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

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Contents

Page

Foreword ..... **iv**

1 Scope **1**

2 Normative references **1**

3 Terms and definitions **1**

4 Principle **1**

5 Instrumentation **1**
5.1 Scanning electron microscope 1
5.2 Tools to calibrate the length measurement function of the SEM software 1

6 Factors influencing the measurement results **2**
6.1 Surface roughness 2
6.2 Taper of cross-section 2
6.3 Specimen tilt 2
6.4 Coating deformation 2
6.5 Rounding of edges of the coating 2
6.6 Plating a protection layer 2
6.7 Etching 2
6.8 Smearing 3
6.9 Poor contrast 3
6.10 Magnification 3
6.11 SEM imaging parameters 3

7 Preparation of cross-sections **3**

8 Calibration of instruments **3**
8.1 General 3
8.2 Photography 4
8.3 Measurement 4

9 Procedure **4**

10 Precision **4**
10.1 General 4
10.2 Repeatability, *r* 4
10.3 Reproducibility limit, *R* 5

11 Expression of results **5**

12 Test report **5**

Annex A (informative) **General guidance on the preparation and measurement of cross-sections** **7**

Annex B (informative) **Details on precision** **10**

Bibliography **12**

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,* 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 analogue 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 *t* µm	Repeatability limit *r* 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 *t* µm	Reproducibility limit *R* 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 9220:2022;

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.

Annex A
(informative)

General guidance on the preparation and measurement of cross-sections

A.1 General

The preparation of specimens and measurements of coating thickness are greatly dependent on individual techniques and there is a variety of suitable techniques available. It is not reasonable to specify only one set of techniques, and it is impractical to include all the suitable techniques. The techniques described in this annex are intended as guidance for metallographers not experienced in measurements of coating thickness.

A.2 Mounting

A.2.1 General

To prevent rounding of the edges of the coating cross-section, the free surface of the coating should be supported so that there is no space between the coating and its support. There are two major approaches to achieve this: overplating the test specimen or gap-free mounting.

A.2.2 Plating a protection layer

Usually the test specimen is overplated with a coating of a metal of similar hardness, at least 10 µm thick. The protection layer should also give an electron signal different from that of the coating.

For hard, brittle coatings (e.g. oxide or chromium coatings), tightly wrapping the test specimen in soft aluminium foil before mounting has proved successful.

If the coating is soft, overplating with a metal that is softer will make polishing more difficult, because the softer metal tends to be polished away more rapidly. However, in some cases, a soft coating can be overplated well with a metal that is harder, e.g. copper, silver or gold should be overplated with nickel.

Overplating of zinc coatings with copper can cause difficulty because of the tendency of dissolved copper to deposit on the coatings during subsequent etching.

Overplating can also be done with autocatalytic processes, e.g. nickel-phosphor can work well on gold.

If a (partially) non-conductive or not sufficiently conductive surface is to be overplated, e.g. photo mask on a coupon of a freshly pattern-plated printed circuit board or a layer of passivation and sealer on zinc, one can try to sputter first a thin gold layer onto the surface of the test specimen and then overplate with nickel (skipping the usual pretreatment steps). Unfortunately, in this case, good adhesion of the overplating cannot be guaranteed, but when successful the results can be good.

When (in the case of passivation layers, sealers, topcoats, etc.) overplating is not possible or not wanted, it is still advisable to sputter a gold layer onto the surface of the test specimen before mounting. It can help distinguish the non-metallic coating from the mounting resin, which both appear quite dark in the back scattered electron image.

A.2.3 Gap-free mounting

Proper cleaning of the test specimen and mounting with (e.g. epoxy) resin systems, which adhere well to the surface of the test specimen, cure at room temperature and do not shrink upon cure, possibly

combined with vacuum impregnation, can help achieve a gap-free encapsulation of the test specimen with mounting resin and, in combination with a suitable polishing sequence, a good edge-retention of the coating to be inspected.

On fully metallic samples, which may be heated to typically 180 °C, phenolic mounting resins and the use of a hot mounting press can also lead to good results. This technique is usually not applicable to test specimens with plastic as a base material or other temperature or pressure sensitive components.

In the case of unavoidable mounting gaps, one can try to fill them with mounting resin by vacuum impregnation: one can interrupt the grinding process at the stage of the fine grinding, fill the gaps by vacuum impregnation and then finish the cross-section.

A.3 Grinding and polishing

A.3.1 General

It is essential to keep the cross-section surface of the mount perpendicular to the coating. This is facilitated by incorporating additional pieces of a similar metal in the plastic mounting near the outer edges, by periodically changing the direction of grinding (rotating through 90°), and by keeping the grinding time and pressure to a minimum. If, before grinding, reference marks are inscribed on the sides of the mount, any inclination from the horizontal is easily measurable.

Grind the mounted test specimens on suitable abrasive paper, using an acceptable lubricant such as water or alcohol, and apply minimum pressure to avoid bevelling the surface. Initial grinding should employ P100 grade or P180 grade abrasive to reveal the true specimen profile and to remove any deformed metal. Subsequently, use grades P240, P400, P800 and P1200 without exceeding grinding times of 30 s to 40 s on each paper. Alter the direction of scratches by 90° for each change of paper.

Polishing should start for 2 min to 3 min on a rotating wheel charged with either 9 µm or 6 µm diamond particles and a suitable lubricant. As for SEM inspections a higher degree of surface finish than for the light microscope (see ISO 1463) is desired, a further treatment, using diamond particles of 1 µm, should follow. It can be advantageous to insert a step using 3 µm diamond particles between 6 µm and 1 µm. Between 9 µm and 1 µm it is necessary. One should not use diamond particles of a different size on one cloth but use separate cloths for each size. Depending on the nature of the test specimen and the mounting resin variations of the above, a general recipe or different recipes can apply.

In some cases, it can be advantageous to finally polish the cross-section with colloidal silica on a highly chemical resistant polishing cloth, for example, made from polychloroprene (after the 1 µm diamond polish). Care should be taken that the silica does not dry and crystallize on the cloth otherwise severe scratches on the cross-section can result. This polishing step can even avoid the need for etching with sometimes hazardous chemicals, e.g. when the base material is aluminium or an aluminium alloy.

After the last grinding step and after polishing, it can be advantageous to place the cross-section in a beaker filled with distilled or de-ionized water for 1 min to 3 min in an ultrasonic bath. Metal smeared into mounting gaps can be partially removed like this and it can help assess, in cases of poor edge retention, whether the cross-section is affected by mounting gaps (see A.2.3 for recommendations how to deal with them) or whether the unexpected observations are true features of the specimen.

If very soft materials are being prepared, abrasive particles can become embedded during grinding. This can be minimized by totally immersing abrasive papers in a lubricant during grinding or by using a copious flow of lubricant. If abrasive particles do become embedded, they can be removed by applying a short, light hand polish with metal polish after grinding and before diamond finishing or by one or more cycles of alternate etching and polishing.

A.3.2 Check for tapering

A convenient way to check for tapering of the cross-section is to mount a small diameter rod or wire with the specimen so that the perpendicular cross-section of the rod is parallel to that of the coating. If a taper is present, the cross-section of the rod will be elliptical.

ISO 1463:2021, Annex B contains a correction formula, which can be used in the case of taper.

A.4 Use of the scanning electron microscope

A.4.1 After the cross-section is finished and before inserting it into the SEM, the surface of the mounting material shall be made electrically conducting to prevent a charge build-up. This is typically done by sputtering a thin layer of a (noble) metal (e.g. gold, palladium, gold-palladium alloy, iridium or chromium) or by flash evaporation of carbon (graphite). When using a conductive mounting resin, sputtering or evaporation of a conductive layer on the finished cross-section is not necessary.

A.4.2 If the image of the cross-section is to be measured, and if the boundaries of the coating cross-section are revealed solely by the photographed contrast between the two materials, the apparent width of the coating cross-section can vary depending on the contrast and brightness settings. The variation can be as great as 10 % without any change in instrument magnification. To minimize the resulting uncertainty, adjust the contrast and brightness so that the image shows surface detail of the materials on either side of each boundary.

A.4.3 Many SEMs are equipped with energy dispersive X-ray spectroscopy (EDS) which can be helpful in identifying metallic coating layers.

A.4.4 The use of backscatter images should be considered default as they deliver a good contrast between metal layers with atomic numbers as close together as 1. Secondary electrons can be used at sufficient contrast.

Annex B
(informative)

Details on precision

B.1 Thickness range of 1 µm

In 2020 seven laboratories in Europe and Asia participated in a round robin test to determine precision data for this document as follows:

— Test specimens: Silicon wafer, sputtered with ≈1 µm of titanium, then sputtered with ≈1 µm of molybdenum as protection layer (see 6.6 and A.2.2); after coating separated to pieces of ≈1 cm × 1 cm.

— Experimental: Each laboratory received one or more pieces from the coated wafer. Metallographic cross-sections were prepared, images were taken in the SEM, and the thickness of the titanium coating was measured and reported.

— Remarks: Two laboratories prepared cross-sections from more than one piece; one laboratory took pictures from one cross-section at two different magnifications and reported the results separately (they differed a bit); one laboratory gave a finished cross-section to another laboratory for a cross-check. The so obtained results were treated in the evaluation as if they came from additional laboratories to base the statistically calculated precision data on more data sets.

— Evaluation: Precision data was calculated according to ISO 5725-2:1994/Cor 1:2002 as well as applying the Q statistics according to ISO 13528:2015. Two outlier labs identified by the Cochran and Grubbs tests were removed (see ISO 5725-2:1994/Cor 1:2002), so that 9 data sets were used for the final evaluation (see Table B.1).

Findings of the round robin test include the following.

— The titanium coating was quite rough (imperfection of the test specimen), so that the precision data is likely an upper limit for the method described in this document.

— The outliers were likely due to pieces with a different thickness or an increased local inhomogeneity of the titanium coating (imperfection of the test specimen) and thus removed from the final data set.

— While the reproducibility standard deviations, s_R, determined according to ISO 5725-2:1994/Cor 1:2002 and with the Q statistics (see ISO 13528:2015) are quite similar, the repeatability standard deviation, s_r, calculated according to ISO 5725-2:1994/Cor 1:2002 is more than double of s_r calculated with the Q statistics (see Table B.1).

Table B.1 — Results of the round robin test for the thickness range of ≈1 µm

Statistics	General mean/ Hampel estimator m nm	Repeatability standard deviation s_r nm	Reproducibility standard deviation s_R nm
ISO 5725-2:1994/ Cor 1:2002	1 011,9	36,8	47,1
Q statistics (see ISO 13528:2015)	1 014,0	16,9	42,6