.1 Surface roughness

If the coating or its substrate is rough relative to the coating thickness, one or both of the interfaces of the coating cross-section can be too irregular to permit accurate measurement of the average thickness in the field of view. In this case, it can be helpful to use software solutions, which can identify the boundary lines of the coating and either determine its area and divide it by the image width or place automatically, for example, 100 measurement lines in order to calculate an average coating thickness.

## 6.2 Taper of cross-section

If the plane of the cross-section is not perpendicular to the plane of the coating, the measured thickness will be greater than the true thickness. For example, an inclination of 10° to the perpendicular will contribute a 1,5 % error.

NOTE This source of error is also known as cosine error in the small-angle approximation.

## 6.3 Specimen tilt

Any tilt of the specimen (plane of cross-section) with respect to the SEM beam can result in an inaccurate measurement.



NOTE 1 If the tilt of the test specimen is different from that used for calibration, inaccuracies can result.

NOTE 2 This source of error is also known as cosine error in the small-angle approximation.



## 6.4 Coating deformation

Detrimental deformation of the coating can be caused by excessive temperature or pressure during the mounting and preparation of cross-sections of soft coatings or coatings that melt at low temperatures, and by excessive abrasion of brittle materials during preparation of cross-sections.

## 6.5 Rounding of edges of the coating

If the edge of the coating cross-section is rounded, i.e. if the coating cross-section is not completely flat up to its edges, the observed thickness can differ from the true thickness. Edge rounding can be caused by improper mounting, grinding, polishing, or etching (see 6.6 and A.2).

## 6.6 Plating a protection layer

Overplating of the test specimen, i.e. plating a protection layer onto the test specimen, serves to protect the coating edges during preparation of cross-sections and thus to prevent an inaccurate measurement. Removal of the coating material during surface preparation for overplating can cause a low thickness measurement.

## 6.7 Etching

Optimum etching will produce a clearly defined and narrow dark line at the interface between the two materials. A wide or poorly defined line can result in an inaccurate measurement.

NOTE Etching is usually applied for the microscopic method (see ISO 1463) and can be useful for relatively thick coatings in the SEM, too, especially when individual layers from the same material need to be distinguished and there is no or too weak material contrast in the back scattered electron image (see 6.9). For (very) thin coatings, etching has often a negative effect on the measurement uncertainty.

### 6.8 Smearing

Polishing can leave smeared metal that obscures the true boundary between two metals and results in an inaccurate measurement. This can occur with soft metals like indium or gold. To help identify whether or not there is smearing, repeat the polishing, etching, and measurement several times. Any significant variation in readings is an indication of possible smearing.

### 6.9 Poor contrast

The visual contrast between metals in an SEM is poor when their atomic numbers are close together. For example, bright and semi-bright nickel layers cannot be discriminable unless their common boundary can be brought out sufficiently by appropriate etching (see 6.7) and SEM techniques.

### 6.10 Magnification

For a given coating thickness, measurement errors tend to increase with decreasing magnification. If practical, the magnification should be chosen so that the field of view is between 1,5 and 3 times the coating thickness. For very thin coatings this is often not practicable; then choose the maximum magnification at which the image of the coating and its boundaries appears still “sharp”.

### 6.11 SEM imaging parameters

The acceleration voltage of the SEM can influence the appearance of the coating in the image. For example, a higher acceleration voltage causes a higher depth from which the signal is collected and can lead to not clearly discernible edges, e.g. at a metal to polymer (e.g. molding resin) interface.



High probe currents can improve the brightness and contrast of the image and increase count rates for energy-dispersive X-ray spectroscopy (EDS), but can at the same time reduce the resolution and thus increase measurement uncertainty.



The settings of brightness, contrast and gamma can influence the appearance of the coating in the image and – especially for thin coatings – the measured thickness.

## 7 Preparation of cross-sections

Prepare the test specimen so that:

a) the cross-section is perpendicular to the plane of the coating;

b) the surface is flat and the entire width of the coating image is simultaneously in focus at the magnification to be used for the measurement;

c) all material deformed by cutting or cross-sectioning is removed;

d) the boundaries of the coating cross-section are sharply defined by no more than contrasting appearance, or by a narrow, well-defined line.

NOTE Further guidance is given in Annex A.

## 8 Calibration of instruments

### 8.1 General

Before use, each instrument (5.1) shall be calibrated with an appropriate tool (5.2) under the same conditions as used for the sample measurement.

Appropriate attention shall be given to the factors listed in Clause 6 to the procedures specified in Clause 9 and to the uncertainty limits of Clause 10. The stability of the calibration shall be checked at regular intervals.

### 8.2 Photography

Capture an image of the certified calibration standard, e.g. the micrometre scale, the graticule, 10 × 10 to 15 × 15 of the metallic bumps in top view or of some of the spherical particles (5.2) with sufficient contrast for later measurement.

Spherical particles (5.2) brought from a suspension onto a clean SEM sample stub tend to agglomerate. Search isolated particles on the sample stub to record the images for calibration. An inappropriate choice of imaging parameters (6.11) can let the calibration fail.

### 8.3 Measurement

**8.3.1** Using the tools of the SEM's software or using a separate image analysis software, to which the image file and its calibration data were imported, measure the left-to-left or right-to-right distance between the lines of the stage micrometre or the graticule (5.2) or the diameter of the spherical particles (5.2).

**8.3.2** Repeat the measurement at minimum three different locations over the entire field of the image.

**8.3.3** The image of the metallic bumps (5.2) needs to be analysed with a software, which can fit circles to the top view of the cylindrical bumps and then determine the distance of their centres.



## 9 Procedure



**9.1** Each instrument (5.1) shall be operated in accordance with the manufacturer's instructions. Appropriate attention shall be given to the factors listed in Clause 6 and to the uncertainty requirements of Clause 10.

**9.2** Capture an image of the test specimen under the same conditions and instrument settings used for the calibration. The boundaries of the coatings shall be clearly and sharply defined. Make an appropriate measurement using the tools of the SEM's software or in a separate image analysis software, to which the image file and its calibration data were imported.

## 10 Precision

### 10.1 General

See Annex B for further information on determining precision.

### 10.2 Repeatability, *r*

Repeatability, *r*, is the value less than or equal to which the absolute difference between two test results obtained under repeatability conditions may be expected to be, with a probability of 95 % (according to ISO 5725-1:1994, 3.16). The repeatability limit, *r*, in accordance with this document and calculated with a probability of 95 %, is given in Table 1 for typical applications of this measurement technique.

**Table 1 — Repeatability limit, *r***

| Application | Thickness<br>*t*<br>µm | Repeatability limit<br>*r*<br>µm |
|---|---|---|
| Cross-section of a Ti coating on a Si wafer | ≈1 | ≈0,05 |
| Cross-section of a polyimide foil | ≈14 | ≈0,5 |
| Cross-section of a polyimide foil | ≈25 | ≈0,5 |

### 10.3 Reproducibility limit, *R*

Reproducibility limit, *R*, is the value less than or equal to which the absolute difference between two test results obtained under reproducibility conditions may be expected to be, with a probability of 95 % (according to ISO 5725-1:1994, 3.20). The reproducibility limit, *R*, in accordance with this document and calculated with a probability of 95 %, is given in Table 2 for typical applications of this measurement technique.

**Table 2 — Reproducibility limit, *R***

| Application | Thickness<br>*t*<br>µm | Reproducibility limit<br>*R*<br>µm |
|---|---|---|
| Cross-section of a Ti coating on a Si wafer | ≈1 | ≈0,12 |
| Cross-section of a polyimide foil | ≈14 | ≈2,0 |
| Cross-section of a polyimide foil | ≈25 | ≈2,0 |



## 11 Expression of results

Depending on the coating thickness and at the operator's convenience, express the results either in millimetres, micrometres, or nanometres with one more digit than significant to prevent rounding errors when calculating statistics.

## 12 Test report

The test report shall contain at least the following information:

a) a reference to this document, i.e. ISO 9220:—;

b) the measured value;

c) identification of the test specimen(s);

d) location of the measurements on the test specimen;

e) a scale bar or an information about the image width superimposed on the SEM image;

f) any unusual features of the measurements that can have affected the results;

g) any deviations from the procedure described in this document;

h) date the measurements were made;

i) name of the individual responsible for the measurements.