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Metallic and other inorganic coatings — Review of methods of measurement of thickness

Revêtements métalliques et autres revêtements inorganiques — Vue d'ensemble sur les méthodes de mesurage de l'épaisseur

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

ISO 3882 was prepared by Technical Committee ISO/TC 107, *Metallic and other inorganic coatings*, Subcommittee SC 2, *Test methods*.

This third edition cancels and replaces the second edition (ISO 3882:1986), which has been technically revised.

Introduction

This International Standard summarizes the various methods used for the measurement of coating thickness and describes their working principles. Methods of measuring coating thickness may be either destructive or non-destructive (see Table 1). The information given in Table 2 will assist in the choice of typical instrumental methods suitable for thickness measurements. For all instrumental methods, manufacturers' instructions should be followed.

The thickness ranges covered by the different methods depend on the coating materials, thickness of the coating, substrates and instruments used (see Table 3); e.g., although X-ray spectrometry can be used to measure the thickness of a chromium coating, thicknesses of 20 µm or more cannot be measured with sufficient precision. Similarly, while magnetic methods may be used to measure the thickness of a gold coating over a magnetic steel substrate, many magnetic instruments do not have the sensitivity to measure accurately thicknesses of gold coatings less than 2 µm.

Where a referee method is required the appropriate coating specification should be consulted.

Metallic and other inorganic coatings — Review of methods of measurement of thickness

1 Scope

This International Standard reviews methods for measuring the thickness of metallic and other inorganic coatings on both metallic and non-metallic substrates (see Tables 1, 2 and 3). It is limited to tests already specified, or to be specified, in International Standards, and excludes certain tests that are used for special applications.

Table 1 — Methods of measuring coating thickness

Non-destructive		Destructive	
Split beam microscope (light section)	ISO 2128 [a]	Microscopical (optical)	ISO 1463
Magnetic	ISO 2178 and ISO 2361	Fizeau multiple-beam interferometry	ISO 3868 [b]
Eddy current	ISO 2360	Profilometric (stylus)	ISO 4518 [b]
X-ray spectrometric	ISO 3497	Scanning electron microscope	ISO 9220
Beta backscatter	ISO 3543	Dissolution methods:	
		Gravimetic strip and weigh method and gravimetric analytical method	ISO 10111
		Coulometric method	ISO 2177

[a] Can be destructive in some applications.
[b] Can be non-destructive in some applications.

2 Normative references

The following referenced documents are indispensable for the application 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 1463, *Metallic and oxide coatings — Measurement of coating thickness — Microscopical method*

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

ISO 2128, *Anodizing of aluminium and its alloys — Determination of thickness of anodic oxide coatings — Non-destructive measurement by split-beam microscope*

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

ISO 2178, *Non-magnetic coatings on magnetic substrates — Measurement of coating thickness — Magnetic method*

ISO 2360, *Non-conductive coatings on non-magnetic basis materials — Measurement of coating thickness — Amplitude-sensitive eddy current method*

ISO 2361, *Electrodeposited nickel coatings on magnetic and non-magnetic substrates — Measurement of coating thickness — Magnetic method*

ISO 3497, *Metallic coatings — Measurement of coating thickness — X-ray spectrometric methods*

ISO 3543, *Metallic and non-metallic coatings — Measurement of thickness — Beta backscatter method*

ISO 3868, *Metallic and other non-organic coatings — Measurement of coating thicknesses — Fizeau-multiple beam interferometry method*

ISO 4518, *Metallic coatings — Measurement of coating thickness — Profilometric method*

ISO 9220, *Metallic coatings — Measurement of coating thickness — Scanning electron microscope method*

ISO 10111, *Metallic and other inorganic coatings — Measurement of mass per unit area — Review of gravimetric and chemical analysis methods*

3 Terms and definitions

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

4 Non-destructive methods

4.1 Split beam microscope (light section) method, ISO 2128

This equipment, originally designed for the measurement of surface roughness, is used for measuring the thickness of transparent and translucent coatings, in particular, anodic oxide coatings on aluminium. A light beam is projected on to the surface at an angle of 45°. Part of the beam is reflected from the surface of the coating and the rest penetrates the coating and is reflected from the coating/metal substrate interface. The distance that separates the two images observed in the eyepiece of the microscope is proportional to the thickness of the coating and can be measured by means of a vernier screw which controls a calibrated graticule. The method can be used where sufficient light is reflected from the coating/metal substrate interface to give a clear image in the microscope. For transparent or translucent coatings, such as anodic oxide films, this method is non-destructive.

For measuring the thickness of opaque coatings, a small area of the coating is removed and in this application the method is destructive. The step between the surface of the coating and the basis metal produces a deflection of the light beam which gives an absolute measure of the coating thickness.

The method is not suitable for hard anodic coatings or for coatings that are very thin (less than 2 µm thick), very thick (greater than 100 µm thick) or rough. It is not suitable for coatings on heavily shot-blasted surfaces. Other methods such as eddy current (ISO 2360), interference microscope (ISO 3868) and microscopical (ISO 1463) may be applicable for thickness measurement where the split beam microscope method cannot be used.

The method is best suited to small parts because of the ease with which they can be set up on the microscope stage.

The measurement uncertainty of the method is usually less than 10 % of the thickness.

4.2 Magnetic methods, ISO 2178 and ISO 2361

Instruments for these methods measure either the magnetic attraction between a magnet and the basis metal, as influenced by the presence of the coating, or the reluctance of a magnetic flux path passing through the coating and the basis metal.

All instruments using magnetic methods are sensitive to the magnetic condition and properties of the test specimen, surface curvature, surface cleanliness, surface roughness, and thickness of the basis metal and of the coating.

These methods are limited in practice to non-magnetic coatings on a magnetic substrate (see ISO 2178) and to electroplated nickel coatings on a magnetic or non-magnetic substrate (see ISO 2361).

The measurement uncertainty of the method is less than 10 % of the thickness or 1,5 µm, whichever is the greater.

4.3 Eddy current method, ISO 2360

ISO 2360 describes an amplitude method and is based on differences in electrical conductivity between coatings and substrates. The method is used primarily for measuring the thickness of non-conductive coatings on non-magnetic metals and of single layer metal coatings on non-conductors. If this method is used for measuring thicknesses of metallic coatings on metallic substrates, great care is necessary if acceptable results are to be obtained.

The method is ideal for rapid determination of anodic coating thickness measurements on aluminium and its alloys and is well suited for use in field measurements. For autocatalytic nickel coatings, this method gives erratic measurements due to variations in conductivity of the coatings with changes in phosphorous content.

ISO 2360 only covers the measurement of the thickness of a non-conductive coating on a non-magnetic basis metal.

The measurement uncertainty of the method is usually less than 10 % of the thickness or 0,5 µm, whichever is the greater.

4.4 X-ray spectrometric methods, ISO 3497

These methods use emission and absorption X-ray spectrometry for determining the thickness of metallic coatings.

X-rays are made to irradiate a fixed area of the coated surface, and the intensity of the secondary radiation emitted by the coating or by the substrate and attenuated by the coating is measured. A correlation exists between the intensity of the X-rays and the coating thickness; this is established using calibration standards.

This method's accuracy is reduced:

— when constituents of the coating are present in the basis metal, and vice versa;

— when more than two coatings are superimposed;

— when the chemical composition of a coating varies greatly from that of the calibration standard.

The method is not applicable above a certain thickness which depends on the atomic numbers and densities of the materials involved.

For autocatalytic nickel coatings this method is only recommended for deposits in the as-plated condition. The phosphorus content of the coating should be known in order to enable calculation of the thickness of the deposit. As the matrix effect due to the distribution of phosphorus in layers of the coating also affects the measurement uncertainty, the calibration standards have to be made under the same conditions as those of the production process.

Instruments capable of measuring the thickness of a coating with an uncertainty of less than 10 % of the thickness are commercially available.

4.5 Beta backscatter method, ISO 3543

This method uses radio isotopes that emit beta rays and detectors that measure the intensity of those beta rays backscattered by the test specimen. The intensity of the backscattered beta rays will be between two values, namely the backscatter intensity of the coating and that of the basis metal. The measurement is only possible if the atomic number of the coating material is sufficiently different from that of the substrate. The

instrument is calibrated using calibration standards having the same coating and substrates as the specimen to be measured. The measured intensity of the beta rays backscattered by the test specimen is used to calculate the mass per unit area of the coating, which, provided that the test specimen is of uniform density, is directly proportional to the thickness.

This method can be used for both thin and heavy coatings, the maximum thickness being a function of the coating material density.

A measurement uncertainty of less than 10 % of the thickness over a wide range of thickness can be achieved using this method.

5 Destructive methods

5.1 Microscopical (optical) method, ISO 1463

In this method, coating thickness is measured on a magnified image of a cross-section of the coating using an optical microscope.

The measurement uncertainty of this method is less than 10 % of the thickness subject to a minimum error of 0,8 µm. However, with careful preparation of the specimen and the application of suitable instruments this method is capable of providing a measurement uncertainty of 0,4 µm under reproducible conditions.

5.2 Fizeau multiple-beam interferometry method, ISO 3868

By completely dissolving a small area of the coating without attacking the substrate, or by masking an area before plating, a step is formed from the surface of the coating to that of its substrate. The height of this step is measured with a multiple-beam interferometer.

This method is particularly useful for measuring the thickness of very thin opaque metal coatings. It is not suitable for vitreous enamel coatings. This method is essentially a laboratory technique and is useful for measuring the thickness of coatings on standards used for calibrating non-destructive test instruments such as beta backscatter and X-ray spectrometry instruments, especially for standards with rather thin (sub-micron) coatings.

This method provides an absolute measure of the microscopic vertical surface variation ranging from 0,002 µm to 0,2 µm. The measurement uncertainty is $\pm$ 0,001 µm.

5.3 Profilometric (stylus) method, ISO 4518

By masking during the coating process, or by dissolving a small area of the coating without attacking the substrate, a step is formed from the surface of the substrate to that of the coating. A stylus is drawn across this step and its height is determined by electronically measuring and recording the motion of the stylus.

Available commercial instruments allow measurements to be made over the range 0,01 mm to 0,000 02 mm (20 nm).

The measurement uncertainty of this method is less than 10 % of the thickness.

5.4 Scanning electron microscope method, ISO 9220

In this method, coating thickness is measured on a magnified image of a cross-section of the coating using a scanning electron microscope. The measurement is made on a conventional micrograph or on a photograph of the video waveform signal for a single scan across the coating. Errors can occur if both the calibration and the measurement are not made over the same segment of the field, as the magnification may not be uniform over the entire field. The magnification also often drifts with time introducing further error in the thickness measurement.

The measurement uncertainty is less than 10 % of the thickness or 0,1 µm, whichever is the greater.

5.5 Dissolution methods

5.5.1 Coulometric method, ISO 2177

The metallic coating thickness is determined by measuring the quantity of electricity consumed in dissolving the coating from a precisely defined area when the article is made anodic in a suitable electrolyte under suitable conditions.

The change in potential that occurs when the underlying material is reached serves to indicate the end-point of the dissolution. The method is applicable to metallic coatings on both metallic and non-metallic substrates.

The measurement uncertainty of the method is less than 10 % of the thickness.

5.5.2 Gravimetric (strip and weigh) method, ISO 10111

In this method, the coating mass is determined by weighing the sample before and after dissolving the coating without attacking the substrate, or by weighing the coating after dissolving the substrate without attack of the coating. The coating has to be of uniform density.

The mass of the coating divided by the density and the area of the coating gives the average coating thickness.

The limitation of the method is that it does not indicate the presence of bare spots or sites with thicknesses lower than the specified minimum in the measuring areas. Additionally, the single value obtained from each measuring area is the mean thickness over that area: there can be no further mathematical analysis of this single value, e.g. for statistical process control purposes.

The measurement uncertainty of this method is less than 5 % of the thickness over a wide range of thicknesses.

5.5.3 Gravimetric (analytical) method, ISO 10111

In this method, the coating mass is determined by dissolving the coating, with or without dissolving the substrate material, and determining the quantity of coating metal by chemical analysis.

The mass of the coating divided by the density and the area of the coating gives the average coating thickness.

The limitations of the method are as follows:

— it is not reliable if the same metal is present in the coating and in the substrate or in the basis metal;

— it does not indicate the presence of bare spots or sites with thicknesses lower than the specified minimum in the measuring areas;

— the single value obtained from each measuring area is the mean thickness over that area: there can be no further mathematical analysis of this single value, e.g. for statistical process control purposes.

The measurement uncertainty of this method is normally less than 5 % of the thickness over a wide range of thicknesses.

Table 2 — Applicability of typical instrumental methods for coating thickness measurement

Substrate	Coating																
	Al and alloys	Ag	AO[a]	Au	Cd	Cr	Cu	Ni[b]	Ni[c]	NM[d]	Pb	Pd	Rh	Sn	Sn-Ni	Sn-Pb	VE[e]	Zn
Al and alloys	—	B C X	E	B C X	B C X	B C X E	B C X	B C X M[f, g]	B C[h] E[f, h] X[h, i]	E	B C X	B X	B X[i]	B C X	BCX	B[j] X C[j]	E	B C X
Ag	B X[k] C	—	X	B C X	C X	B C X E	B C X	B C X M[f, g]	B C[h, i] X E	B E X[i]	B C X	X[i]	X	C X	X	B[j] X	E X[i]	B C X
Cu and alloys	B X[k]	B C X	E	B C X	B C X	B C X E	C[l] X	C X M[f, g]	C[h] E[f] X[i]	B E X[i]	B C X	B X	B X	B C X	C X	B[j] X C[j]	E X[i]	C X
Mg and alloys	B X[k]	B X	E	B X	B X	B X	B X	B X M[f, g]	B X[h] E[f, h]	E	B X	B X	B X	B X	X	B[j] X	E	B X
Ni	B X M[k]	B C X M[k]	B	B C X M[k]	B C X M[k]	B C X M[k]	C X M[k]	S	—	M[k] X[i]	B C X M[k]	B X M[k]	B X M[k]	B C X M[k]	X M[k]	B[j] C[j] X M[k]	M[k] X[i]	C X M[k]
Ni-Co-Fe	B M X[i, k]	B M C X	X	B C M X	B C M X	C M X	C M X	C X[i]	C X[i] M[f]	B M X[i]	B C M X	B M X	B M X	B M C X	X	B M X C[j]	X[i] M	M C X
NM[d]	B E X	B C X E[m]	—	B C X E[m]	B C X E[m]	B C X E[m]	B C X E[m]	B C X M[f, g]	B C[h] X[m] E[f, h]	—	B C X E[m]	B X E[m]	B X E[m]	B C X E[m]	X E[m]	B[j] C[j] X E[m]	—	B C X E[m]
Steels[o]	B M X[k]	B C M X	X	B C M X	B C M X	C M X[h]	C M X	C X M[f, g]	C[h] M X[i]	B E M X[i]	B C M X	B M X	B M X	B C M X	X	B[j] M X C[j]	M X[i]	B C M X
Steels[p]	B X[k]	B C X E[m]	X	B C X E[m]	B C X	C X[n]	C X E[m]	C X M[f, g]	C[h] X[h, i]	B E X[i]	B C X	B X	B X	B C X	X C	B[j] X C[j]	E X[i]	B C X
Ti	B[i] X[k]	B C X E[m]	X	B C X E[m]	B C X	C X	B C X E[m]	B C X M[f, g]	B C[h] X[i]	B E	B X	B X	B X	B C X	—	B[j] C X	X[i]	B C X
Zn and alloys	B X[k]	B C X	X	B X	B X	C X	C X	C X M[f, g]	C X[h, i]	B E X[i]	B X	B X	B X	B C X	—	B[j] X	E[j] X[i]	—

Table 2 *(continued)*

The methods are indicated by the following letters: B = beta backscatter, ISO 3543; C = coulometric, ISO 2177; E = eddy current, ISO 2360; M = magnetic/electromagnetic induction, ISO 2178, for nickel coating (see footnote g); S = step test; X = X-ray spectrometry (ISO 3497). NOTE 1 This table provides general guidance only. The applicability of a method can be a function of an individual instrument and of other variables such as coating and substrate thickness. For details of the methods, reference should be made to the appropriate clauses of this International Standard as well as to the relevant International Standard for each method of measurement. Where proprietary instruments are used special care should be exercised to ensure that specific manufacturer's instructions and procedures are followed, particularly in matters concerning calibration. NOTE 2 The thickness of metallic and non-metallic coatings can also be measured by the microscopic method (ISO 1463) and the scanning electron microscopic method (ISO 9220). For applicability of the gravimetric (ISO 10111), interferometric (ISO 3868), profilometric (ISO 4518) and split beam microscopic (ISO 2128) methods, see the appropriate clauses of this International Standard and the relevant International Standard for each method of measurement.
a Anodic oxide.
b Nickel (non-autocatalytic).
c Autocatalytic nickel.
d Non-metal.
e Vitreous and porcelain enamel.
f Method is sensitive to permeability variations of the coating.
g Magnetic method instruments (ISO 2361) can be used for thickness measurement of some nickel coatings.
h Method is sensitive to variations in the phosphorous/boron content of the coating.
i Measurement is possible but measuring error may be greater than normal.
j Method is sensitive to alloy composition.
k Only when substrate is sufficiently thick.
l Only on CuZn, CuBe, CuSnAl.
m Method is sensitive to conductivity variations of the coating.
n Not for chromium steels.
o Magnetic, corrosion resisting steels.
p Non-magnetic steels.

Table 3 — Representative thickness ranges of coating thickness measuring instruments

Instrument type	Representative thickness [a] [b] µm	Relevant International Standard
Magnetic for non-magnetic coatings on steel	5 to 7 500	ISO 2178
Magnetic for nickel coatings	1 to 125	ISO 2361
Eddy current	5 to 2 000	ISO 2360
X-ray spectrometric	0,25 to 25	ISO 3497
Beta backscatter	0,1 to 1 000	ISO 3543
Split beam microscope	2 to 100	ISO 2128
Coulometric	0,25 to 100	ISO 2177
Microscopical	4 to several hundreds	ISO 1463
Profilometric	0,002 to 100	ISO 4518
Scanning electron microscope	1 to several hundreds	ISO 9220

NOTE 1 The thickness ranges indicated are representative for:

— standard models of commercially available instruments;

— using large, flat, and smooth test specimens;

— for commonly used electroplated, autocatalytically deposited, anodized or ceramic coatings;

— for measurements made with reasonable care and effort.

Actual ranges depend on such factors as size, shape, coating material and substrate material of the test specimen and make and model of the instrument. The stated ranges can often be extended by modification of the instrument or of the measuring techniques. Individual instruments may not cover the entire range given for that instrument type.

NOTE 2 In general, at a thickness of one tenth the thickness given for the lower end of the range, a measurement uncertainty of 100 % of the thickness can be expected. Thus the microscopical method has a measurement uncertainty of about one tenth of 4 µm, i.e. 0,4 µm.

[a] The values presented in this table were provided by the instrument manufacturers for general guidance only.

[b] Thickness ranges having an uncertainty of less than 10 % of the thickness.

Bibliography

[1] CLARKE, M. and DUTTA, P.K., *Transactions of the Institute of Metal Finishing*, 1966, **44**(1), 9

[2] HARBULAK, E.P., Simultaneous thickness and electrochemical potential determination of individual layers in multilayer nickel deposits, *Plating and surface finishing*, February 1980, **67**(2), 49-54

[3] JOFFE, B.B., *Plating and surface finishing*, September 1983, **40**

[4] LATTER, T.D.T., *British journal of NDT*, July 1989, **31**(7), 372

[5] KOMATSU, H., *Interferometry: Principles and applications of two-beam and multiple-beam interferometry*, Sendai, Japan, Institute of Materials Research, Tohoku University

[6] RAY, G.P., *Thickness testing of electroplated and related coatings*, Electrochemical Publications Ltd., Asahi House, 10 Church Road, Port Erin, Isle of Man, British Isles, 1993, ISBN 0 901150 27 4. Available from the Institute of Metal Finishing, Exeter House, 48 Holloway Head, Birmingham B1 1NQ, UK

[7] BIKULCIUS, G., *Plating and surface finishing*, August 1997, **84**(8), 30

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