SLOVENSKI STANDARD
SIST EN ISO 3497:2002

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Kovinske prevleke - Merjenje debeline prevleke - Rentgenska spektrometrijska metoda (ISO 3497:2000)

Metallic coatings - Measurement of coating thickness - X-ray spectrometric methods (ISO 3497:2000)

Metallische Schichten - Schichtdickenmessung - Röntgenfluoreszenz-Verfahren (ISO 3497:2000)

Revetements métalliques - Mesurage de l'épaisseur du revetement - Méthodes par spectrométrie de rayons X (ISO 3497:2000)

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ICS:

17.040.20	Lastnosti površin	Properties of surfaces
25.220.40	Kovinske prevleke	Metallic coatings

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English version

Metallic coatings - Measurement of coating thickness - X-ray spectrometric methods (ISO 3497:2000)

Revêtements métalliques - Mesurage de l'épaisseur du revêtement - Méthodes par spectrométrie de rayons X (ISO 3497:2000)

Metallische Schichten - Schichtdickenmessung - Röntgenfluoreszenz-Verfahren (ISO 3497:2000)

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This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Management Centre has the same status as the official versions.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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Foreword

The text of the International Standard ISO 3497:2000 has been prepared by Technical Committee ISO/TC 107 "Metallic and other inorganic coatings" in collaboration with 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of an identical text or by endorsement, at the latest by June 2001, and conflicting national standards shall be withdrawn at the latest by June 2001.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

Endorsement notice

The text of the International Standard ISO 3497:2000 was approved by CEN as a European Standard without any modification.

INTERNATIONAL STANDARD

ISO 3497

Third edition
2000-12-15

Metallic coatings — Measurement of coating thickness — X-ray spectrometric methods

Revêtements métalliques — Mesurage de l'épaisseur du revêtement — Méthodes par spectrométrie de rayons X

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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 3.

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 International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 3497 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 3497:1990), which has been technically revised.

Annex A of this International Standard is for information only.

Metallic coatings — Measurement of coating thickness — X-ray spectrometric methods

1 Scope

WARNING Problems concerning protection of personnel against X-rays are not covered by this International Standard. For information on this important aspect, reference should be made to current international and national standards, and to local regulations, where these exist.

1.1 This International Standard specifies methods for measuring the thickness of metallic coatings by the use of X-ray spectrometric methods.

1.2 The measuring methods to which this International Standard applies are fundamentally those that determine the mass per unit area. Using a knowledge of the density of the coating material, the results of measurements can also be expressed as linear thickness of the coating.

1.3 The measuring methods permit simultaneous measurement of coating systems with up to three layers, or simultaneous measurement of thickness and compositions of layers with up to three components.

1.4 The practical measurement ranges of given coating materials are largely determined by the energy of the characteristic X-ray fluorescence to be analysed and by the acceptable measurement uncertainty and can differ depending upon the instrument system and operating procedure used.

2 Terms and definitions

For the purposes of this International Standard, the following terms and definitions apply.

2.1
X-ray fluorescence
XRF
secondary radiation occurring when a high intensity incident X-ray beam impinges upon a material placed in the path of the incident beam

NOTE The secondary emission has wavelengths and energies characteristic of that material.

2.2
intensity of fluorescent radiation
radiation intensity, x, measured by the instrument, expressed in counts (radiation pulses) per second

2.3
saturation thickness
thickness that, if exceeded, does not produce any detectable change in fluorescent intensity

NOTE Saturation thickness depends upon the energy or wavelength of the fluorescent radiation, density and atomic number of the material and on the angle of incident and fluorescent radiation with respect to the surface of the material.

2.4
normalized intensity
x_n
ratio of the difference in intensity obtained from a coated specimen, x, and an uncoated substrate material, x_0, and the difference obtained from a material of thickness equal to or greater than the saturation thickness, x_s (see 2.3) and an uncoated substrate material, x_0, all measured under the same conditions

NOTE 1 The mathematical relationship is given by:

$$x_n = \frac{x - x_0}{x_s - x_0}$$

where

x is the intensity obtained from the coated specimen;

x_0 is the intensity obtained from uncoated substrate material;

x_s is the intensity obtained from a material of thickness equal to or greater than the saturation thickness.

NOTE 2 The normalized intensity is independent of measurement and integration time, and intensity of the excitation (incident radiation). The geometric configuration and the energy of the excitation radiation can influence the normalized count rate. The value of x_n is valid between 0 and 1.

2.5
intermediate coatings
coatings that lie between the top coating and the basis material and are of thicknesses less than saturation for each of the coatings

NOTE Any coating lying between the top coating and the basis material (substrate) and having a thickness above saturation should itself be considered the true substrate since the material under such a coating will not affect the measurement and can be eliminated for measurement purposes.

2.6
count rate
number of radiation pulses recorded by the instrument per unit time (see 2.2).

2.7
basis material
basis metal
material upon which coatings are deposited or formed

[ISO 2080:1981, definition 134]

2.8
substrate
material upon which a coating is directly deposited

NOTE For a single or first coating the substrate is identical with the **basis material**; for a subsequent coating the intermediate coating is the substrate.

[ISO 2080:1981, definition 630]

3 Principle

3.1 Basis of operation

A relationship exists between mass per unit area of the coating (and thus the linear coating thickness if the density is known) and the secondary radiation intensity. This relationship, for any practical instrument system, is first established by calibrating using calibration standards having coatings of known mass per unit area. If the coating material density is known, such standards can have coatings given in linear thickness units, provided that the actual density value is also given.

NOTE The coating material density is the density as-coated, which may or may not be the theoretical density of the coating material at the time the measurement is made. If this density differs from the density of the calibration standards, a factor that reflects this difference is used and documented in the test report.

The fluorescent intensity is a function of the atomic number of the elements. Providing the top coating, intermediate coating (if present) and the substrate are of different elements or a coating consists of more than one element, these elements will generate radiation characteristics for each of them. A suitable detector system can be adjusted to select either one or more energy bands, enabling the equipment to measure thickness and/or composition of either the top coating or the top and some intermediate coatings simultaneously.

3.2 Excitation

3.2.1 General

The measurement of the thickness of coatings by X-ray spectrometric methods is based on the combined interaction of the coating (or coatings) and substrate with an intense, often narrow, beam of polychromatic or monochromatic X-radiation. This interaction results in generating discrete wavelengths or energies of secondary radiation which are characteristic of the elements composing the coating(s) and substrate.

The generated radiation is obtained from a high voltage X-ray tube generator or from suitable radioisotopes.

3.2.2 Generation by a high voltage X-ray tube

Suitable excitation radiation will be produced by an X-ray tube if sufficient potential is applied to the tube and stable conditions apply. Applied voltages are in the order of 25 kV to 50 kV for most thickness requirements but voltages down to 10 kV may be necessary in order to measure low atomic number coating materials. For some applications the use of a primary filter, located between the X-ray tube and the specimen, decreases the measurement uncertainty.

The chief advantages of this method of excitation are

— the ability to create, by collimation, a very high intensity beam on a very small measurement area;

— the ease of control for personnel safety requirements;

— the potential stability of emission obtainable by modern electronic methods.

3.2.3 Generation by a radioisotope

Only a few radioisotopes emit gamma radiation in the energy band suitable for coating thickness measurement.

Ideally, the excitation radiation is of slightly higher energy (shorter in wavelength) than the desired characteristic X-rays. The advantages of radioisotope generation include the possibility of a more compact construction of the instrument, due mainly to there being no need for cooling. In addition, the radiation, unlike that from high voltage X-ray generators, is essentially monochromatic and there is low background intensity.