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

(English Translation)

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1 Scope

Warning. This standard does not cover the issue of personnel protection against

X-ray radiation. Information on this important aspect can be found in current international and national standards and local regulations.

1.1
This standard specifies the method for measuring the thickness of metallic coatings using X-ray spectroscopy.

1.2
The measurement method used in this standard is essentially a method for determining the mass per unit area. If the density of the coating material is known, the measurement result can also be expressed as the linear thickness of the coating layer.

1.3
This measurement method can simultaneously measure a three-layer coating system, or simultaneously measure the thickness and composition of three components.

1.4 The actual measurement range for a given coating material depends primarily on

the energy of the characteristic X-ray fluorescence being analyzed and the permissible measurement uncertainty; it varies depending on the instrumentation and operating procedures used.

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2 Terms and definitions

The following terms and definitions apply to this standard.

2.1

X-ray fluorescence (XRF)

Secondary radiation produced when high-intensity incident X-rays strike a material placed in the path of the incident beam.

Note: This secondary emission has the wavelength and energy characteristics of the material.

2.2

Intensity of fluorescent radiation the material beneath such a coating does not affect the measurement and can be disregarded during measurement.

2.6

Count rate

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

2.7

Basis material

Basis metal

The material on which a coating is deposited or formed [SO 2080.1981, Definition

134].

2.8

Substrate

The material directly deposited by a coating layer [SO 2080.1981, Definition 630].

Note: For a single or first coating layer, the substrate is the same as the base material; for subsequent coating layers, the intermediate coating layer is the substrate.

3.1 Operating mechanism

There is a relationship between the mass per unit area of the coating layer (if the density is known, then it is the linear thickness of the coating layer) and the intensity of secondary radiation. For any practical instrument system, this relationship is first determined by calibration, using a standard block with a known mass per unit area of the coating layer. If the density of the coating layer material is known, meanwhile the actual density is given, then such a standard block can provide the linear thickness of the coating layer.

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Note: The density of the coating layer material is the density under coating conditions, not necessarily the theoretical density of the coating layer material at the time of measurement. If this density differs from the density of the calibration standard, a coefficient reflecting this difference shall be used and noted in the test report.

Fluorescence intensity is a function of the atomic number of the element. If the surface coating layer, intermediate coating layer (if present), substrate are composed of different elements, OR if a coating layer is composed of more than one element, these elements will produce their own radiation characteristics. An appropriate detector system can be adjusted to select one or more energy bands, allowing the device to measure the thickness and composition of both the surface coating layer and some intermediate coating layers simultaneously.

3.2.1 General requirements

X-ray spectroscopic methods for determining coating layer thickness are based on the interaction of a strong, narrow beam of polychromatic or monochromatic X-rays with the substrate and coating layer. This interaction produces secondary radiation with discrete wavelengths and energies, which possess the elemental characteristics of the coating layer and substrate.

Such radiation can be produced by a high-pressure X-ray tube generator or a suitable radioactive isotope.

3.2.2 Generation by a high-voltage X-ray tube

Under stable conditions, if a sufficient potential is applied to the X-ray tube, appropriate excitation radiation can be produced. Most thickness measurements require an applied voltage of approximately 25 kV ~ 50 kV, but for measuring low atomic number coating materials, it may be necessary to reduce the voltage to 10 kV. The measurement uncertainty is reduced by using a primary color filter installed between the X-ray tube and the specimen.

The main advantages of this excitation method are.

- A very strong radiation beam can be generated on a very small measurement surface through collimation;

- Personnel safety requirements are easily guaranteed;

- Sufficiently stable emission can be obtained using modern electronic methods.

3.2.3 Generation by radioactive isotopes

Only a few radioactive isotopes emit gamma rays in an energy band, which is suitable for coating thickness measurement. Ideally, the excitation radiation energy is slightly higher

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(slightly shorter wavelength) than the required characteristic X-ray energy. The advantage of radioactive isotope excitation is a more compact instrument structure, mainly because no cooling is required. Furthermore, unlike high-voltage X-ray tube generators, its radiation is monochromatic and has a low background intensity.

Compared to the X-ray tube method, its main technical disadvantages are.

- The obtained intensity is much lower, making small-area measurements impossible;

- Some radioactive isotopes have short half-lives;

- High-intensity radioactive isotopes pose personnel protection issues (high-voltage

X-ray tubes can be easily shut down).

3.3.1 General requirements

Secondary radiation generated on the surface of the coating layer by X-ray irradiation usually contains components other than those required for coating layer thickness measurement. The desired components can be separated using wavelength dispersion or energy dispersion.

3.3.2 Wavelength dispersion

The wavelength characteristics of the coating layer or substrate can be selected using a crystal spectrometer. Typical characteristic radiation data for commonly used crystals can be found in publications of authoritative institutions in various countries.

3.3.3 Energy dispersion

X-ray quanta are usually expressed in terms of wavelength or equivalent energy. The relationship between wavelength and energy is.

Where.

lambda - Wavelength, in nanometers (nm);

E - Energy, in kiloelectron volts (keV).

3.4 Detection

The detector used in the wavelength dispersion system consists of a gas-filled tube, a solid-state detector, or a scintillation counter connected to a photomultiplier.

The most suitable detector for receiving fluorescence photons in the energy dispersion system is selected by the instrument designer according to the application.

Measurements can be performed in a normal atmosphere within the energy band range of 1.5 keV ~ 100 keV, without the need for helium or a vacuum.

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