



SLOVENSKI STANDARD
oSIST prEN ISO 9220:2021
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Kovinske prevleke - Merjenje debeline prevleke - Postopek z vrstičnim elektronskim mikroskopom (ISO/DIS 9220:2021)

Metallic coatings - Measurement of coating thickness - Scanning electron microscope method (ISO/DIS 9220:2021)

Metallische Überzüge - Messung der Schichtdicke - Verfahren mit Rasterelektronenmikroskop (ISO/DIS 9220:2021)

Revêtements métalliques - Mesurage de l'épaisseur de revêtement - Méthode au microscope électronique à balayage (ISO/DIS 9220:2021)

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25.220.40	Kovinske prevleke	Metallic coatings

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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

ICS: 25.220.40

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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

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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.

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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For an explanation of the voluntary nature of standards, 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 www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 107, *Metallic and other inorganic coatings*.

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

The main changes compared to the previous edition 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 [Subclause 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 coatings (hereafter also other inorganic coatings are meant) by examination of cross-sections with a scanning electron microscope (SEM). The method can be used for thicknesses up to several millimetres, but for such thick coatings it is usually more practical to use a light microscope (ISO 1463) when applicable. The lower thickness limit depends on the achieved measurement uncertainty (see [Clause 10](#)).

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 1463, *Metallic and oxide coatings — Measurement of coating thickness – Microscopical method*

ISO 2064, *Metallic and other non-organic coatings – Definitions and conventions concerning the measurement of thickness*

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 <http://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 crosssection of the coating for metallographic 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 (SEM)

Suitable instruments are available commercially.

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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. Either a stage micrometer, 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 micrometer to a few micrometers can be used, all of which are commercially available. They shall 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 bounding the coating cross-section may 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 e. g. 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.

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6.3 Specimen tilt

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

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NOTE 1 If the tilt of the test specimen is different from that used for calibration, inaccuracies may 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 may differ from the true thickness. Edge rounding can be caused by improper mounting, grinding, polishing, or etching (see 6.6 and A.1).

6.6 Overplating

Overplating of 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 (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 (6.9). For (very) thin coatings etching has often a negative effect on the measurement uncertainty.

6.8 Smearing

Polishing may leave smeared metal that obscures the true boundary between two metals and results in an inaccurate measurement. This may 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 may not be discriminable unless their common boundary can be brought out sufficiently by appropriate etching (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](#).

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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 micrometer 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 micrometer 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 centers.

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.