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

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up 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.

Draft International Standards adopted by the Technical Committees are circulated to the Member Bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 3882 was drawn up by Technical Committee ISO/TC 107, *Metallic and other non-organic coatings*, and was circulated to the Member Bodies in August 1975.

It has been approved by the Member Bodies of the following countries :

Australia	Ireland	Spain
Austria	Israel	Sweden
Brazil	Italy	Switzerland
Bulgaria	Japan	Turkey
Czechoslovakia	Mexico	United Kingdom
France	New Zealand	U.S.A.
Germany	Poland	U.S.S.R.
Hungary	Romania	
India	South Africa, Rep. of	

No Member Body expressed disapproval of the document.

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

1 SCOPE AND FIELD OF APPLICATION

This International Standard reviews methods of measurement of 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; it does not deal with certain tests which are employed for special applications.

The terms used in this International Standard are in accordance with ISO 2064.

The methods of measurement of thickness can be divided into destructive and non-destructive methods (see table 1).

A summary of the various methods is given in the following clauses which describe working principles, while the information given in table 2 will assist in the choice of the method which is most suited to a particular purpose.

2 REFERENCES

ISO 1463, *Metallic and oxide coatings — Measurement of thickness by microscopical examination of cross-sections.*

ISO 2064, *Metallic and other non-organic coatings — Definitions and conventions concerning the measurement of the 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 metallic and vitreous or porcelain enamel coatings on magnetic basis metals — Measurement of coating thickness — Magnetic method.*

ISO 2360, *Non-conductive coatings on 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 back-scatter method.*[1)]

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

3 NON-DESTRUCTIVE METHODS

3.1 Magnetic methods

Methods of this type 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 accuracy of the method is normally better than ± 10 % or ± 0,7 µm, whichever is greater.

These methods, in practice, are limited 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).

3.2 Eddy current method

This method is based on differences in electrical conductivity between the coating and the substrate. It is used primarily for the measurement of thickness of non-conductive coatings on metals, and of metals on non-conductors. When this method is used for the measurement of thickness of metallic coatings on a metallic substrate, extreme caution should be observed.

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

The accuracy of the method is normally better than ± 10 % or ± 0,7 µm, whichever is greater.

3.3 X-ray spectrometry methods

These methods use emission and absorption X-ray spectrometry for determining the thickness of metallic coatings up to about 15 µm. This limit will depend on the coating material and the equipment used.

1) At present at the stage of draft.

When X-rays hit a fixed area of the coated surface, the intensity of the secondary radiation emitted by the coating, or by the substrate attenuated by the coating, is measured. A correlation exists between the intensity of the X-rays and the coating thickness.

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.

Instruments capable of measuring thickness of coatings with an accuracy of ± 10 % are commercially available.

Minimum measurable thickness : generally a few hundredths of a micrometre (see ISO 3497).

3.4 Beta back-scatter method

The beta-ray gauge employs a radio-isotope which emits beta-rays, and a detector for measuring the intensity of the beta-rays back-scattered by the test specimen. Some of the beta-rays entering the metal collide with atoms of the metal and are refelcted back towards the source. The intensity of these back-scattered beta-rays is a function of the coating thickness, and the measurement is possible provided that the atomic number of the coating metal is sufficiently different from that of the basis metal.

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. In practice, high atomic number coatings, such as gold, can be measured up to 50 μm, while low atomic number coatings, such as copper or nickel, can be measured up to about 300 μm.

Normally an accuracy better than ± 10 % can be obtained by this method, over a wide range of thicknesses (see ISO 3543).

3.5 Split-beam microscope method

A beam of monochromatic light, having passed through the objective of a microscope, is directed onto the surface of the sample, generally at an angle of 45° to the perpendicular. The beam is reflected at the surface and by using another objective a line is visible. The light rays are reflected by an amount which is related to the roughness of the surface. To measure the coating thickness a small area of the coating has to be removed, except in the case of transparent coatings. The step between the surface of the coating and the basis metal produces a deflection of the light beam. The difference in the deflection is an absolute measure of the coating thickness.

For transparent coatings such as anodic oxide films, this method is non-destructive (see ISO 2128).

This method is useful for coatings of thickness greater than or equal to 5 μm.

4 DESTRUCTIVE METHODS

4.1 Dissolution methods

4.1.1 *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 attacking the coating.

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

The accuracy of the gravimetric method is normally better than 5 % over a wide range of thicknesses.

4.1.2 *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 accuracy of these methods is normally better than 5 % over a wide range of thicknesses.

The method cannot be used accurately if the same metal is present in the coating and in the substrate or in the basis metal.

4.1.3 *Coulometric method*

Coating thickness may be 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 occurring when the underlying material is reached serves to indicate the end-point of the dissolution.

The accuracy of the method is normally better than ± 10 % over a range of 0,2 to 50 μm (see ISO 2177).

4.2 Microscopical method

In the microscopical method the coating thickness is measured on a magnified image of a cross-section of the coating.

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

4.3 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, the height of which is detemined by electronically measuring and recording the motion of the stylus.

The accuracy of the method is normally better than ± 10 % over a wide range of thicknesses.

Thicknesses of the order of a few hundredths of a micrometre can be measured by this method.

4.4 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 accuracy of the multiple-beam method is normally better 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 back-scatter 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-sections (ISO 1463) Profilometric method[2] Interference microscope methods[2] (ISO 3868)

1) May be destructive in some applications.

2) May be non-destructive.

TABLE 2 — **Applicability of different coating thickness measuring methods**
(Based on current practice in 1973)

Substrates \ Coatings	Copper	Nickel	Chromium	Auto catalytic nickel	Zinc	Cadmium	Gold	Palladium	Rhodium	Silver	Tin	Lead	Tin-lead alloys	Anodic oxides	Non-metals	Vitreous and porcelain enamels
Magnetic steel (incl. corrosion-resisting steel)	CM	CM[1]	CM	C[2]M[1]	BCM	BCM	BM	BM	BM	BCM	BCM	BCM	B[3]C[3]M	—	BM	M
Non-magnetic stainless steels	CE[4]	CM[1]	C	BC[2]	BC	BC	B	B	B	BCE[4]	BC	BC	B[3]C[3]	—	BE	E
Copper and alloys	C only on brass and Cu-Be	CM[1]	C	C[2]	C	BC	B	B	B	BC	BC	BC	B[3]C[3]	—	BE	E
Zinc and alloys	C	M[1]	—	—	—	B	B	B	B	B	B	B	B[3]	—	BE	—
Aluminium and alloys	BC	BCM[1]	BC	BC[2]E[1][2]	BC	BC	B	B	B	BC	BC	BC	B[3]C[3]	E	E	E
Magnesium and alloys	B	BM[1]	B	B	B	B	B	B	B	B	B	B	B[3]	—	E	—
Nickel	C	—	C	—	C	BC	B	B	B	BC	BC	BC	B[3]C[3]	—	BE	—
Silver	B	BM[1]	B	B	B	—	B	—	—	—	—	BC	B[3]	—	BE	E
Nickel-cobalt-iron alloys	M	CM[1]	M	C[2]M[1]	BM	BM	BM	BM	BM	BM	BM	BCM	B[3]C[3]M	—	BM	—
Non-metals	BCE[4]	BCM[1]	BC	BC[2]	BC	BC	B	B	B	BC	BC	BC	B[3]C[3]	—	—	—
Titanium	B	BM[1]	—	BE[1][2]	B	B	B	B	B	B	B	B	B[3]	—	BE	—

B = Beta back-scatter

C = Coulometric

E = Eddy current

M = Magnetic

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

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

3) Method is sensitive to alloy composition.

4) Method is sensitive to conductivity variations of the coating.

This table indicates the most commonly used test methods for the measurement of coating thicknesses. In addition to these methods, other methods can also be used. These methods are : the microscopical, X-ray spectrometry, profilometry, interferometry, split-beam, gravimetric and analytical determination, which are applicable to most coating-substrate combinations.

Each of these methods has limitations, and these limitations are described in the pertinent International Standards, national standards, operating instructions for coating thickness gauges, books on electroplating, and the text of this International Standard.