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Metallic and non-metallic coatings — Measurement of thickness — Beta backscatter method

Revêtements métalliques et non métalliques — Mesurage de l'épaisseur — Méthode par rétrodiffusion des rayons beta

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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.

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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 3543 was prepared by Technical Committee ISO/TC 107, *Metallic and other inorganic coatings*, Subcommittee SC 2, *Methods of inspection and coordination of test methods*.

This second edition cancels and replaces the first edition (ISO 3543:1981), which has been technically revised.

Annex A of this International Standard is for information only.

Metallic and non-metallic coatings — Measurement of thickness — Beta backscatter method

1 Scope

WARNING Beta backscatter instruments used for the measurement of coating thicknesses use a number of different radioactive sources. Although the activities of these sources are normally very low, they can present a hazard to health, if incorrectly handled. Therefore, reference should be made to current international and national standards, where these exist.

This International Standard specifies a method for the non-destructive measurement of coating thicknesses using beta backscatter gauges. It applies to both metallic and non-metallic coatings on both metallic and non-metallic substrates. To make use of this method, the atomic numbers or equivalent atomic numbers of the coating and the substrate need to differ by an appropriate amount.

NOTE Since the introduction of the X-ray fluorescence method (ISO 3497), the beta backscatter method has been used less and less for the measurement of coating thickness. However, because of its lower cost, it is still a very useful method of measurement for many applications. In addition it has a wider measuring range.

2 Terms and definitions

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

2.1
radioactive decay
spontaneous nuclear transformation in which particles or gamma radiation are emitted or X-radiation is emitted following orbital electron capture, or the nucleus undergoes spontaneous fission

[ISO 921:1997, definition 972]

2.2
beta particle
electron or positron which has been emitted by an atomic nucleus or neutron in a nuclear transformation

[ISO 921:1997, definition 81]

2.3
beta-emitting isotope
beta-emitting source
beta emitter
material, the nuclei of which emit beta particles

NOTE 1 It is possible to classify beta emitters by the maximum energy level of the particles that they release during their disintegration.

NOTE 2 Table A.1 lists some isotopes used with beta backscatter gauges.

2.4

electron-volt

unit of energy equal to the change in energy of an electron in passing through a potential difference of 1 V

NOTE 1 $1\ \text{eV} = 1{,}602\ 19 \times 10^{-19}\ \text{J}$

[ISO 921:1997, definition 393]

NOTE 2 Since the electron-volt is too small for the energies encountered with beta particles, the mega-electron-volt (MeV) is commonly used.

2.5

activity

disintegration rate

number of spontaneous nuclear disintegrations occurring in a given quantity of material during a suitably small interval of time divided by that interval of time

[ISO 921:1997, definition 23]

NOTE 1 In beta backscatter measurements a higher activity corresponds to a greater emission of beta particles.

NOTE 2 The SI unit of activity is the becquerel (Bq). The activity of a radioactive element used in beta backscatter gauges is generally expressed in microcuries (µCi) (1 µCi = $3{,}7 \times 10^4$ Bq, which represents $3{,}7 \times 10^4$ disintegrations per second).

2.6

radioactive half-life

time required for the activity to decrease to half its value by a single radioactive decay process

[ISO 921:1997, definition 975]

2.7

scattering

process in which a change in direction or energy of an incident particle or incident radiation is caused by a collision with a particle or a system of particles

[ISO 921:1997, definition 1085]

2.8

backscatter

scattering as a result of which a particle leaves a body of matter from the same surface at which it entered

NOTE Radiations other than beta rays are emitted or backscattered by a coating and substrate and some of these can be included in the backscatter measurement. In this International Standard the term “backscatter” is used to mean the total radiation measured.

2.9

backscatter coefficient (of a body)

R

ratio of the number of particles backscattered to that entering the body

NOTE The value of R is independent of the activity of the isotope and of the measuring time.

2.10
backscatter count

2.10.1
absolute backscatter count

X

number of particles backscattered during a fixed interval of time, and received by a detector

NOTE X depends on the activity of the isotope, the measuring time, the geometric configuration of the measuring system and the properties of the detector. The count produced by the uncoated substrate is generally designated by X_o, and that of the coating material by X_s. To obtain these values, both these materials have to be available with a thickness greater than the saturation thickness (see 2.13).

2.10.2
normalized backscatter count

X_n

quantity that is independent of the activity of the isotope, the measuring time and the properties of the detector and defined by the equation:

$$X_\text{n} = \frac{X - X_\text{o}}{X_\text{s} - X_\text{o}}$$

where

X_o is the absolute backscatter count of the saturation thickness of the substrate material;

X_s is the absolute backscatter count of the saturation thickness of the coating material;

X is the absolute backscatter count of the coated specimen;

each of these counts being taken over the same interval of time

NOTE 1 The value of X_n is valid between 0 and 1.

NOTE 2 For simplicity, it is often advantageous to express the normalized backscatter count as a percentage by multiplying X_n by 100.

2.11
normalized backscatter curve

curve obtained by plotting the coating thickness as a function of X_n

2.12
equivalent (apparent) atomic number

for a material, which can be an alloy or a compound, the atomic number of an element that has the same backscatter coefficient R as the material

2.13
saturation thickness

minimum thickness of a material that, if exceeded, does not produce a change in backscatter

NOTE Figure A.1 shows saturation thickness, s, plotted as a function of density for different isotopes.

2.14
sealed source
radioactive source sealed in a container or having a bonded cover, the container or cover being strong enough to prevent contact with and dispersion of the radioactive material under the conditions of use and wear for which it was designed

[ISO 921:1997, definition 1094]

NOTE Also referred to as "sealed isotope".

2.15
aperture
opening of the mask abutting the test specimen and that determines the size of the area on which the coating thickness is to be measured

NOTE This mask is also often referred to as a platen, an aperture platen or a specimen support.

2.16
source geometry
spatial arrangement of the source, the aperture and the detector, with respect to each other

2.17
dead time
time period during which a Geiger-Müller detector is unresponsive to the receipt of further beta particles

2.18
resolving time
recovery time of the Geiger-Müller detector tube and associated electronic equipment during which the counting circuit is unresponsive to further pulses

2.19
basis material
basis metal
material upon which coatings are deposited or formed

[ISO 2080:1981, definition 134]

2.20
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

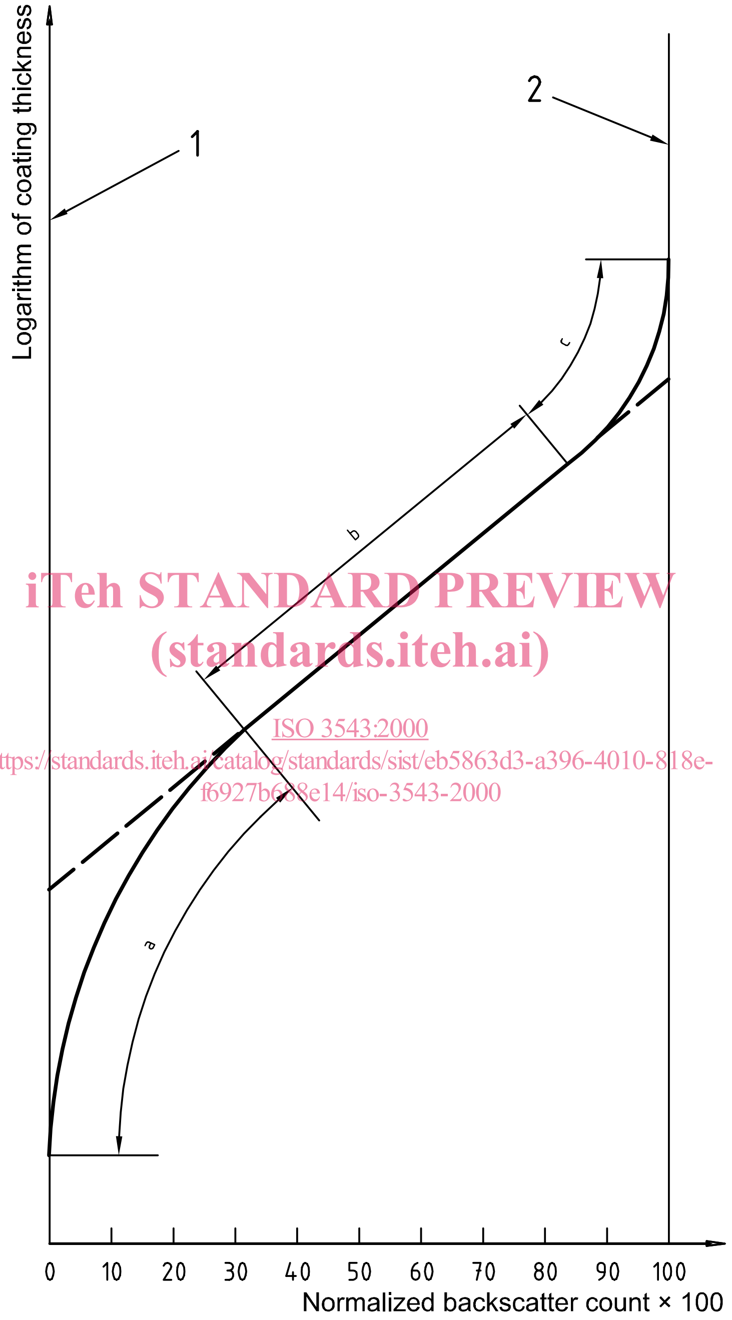
[ISO 2080:1981, definition 630]

3 Principle

When beta particles impinge upon a material, a certain portion of particles is backscattered. This backscatter is essentially a function of the atomic number of the material.

If the body has a surface coating, and if the atomic numbers of the substrate and of the coating material are sufficiently different, the intensity of the backscatter will be between two limits: the backscatter intensity of the substrate and that of the coating. Thus, with proper instrumentation and, if suitably displayed, the intensity of the backscatter can be used for the measurement of mass per unit area of the coating, which, provided that it is of uniform density, is directly proportional to the thickness, i.e., to the mean thickness within the measuring area.

The curve expressing coating thickness versus beta backscatter intensity is continuous and can be subdivided into three distinct regions as shown in Figure 1, on which the normalized count, X_n, is plotted on the x-axis, and the logarithm of the coating thickness on the y-axis. In the range $0 \leqslant X_n \leqslant 0{,}3$ the curve is essentially linear. In the range $0{,}3 \leqslant X_n \leqslant 0{,}8$ the curve is nearly logarithmic; this means that, when drawn on semi-logarithmic graph paper, as in Figure 1, the curve approximates a straight line. In the range $0{,}8 \leqslant X_n \leqslant 1$ the curve is nearly hyperbolic.



Key

1 Substrate with saturation thickness

2 Coating with saturation thickness

a Approximately linear

b Approximately logarithmic

c Approximately hyperbolic

Figure 1 — Typical normalized backscatter curve

4 Apparatus

4.1 Beta backscatter gauge, comprising:

a) a radiation source (isotope) emitting mainly beta particles having an energy appropriate to the coating thickness to be measured;

b) a probe or measuring system with a range of apertures that limit the beta particles to the area of the test specimen on which the coating thickness is to be measured, and containing a detector capable of counting the number of backscattered particles, for example a Geiger-Müller counter (or tube);

c) a readout instrument where the intensity of the backscatter is displayed;

d) a readout instrument display, which can be in the form of a meter reading or a digital readout, either proportional to the absolute count or to the absolute normalized count or to the coating thickness expressed either in thickness units or in mass per unit area.

5 Factors relating to measurement uncertainty

5.1 Counting statistics

Radioactive decay takes place in a random manner. This means that, during a fixed time interval, the number of beta particles backscattered will not always be the same. This gives rise to statistical errors inherent in radiation counting. In consequence, an estimate of the counting rate based on a short counting interval (for example, 5 s) can be appreciably different from an estimate based on a longer counting period, particularly if the counting rate is low. To reduce the statistical error to an acceptable level, the counting interval has to be long enough to accumulate a sufficient number of counts.

For counts normally made, the standard deviation, σ, will closely approximate the square root of the absolute count, that is $\sigma = \sqrt{X}$; in 95 % of all cases, the true count will be within $X \pm 2\,\sigma$. To judge the significance of the precision, it is often helpful to express the standard deviation as a percentage of the count, that is $100\sqrt{X}/X$, or $100/\sqrt{X}$. Thus, a count of 100 000 will give a value 10 times more precise than that obtained with a count of 1 000. Whenever possible, a counting interval shall be chosen that will provide a total count of at least 10 000, which would correspond to a standard deviation of 1 % arising from the random nature of radioactive decay.

Direct-reading instruments are also subject to these statistical random errors. However, if these instruments do not permit the display of the actual count rate, one way to determine the measuring precision is to make a large number of repetitive measurements at the same location on the same coated specimen, and to calculate the standard deviation by conventional means.

NOTE The precision of a thickness measurement by beta backscatter is always less than the precision described in 5.1, as it also depends on the other factors described in 5.2 to 5.17.

5.2 Coating and substrate materials

As the backscatter intensity of a measurement depends on the atomic numbers of the substrate and of the coating, the uncertainty of the measurement will depend to a large extent on the difference between these atomic numbers; thus, with the same measuring parameters, the greater this difference, the more accurate the measurement will be.

As a guide, for most applications, the difference in atomic numbers should be at least 5. For materials with atomic numbers below 20, this difference may be reduced to 25 % of the higher atomic number; for materials with atomic numbers higher than 50, this difference should be at least 10 % of the higher atomic number. Most unfilled plastics and related organic materials (for example photoresists) may be assumed to have an equivalent atomic number close to 6.

NOTE Table A.2 gives the atomic numbers of some typical coatings and substrate materials.

5.3 Aperture

Despite the collimated nature of the sources used in commercial backscatter gauges, the backscatter recorded by the detector is nearly always the sum of the backscatter produced by the test specimen exposed through the aperture and that of the specimen support. It is therefore, advantageous to use for the platen construction a material with a low atomic number, and to select the largest aperture possible. However, measuring errors will still occur if the edges of the aperture opening are worn or damaged, or if the test specimen does not properly contact these edges.

Because the measuring area on the test specimen has to be constant to prevent the introduction of another variable, namely the dimensions of the test specimen, the aperture shall be smaller than the area of the surface on which the measurement is made.

5.4 Coating thickness

5.4.1 In the logarithmic range, the “relative measuring error” is nearly constant, and has its smallest value.

5.4.2 In the linear range, the “absolute measuring error”, expressed in mass per unit area or thickness, is nearly constant, which means that as the coating thickness decreases, the relative measuring error increases. At, or near, X_n = 0,3, the relative errors of the linear and logarithmic ranges are about the same. This means that the relative error at this point can, for all practical purposes, be used to calculate the absolute error over the entire linear range.

5.4.3 In the hyperbolic range, the measuring error is always large, because a small variation in the intensity of the beta backscatter will produce a large variation in the measured value of coating thickness.

5.5 Resolving time of the detector

Because of the dead time (see 2.17) of the Geiger-Müller tube, the count indicated by the readout instrument is always less than the actual number of backscattered beta particles that would otherwise be counted. This does not diminish the measuring accuracy, unless the count rate is excessively high.

5.6 Source geometry

The greatest measuring accuracy is obtained with the source placed in a particular position with respect to the test specimen. This position depends on the collimation of the beam of beta particles from the source, and the location, form and size of the aperture. If possible, most of the backscattered radiation should be from the test specimen, and not from the platen. In general, the measuring uncertainty is reduced to a minimum when the isotope is mounted on the aperture platen, where it can be adjusted to an optimum position. The source shall be mounted in accordance with the manufacturer’s instructions.

5.7 Curvature

This test method is sensitive to the curvature of the test specimen. However, the normalized backscatter curve remains the same if the surface of the test specimen does not protrude into the aperture of the platen by more than 50 µm or if the calibration is made using standards with the same curvature as the test specimen. By using specially selected aperture platens or masks, where the isotope is pre-mounted in a fixed optimum position, it is possible to obtain nearly identical readings on flat and curved specimens. This permits the use of flat calibration standards for the measurement of curved specimens.

In most cases, the relationship between maximum aperture size and specimen surface curvature is specific to the individual instrument design. These details shall, therefore, be obtained from manufacturer’s data.