INTERNATIONAL STANDARD

ISO 4518

Second edition
2021-04

Metallic coatings — Measurement of coating thickness — Profilometric method

Revêtements métalliques — Mesurage de l'épaisseur de revêtement — Méthode profilométrique

Reference number
ISO 4518:2021(E)

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Published in Switzerland

Contents

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, 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 second edition cancels and replaces the first edition (ISO 4518:1980), which has been technically revised. The main changes compared with the previous edition are as follows:

— optical profilometers such as confocal microscopes or interference microscopes have been added as alternatives to stylus instruments for the measurement of the step height;

— a description of more modern stylus profilometers has been added.

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 — Profilometric method

1 Scope

This document specifies a method for the measurement of metal coating thickness by first forming a step between the surface of the coating and the surface of its substrate and then measuring the step height using a profile recording instrument. It covers the instrumentation characteristics and the procedure appropriate to this specific application of profilometric methods.

The method is applicable to the measurement of thicknesses of metal coatings from 0,01 µm to 1 000 µm on flat surfaces and, if appropriate precautions are taken, on cylindrical surfaces. It is highly suitable for the measurement of minute thicknesses but, for thicknesses of less than 0,01 µm, surface flatness and surface smoothness are very critical and, accordingly, the method is not suitable for use down to the lowest level of measurement usual for electronic stylus instruments. The method is suitable for measuring coating thicknesses when preparing coating thickness reference standards.

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 2177, *Metallic coatings — Measurement of coating thickness — Coulometric method by anodic dissolution*

3 Terms and definitions

No terms and definitions are listed in this document.

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/

4 Principle

Formation of a step either by dissolving part of the coating (acceptance testing) or by masking a portion of the substrate prior to coating (production inspection). Measurement of the height of the step using a profile recording instrument.

5 Instrumentation — Operational parameters and measurement characteristics

5.1 Types of profile recording instruments

Any of the following three types may be used:

a) contact stylus instruments, known as “surface analysers” and “surface profile recorders”, generally used to measure surface roughness but which, for the purposes of this document, are used to record the profile of a step;

b) inductive measuring probe or displacement sensor (incremental or absolute) capable of recording the profile of a step;

c) optical profilometers, i.e. white-light interference microscopes or confocal microscopes (ordinary, laser scanning or chromatic), which are generally used to measure surface topographic information and 3D surface roughness but which, for the purposes of this document, are used to record and evaluate the profile of a step.

Stylus instruments can have a greater utility, being suitable for roughness and form measurements, while measuring probes or displacement sensors can be simpler in construction. Stylus instruments designed only for roughness generally cover a range of coating thicknesses from 0,005 µm to 250 µm. Other contact instruments cover up to approximately 1 000 µm.

5.2 Stylus instruments

These instruments are used to record the profile of a surface and have the following components.

5.2.1 A pick-up with a conical or pyramidal stylus, having an included angle of 1,05 rad (60°) or 1,57 rad (90°) and a nominal tip radius, in the direction of the traverse, of 2 µm, 5 µm, 10 µm or 50 µm. The force of contact on the test surface shall not exceed the appropriate value given in Table 1.

Table 1 — Maximum force on stylus

Nominal value of stylus tip radius, µm	2	5	10	50[b]
Maximum static force at the mean level of the stylus, mN[a]	0,7	4	16	10[b]

[a] 1 mN ≈ 0,1 gf.

[b] Values useful for low-hardness metals such as tin.

5.2.2 A traverse unit, that moves the pick-up relative to a datum skid or, in those cases where the skid can result in damage to the surface or introduce distortion of the step to be measured, a datum surface having nominal form of the profile.

5.2.3 A component that unites the amplifier and the recording instrument, in an electronic controller and a computer software, which can display and print the digitized data at any desirable vertical and horizontal magnification (zoom).

Old-fashioned instruments with purely analogous measuring value processing are equipped with an amplifier with firm amplifying steps and a recording instrument that plots the amplified values (V_v) over the amplified values of the horizontal movement of the traverse unit (V_h).

5.2.4 Profile recording instruments with the following properties:

— traverse length: 1 mm to 100 mm;

— range of thickness measurement: 0,005 µm to 250 µm;

— height resolution (dependent on the range of measurement): 0,000 5 µm to 1 µm.

5.3 Inductive measuring probes or displacement sensors

5.3.1 The design of inductive measuring probes or displacement sensors is very similar to that of the electronic stylus instruments (see 5.2), the principal difference being that the large-radius stylus does not plot the microprofile of the surface.

5.3.2 Typical examples of the operational parameters and the measuring characteristics of an inductive measuring probe or a displacement sensor are as follows.

Operational parameters:

— a linearity of not less than 0,5 %;

— a table providing a rectilinear motion to the surface to be traversed;

— suitable amplifiers;

— using the following operational parameters:

  — radius of stylus: 250 µm;

  — maximum magnification: × 50 000;

  — static stylus force: 0,12 N.

Measurement characteristics:

— traverse length: 100 mm;

— range of thickness measurement: 1 µm to 1 000 µm;

— height resolution (dependent on the range of measurement): 0,02 µm to 20 µm.

5.4 Optical profilometers

Optical profilometers are based on the technology of optical microscopes, use similar objectives and thus have similar lateral resolution but, due to the interference or confocal technique and digital data evaluation, they have a good height resolution.

Typical examples of the measuring characteristics of optical profilometers are as follows.

For white-light interference microscopes:

— range of thickness measurement: 10 × *Ra* to 50 % of the maximum measurable height difference (depending on the instrument);

— height resolution: 0,5 nm.

For confocal microscopy:

— range of thickness measurement: 10 × *Ra* to 50 % of the maximum measurable height difference (depending on the instrument);

— height resolution: 0,5 nm.

For laser-scanning confocal microscopes:

— range of thickness measurement: 10 × *Ra* to 50 % of the maximum measurable height difference (depending on the instrument);

— height resolution: 0,5 nm.

For chromatic confocal microscopes:

— range of thickness measurement: 10 × *Ra* to 50 % of the maximum measurable height difference (e.g. 0,15 mm or 0,5 mm);

— the height resolution depends on the selected sensor and the measurable height difference: the larger the working range, the worse the resolution.

6 Factors relating to accuracy

6.1 Profile record

Because the thickness measurement is made from the recorded profile, errors will arise if the recording does not give a faithful reproduction of the step at a suitable magnification. Inaccurate recordings can reflect the quality or improper adjustment of the recording instrument.

6.2 Vertical magnification (only for instruments with chart recorders)

If the vertical magnification is too low, measurement precision will be poor. It should be set to take maximum advantage of the chart width.

6.3 Graphical measurements

If the test surface is not parallel to the reference (datum) surface, the recording of the horizontal surface is sloped with respect to the chart grid. The vertical portion of the step is also sloped but it can still be vertical on the chart grid, depending on the vertical and horizontal magnifications, on the radius of the stylus and finally on the height of the step (i.e. thickness). When the profile is sloped, a common error is to measure the perpendicular distance between the mean lines of the profile without correcting for the differences between the horizontal and vertical magnifications.

Modern instruments with software-based evaluation can correct for such effects.

To avoid these errors or additional mathematical calculations, the datum and test surfaces shall be parallel. This may be accomplished using an appropriate jig or fitting.

6.4 Applied force (only for profilometers with stylus)

If the force on the stylus is too high, the stylus produces a scratch or deformation that can introduce a measurement error. The force should be kept to a minimum and generally should not exceed the appropriate values given in Table 1.

6.5 Stylus diameter and surface roughness

If a small diameter stylus is used on a rough surface, the step height can be difficult to measure accurately because of poor definition of the extremes of the recorded step. A large diameter stylus minimizes this difficulty.

If the substrate and coating surfaces are of different roughness, the recorded step profile can be misleading to the extent that the stylus rides the high spots more on one surface than the other because of different peak to peak spacings. A small diameter stylus tends to reduce this error.

A small diameter stylus equipped with an electronic filter to smooth out the profile can be advantageous but can round the corners of the step profile.

6.6 Surface roughness

In principle, the roughness of a recorded substrate (peak to valley height of the surface profile) should not exceed 10 % of the step height.

When optical profilometers are used, the lateral dimensions of the surface profile elements (e.g. the mean width of the profile elements, *RSm*) shall exceed a few times the wavelength of the light, otherwise a plane cannot be determined.

6.7 Vibrations

Vibrations can cause irregularities or noise in the recorded profile, making accurate measurement difficult. This effect should be minimized by insulating the equipment from vibrations. In principle, the peak to valley height should not exceed 10 % of the step height.

6.8 Surface curvature

Surface curvature can interfere with accurate measurement of the recorded step profile. The measurement should be carried out on as flat a surface as practicable. If it must be carried out on a curved surface, the traverse of the stylus should be in the direction of least curvature, e.g. parallel to the axis of a cylinder. (This implies that the step should be parallel to the direction of maximum curvature.)

6.9 Cleanliness

Any foreign matter such as dirt, grease and corrosion products can lead to erroneous measurements. The surfaces to be measured should be cleaned, and the laboratory air should be reasonably free of dust and dirt.

6.10 Temperature

Temperature variations can affect the measurement. Therefore, the temperature should be uniform and reasonably stable.

6.11 Step configuration

A poorly defined step (e.g. excessive rounding of edges) can make an accurate measurement difficult by obscuring the levels of the upper and lower portions of the steps. The steps should be reasonably well defined.

6.12 Datum reference (only for profilometers with stylus)

The stylus holder rides on a datum skid or on a datum surface, and the vertical motion of the stylus relative to the datum skid or surface is recorded. The datum skid is a rounded surface that rides over the surface of the test specimen, and the datum surface is part of the equipment independent of the test specimen.

The faithfulness of a recording will depend on the quality (smoothness and straightness) of the datum reference.

6.13 Calibration

The thickness measurement will be not better than the uncertainty of the instrument calibration and that of the step height standard used to calibrate the instrument. The calibration can change, and the frequency with which calibration is required has to be learned by experience. Though the instrument can be carefully calibrated, an error of 2 % can exist because of nonlinearity of the instrument response. To minimize this error, the instrument can be calibrated at two points closely bracketing the step height to be measured.

7 Calibration

7.1 Calibrate the instrument in accordance with the manufacturer's instructions, paying appropriate attention to the factors listed in Clause 6.

7.2 The step height of the standards used to calibrate the instrument should be known with an uncertainty of less than 5 %. However, for steps of less than 0,1 µm, the uncertainty can be considerably greater than 5 %.

7.3 Repeat the calibration at regular intervals (see 6.13), and whenever a calibration change is suspected.

8 Measuring procedure

8.1 Preparation of step

8.1.1 Remove a portion of the coating without any attack on its substrate. The top of the step shall not be marred or attacked in any way, and the bottom of the step shall be free of any traces of the coating. Suitable methods of producing steps are given in 8.1.2, 8.1.3 and 8.1.4.

8.1.2 Using a suitable material, mask all of the coating except the area to be dissolved. Dissolve the exposed coating in a suitable reagent that will not attack the substrate, and then remove all traces of the masking material.

8.1.3 Dissolve a small area of the coating with an electrolytic cell of the same type as those used for the coulometric measurement of thickness by anodic dissolution in accordance with ISO 2177.

A single traverse of the stylus across the diameter of the circle produced by the small cell can provide a profile of two steps.

In order to be consistent with the definition in ISO 2064 of minimum thickness as measured over a reference area of about 1 cm^2, it is recommended to remove four small circular areas of coating within a 1 cm square, see Figure 1, record the step profile and measure the step height nearest each of the four corners of the square.

Dimensions in cm

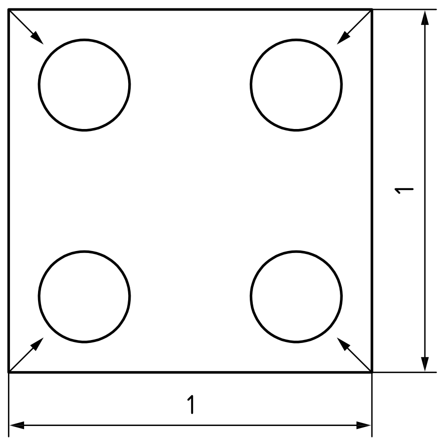

Figure 1 — Four small circular areas of removed coating within a square of 1 cm side length

8.1.4 In some cases, the step can be formed by masking a portion of the substrate prior to coating. The masked area shall be sufficiently small (diameter of the order of 1 mm or 2 mm) so that edge build-up will not interfere with the measurement.

