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

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

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.

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.

International Standard ISO 3497 was prepared by Technical Committee ISO/TC 107, *Metallic and other inorganic coatings*.

This second edition cancels and replaces the first edition (ISO 3497:1976), which has been technically revised.

Annex A forms an integral part of this International Standard.

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

1 Scope

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

These methods permit the simultaneous measurement of some 3-layer systems.

1.2 The measuring methods to which this International Standard applies are fundamentally ones which 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 practical measurement ranges of given coating materials are largely determined by the acceptable measurement uncertainty and may differ depending upon the instrument system and operating procedure used. A table of typical ranges for common materials is given in annex A.

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

2 Definitions

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

2.1 X-ray fluorescence (XRF): The secondary radiation occurring when a high intensity incident X-ray beam impinges upon a material placed in the path of the incident beam. The secondary emission has wavelengths and energies characteristic of that material.

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

2.3 normalized intensity, I_n: The ratio of the difference in intensity obtained from a coated specimen and an uncoated substrate material, and the difference obtained from a material of thickness equal to or greater than the saturation thickness (see 2.4) and an uncoated substrate material, all measured under the same conditions. This is given by

$$\frac{I_e - I_o}{I_s - I_o}$$

where

I_e is the intensity obtained from the coated specimen;

I_o is the intensity obtained from uncoated substrate material;

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

This variable is independent of measurement and integration time, and intensity of the exciting (incident) radiation. The geometric configuration and the energy of the exciting radiation can influence the normalized count rate.

2.4 saturation thickness: The thickness that, if exceeded, will not produce any detectable change in fluorescent intensity.

NOTE 1   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.

2.5 intermediate coating: Those coatings that lie between the top coating and the basis material and

are of thicknesses less than saturation for each of the coatings.

NOTE 2   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 measurement and can be eliminated for measurement purposes.

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

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 calibration 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 3   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.

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 they will generate radiation characteristic of each element. A suitable detector system can be adjusted to select either one or more energy bands, enabling the equipment to measure 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 the generation of discrete wavelengths and 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 exciting 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. The chief advantages of this method of excitation are the ability to create, by collimation, a very high intensity beam onto a very small measurement area, the ease of control for personnel safety requirements and 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 exciting radiation is slightly more energetic (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 a low background intensity.

The major technical disadvantage is the much lower intensity obtainable which prohibits measurements on small areas when compared with the X-ray tube method, the short half-life of the radioisotopes and the personnel protection problems associated with high intensity radioisotopes (the high voltage X-ray tube can be simply 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. Separation of the desired components is done 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 is 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 kilo-electronvolts, is given by

$$\lambda \cdot E = 1,2396$$

3.4 Detection

3.4.1 The type of detector used for wavelength dispersive systems is usually a gas filled tube or scintillation counter interconnected with a photo-multiplier.

3.4.2 The most suitable detector for use in energy dispersive systems to receive the fluorescent photons 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 the different characteristic energies pass into the proportional counter detector tube and then onto a multi-channel analyser which is adjusted to select the correct energy band (or bands).

3.5 Thickness measurement

There are two X-ray methods for measurement of thickness as follows.

a) **Emission method.** If the intensity of the characteristic radiation from the coating is measured, the intensity will increase with increasing thick-ness up to the saturation thickness. See figure 1a).

b) **Absorption method.** If the intensity of the characteristic radiation from the substrate is measured, the intensity will decrease with increasing thickness. See figure 1b).
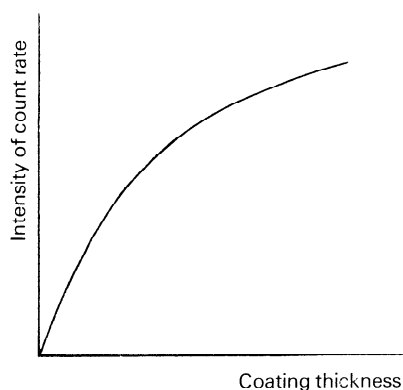
When the X-ray emission method is used, the equipment is adjusted to receive a selected band of energies characteristic of the coating material. Thus thin coatings produce low intensities while thick coatings produce high intensities.

The X-ray absorption method uses the band of energies characteristic of the substrate material. Thus thin coatings result in high intensities while thick coatings produce low intensities. In practice, care has to be taken to ensure that no intermediate coating is present.
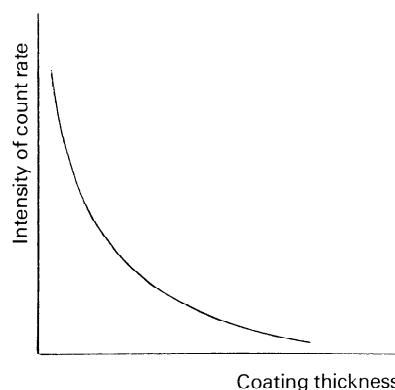
It is possible to combine X-ray absorption and emission when coating thicknesses are expressed as a ratio of the respective intensities of substrate and coating materials. Measurements by this ratio method are largely independent of the distance between test specimen and detector.

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

In all methods, the intensity of secondary radiation is recorded as pulses, usually taken over a preselected fixed time period. The normalized count-rate system is used in many commercially available instruments adjusted so that the count-rate characteristic of the uncoated substrate is zero and that from an infinitely thick sample of the coating material is unity. All measurable thickness therefore produce count rates which lie within the normalized count-rate range of 0 to 1. See figure 2.

a) X-ray emission method b) X-ray absorption method

Figure 1 — Relationship between intensity of count rate and coating thickness
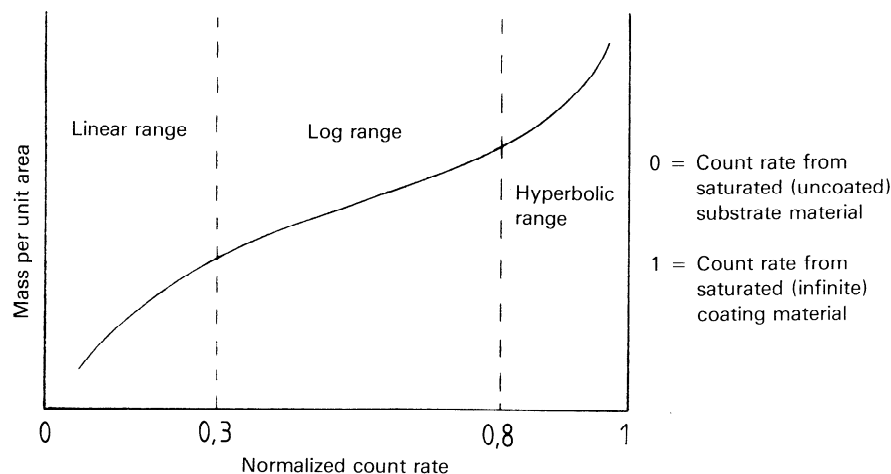
Figure 2 — Relationship between mass per unit area and normalized count rate

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 at 0,3 and 0,8. Other standards may be necessary with some equipment 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 at the thin end of the range by suitable use of standards having thicker coatings but lower uncertainties.

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). Where the uncoated substrate/infinite coating count-rate ratio is 3 : 1 (for coating/substrate combinations having similar energies) it is often necessary 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.

3.6 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.

3.7 Alloy composition thickness measurement

Certain alloys and compounds, for example tin-lead, can be measured simultaneously for composition and thickness. Since the thickness measurement of an alloy or compound is dependent upon alloy composition, it is mandatory either to know or assume the composition before thickness measurement or to be able to measure composition.

NOTE 4   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

Fluorescent X-ray equipment suitable for measuring coating thickness in accordance with this International Standard is available commercially. Equipment designed specifically for coating thickness measurement is of the energy dispersive kind and usually comes with a microprocessor for converting the intensity measurement to mass per unit area or thickness, for storing calibration data, and for computing various statistical measurements. See figure 3.

NOTE 5   The essential components of an X-ray fluorescence coating thickness measuring apparatus include a primary X-ray source collimator, a support for the test specimen, a detector and an evaluating system. The source, collimator and detector are usually in a geometrically fixed relation with each other. If the atomic numbers of the coating and substrate materials are very close, it may be necessary to introduce an absorber which will absorb the characteristic fluorescent energy of one of the materials, for example the substrate.