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Non-conductive coatings on non-magnetic electrically conductive base metals — Measurement of coating thickness — Amplitude-sensitive eddy-current method

Revêtements non conducteurs sur matériaux de base non magnétiques conducteurs de l'électricité — Mesurage de l'épaisseur de revêtement — Méthode par courants de Foucault sensible aux variations d'amplitude

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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*.

This fourth edition cancels and replaces the third edition (ISO 2360:2003), which has been technically revised.

Non-conductive coatings on non-magnetic electrically conductive base metals — Measurement of coating thickness — Amplitude-sensitive eddy-current method

1 Scope

This document specifies a method for non-destructive measurements of the thickness of non-conductive coatings on non-magnetic electrically conductive base metals, using amplitude-sensitive eddy-current instruments.

In this document, the term “coating” is used for materials such as, for example, paints and varnishes, electroplated coatings, enamel coatings, plastic coatings, claddings and powder coatings. This method is particularly applicable to measurements of the thickness of most oxide coatings produced by anodizing, but is not applicable to all conversion coatings, some of which are too thin to be measured by this method (see Clause 6).

This method can also be used to measure non-magnetic metallic coatings on non-conductive base materials. However, the phase-sensitive eddy-current method specified in ISO 21968 is particularly usable to this application and can provide thickness results with a higher accuracy (see Annex A).

This method is not applicable to measure non-magnetic metallic coatings on conductive base metals. The phase-sensitive eddy-current method specified in ISO 21968 is particularly useful for this application. However, in the special case of very thin coatings with a very small conductivity, the amplitude-sensitive eddy-current method can also be used for this application (see Annex A).

Although the method can be used for measurements of the thickness of coatings on magnetic base metals, its use for this application is not recommended. In such cases, the magnetic method specified in ISO 2178 can be used. Only in case of very thick coatings above approximately 1 mm, the amplitude-sensitive eddy-current method can also be used for this application (see Annex A).

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 2064, *Metallic and other inorganic coatings — Definitions and conventions concerning the measurement of thickness*

ISO 4618, *Paints and varnishes — Terms and definitions*

ISO/IEC Guide 98-3, *Uncertainty of measurement — Part 3: Guide to the expression of uncertainty in measurement (GUM:1995)*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 2064 and ISO 4618 and the following apply.

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

— IEC Electropedia: available at http://www.electropedia.org/

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

3.1
adjustment of a measuring system
set of operations carried out on a measuring system so that it provides prescribed indications corresponding to given values of a quantity to be measured

Note 1 to entry: Adjustment of a measuring system can include zero adjustment, offset adjustment, and span adjustment (sometimes called gain adjustment).

Note 2 to entry: Adjustment of a measuring system should not be confused with calibration, which is a prerequisite for adjustment.

Note 3 to entry: After an adjustment of a measuring system, the measuring system must usually be recalibrated.

Note 4 to entry: Colloquially, the term “calibration” is frequently, but falsely, used instead of the term “adjustment”. In the same way, the terms “verification” and “checking” are often used instead of the correct term “calibration”.

[SOURCE: ISO/IEC Guide 99:2007, 3.11 (also known as “VIM”)]

3.2
calibration
operation that, under specified conditions, in a first step, establishes a relation between the quantity values with measurement uncertainties provided by measurement standards and corresponding indications with associated measurement uncertainties and, in a second step, uses this information to establish a relation for obtaining a measurement result from an indication

Note 1 to entry: A calibration may be expressed by a statement, calibration function, calibration diagram, calibration curve, or calibration table. In some cases, it may consist of an additive or multiplicative correction of the indication with associated measurement uncertainty.

Note 2 to entry: Calibration should not be confused with adjustment of a measuring system, often mistakenly called “self-calibration”, nor with verification of calibration.

Note 3 to entry: Often, the first step alone in the above definition is perceived as being calibration.

[SOURCE: ISO/IEC Guide 99:2007, 2.39 (also known as “VIM”)]

4 Principle of measurement

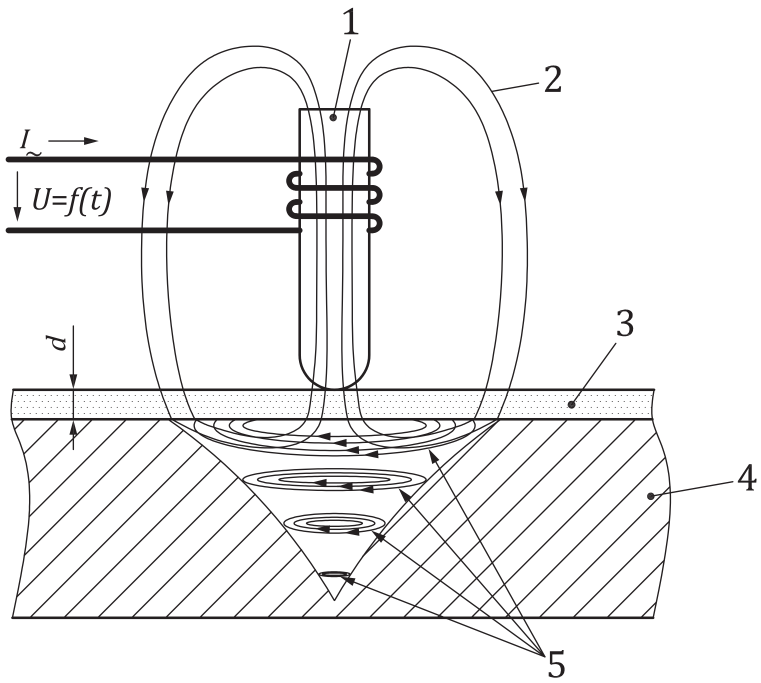
Eddy-current instruments work on the principle that a high frequency electromagnetic field generated by the probe system of the instrument will produce eddy-currents in the base metal beneath the coating on which the probe is placed (see Figure 1). These induced currents cause a change of the electromagnetic field surrounding the probe coil and therefore result in a change of the amplitude of the probe coil impedance. The induced eddy-current density is a function of the distance between the generating coil and the base metal surface. Consequently, this impedance change can be used as a measure of the thickness of the coating on the conductor by means of a calibration with reference standards (see also Annex A).

In order to measure a change of the coil impedance amplitude, the test coil is usually part of an oscillator circuit with a resonant frequency determined by the coil inductance and resistance. A change of the coil impedance amplitude results in a shift of the resonant frequency. Consequently, the measured resonant frequency is a measure of the coating thickness. The values are either pre-processed by digital means or are directly displayed on a usefully scaled gauge.

The probe and measuring system/display may be integrated into a single instrument.

NOTE 1 Annex C describes the basic performance requirements of the equipment.

NOTE 2 Factors affecting measurement accuracy are discussed in Clause 5.



Key

1	ferrite core of the probe	5	induced eddy-current
2	high frequency electromagnetic field	$I_\sim$	exciting current
3	non-conductive coating	*t*	coating thickness
4	base metal	$U = f(t)$	measurement signal

Figure 1 — Amplitude-sensitive eddy-current method

5 Factors affecting measurement uncertainty

5.1 Basic influence of the coating thickness

The sensitivity of a probe, i.e. the measurement effect, decreases with increasing thickness within the measurement range of the probe. In the lower measurement range, this measurement uncertainty (in absolute terms) is constant, independent of the coating thickness. The absolute value of this uncertainty depends on the properties of the probe system and the sample materials, e.g. the homogeneity of the base metal conductivity, the base metal roughness and the sample surface roughness. In the upper measurement range, the uncertainty becomes approximately a constant fraction of the coating thickness.

5.2 Electrical properties of the base metal

The conductivity of the base metal determines the induced eddy-current density for a given probe system and frequency. Consequently, the base metal conductivity causes the measurement effect for this method. The relationship between coating thickness and the measured value depends strongly on the conductivity of the base metal. Consequently, calibration procedures and measurements shall be made on the same material. Different materials with different conductivities as well as local fluctuations of the conductivity or variations between different samples can cause (more or less) errors in the thickness reading.

NOTE There are instruments and probes available that are capable of automatically compensating the base metal conductivity influence thus avoiding the resulting thickness error.

5.3 Geometry: Base metal thickness

Generation of eddy currents by the coil's magnetic field in the depth of the base metal is obstructed if the base metal thickness is too small. This influence can only be neglected above a certain critical minimum base metal thickness.

Therefore, the thickness of the base metal should always be higher than this critical minimum base metal thickness. An adjustment of the instrument can compensate for errors caused by thin base metal. However, any variation in thickness of the base metal can cause increased uncertainty and errors.

The critical minimum base metal thickness depends on both the probe system (frequency, geometry) and the conductivity of the base metal. Its value should be determined experimentally, unless otherwise specified by the manufacturer.

NOTE A simple experiment to estimate the critical minimum base metal thickness is described in D.3.

However, in the absence of any other information, the required minimum base metal thickness, t_{min}, can be estimated from Formula (1).

$$t_{min} = 3 \cdot \delta_0 \quad (1)$$

where

δ_0 is the standard penetration depth of the base metal (see A.1).

5.4 Geometry: Edge effects

The induction of eddy currents is obstructed by geometric limitations of the base metal (e.g. edges, drills and others). Therefore, measurements made too near to an edge or corner may not be valid unless the instrument has been specifically adjusted for such measurements. The necessary distance in order to avoid an impact of the edge effect depends on the probe system (field distribution).

NOTE 1 A simple experiment to estimate the edge effect is described in D.2.

NOTE 2 When compared with the phase-sensitive method of ISO 21968, the amplitude-sensitive eddy-current instruments can be substantially more affected by edge effects.

5.5 Geometry: Surface curvature

The propagation of the magnetic field and consequently the induction of eddy currents are affected by the surface curvature of the base metal. This influence becomes more pronounced with decreasing radius of the curvature and decreasing coating thickness. In order to minimize this influence, an adjustment should be performed on a base metal with the same geometry.

The influence of surface curvature depends considerably on the probe geometry and can be reduced by reducing the sensitive area of the probe. Probes with very small sensitive areas are often called microprobes.

NOTE 1 There are instruments and probes available that are capable of automatically compensating the base metal surface curvature influence thus avoiding the resulting thickness error.

NOTE 2 A simple experiment to estimate the effect of surface curvature is described in D.4.

5.6 Surface roughness

Measurements are influenced by the surface topography of the base metal and the coating. Rough surfaces can cause both systematic and random errors. Random errors can be reduced by making multiple measurements, each measurement being made at a different location, and then calculating the average value of that series of measurements.

In order to reduce the influence of roughness, a calibration should be carried out with an uncoated base metal with a roughness equivalent to the coated sample base metal.

If necessary, the definition of the average coating thickness that is used should be stated between the supplier and client.

NOTE When compared with the phase-sensitive method of ISO 21968, the amplitude-sensitive eddy-current measurement can be more affected by base metal roughness.

5.7 Cleanliness: Lift-off effect

If the probe is not placed directly onto the coating, the gap between the probe and coating (lift-off) will affect the measurement as if it were an additional coating. Lift-off can be produced unintentionally due to the presence of small particles between the probe and coating. The probe tip shall frequently be checked for cleanliness.

5.8 Probe pressure

The pressure that the probe exerts on the test specimen can affect instrument reading and shall always be the same during adjustment and measurements.

The influence of the probe pressure is more pronounced in case of soft coatings because the probe tip can be indented into the coating. Therefore, the probe pressure should be as small as possible. Most commercially available instruments are equipped with spring loaded probes, which ensure a constant pressure during the placement. A suitable auxiliary device should be used in case the probe is not spring loaded.

NOTE 1 The contact pressure and the probe tip indentation depth can be reduced by reducing the applied load force or by using a probe with a larger diameter of the probe tip.

NOTE 2 An indentation of the probe tip into soft coatings can be reduced by placing a protective foil with known thickness onto the coated surface. In this case, the coating thickness is the measured thickness minus the foil thickness. This procedure is not applicable when measuring non-magnetic metallic coatings on non-conductive base materials.

5.9 Probe tilt

Unless otherwise instructed by the manufacturer, the probe shall be applied perpendicularly to the coating surface as tilting the probe away from the surface normal can cause measurement errors.

The risk of inadvertent tilt can be minimized by the probe design or by the use of a probe-holding jig.

NOTE Most commercially available instruments are equipped with spring loaded probes, which ensure a perpendicular placement on the sample surface.

5.10 Temperature effects

As temperature changes affect the characteristics of the probe, it should be used under approximately the same temperature conditions as when the instrument was calibrated.

NOTE 1 The influence of temperature variations can be reduced by a temperature compensation of the probe. The manufacturer's specification is taken into account.

NOTE 2 Temperature differences between the probe, electronics of the instrument, environment and sample can cause large thickness errors. One example is the thickness measurement of hot coatings.

Most metals change their electrical conductivity with temperature. Because the measured coating thickness is influenced by changes in the electrical conductivity of the base metal, large temperature changes should be avoided (see 5.2).

5.11 Intermediate coatings

The presence of an intermediate coating can affect the measurement of the coating thickness if the electrical characteristics of that intermediate coating differ from those of the coating or base metal. If a difference does exist, then the measurements will, in addition, be affected by an intermediate coating thickness of less than t_{min}. If the thickness is greater than t_{min}, then the intermediate coating, if non-magnetic, can be treated as the base metal (see 5.3).

5.12 External electromagnetic fields

The measurement results can be influenced by strong electromagnetic interfering fields. In cases showing unexpected results or a strong variation of results, which cannot be explained by other factors, this influence should be taken into account. In this situation, a comparison measurement should be carried out at a location without interfering fields.

6 Calibration and adjustment of the instrument

6.1 General

Prior to use, every instrument shall be calibrated or adjusted according to the instructions of the manufacturer by means of suitable thickness reference standards and base metal. The material, geometry, and surface properties of the base metal used for calibration or adjustment should be similar to those for the test specimens in order to avoid deviations caused by the factors described in Clause 5. Otherwise, these influences shall be considered in the estimation of the measurement uncertainty.

During calibration or adjustment, the instruments, standards and base metal should have the same temperature as the test specimens to minimize temperature induced differences.

In order to avoid the influence of instrument drift, periodic control measurements with reference standards or control samples are recommended. If required, the instrument has to be re-adjusted.

NOTE Most instruments automatically adjust themselves during a function called “calibration”, carried out by the operator, whereas the result of the calibration is often not obvious.

6.2 Thickness reference standards

Thickness reference standards for calibration and adjustment are either coated base metals or foils, which are placed onto uncoated base metals.

Foils and coatings shall be non-conductive and non-magnetizable. Thickness values of the reference standards and their associated uncertainties shall be known and unambiguously documented. The surface area for which these values are valid shall be marked. The thickness values should be traceable to certified reference standards.

The uncertainties shall be documented with their confidence level, e.g. U (95 %), i.e. the probability, that the “true” value is within the reported uncertainty interval around the documented thickness value, is minimum 95 %.

Prior to use, foils and coatings are to be checked visually for damage or mechanical wear as this would cause an incorrect adjustment and thus systematic deviation of all measurement values.

In most cases, the foil material is plastic. In contrast to the magnetic method (see ISO 2178), conductive materials, e.g. copper alloys, cannot be used because in such foils, eddy currents can be induced. They would affect the measurement and cause thickness errors.

NOTE When measuring non-magnetic metallic coatings on non-conductive base materials, the situation is “inverted”.

The use of foils as reference standards, compared to selected coated base metals, benefits from the possibility of placing the foils directly on each base metal. The geometry influence and other factors are already considered within the adjustment.

However, by placing the probe on foils, elastic or plastic deformation may occur, which can affect the measurement result. Moreover, any gap between the pole of the probe, foil and base metal has to be avoided. Especially for concave specimens, or if the foil is wrinkled or bent, the usually low pressure of the spring loaded guiding sleeve of the probe may not be sufficient to ensure there is no gap.

Possible elastic or even plastic deformation of a reference foil depends on the applied force of the probe and the probe tip diameter (see 5.9). Consequently, the calibration of such reference foils should be carried out with comparable values of the applied force and tip diameter to avoid indentation differences during the probe calibration. In this way, respective indentation errors are already taken into account in the foil thickness value, i.e. this value can be smaller than the unaffected geometric thickness. The values of both the applied force and the tip diameter used at the foil calibration should be known from the reference foil manufacturer so that possible thickness errors can be estimated.

6.3 Methods of adjustment

Adjustment of the coating thickness gauges is executed by placing the probes on uncoated and/or one or more coated pieces of base metal with known coating thickness. Depending on the instrument types, instructions of the manufacturer and on the functional range of the instrument under use, adjustments can be carried out on the following items:

a) a piece of uncoated base metal;

b) a piece of uncoated base metal and a piece of coated base metal with defined coating thickness;

c) a piece of uncoated base metal and several pieces of coated base metal with defined but different coating thickness;

d) several pieces of coated base metal with defined but different coating thickness.

According to 6.2, the term “coated base metal” includes foils placed onto uncoated base metal.

The stated adjustment methods may lead to different accuracies of the measuring results. Thus, a method that best fits the given application and leads to the desired accuracy should be used. The measuring uncertainty that can be achieved by the different adjustment methods depends on the evaluation algorithm of the gauges as well as on the material, geometry and surface condition of the standards and of the base metals to be measured. If the desired accuracy is not achieved by one method, a different adjustment method may lead to better results. In general, the measuring uncertainty can be reduced by increasing the number of adjustment points and the better and closer the adjustment points cover the expected thickness interval of the coating to be measured.

NOTE 1 The process that is used to adapt the probe to the given base metal by placing the probe onto the uncoated base metal, is often called “zeroing” or “zero point calibration”. However, even this procedure is an “adjustment” or part of an adjustment process as defined by this document.

NOTE 2 Depending on how many pieces of coated and uncoated base metals are used to adjust the instrument, the corresponding adjustment method is often called “single-point”, “two-point” or “multiple-point adjustment”.

The measurement uncertainty resulting from an adjustment of the instrument cannot be generalized to all subsequent measurements. In each case, all specific and additional influencing factors need to be considered in detail, see Clause 5 and Annex D.

NOTE 3 Some types of gauges permit resetting the instrument to an original adjustment of the manufacturer. This adjustment is valid for the manufacturer’s uncoated or coated reference standards only. If these standards or the same types of standards are used to check the instrument after a period of use, any deterioration of gauge and probes, e.g. wear of the probe by abrasion of the contact pole, can be recognized by observing deviations of the measuring results.

7 Measurement procedure and evaluation

7.1 General

Every instrument shall be operated according to the manufacturer's instructions especially considering the factors affecting measurement accuracy discussed in Clause 5.

Before using the instrument and after changes affecting the measurement accuracy (see Clause 5), the adjustment of the instrument shall be checked.

To ensure that the instrument measures correctly, it shall be calibrated with valid standards at the place of inspection each time:

a) the instrument is put into operation,

b) material and geometry of the test specimens are changed, or

c) other conditions of the inspection have changed (e.g. temperature) whose effects are not known.

As not all changes of measurement conditions and their influences on the measurement accuracy can be immediately recognized (e.g. drift, wear of the probe), the instrument should be calibrated at regular time intervals while in use.

7.2 Number of measurements and evaluation

The coating thickness should be determined as the arithmetic mean of several single values, which are measured in a defined area of the coating surface. In addition to the mean, the standard deviation should be reported (see Annex B). The random part of the measurement uncertainty can be reduced by increasing the number of measurements. If not otherwise specified or agreed upon, it is recommended to measure at least five single values (depending on the application).

NOTE 1 From the standard deviation, a variation coefficient *V* can be calculated. *V* corresponds to the relative standard deviation (e.g. in percent) and enables a direct comparison of the standard deviation for different thicknesses.

NOTE 2 The total scatter of the measurement is composed of the scatter of the instrument itself and the scatter caused by the test specimen. The standard deviation of the operator and probe in the measured thickness range is determined by repeated measurements at the same location, if necessary with the help of an auxiliary device for placing the probe.

When measuring on rough coating surfaces or on test specimens with known large thickness gradients (e.g. due to their size and/or their shape), the reason for deviations between the single measurements should be determined by a series of measurements.

8 Uncertainty of the results

8.1 General remarks

A complete evaluation of the uncertainty of the measured thickness shall be carried out in accordance with ISO/IEC Guide 98-3. Details of the background of the expression of the uncertainty are summarized in Annex B and a typical example of this calculation is described in Annex F.

Uncertainty of the thickness measuring result is a combination of uncertainties from a number of different sources. Important sources that should be considered include the following:

a) uncertainty of the calibration of the instrument;

b) stochastic influences affecting the measurement;

c) uncertainties caused by factors summarized in Clause 5;

d) further influences, drifts, digitalization effects and other effects.

All uncertainty components shall be estimated and summarized to the combined standard uncertainty as described in ISO/IEC Guide 98-3, see Annex B.

A possible procedure for the estimation of the uncertainty is given in the following simplified approach (see 8.2 to 8.5).

The single uncertainty components of the listed sources are dependent on the respective measurements, the properties of the samples measured, the instrument, the environmental condition, etc. and can show large differences for different applications. Therefore, the single uncertainty components shall be estimated for each measurement in all detail. The quality of the uncertainty is determined by the quality of the estimation of all uncertainty components. Missing components result in incorrect uncertainty estimations and consequently in incorrect thickness results.

In particular, the factors listed in Clause 5 can result in large uncertainty values and should be minimized by an adjustment if possible.

NOTE In addition to the need to express the uncertainty in the result, the analysis of possible uncertainty components provides detailed information in order to improve the measurement.

8.2 Uncertainty of the calibration of the instrument

If no other information is given, the current uncertainty of an instrument can be estimated within a limited thickness range by realization of n repeated measurements on a given reference standard with known thickness, t_r, and uncertainty, U_r (k = 2). The measurement result is the arithmetic mean value, $\overline{t}_m$, of the measured thickness values with the standard deviation, $s(t_m)$. The quality of the calibration is determined by the ratio, E, of the resulting difference, $\left|\overline{t}_m - t_r\right|$, and the combined uncertainty of the calibration measurement. This uncertainty (denominator of E, k = 2) is considered to be caused by the stochastic error of the measurement with n repeats (compare to 8.3) and the given reference standard uncertainty, U_r. In case of $E \leq 1$, the calibration is valid and cannot be further improved by means of this reference standard, i.e. the difference cannot be distinguished from the uncertainty. Therefore, the standard uncertainty of the calibration, u_{cal} (k = 1), is given by the combined uncertainty of the verification measurement but with respect to the 1 sigma level (k = 1).

However, in the case of $E > 1$, a significant deviation of the calibration within the uncertainty is detected and an adjustment of the instrument should be carried out in order to improve the calibration accuracy.

See Formulae (2) and (3):

$$E = \frac{\left|t_r - \overline{t}_m\right|}{2 \times u_{cal}} \tag{2}$$

$$u_{cal} = \sqrt{\left[t(68{,}27\,\%, n-1) \times \frac{s(t_m)}{\sqrt{n}}\right]^2 + \left[0{,}5 \times U_r\right]^2} \tag{3}$$

NOTE 1 In case the tolerance, T, of the reference standard is given instead of U_r, i.e. ($t_r \pm T$), for example in a certificate of a certified reference material, the respective standard uncertainty (for 68,3 % confidence level) can be calculated as $U_r = \frac{T}{\sqrt{3}}$ and the expanded uncertainty (for 95,4 % confidence level) as $U_r\,(k=2) = 1{,}653 \times \frac{T}{\sqrt{3}}$. The deviation from the usual factor 2 for normal distribution is due to the fact that tolerances follow rectangular distributions.

The calibration uncertainty u_{cal} is only valid in a small thickness range around t_r. In the case of a larger thickness range of interest, the uncertainty u_{cal} should be estimated on both sides of the thickness range. The linear interpolation between both values gives the uncertainty of interest as a function of the thickness.