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Metallic and other non-organic coatings — Review of methods of measurement of thickness

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

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Foreword

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This second edition cancels and replaces the first edition (ISO 3882-1976), table 2 of which has been technically revised.

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Metallic and other non-organic coatings — Review of methods of measurement of thickness

0 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 and table 3 will assist in the choice of the method most suited to a particular purpose.

The thickness ranges covered by the different methods depend on the coating materials, substrates and instruments used.

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1 Scope and field of application

This International Standard reviews methods for measuring the thickness of metallic and other non-organic coatings on both metallic and non-metallic substrates. It is limited to tests already specified, or to be specified, in International Standards, and excludes certain tests which are employed for special applications.

2 References

ISO 1463, *Metallic and oxide coatings — Measurement of coating thickness — Microscopical method.*

ISO 2064, *Metallic and other non-organic 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 metals — Measurement of coating thickness — 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.*

3 Definitions

For the purpose of this International Standard, the definitions of ISO 2064 apply.

4 Non-destructive methods

4.1 Magnetic methods

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.

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

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

4.2 Eddy current method

This method is based on differences in electrical conductivity between coatings and substrates. It is used primarily for measuring the thicknesses 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.

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 % or 0,5 µm, whichever is the greater.

4.3 X-ray spectrometric methods

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

X-rays are made to hit 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.

The X-ray method is generally applicable but its accuracy is reduced in the following situations:

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

b) when more than two coatings are superimposed;

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

It is not applicable above a certain thickness which depends on the atomic number and the mass of the metal.

Instruments capable of measuring thicknesses of coating with an uncertainty of less than 10 % are commercially available (see ISO 3497).

4.4 Beta backscatter method

Beta ray gauges use radioisotopes which emit beta rays and detectors for measuring the intensity of the beta rays backscattered by the test specimen. The intensity of the backscattered beta rays will be between two values, 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 with 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 latter is of uniform density, is directly proportional to the thickness.

The method can be used for measuring both thin and heavy coatings, the maximum thickness being a function of the atomic number of the coating.

Usually a measurement uncertainty of less than 10 % over a wide range of thicknesses (see ISO 3543) can be obtained by this method.

4.5 Split-beam microscope (light section) method (see ISO 2128)

This equipment was originally designed for the measurement of surface roughness, but it is also 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 while the other part penetrates the coating and is reflected from the coating/metal substrate interface. The distance which 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 has to be 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 measurement uncertainty of the method is usually less than 10 %.

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5 Destructive methods

5.1 Dissolution methods

5.1.1 Gravimetric method (stripping and weighing)

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 should 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 measurement uncertainty of the gravimetric method is normally less than 5 % over a wide range of thicknesses.

5.1.2 Analytical 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 measurement uncertainty of this method is normally less than 5 % over a wide range of thicknesses.

The method may not be reliable if the same metal is present in the coating and in the substrate or in the basis metal.

5.1.3 Coulometric method

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 which 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 normally less than 10 % (see ISO 2177).

5.2 Microscopical (optical) method

In the microscopical method, coating thicknesses are measured on a magnified image of a cross-section of the coating.

Normally the measurement uncertainty of this method is less than 10 % subject to a minimum error of 0,8 µm (see ISO 1463).

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5.3 Profilometric method (stylus method)

By masking during the coating process, or by dissolving a small area of the coating without attacking its 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.

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The measurement uncertainty of the method is normally less than 10 % over a wide range of thicknesses (see ISO 4518).

5.4 Interference microscope method

The thickness of the coating is measured by directing a monochromatic light beam upon a step of the deposit. This step is obtained by masking an area of the substrate before deposition or by masking the coating before dissolution of the unmasked portion.

A step in the specimen surface causes a shift in the fringe pattern. A shift of one full fringe spacing is equivalent to a vertical displacement of one-half of the wavelength of the monochromatic light. The whole and fractional number of fringe spacings occupied by the fringe shift is determined with an eyepiece micrometer.

For a description of a multiple-beam interferometry method, see ISO 3868.

The measurement uncertainty of the multiple-beam method is normally less than 0,01 µm.

Table 1 — List of methods of coating thickness measurement

Non-destructive methods	Destructive methods
Magnetic (ISO 2178 and ISO 2361) Eddy current (ISO 2360) X-ray spectrometry method (ISO 3497) Beta backscatter method (ISO 3543) Split-beam microscope[1] (ISO 2128)	Dissolution methods: — gravimetric (chemical dissolution) — analytical determination of dissolved metal — coulometric method (ISO 2177) Microscopical examination of cross-section (ISO 1463) Profilometric method[2] (ISO 4518) Interference microscope methods[2] (ISO 3868)

1) May be destructive in some applications.

2) May be non-destructive.

Table 2 — Applicability of different instrumental methods of measuring coating thicknesses
(based on current practice in 1983)

Substrates \ Coatings	Aluminium and alloys	Anodic oxides	Cadmium	Chromium	Copper	Gold	Lead	Nickel	Nickel autocatalytic	Non-metals	Palladium	Rhodium	Silver	Tin	Tin-lead alloy	Vitreous and porcelain enamels	Zinc
Aluminium and alloys	—	E	BC	BC	BC	B	BC	BCM[1]	BC[2]E[1)2)]	E	B	B	BC	BC	B[3]C[3]	E	BC
Copper and alloys	—	E	BC	C	C only on brass and Cu-Be	B	BC	CM[1]	C[2]M[1]	BE	B	B	BC	BC	B[3]C[3]	E	C
Magnesium and alloys	—	E	B	B	B	B	B	BM[1]	B	E	B	B	B	B	B[3]	—	B
Nickel	—	—	BC	BC	C	B	BC	—	—	BE	B	B	BC	BC	B[3]C[3]	—	C
Nickel-cobalt-iron alloys (for example Kovar)	—	—	BM	M	M	BM	BCM	CM[1]	C[2]M[1]	BM	BM	BM	BM	BM	B[3]C[3]M	—	BM
Non-metals	BE	—	BC	BC	BC	B	BC	BCM[1]	BC[2]	—	B	B	BC	BC	B[3]C[3]	—	BC
Silver	—	—	—	B	B	B	BC	BM[1]	B	BE	—	—	—	—	B[3]	E	B
Steels, magnetic	BM	—	BCM	CM	CM	BM	BCM	CM[1]	C[2]M[1]	BM	BM	BM	BCM	BCM	B[3]C[3]M	M	BCM
Steels, non-magnetic	B	—	BC	C	C	B	BC	CM[1]	B[2]C[2]M[1]	BE	B	B	BC	BC	B[3]C[3]	E	BC
Titanium	—	—	B	—	B	B	B	BM[1]	B	BE	B	B	B	B	B[3]	—	B
Zinc and alloys	—	—	B	B	C	B	B	M[1]	—	BE	B	B	B	B	B[3]	—	—

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Key

B is beta backscatter; C is coulometric; E is eddy current; M is magnetic.

1) Method is sensitive to permeability variations of the coating.

2) Method is sensitive to variations in the phosphorus or boron content of the coating.

3) Method is sensitive to alloy composition.

NOTE — This table indicates the most commonly used instrumental methods for the measurement of coating thicknesses. The instrument manufacturers' instructions should be taken as guides to the practical limitations of the methods. In the case of the coulometric method other combinations not listed in the table may be tested with some existing electrolyte or new electrolytes may be developed for them. In addition to these methods, other methods may be used. These are as follows: microscopical, X-ray spectrometry, profilometry, interferometry, split-beam microscope, gravimetric, and analytical determination, which are applicable to most coating/substrate combinations.

Table 3 — Representative thickness ranges of coating thickness measuring instruments

The thickness ranges indicated are representative for:

a) standard models of commercially available instruments;

b) using large, flat, and smooth test specimens;

c) for commonly used electroplated, autocatalytically deposited, anodized, or ceramic coatings; and

d) 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 instrument. The stated ranges can often be extended by modification of the instrument or of the measuring techniques. Any individual instrument may not cover the entire range given for that instrument type.

Instrument type	Representative thickness (μm) range for measurements having an uncertainty of less than 10 %[1)]	Relevant ISO documents
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 spectrometry	0,25 to 65	ISO 3497
Beta backscatter	0,1 to 100	ISO 3543
Split beam	5 to several hundreds	ISO 2128
Coulometric	0,25 to 100	ISO 2177
Microscopical	8 to several hundreds	ISO 1463
Profilometric	0,01 to 100	ISO 4518

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1) The values presented in table 3 were provided by instrument manufacturers in 1982.

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NOTE — In general, at a thickness of one-tenth of the thickness given for the lower end of the range, one can expect a measurement uncertainty of about 100 %. Thus the microscopical method has an absolute uncertainty of about one-tenth of 8 μm or 0,8 μm.

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