INTERNATIONAL STANDARD

ISO 1463

Fourth edition
2021-05

Metallic and oxide coatings — Measurement of coating thickness — Microscopical method

Revêtements métalliques et couches d'oxyde — Mesurage de l'épaisseur de revêtement — Méthode par coupe micrographique

Reference number
ISO 1463:2021(E)

COPYRIGHT PROTECTED DOCUMENT

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

Published in Switzerland

Contents

Page

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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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

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, including for corrosion protection and corrosion testing of metals and alloys,* in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This fourth edition cancels and replaces the third edition (ISO 1463:2003), which has been technically revised.

The main changes compared with the previous edition are as follows:

— digital image processing for light microscopes has been added;

— further hints and methods for the preparation of microsections have been added;

— one hazardous etching recipe has been removed from Annex C.

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 and oxide coatings — Measurement of coating thickness — Microscopical method

WARNING — The use of this document can involve hazardous materials, operations and equipment. This document does not purport to address all of the safety problems associated with its use. It is the responsibility of users of this document to take appropriate measures to ensure the safety and health of personnel prior to the application of the document.

1 Scope

This document specifies a method for the measurement of the local thickness of metallic coatings, oxide layers, and porcelain or vitreous enamel coatings, by the microscopical examination of cross-sections using an optical microscope.

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 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 portion of the test specimen is cut out and mounted. The mounted cross-section is prepared by suitable techniques of grinding, polishing and etching. The thickness of the coating cross-section is measured by means of a calibrated scale.

NOTE These techniques will be familiar to experienced metallographers, but some guidance is given in Clause 5 and in Annex A for less experienced operators.

5 Factors relating to measurement uncertainty

5.1 Surface roughness

If the coating or its substrate has a rough surface, one or both of the interfaces bounding the coating cross-section could be too irregular to permit accurate measurement (see A.6).

5.2 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, e.g. an inclination of 10° to the perpendicular will contribute a 1,5 % uncertainty.

NOTE B.1 provides guidance on the taper of a cross-section.

5.3 Deformation of coating

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

5.4 Rounding of edge of 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 true thickness cannot be observed microscopically. Edge rounding can be caused by improper mounting, grinding, polishing or etching. It is usually minimized by overplating the test specimen before mounting (see A.2).

5.5 Overplating

Overplating of the test specimen protects the coating edges during preparation of cross-sections and thus prevents erroneous measurement. Removal of coating material during surface preparation for overplating can result in a low thickness measurement.

5.6 Etching

Optimum etching produces a clearly defined and narrow dark line at the interface of two metals. Excessive etching produces a poorly defined or wide line that can result in erroneous measurement.

5.7 Smearing

Improper polishing or overplating with a softer metal can cause smearing of one metal over the other metal, obscuring the boundary between the coating and the substrate. This problem can be alleviated by repeating the preparation of the cross-section of the coated metal until repeatability of the thickness measurement (see A.3 and A.5) is obtained and also by overplating with a harder metal.

5.8 Magnification

For any given coating thickness, measurement uncertainty generally increases with decreasing magnification. The magnification should be chosen so that the field of view is between 1,5 × and 3 × the coating thickness.

5.9 Calibration of stage micrometer

Any uncertainty in calibration of the stage micrometer will be reflected in the measurement of the specimen. A suitable, traceable length standard shall be used.

5.10 Calibration of the microscope's length measuring device

5.10.1 Micrometer eyepiece

A filar micrometer eyepiece provides a satisfactory means of making the measurement of the specimen. The measurement will be no more accurate than the calibration of the eyepiece. As calibration is operator dependent, the eyepiece shall be calibrated by the person making the measurement.

Repeated calibrations of the micrometer eyepiece can be reasonably expected to have a spread of less than 1 %. The distance between the two lines of a stage micrometer used for the calibration shall be known to within 0,2 µm or 0,1 %, whichever is the greater.

Some image splitting micrometer eyepieces have a nonlinearity that introduces an uncertainty of up to 1 % for short measurement distances.

Uncertainties can be introduced by backlash in the movement of the micrometer eyepiece. To avoid this uncertainty, ensure that the final motion during alignment of the hairline is always made in the same direction.

5.10.2 Digital image processing

Microscopes with a triocular tube, camera adapters with projecting lens and digital cameras connected to a computer with software for image capturing and processing are nowadays state of the art. Similar to 5.10.1, the measurement will be no more accurate than the adjustment and calibration of the length measurement function (combination of hardware and software).

For adjustment, digital images from the stage micrometer (in both directions parallel to the x- and y-axis of the image) are recorded for every combination of objective, if applicable intermediate magnification changer, and resolution setting of the camera (full resolution and typical settings of pixel binning). The length in object space represented by a pixel of the digital image is calculated by measuring a known distance on the stage micrometer with the respective function of the software and is then saved in the software. Usually after such an adjustment, the images are recorded "calibrated", i.e. with the µm/pixel factor assigned to the image, by selecting the objective, if applicable the intermediate magnification changer, and the pixel setting of the camera in the software at the time of capturing the image.

The adjustment and/or calibration are usually stable for long time. Furthermore, they are not operator dependent as long as no changes are applied to the tube, if applicable an intermediate magnification changer, the camera adapter or the camera itself, and as long as the same resolution of the camera (number of pixels in x and y direction) is used for adjustment and/or calibration and for measurement. Normally, it is sufficient to record in regular time intervals images from the stage micrometer and measure known distances. When the deviation between the measured length and the certified length is less than a reasonably defined uncertainty limit for length measurements, which the laboratory wants to achieve, e.g. 1 %, the calibration is still valid and no re-adjustment is necessary.

5.11 Uniformity of magnification

Uncertainties can occur if the magnification is not uniform over the entire field of view. Thus, ensure that both the calibration and the measurement are made over the same portion of the field of view with the measured boundaries centred about the optical axis.

5.12 Lens quality

As lack of sharpness of the image contributes to the uncertainty of the measurement, ensure that good quality lenses are used.

NOTE Sometimes, image sharpness can be improved by using monochromatic light.

5.13 Orientation of measuring lines

Ensure that the movement of the hairline of the eyepiece for alignment or the measuring line of a digital image processing software is perpendicular to the boundaries of the coating cross-section, e.g. 10° misalignment will contribute a 1,5 % uncertainty.

5.14 Tube length

A change in tube length causes a change in magnification and, if this change occurs between the time of calibration and the time of measurement, the measurement will be in uncertainty. Take care to avoid

a change in tube length, which can occur when the eyepiece is repositioned within the tube, when the focus of the eyepiece tube is changed, when the camera adapter is repositioned or changed and, for some microscopes, when the fine focus is adjusted.

6 Preparation of cross-sections

Prepare, mount, grind, polish and etch the specimen so that

a) the cross-section is perpendicular to 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, and

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 Clause 5 and in Annex A. Some typical etchants are described in Annex C.

7 Measurement

7.1 Give appropriate attention to the factors listed in Clause 5 and Annex A.

7.2 Calibrate the microscope and its measuring device with a certified or calibrated stage micrometer.

7.3 Measure the width of the image of the coating cross-section on at least five points distributed along a length of the cross-section.

NOTE Guidance on the measurement of taper of cross-section and of tooth-constructed coatings is given in Annex B.

8 Measurement uncertainty

The microscope and associated equipment, its use, its calibration and the method of preparation of the cross-section shall be chosen so as to allow the coating thickness to be determined to within 1 µm or 10 %, whichever is the greater, of the actual coating thickness. The method is capable of giving an absolute measurement uncertainty of 0,8 µm, and, for thicknesses greater than 25 µm, a reasonable measurement uncertainty of 5 % or better (see also B.3). However, with careful preparation of the specimen and the application of suitable instruments, this method is capable of providing a measurement uncertainty of 0,4 µm under reproducible conditions.

9 Test report

The test report shall include the following information:

a) a reference to this document, i.e. ISO 1463:2021;

b) the identity of the test specimen;

c) the results of the test, indicating

   1) the location on the coated item at which the cross-section was made,

   2) the measured thickness, in micrometres (millimetres if greater than 1 mm) at each point (see 7.3), and

3) the local thickness, i.e. the arithmetic mean of the measured thicknesses;

d) any deviations from the procedure specified;

e) any unusual features (anomalies) observed during the test;

f) the date of test.

Annex A
(informative)

Guidance on the preparation and measurement of cross-sections

A.1 General

The preparation of test specimens and measurement 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 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 edge 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 specimen or gap-free mounting.

A.2.2 Overplating

Usually the specimen is overplated with a coating of a metal of similar hardness, at least 10 µm thick.

For hard, brittle coatings (e.g. oxide or chromium coatings), tightly wrapping the 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 or cadmium coatings with copper can cause difficulty because of the tendency of dissolved copper to deposit on the coatings during subsequent etching. It is better to overplate zinc with cadmium and vice versa.

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 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 specimen before mounting. It can help distinguish the non-metallic coating from the mounting resin, which both appear dark in the microscope. It does not matter that the gold layer would be thinner than the resolution limit of the microscope; one can see it nevertheless as a bright line of undefinable thickness.

A.2.3 Gap-free mounting

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

with vacuum impregnation, can help achieve a gap-free encapsulation of the 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 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

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 near the outer edges of the plastic mounting, 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 of the surface. Initial grinding should employ P100 grade or P180 grade abrasive to reveal the true test 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. A final polish for 2 min to 3 min on a rotating wheel charged with either 9 µm or 6 µm diamond paste particles and a suitable lubricant should suffice to remove scratches before final examination. If an especially high degree of surface finish is required, a further treatment, using diamond paste of 1 µm particles, may be used. It can be advantageous to insert a step using 3 µm diamond paste 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 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, e. g. 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 may 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.4 Etching

Etching is usually advisable in order to promote contrast between the metal layers to remove traces of smeared metal and to develop a fine line at the boundary of the coating. Some typical etchants are given in Annex C.

A.5 Alternative to the classical metallographic method described in A.2 to A.4

Ion beam milling (or ion beam cutter) devices working with a broad argon-ion beam can be used to directly produce cross-sections or to enhance mechanically prepared samples. It is possible to prepare cross-sections of hard, soft, porous and heterogeneous materials without the smearing of soft materials or disruptions of brittle materials.

A.6 Measurement

The measuring device may be a digital image processing software, a filar micrometer or a micrometer eyepiece. The latter has lower precision. When observing and measuring through the eyepiece, an image splitting eyepiece is advantageous for thin coatings on rough substrate surfaces. Measurement of the image projected on a ground glass plate is usually less satisfactory because of the lack of sharpness of the image and poor legibility of the ruler when the projected image is visible.

When observing through the eyepiece, the measuring device should be calibrated at least once before and once after a measurement, unless repeated experience indicates otherwise. For digital image processing, weekly, monthly or quarterly calibration intervals can usually be proven to be sufficient.

When making calibration and coating measurements

— both should be made by the same operator (less critical for digital image processing),

— the stage micrometer and the coating should be centred in the field, and

— each measurement at a point should be made at least twice and averaged.

For critical and referee measurements, all steps for the preparation of cross-sections and measurement of coating thickness, from grinding with P1200 grade or coarser abrasive up to and including the determination of coating thickness, should be performed at least twice. With good techniques and equipment, and smooth coating and substrate surfaces, repeatability within 2 % or 0,5 µm, whichever is the greater, is reasonable.

Some microscopes are subject to a spontaneous movement of the stage relative to the objective, possibly due to non-uniform thermal effects from the light source. Such a movement during the measurement can cause an erroneous measurement at moderate and high magnifications. This can be minimized by completing the measurement quickly and by measuring each interval twice, once from left to right and once from right to left. Digital image processing is usually not affected by this effect. If the stage moves during image capture, the image would be distorted, discarded and one would anyway take it again.

Annex B
(informative)

Taper of cross-section and measurement of tooth-constructed coatings

B.1 Taper of cross-section

If the sample position deviates from the perpendicular (see Figure B.1), higher measuring values will be obtained (see 5.2).

The coating thickness d can be calculated using Formula (B.1):

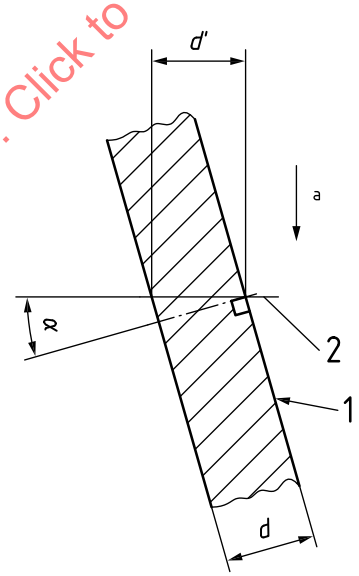
$$d = d' \cos \alpha \tag{B.1}$$

where

d is the coating thickness when $\alpha = 0$;

α is the deviation of the cross-section from the perpendicular of the coating surface, in degrees;

d' is the measured coating thickness when $\alpha \neq 0$.



Key

1 coating surface
2 section
[a] Direction of observation.

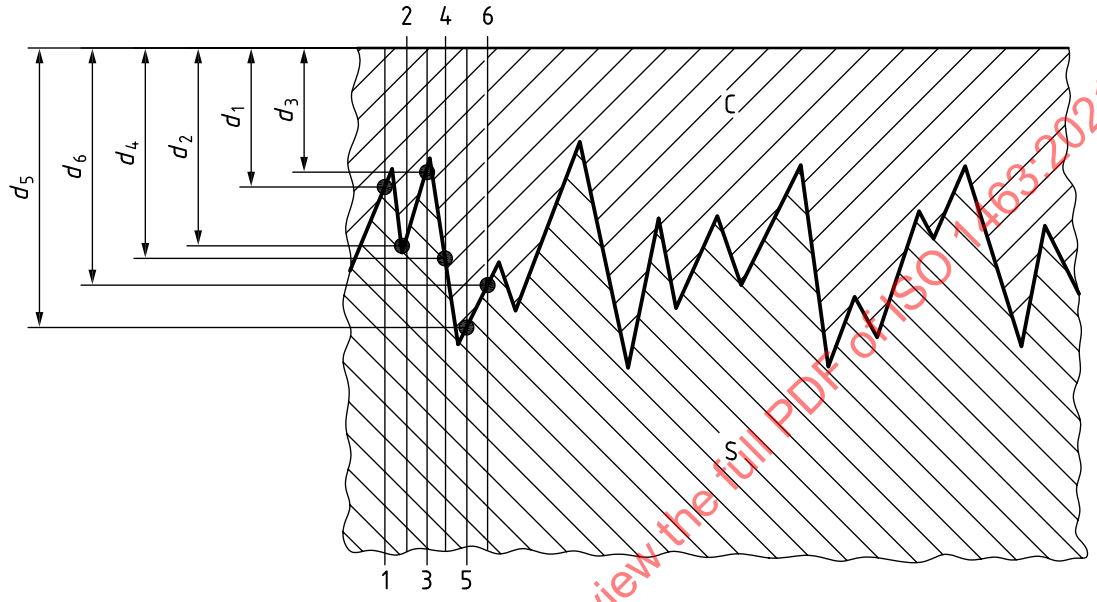
Figure B.1 — Deviation of the cross-section by the angle α

B.2 Measurement of tooth-constructed coatings

B.2.1 Principle

This method can be used for determining the coating thickness for coatings bounded by a tooth construction, e.g. thermochemically produced boride-nitride coatings.

The coating thickness is magnified 200 × and measured by a screen line distance of 2 mm between the boundary line of the coating over a suitable total length of, for example, 100 mm (see Figure B.2).



Key

C coating

S substrate

Figure B.2 — Diagrammatic representation of the thickness determination of coatings with tooth construction

B.2.2 Evaluation

The arithmetical average value for coatings with tooth construction is calculated from single values. The standard deviation provides an indication of the irregularities of the boundary surface (i.e. the degree of tooth construction).

B.3 Empirical values for the standard deviation of measurements obtained by light microscopy

Under repeatability conditions, the standard deviation, σ, is 0,3 µm. Under reproducibility conditions, the standard deviation is 0,8 µm.

Based on the stated standard deviations, Table B.1 indicates the confidence interval, q, of the local coating thickness. The values in the table apply to a statistical certainty of 95 % calculated with Formula (B.2) (simplified notation):

$$q = \pm \frac{1{,}96}{\sqrt{n}} \times \sigma \tag{B.2}$$

where