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

Reference number
ISO 3497:2000(E)

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ISO 3497:2000
https://standards.iteh.ai/catalog/standards/sist/653c536e-966d-40e2-b6e0-
1cc23505a968/iso-3497-2000

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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.

The major technical disadvantages when compared with the X-ray tube method are

⸺ the much lower intensity that is obtained, which prohibits measurements on small areas;

⸺ the short half-life of some radioisotopes;

⸺ personnel protection problems associated with high intensity radioisotopes (the high voltage X-ray tube can simply be switched off).

3.3 Dispersion

3.3.1 General

The secondary radiation resulting from the exposure of a coated surface to X-ray radiation often contains components additional to those required for the measurement of coating thickness. The desired components are separated by either wavelength or energy dispersion.

3.3.2 Wavelength dispersion

The wavelength characteristic of either coating or substrate is selected using a crystal spectrometer. Typical characteristic emission data for commonly used crystals are available in published form from various national authorities.

3.3.3 Energy dispersion

X-ray quanta are usually specified in terms of wavelength or equivalent energies. The relationship between the wavelength, λ, in nanometres, and energy E, in kiloelectron-volts (keV), is given by

$$\lambda \times E = 1{,}239\ 842\ 7.$$

3.4 Detection

The type of detector used for wavelength dispersive systems can be a gas-filled tube, a solid state detector or scintillation counter connected to a photomultiplier.

The most suitable detector for receiving fluorescent photons and used in energy dispersive systems is selected by the instrument designer according to the application. In the energy band of about 1,5 keV to 100 keV, measurements can be made in normal atmosphere without helium gas or vacuum.

Fluorescent radiation of different characteristic energies passes into the energy dispersive detector and then on to a multi-channel analyser that is adjusted to select the correct energy band.

3.5 Thickness measurement

3.5.1 Emission method

If the intensity of the characteristic radiation from the coating is measured, the intensity increases with increasing thickness up to the saturation thickness. See Figure 1 a).

When the X-ray emission method is used, the equipment is adjusted to receive a selected band of energies characteristic of the coating material. Hence thin coatings produce low intensities and thick coatings produce high intensities.

3.5.2 Absorption method

If the intensity of the characteristic radiation from the substrate is measured, the intensity decreases with increasing thickness. See Figure 1 b).

The X-ray absorption method uses the band of energies characteristic of the substrate material. Hence thin coatings result in high intensities and thick coatings produce low intensities. In practice, care has to be taken to ensure that no intermediate coating is present.

The absorption characteristic is similar to the inverse of the emission characteristic.
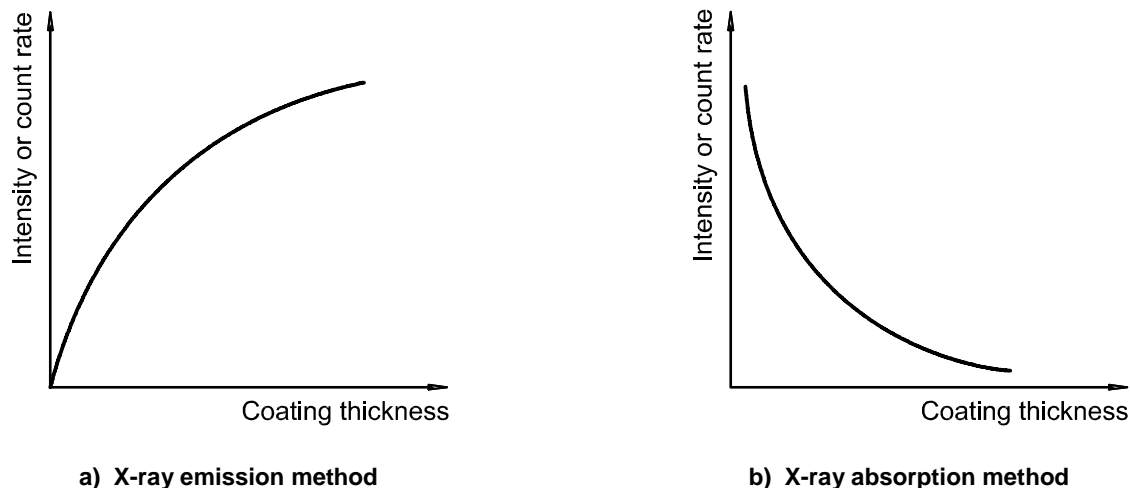

a) X-ray emission method

b) X-ray absorption method

Figure 1 — Schematic illustrations of the relationship between intensity or count rate and coating thickness

3.5.3 Ratio method

It is possible to combine X-ray absorption and emission when coating thicknesses are expressed as a ratio of the respective intensities of substrate and coating materials. Measurements by the ratio method are largely independent of the distance between the test specimen and the detector.

3.5.4 Measurement

For both methods described in 3.5.1 and 3.5.2, the normalized count-rate system is usually used in many commercially available instruments adjusted so that the count-rate characteristic of the uncoated substrate is zero and that of an infinitely thick sample of the coating material is unity. All measurable thicknesses therefore produce count rates that lie within the normalized count-rate range of 0 to 1. See Figure 2.

In all cases, the best or most sensitive range of measurement lies approximately between 0,3 and 0,8 on the normalized count-rate scale. Thus for best measurement accuracy over the whole thickness range, it is advantageous to use calibration standards having count-rate characteristics of 0,3 and 0,8. With some equipment other standards may be necessary in order to ensure precision at other thicknesses. Since the relative uncertainty of calibration of standards increases as thickness decreases, it is essential to establish the correct mathematical relationship for the thin end of the range by suitable use of standards having thicker coatings but lower uncertainties.
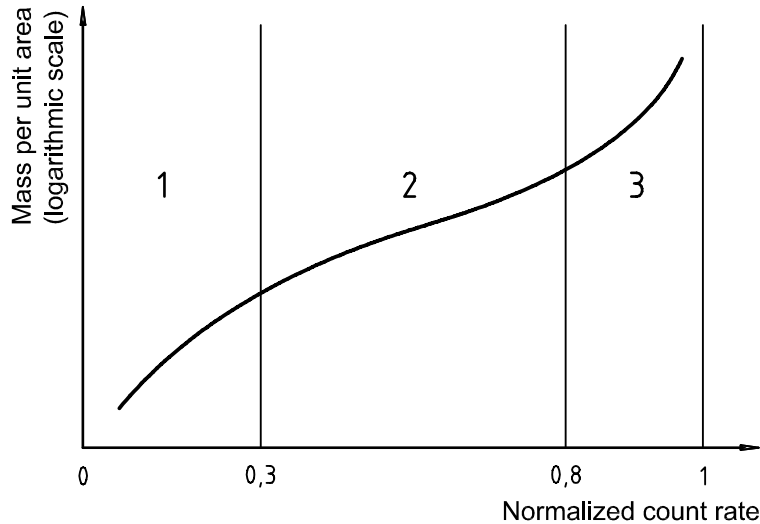
Key

1 Linear range

2 Logarithmic range

3 Hyperbolic range

NOTE   0 = Count rate from saturated (uncoated) substrate material; 1 = count rate from saturated (infinite) coating material.

Figure 2 —Schematic illustration of the relationship between mass per unit area and normalized count rate

3.6   Absorber for secondary radiation

When measuring coating/substrate material combinations that have widely differing energies (energy dispersive systems), the ratio of saturated coating to uncoated substrate count-rate characteristics is very high (10:1 is typical). In such cases, it is not always essential to have calibration standards having a similar or the same substrate (since the substrate material will not radiate in the same energy band as the coating material). When the uncoated substrate/infinite coating count-rate ratio is 3:1 (for coating/substrate combinations having similar energies) it is often helpful to use an "absorber" selected to absorb the radiation of one of the materials, usually that of the substrate material. This absorber is usually placed manually or automatically between the surface being measured and the detector.

3.7   Mathematical deconvolution

When using a multi-channel analyser a mathematical deconvolution of the secondary radiation spectra can be used to extract the intensities of the characteristic radiation. This method can be used when the energies of the detected characteristic radiations do not differ sufficiently, e.g. characteristic radiation from Au and Br. This method is sometimes described as 'numerical filtering' in order to distinguish it from the filtering method (see 3.6).

3.8   Multilayer measurements

It is possible to measure more than one coating layer provided that the characteristic X-ray emissions of the inner layers are not completely absorbed by the outer layers. In an energy dispersive system the multi-channel analyser is set to receive two or more distinct energy bands characteristic of two or more materials.

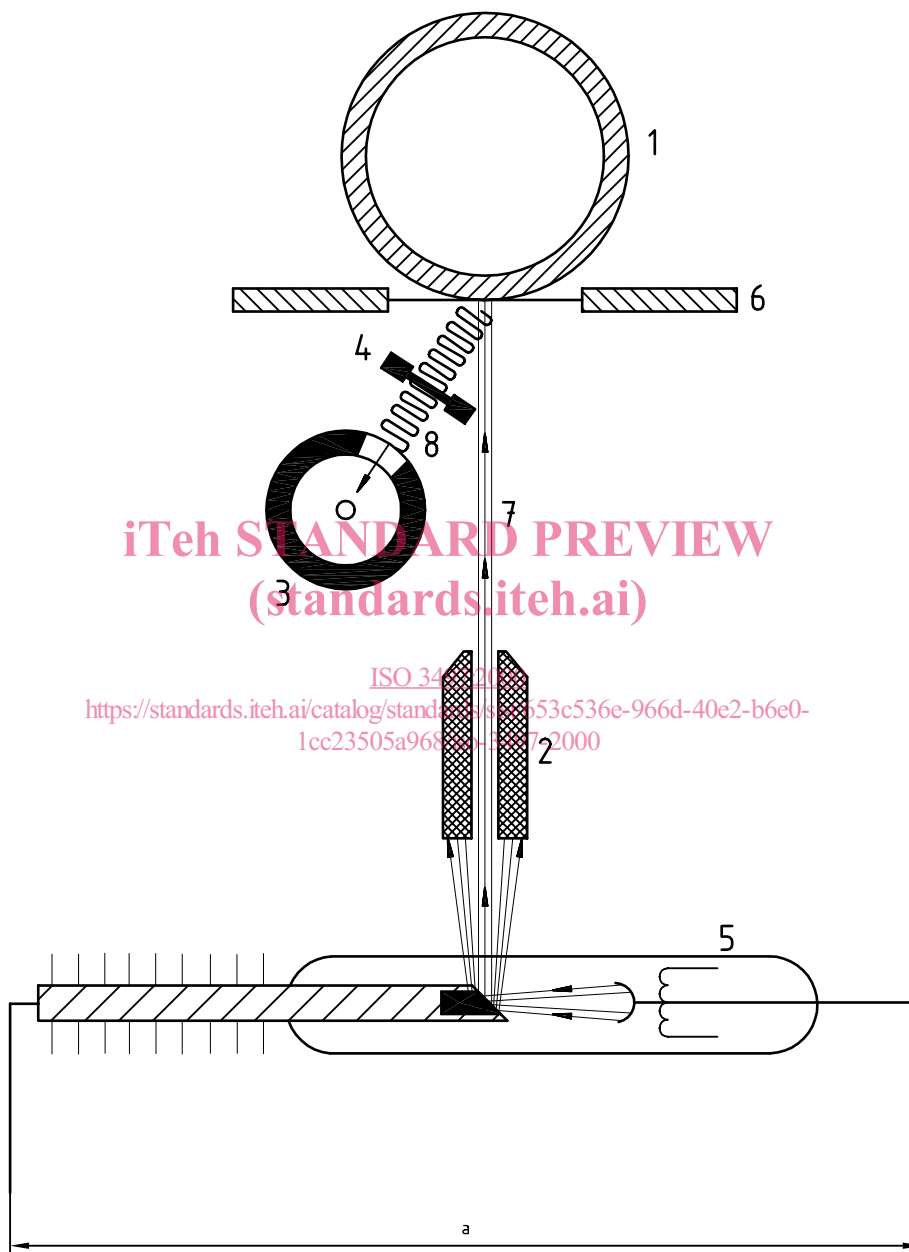## 3.9   Alloy composition thickness measurement

Certain alloys and compounds, for example Sn-Pb, can be measured simultaneously for composition and thickness. In some cases this method can also be used under the conditions described in 3.8, e.g., Au on Pd/Ni on a Cu alloy substrate. Since the thickness measurement of an alloy or compound is dependent upon alloy composition, it is essential either to know or assume the composition before thickness measurement or to be able to measure the composition.

NOTE   Assumed compositions can introduce errors in thickness measurements.

Some coatings can form alloys by interdiffusion with the substrate. The presence of such alloy layers can add to the measurement uncertainty.

4 Apparatus

See Figures 3, 4 and 5.

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Key

1	Test specimen	4	Absorber	7	Incident X-ray beam
2	Collimator	5	X-ray generator	8	Characteristic fluorescent X-ray beam for detection and analysis
3	Detector	6	Specimen support		

a   High voltage

Figure 3 — Schematic representation of an X-ray tube