
**Metallic coatings on non-metallic basis
materials — Measurement of coating
thickness — Micro-resistivity method**

*Revêtements métalliques sur matériaux non-métalliques — Mesurage
de l'épaisseur des revêtements — Méthode utilisant la micro-
résistivité*

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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).

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).

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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, 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).

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 on non-metallic basis materials — Measurement of coating thickness — Micro-resistivity method

1 Scope

This document specifies a method for non-destructive measurements of the thickness of conductive coatings on non-conductive base materials. This method is based on the principle of the sheet resistivity measurement and is applicable to any conductive coatings and layers of metal and semiconductor materials. In general, the probe has to be adjusted to the conductivity and the thickness of the respective application. However, this document focuses on metallic coatings on non-conductive base materials (e.g. copper on plastic substrates, printed circuit boards).

This method is also applicable to thickness measurements of conductive coatings on conductive base materials, if the resistivity of the coating and the base material is significantly different. However, this case is not considered in this document.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

No terms and definitions are listed in this document.

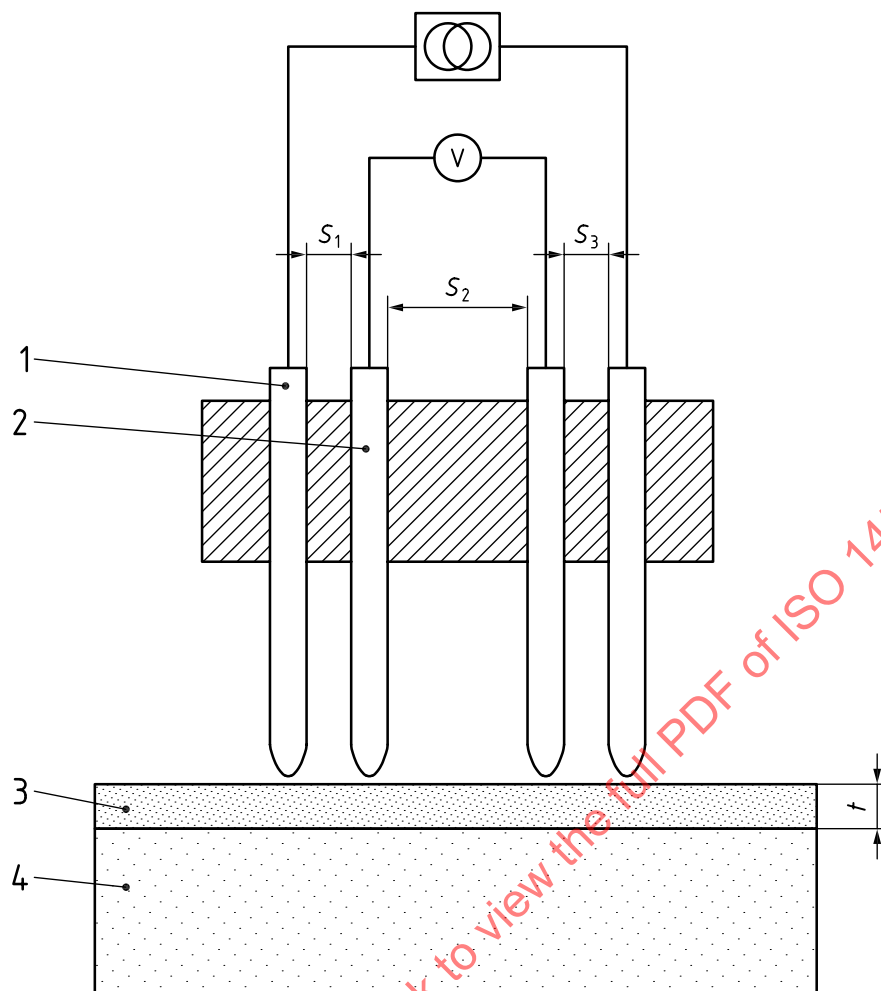
ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <http://www.electropedia.org/>

4 Measurement principle

The sheet resistivity method uses the so-called “four-point probe” as shown in [Figure 1](#). A row of four spring-loaded metal tips are placed in contact with the surface of the conductive coating. The tip distances between the outer and inner tips, S_1 and S_3 , are equal. Usually, a constant current is passed through the two outer contacts (labelled as 1). The introduced current penetrates the conductive material of the coating with the resistivity ρ . The resulting voltage drop is measured across the two inner contacts (labelled as 2).

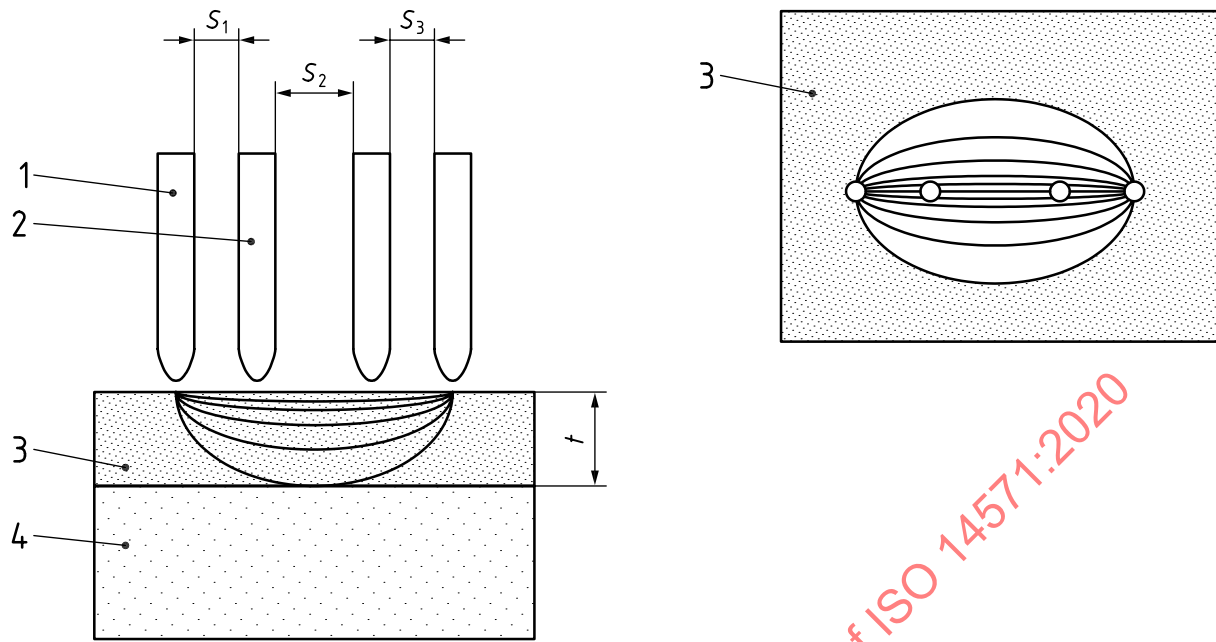
In general, the flow of the introduced current is non-uniformly distributed over the cross-section of the coating and is not parallel to the coating (see [Figure 2](#)). The current density decreases with increasing distance from the direct line between the outer contacts labelled as 1 (with depth and width). If the current is effectively limited by the thickness of the coating, the voltage drop between the inner contacts labelled as 2 is a measure of the thickness.



Key

- 1 outer contacts of the probe
- 2 inner contacts of the probe
- 3 conductive coating
- 4 non-conductive base material
- t coating thickness

Figure 1 — Schematic representation of the sheet resistivity method

**Key**

- 1 outer contacts of the probe
- 2 inner contacts of the probe
- 3 conductive coating
- 4 non-conductive base material
- t coating thickness

Figure 2 — Schematic representation of the non-uniformly distributed current within the coating

The measured voltage drop depends on the resistivity of the metallic coating, on the probe geometry (distance of the four probe contacts S_1, S_2, S_3), the applied current and the thickness of the coating. If the resistivity of the coating can be expected to be homogenous and the thickness is sufficiently small, the measured voltage drop is determined only by the unknown thickness and the applied current. In general, there is no simple and practical equation to calculate the thickness as a function of the material resistivity, the probe geometry and the measured voltage and current. However, there are some well-known approximations for practical use in certain cases. Particularly in the case of equal tip distances ($S_1 = S_2 = S_3 = S$) and for a thickness to probe spacing ratio $t/S < 0,5$, the coating thickness, t , in micrometres, can be calculated using [Formula \(1\)](#), when $t/S < 0,5$:

$$t = \rho \frac{I \ln(2)}{V \pi} \quad (1)$$

where

- ρ is the resistivity of the coating, in $\mu\Omega \cdot m$;
- V is the potential difference across the inner probe tips, in volts;
- I is the current passed through the outer probe tips, in amperes;
- S is the equal probe tip spacing ($S = S_1 = S_2 = S_3$).

Usually the supplied current I is held constant. Therefore, the coating thickness is inversely proportional to the measured voltage:

$$t = \frac{C}{V} \quad (2)$$

where C is a constant $0,221 \rho l$.

[Formula \(2\)](#) is the basis for many applications in the above case. In general, suitable correction functions for [Formula \(2\)](#) are necessary if the prerequisite of a ratio $t/S < 0,5$ or an equal probe tip spacing is not satisfied.

Because the introduced current decreases with increasing penetration depth, a sufficiently thick coating does not limit the current and the coating appears to be of infinite thickness to this method. The wider the probe spacing the deeper the current penetrates into the conductive material. Consequently, the measurement range is determined by the probe spacing for a given coating material. The probe geometry (tip spacing) has to be adjusted with respect to the conductivity and the expected thickness range of the application of interest. Furthermore, the sensitivity of this method decreases with increasing thickness.

The application of [Formula \(2\)](#) is also limited by very thin coatings because the resistivity is expected to be constant and not a function of the thickness. However, for very thin coatings, the resistivity starts to increase and below a critical thickness this increase of the resistivity is strongly pronounced. Typical values of this critical thickness are in the range of approximately 10 nm to 300 nm for metals. For measurements in this range and below this critical thickness, a special calibration or additional correction functions are necessary.

Because the introduced current decreases with increasing distance from the contacts of the probe, the current flow is not affected by a sample width wider than a critical width. Therefore, the sample width has to be wider than this critical width. Otherwise, the measured thickness becomes a function of the sample width and the sample width has to be considered in addition. The probe spacing also determines the value of the critical width for a given coating material.

5 Factors affecting measurement uncertainty

5.1 Range of measurement

The measurable thickness range is determined by the probe geometry (tip distance) and the conductivity of the coating. The probe geometry has to be adjusted to the thickness range of interest.

Usually the manufacturer provides the uncertainty of the respective probe for the recommended thickness range.

5.2 Coating resistivity

Measurements will be affected by the resistivity of the coating if the resistivity of the coating differs from the resistivity of the calibration standard(s) used to calibrate the instrument. A 5 % difference in resistivity will result in a 5 % error unless this difference is accounted for in the calibration procedure.

Furthermore, a homogenous resistivity throughout the coating is expected for this method. The measurement will be affected by a resistivity variation of the coating. This can be caused by composition variation of the coating, by coating defects (e.g. cracks, porosity, voids, inclusions) or by a surface preparation or contamination.

5.3 Width of the sample

Below a critical width, determined by probe design (tip spacing) and to a lesser degree on the electrical conductivity of the metallic coating, the coating thickness measurement becomes dependent upon

the width of the electrical current path (e.g. conductive track width of printed circuit boards). The instrument shall therefore be calibrated using calibration standards of the width to be measured or appropriate correction functions shall be used.

NOTE 1 An exact positioning of the probe in the middle of the sample (e.g. conductive track) and parallel to its direction is necessary to avoid measurement errors. Usually special probe positioning systems or probe guides are provided by the manufacturers.

NOTE 2 If the critical path width is not known, or for some reason is unobtainable, it can be obtained using a number of reference standards having the same thickness (made from the same piece of uniform material), but of different known widths (see [Annex A](#)).

5.4 Curvature

Sharp or small radii of curvature will greatly affect the thickness measurement. This effect is minimized if the probe is placed on the surface so that its axis is parallel to that of the curved surface. Alternatively, calibration standards of the same curvature can be used. The influence decreases with increasing radii of curvature.

5.5 Surface roughness

Measurements are affected by surface topography of the metallic coating. Rough surfaces can cause thickness measurement errors. In such cases, it is strongly recommended to perform a sufficient number of measurements at different locations on the sample and using the mean together with the standard deviation as a representative thickness value of the coating.

5.6 Temperature

A temperature change between calibration and measurement causes errors of the measured thickness because the resistivity of the coating varies with temperature. This temperature influence is important especially if the resistivity temperature coefficient of the coating material is high (e.g. Cu: $\alpha = 0,0039 \text{ K}^{-1}$). Therefore, the temperature of the sample should be measured and the thickness should be corrected with respect to temperature. Some manufacturers provide instruments with a temperature sensor and an automatic temperature correction for this purpose.

5.7 Probe contact pressure

The pressure with which the probe contacts are applied to the test specimen can affect the instrument readings. The applied pressure should therefore be made constant and as low as possible to minimize sample damage but still steady to ensure a good repeatability (reliable contact to the coating). This is achieved in practice by using a constant pressure probe having tips supported by adapted springs. The shape of the tips can be sharpened or rounded with respect to the coating material to achieve a reliable contact.

The current through the two outer tips should be applied only if the contact of the tips is established in order to avoid possible damages of the surface.

6 Calibration of instruments

6.1 General

Before use, each instrument shall be calibrated in accordance with the manufacturer's instructions, using suitable calibration standards. Appropriate attention shall be given to the factors listed in [Clause 5](#) and to the procedures of [Clause 7](#).

6.2 Calibration standards

Calibration standards of known coating thickness, uniform and homogenous coating resistivity and sufficient width (above the critical width) shall be used.

At least two or more calibration standards having different but known thicknesses appropriately distributed over the thickness range of interest shall be used for instrument calibration. Two calibration standards are the minimum if a tight linear calibration function between the thickness and the reciprocal value of the measured voltage drop can be used [see [Formula \(2\)](#)]. However, usually more than two calibration standards are recommended to reduce the uncertainty of the calibration function of the instrument.

The temperature shall be taken into account.

For calibration with calibration standards below the critical width, consider [5.3](#), NOTE 2 and [Annex A](#).

6.3 Verification

The electrical properties of the coating material being measured shall be similar to those of the coating of the standards. If the thickness being measured by this method is different from that measured by another independent method, the electrical properties can be expected to be different.

Whether or not the conductor width being measured is above or below the critical width is verified by taking measurements on progressively narrower conductor widths produced by systematically removing material from that conductor (see also [Annex A](#)).

7 Procedure

7.1 General

Operate each instrument in accordance with the manufacturer's instructions, giving appropriate attention to the factors listed in [Clause 5](#).

To ensure proper instrument performance, verify the calibration of the instrument at the test site each time the instrument is put into service and at sufficient intervals during use (respective recommendations of the manufacturer should be taken into account).

Observe the precautions given in [7.2](#) to [7.5](#).

7.2 Width of the sample

If the width of the sample (e.g. conductive track) is less than the critical width, ensure that the instrument has been calibrated using standards of similar width to the material to be measured or use appropriate correction functions to consider the path width.

7.3 Curvature

If curved surfaces are to be measured, ensure that either such a curved surface has no effect on measurement, or use calibration standards of similar curvature.

7.4 Number of measurements

It is advisable to always take several measurements at each location of the sample and to use the calculated mean value of these measurements. Both the repeatability of the instrument and the uniformity (or lack of it) of the material can be calculated from the measurement data. The required minimum number of measurements depends on the quality of the coating (e.g. homogeneity of thickness and resistivity, surface roughness) and also on the repeatability of the instrument at the thickness

of interest (instrument repeatability decreases with increasing thickness for a given quality of the coating).

7.5 Surface cleanliness

Before making measurements, remove any foreign matter such as dirt, grease and corrosion products from the surface to be measured, taking care not to remove any coating material.

8 Accuracy requirements

The instrument, its calibration, the uncertainty of the calibration standards, its operation and the sample preparation shall be such that the coating thickness can be determined to the possible uncertainty given by the manufacturer of the instrument. Modern 4-point-probe systems are capable to provide an accuracy of approximately 1 % to 2 % of the measured thickness value within the recommended thickness range. In each case, it is advisable to take the mean of multiple measurements to reduce the uncertainty of the measurement.

9 Test report

The test report shall include the following information:

- a) all information necessary for identification of the sample tested;
- b) a reference to this document, i.e. ISO 14571;
- c) details of the test procedure, including:
 - 1) the area, in square centimetres, over which the measurements were made;
 - 2) the location(s) of the test area(s) on each specimen;
 - 3) the number of test specimens measured;
 - 4) an identification of the instrument, its probe, and standards used for the tests, including reference to the validation certification of the equipment;
- d) the name of the operator and the testing laboratory;
- e) the results of the test expressed as t , in micrometers, including number of measurements, the results of the individual determinations and the mean for each reported measurement;
- f) any deviations from the procedure specified;
- g) any unusual features (anomalies) observed during the test;
- h) any circumstances or conditions thought likely to affect the results or their validity;
- i) the date of the test.