GB

NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

ICS 77.040.99

H 21

GB/T 34190-2017

Surface coating weight (thickness) of electrical steel - X-ray spectrometric method

电工钢表面涂层的重量（厚度）

X 射线光谱测试方法

Issued on: September 07, 2017 **Implemented on: June 01, 2018**

Issued by: General Administration of Quality Supervision, Inspection and Quarantine;
Standardization Administration Committee.

Table of Contents

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1 Scope

This Standard specifies the method that uses X-ray spectrometric method to test the surface coating weight (thickness) of electrical steel.

This Method is for measuring the coating weight per unit area of a sample at room temperature. The measurement results can also be expressed by the thickness of the coating.

This Standard is applicable to the electrical steel that contains stable, undisturbed, X-ray fluorescence intensity characteristic elements in surface coating. The measurement range of a given coating material depends primarily on the available characteristic X-ray fluorescence intensity and acceptable measurement uncertainty. And it varies depending on the instrument and measurement method used.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

GB/T 16921, *Metallic coatings - Measurement of coating thickness - X-ray spectrometric methods*

3 Terms and definitions

For the purposes of this document, the terms and definitions defined in GB/T 16921 as well as the followings apply.

3.1 substrate

a material coated with a coating on its surface

3.2 surface coating

material coated on an electrical steel substrate mainly for insulation protection, produce high intensity X-ray fluorescence. This action produces secondary rays that characterize the elements in the coating and substrate. These secondary rays have discrete wavelengths or radioactivity. Generally, primary X-rays are generated by high-pressure X-ray tubes.

5.1.2 Excitation

With sufficient energy and stable environment for the X-ray tube, it shall produce primary X-rays. The application voltage of most thickness measuring equipment is 25kV ~ 50kV. However, low atomic number coating materials may drop to 10kV. In general, a primary filter is added between the X-ray tube and the sample to reduce the potential deviation of the measurement.

The main features of this type of stimulation are as follows:

a) produce parallel, high-energy beams on very small measurement areas;

b) easy to meet personal safety protection requirements;

c) obtain energy-stable scattered rays by electronic technology.

5.2 X-ray dispersion

5.2.1 General

X-ray fluorescence scattered on the surface of the coating usually contains many components in coating thickness measurement. Generally, separate the required ray components by wavelength or energy dispersion.

5.2.2 Wavelength dispersion

Screen characteristic wavelength ray of a coating or substrate with crystal spectrometer.

5.2.3 Energy dispersion

X-rays are usually determined by wavelength or equivalent energy. The relationship between wavelength λ and energy E is as shown in formula (1):

$$\lambda \times E = 1.239\,842\,7 \quad \cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots (1)$$

Where,

λ - X-ray wavelength, in nanometers (nm);

E - X-ray photon energy, in thousand electron volts (keV).

5.3 X-ray detection

The detector for the wavelength dispersion system can be a proportional counter or a scintillation counter.

Detectors for energy dispersive systems that are suitable for receiving fluorescent photons are generally selected by the manufacturer based on the application of the device. In the range of energy from 1.5keV ~ 100keV, it is generally measured under atmospheric conditions. It does not require vacuum or helium environment.

After the fluorescent ray with different energy characteristics enters the energy dispersive detector, the multi-channel analyzer shall detect the characteristic line.

5.4 Coating weight (thickness) measurement

5.4.1 Scattering method

When using the scattering method, select a suitable line from the multiple characteristic lines of the coating element, for example, Cr-Kα line in chromate coating. Detect its fluorescence intensity with a detection system.

In general, a characteristic element refers to an element with a large content in the coating, a large atomic number, and no or negligible in the substrate. The zero point of the coating weight is the intensity of the scattered line produced only when the uncoated sample is measured, known as background. The maximum point of coating weight is the intensity of the scattered lines produced when measuring an "infinite" thick sample. Generally it is 1.5 or 2.0 times the usual measurement range.

In general, the intensity of scattered X-ray fluorescence depends mainly on the excitation energy, the atomic number of the characteristic elements of the coating, the irradiation area of the primary ray of the sample, and the coating weight per unit area. If other influencing factors are constant, the X-ray fluorescence intensity is a function of the weight per unit area or thickness of the coating.

When measuring the characteristic ray intensity of an electrical steel coating, the thickness of the coating shall increase to a saturation thickness. The intensity of the fluorescent ray is continuously increased, as shown in Figure 1a).

5.4.2 Absorption method

When using the absorption method, select a suitable line from a plurality of characteristic lines of the substrate element. Detect its fluorescence intensity with a detection system. X-ray fluorescence intensity reaches a maximum when the substrate is uncoated. As the weight of the coating increases, the intensity selected as much as possible.

Since the coating may contain multiple dopants, other alloying elements are present in the substrate. It is easy to produce interference spectrum lines that are close to or the same as the characteristic line, which shall be eliminated as much as possible in the measurement.

Since the actual coating density may differ from the standard sample density, the measured value shall generally be multiplied by the density correction factor as shown in formula (2).

$$C = C_m \times \frac{\rho_s}{\rho_c} \qquad \cdots\cdots (2)$$

Where,

C - coating weight per unit area, in grams per square meter or micron (g/m^2 or μm);

C_m - actual measured coating weight per unit area, in grams per square meter or micron (g/m^2 or μm);

ρ_s - coating density of standard sample, in kilograms per cubic meter (kg/m^3);

ρ_c - coating density of sample to be tested, in kilograms per cubic meter (kg/m^3).

Oil stains, impurities and other contaminants appear on the surface of the coating, which may cause deviations in measurement results. Therefore, the surface treatment shall be performed for the sample to be tested.

If the surface of the sample is curved, a collimator or a bunching aperture shall be used to reduce the effect of the bending curvature.

In addition, it is necessary to ensure the stability of the device. For example, change the intensity of the primary beam by the X-ray tube current.

8 Measuring process

8.1 Check the stability of the test device before measurement. And pay attention to the influencing factors in Clause 6.

8.2 Define the measurement area and measurement speed of a single measurement point as needed. Generally, when measuring online, the measurement area of a single measurement point shall be no less than 1000 mm^2; the horizontal scanning speed shall be less than 25 mm/s; the test time of a single measuring point is not less than 4s. For off-line measurement, the measurement area of a single measurement point shall be no less than 500

Annex A

(informative)

Production of coating weight standard sample

The measurement of coating weight through standard analysis curve requires a complete set of standard samples with gradients. Standard sample uniformity, set value deviation, etc. shall lead to fluctuations in measurement results.

A.1 Preparation of standard sample

Firstly, according to the selected measurement method, determine the characteristic elements of the measurement. The scattering method generally selects elements with a large content in the surface coating, a large atomic number, and no or negligible in the substrate. The absorption method generally selects elements with a large content in the substrate, a large atomic number, and no or negligible in the coating.

Produce 200mm×200mm (size can be customized according to equipment requirements) electrical steel substrate. Firstly, perform pre-treatment for steel plate to make the surface of the substrate clean and free of oil. The treatment method and treatment time are adjusted and controlled according to the actual situation to prevent excessive corrosion of the steel plate, causing pores, diffuse reflection, etc. on the surface of the steel plate. Then according to the actual condition of the coating liquid and the production process, the coating liquid of different thickness is uniformly coated on the steel substrate and is solidified. Record the actual coating amount. Convert to the coating weigh per unit area.

NOTE: The actual coating amount can be determined by the difference in sample weight before and after coating, with a ten thousandth balance. It can also be converted according to the relationship between the surface coating density and the coating liquid density. Determine the surface coating weight. If there is a certain conversion relationship between the surface coating density and the coating liquid density, the conversion factor can also be directly added.

Process uniformity "evaluation sample" around the prepared sample area of the standard sample to be determined. The number of samples can be determined by the actual situation, at least 4 blocks, as shown in Figure A.1. Repeat 11 measurements of the characteristic element ray intensity of the coating or substrate in the "evaluation sample". If the ray intensity measurement deviation of all "evaluation samples" is within 3%, the intermediate area of the sample can be used as a standard sample.

------ The End ------