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

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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

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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).

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:

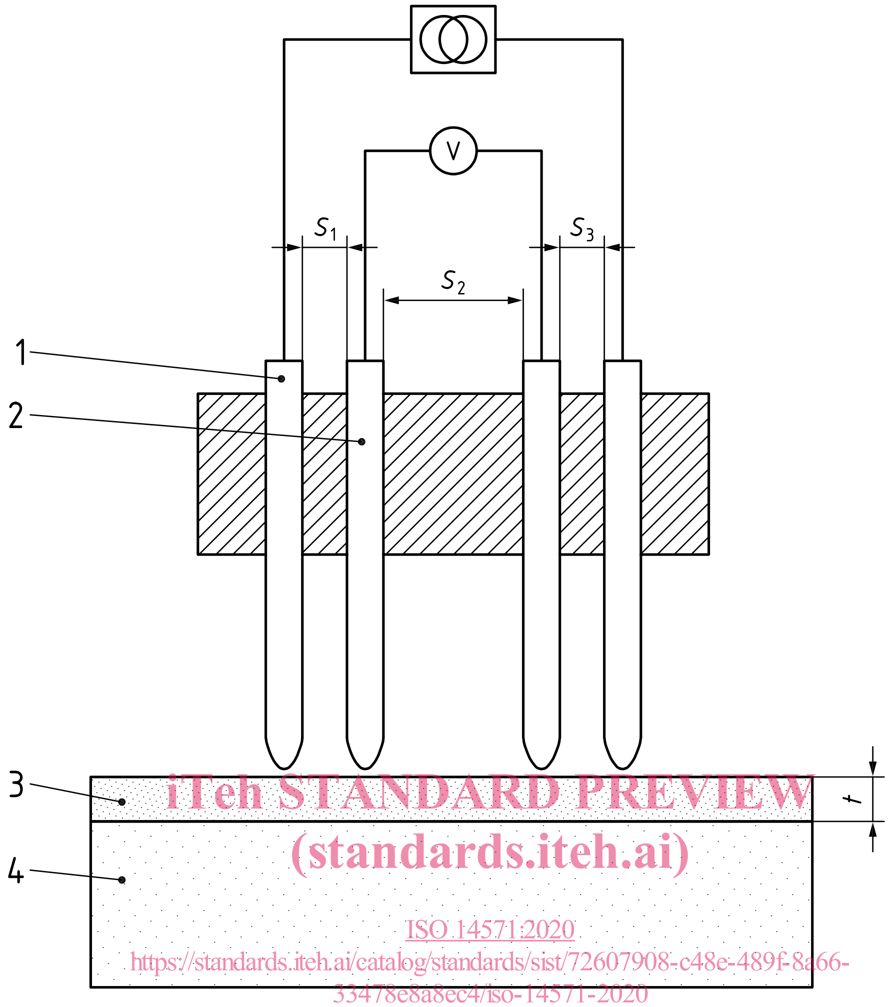
— ISO Online browsing platform: available at https://www.iso.org/obp

— 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

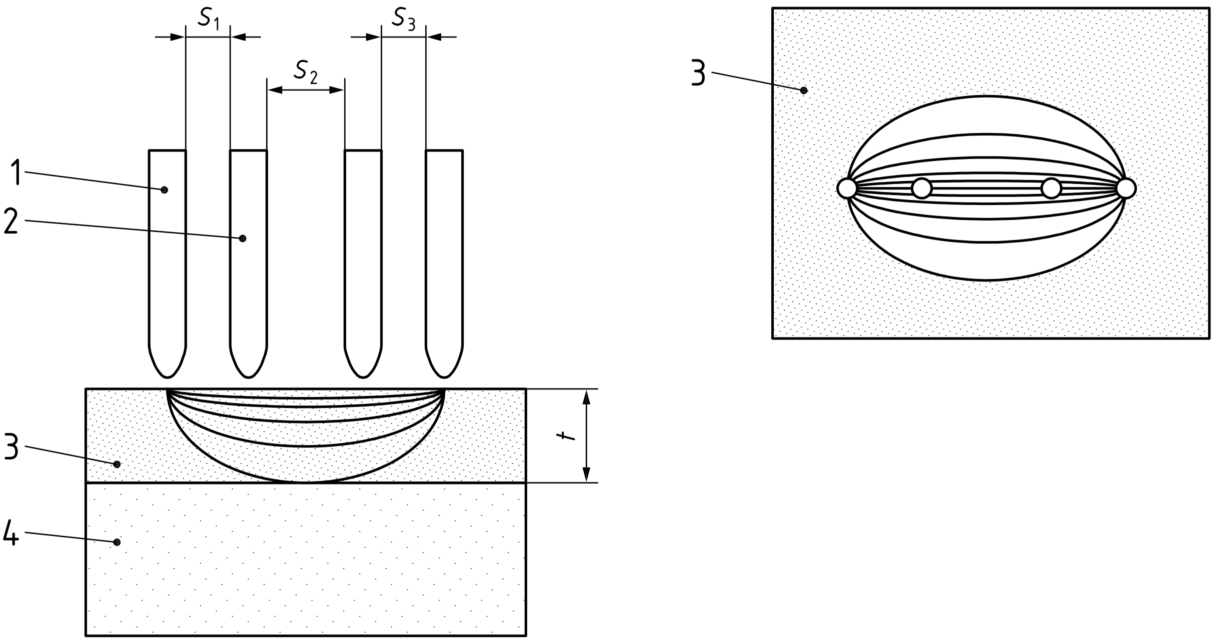


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Figure 2 — Schematic representation of the non-uniformly distributed current within the coating

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 $t/S < 0{,}5$:

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

where

ρ is the resistivity of the coating, in µΩ·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 ρI.

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: α = 0,003 9 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.