BS EN 14571:2005

Metallic coatings on nonmetallic basis materials — Measurement of coating thickness — Microresistivity method

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ICS 25.220.40; 17.040.20

British Standards

National foreword

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English version

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Revêtements métalliques sur matériaux non-métalliques - Mesurage de l'épaisseur des revêtements - Méthode utilisant la microrésistivité

Metallische Überzüge auf nichtmetallischen Grundwerkstoffen - Schichtdickenmessung - Mikro-Widerstand-Verfahren

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Foreword

This document (EN 14571:2005) has been prepared by Technical Committee CEN/TC 262 “Metallic and other inorganic coatings”, the secretariat of which is held by BSI.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by October 2005, and conflicting national standards shall be withdrawn at the latest by October 2005.

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1 Scope

This document describes a method for nondestructive measurements of the thickness of conductive coatings on nonconductive 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 focusses on metallic coatings on nonconductive base materials (e.g. Copper on plastic substrates, printed circuit boards).

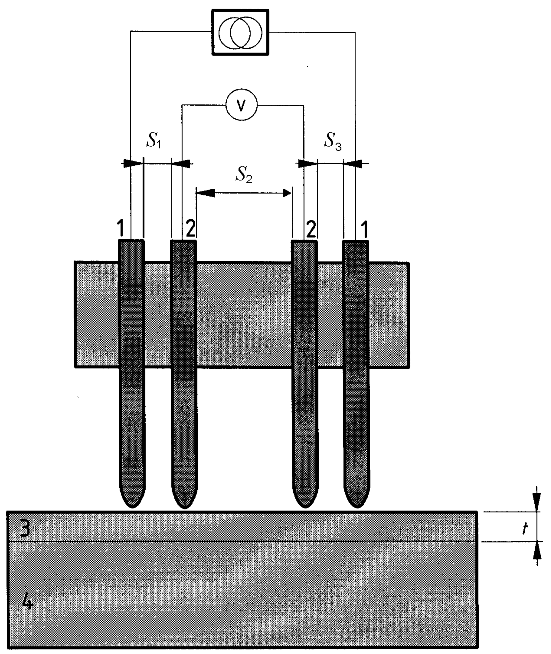
NOTE 1 This method also applies to the measurement of through-hole copper thickness of printed circuit boards. However, for this application a probe geometry different from the one described in this document is necessary.

NOTE 2 This method is also applicable for thickness measurements of conductive coatings on conductive base materials, if the resistivity of the coating and the base material is different. This case is not considered in this document.

2 Measurement principle

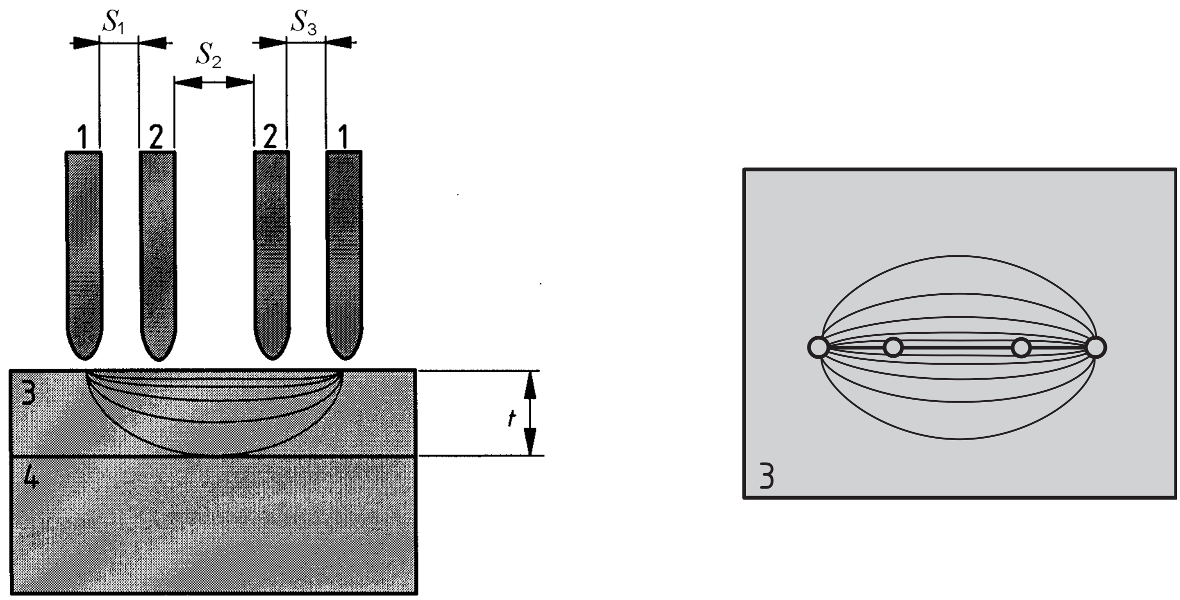
The sheet resistivity method uses the so called four-point probe as shown in Figure 1. A row of four spring-loaded metal tips are placed in contact with the surface of the conductive coating. The tip distances between the outer and inner tips S_1 and S_3 are equal. Usually a constant current is passed through the two outer contacts (4 and 7). 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 (5 and 6).

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 contacts 4 and 7 (with depth and width). If the current is effectively limited by the thickness of the coating, the voltage drop between 5 and 6 is a measure of the thickness.



Key
1 Outer contact of the probe
2 Inner contact of the probe
3 Conductive coating
4 Nonconductive base material
t Coating thickness

Figure 1 — Schematic representation of the sheet resistivity method



Key

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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 4 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. Especially 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 the equation:

$$t = \rho \frac{I}{V} \frac{ln(2)}{\pi} \qquad \left[when \quad \frac{t}{S} < 0{,}5 \right] \tag{1}$$

where

ρ is the resistivity coating, in ohm.m;

V is the potential difference across the inner probe tips, in Volts;

I current passed through outer probe tips, in amps;

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 :

$$t = \frac{C}{V} \tag{2}$$

where

C is a the constant $0{,}221\rho I$

Equation (2) is the basis for many applications in the above case. In general suitable correction functions for Equation (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 infinite 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 Equation (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 thicknesses 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 in width, 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.

3 Factors affecting measurement uncertainty

3.1 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.

3.2 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.

3.3 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 may 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).

3.4 Curvature

Sharp or small radii of curvature will greatly affect the thickness measurement. This effect is minimised 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.

3.5 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.

3.6 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=0,0039 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.

3.7 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 minimise 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 applied only if the contact of the tips is established in order to avoid possible damages of the surface.

4 Calibration of instruments

4.1 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 3 and to the procedures of Clause 5.

4.2 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 Clause 2, Equation 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 3.3, Note 2 and Annex A.

4.3 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 then 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).

5 Procedure

5.1 General

Operate each instrument in accordance with the manufacturers instructions giving appropriate attention to the factors listed in Clause 3.

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 5.2 to 5.5.

5.2 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.

5.3 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.

5.4 Number of measurements

It is advisable always to 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).

5.5 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.

6 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.

7 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 (EN 14571:2005);

c) details of the test procedure, including:

    i) the area, in square centimetres, over which the measurements were made;

    ii) the location(s) of the test area(s) on each specimen;

    iii) the number of test specimens measured;

    iv) 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.

Annex A
(normative)

Method for determining the critical current path width

A.1 General

As mentioned in 3.3. the thickness measurement is affected by the width of the sample, if the width is smaller than a critical width. The critical width is determined by the probe geometry (tip spacing) and the coating material. Therefore, if measuring very narrow metallic conductors and the critical width is not known from the manufacturer it is advisable to ensure that the width is not another variable in addition to the coating thicknesses to be measured. If the width of the sample being measured is above the critical width then the width can be said to be constant and can be ignored.

Usually the critical width should be given by the manufacturer for the respective probe geometry and typical conductivities.

A.2 Sample preparation

To establish the critical width for a particular instrument, probe geometry and coating material, it is necessary to have several samples of the coating material in question which are of the same thickness (made from the same sample of uniform thickness), but of different widths.

A first sample is necessary with an expected width W_a well above the critical width. This width W_a should be wider than at least 3 times the entire probe spacing (3 times the sum S_1+S_2+S_3). Then a second sample of the same coating thickness and having a known width of about W_b =W_a/2 should then be prepared.

A.3 Measurement

Calibrate the instrument using the sample having width W_a, then measure the W_b sample. If the W_b sample is above the critical width the thickness measurement should be the same (within the uncertainty limits of the method). Note that the mean of several measurements on each sample should always be used.

Now produce further similar samples between e.g. W_d = W_a/10 and W_c = W_a/3 in width and in appropriate increments, all having the same thickness. Without recalibrating the instrument measure each of the additional samples in turn, commencing with the W_c sample. When the thickness measurement changes from the known value already established, the critical width is found. If it is necessary to find the critical width very precisely, samples having smaller increments would be necessary.

NOTE To determine whether or not a sample piece of coating material is of uniform thickness, the instrument should be calibrated on the largest possible surface area of that material, away from any edge, surface projection, or any blemish or other discontinuity likely to cause errors (see Clause 3). The thickness value used for calibration should be the nominal thickness of that sample.

Then take multiple measurements at several different locations across the surface of the sample and compare the mean values of thickness of each location. The difference in thickness between each location should not be much greater than the uncertainty at any one location. The smaller this difference the more uniform the coating of the sample.

Bibliography

[1] Smith, F.M.; *Measurement of Sheet Resistivity with the Four-Point Probe*; Bell System Technical Journal; Vol. 37; 1958; p 711-718

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