Designation: B 567 – 98

Standard Test Method for Measurement of Coating Thickness by the Beta Backscatter Method[1]

This standard is issued under the fixed designation B 567; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This test method covers the beta backscatter gages for the nondestructive measurement of metallic and nonmetallic coatings on both metallic and nonmetallic substrate materials.

1.2 The test method measures the mass of coating per unit area, which can also be expressed in linear thickness units provided that the density of the coating is known.

1.3 The test method is applicable only if the atomic numbers or equivalent atomic numbers of the coating and substrate differ by an appropriate amount (see 7.2).

1.4 Beta backscatter instruments employ a number of different radioactive isotopes. Although the activities of these isotopes are normally very low, they can present a hazard if handled incorrectly. This standard does not purport to address the safety issues and the proper handling of radioactive materials. It is the responsibility of the user to comply with applicable State and Federal regulations concerning the handling and use of radioactive material. Some States require licensing and registration of the radioactive isotopes.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *International standard:*

ISO 3543: Metallic and Nonmetallic Coatings—Measurement of Thickness—Beta Backscatter Method

3. Terminology

3.1 *Descriptions of Terms:*

3.1.1 *activity*—the nuclei of all radioisotopes are unstable and tend to change into a stable condition by spontaneously emitting energy or particles, or both. This process is known as radioactive decay. The total number of disintegrations during a suitably small interval of time divided by that interval of time is called "activity." Therefore, in beta backscatter measurements, a higher activity corresponds to a greater emission of beta particles. The activity of a radioactive element used in beta backscatter gages is generally expressed in microcuries (1 μCi = 3.7×10^4 disintegrations per second).

3.1.2 *aperture*—the opening of the mask abutting the test specimen. It determines the size of the area on which the coating thickness is measured. This mask is also referred to as a platen, an aperture plate, a specimen support, or a specimen mask.

3.1.3 *backscatter*—when beta particles pass through matter, they collide with atoms. Among other things, this interaction will change their direction and reduce their speed. If the deflections are such that the beta particle leaves the body of matter from the same surface at which it entered, the beta particle is said to be backscattered.

3.1.4 *backscatter coefficient*—the backscatter coefficient of a body, *R*, is the ratio of the number of beta particles backscattered to that entering the body. *R* is independent of the activity of the isotope and of the measuring time.

3.1.5 *backscatter count:*

3.1.5.1 *absolute backscatter count*—the absolute backscatter count, *X*, is the number of beta particles that are backscattered during a finite interval of time and displayed by the instrument. *X* will, therefore, depend on the activity of the source, the measuring time, the geometric configuration of the measuring system, and the properties of the detector, as well as the coating thickness and the atomic numbers of the coating and substrate materials. X_0 is the count produced by the uncoated substrate, and *Xs*, that of the coating material. To obtain these values, it is necessary that both these materials are available with a thickness greater than the saturation thickness (see 3.1.12).

3.1.5.2 *normalized backscatter*—the normalized backscatter, x_n, is a quantity that is independent of the activity of the source, the measuring time, and the properties of the detector. The normalized backscatter is defined by the equation:

$$x_n = \frac{X - X_0}{X_s - X_0}$$

where:

X_0 = count from the substrate,

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X_s = count from the coating material, and
X = count from the coated specimen, and each count is for the same interval of time.

Because X is always $\geq X_0$ and $\leq X_s$, x_n can only take values between 0 and 1. (For reasons of simplicity, it is often advantageous to express the normalized count as a percentage by multiplying x_n by 100.)

3.1.5.3 *normalized backscatter curve*—the curve obtained by plotting the coating thickness as a function of x_n.

3.1.6 *beta particles*—beta particles or beta rays are high-speed electrons that are emitted from the nuclei of materials undergoing a nuclear transformation. These materials are called beta-emitting isotopes, beta-emitting sources, or beta emitters.

3.1.7 *coating thickness*—in this test method, coating thickness refers to mass per unit area as well as geometrical thickness.

3.1.8 *dead time or resolving time*—Geiger-Müller tubes used for counting beta particles have characteristic recovery times that depend on their construction and the count rate. After reading a pulse, the counter is unresponsive to successive pulses until a time interval equal to or greater than its dead time has elapsed.

3.1.9 *energy*—it is possible to classify beta emitters by the maximum energy of the particles that they release during their disintegration. This energy is generally given in megaelectronvolts, MeV.

3.1.10 *equivalent (or apparent) atomic number*— the equivalent atomic number of an alloy or compound is the atomic number of an element that has the same backscatter coefficient as the material.

3.1.11 *half-life, radioactive*—for a single radioactive decay process, the time required for the activity to decrease by half.

3.1.12 *saturation thickness*—the minimum thickness of a material that produces a backscatter that is not changed when the thickness is increased. (See also Appendix X1.)

3.1.13 *sealed source or isotope*—a 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.

3.1.14 *source geometry*—the spatial arrangement of the source, the aperture, and the detector with respect to each other.

4. Summary of Test Method

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

4.2 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, if the density remains the same, is directly proportional to the thickness.

4.3 The curve expressing coating thickness (mass per unit area) versus beta backscatter intensity is continuous and can be subdivided into three distinct regions, as shown in Fig. 1. The normalized count rate, x_n, is plotted on the X-axis, and the logarithm of the coating thickness, on the Y-axis. In the range $0 \leq x_n \leq 0.35$, the relationship is essentially linear. In the range $0.35 \leq x_n \leq 0.85$, the curve is nearly logarithmic; this means that, when drawn on semilogarithmic graph paper, as in Fig. 1, the curve approximates a straight line. In the range $0.85 \leq x_n \leq 1$, the relationship is nearly hyperbolic.

4.4 Radiation other than the beta rays are emitted or backscattered by the coating or substrate, and may be included in the backscatter measurements. Whenever the term backscatter is used in this method, it is to be assumed that reference is made to the total radiation measured.

5. Significance and Use

5.1 The thickness or mass per unit area of a coating is often

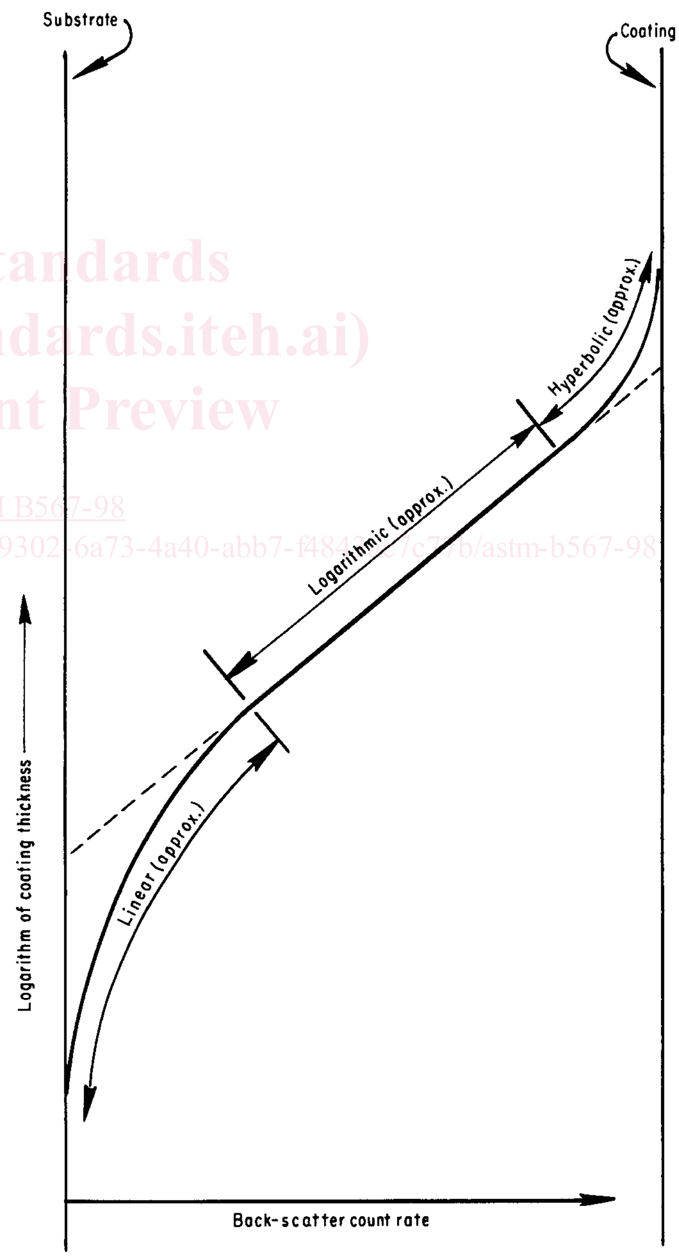

FIG. 1 Normalized Backscatter

critical to its performance.

5.2 For some coating-substrate combinations, the beta backscatter method is a reliable method for measuring the coating nondestructively.

5.3 The test method is suitable for thickness specification acceptance if the mass per unit area is specified. It is not suitable for specification acceptance if the coating thickness is specified and the density of the coating material can vary or is not known.

6. Instrumentation

6.1 In general, a beta backscatter instrument will comprise: (*1*) a radiation source (isotope) emitting primarily beta particles having energies appropriate to the coating thickness to be measured (see Appendix X2), (*2*) 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)), and (*3*) a readout instrument where the intensity of the backscatter is displayed. The display, in the form of a meter reading or a digital readout can be: (*a*) proportional to the count, (*b*) the normalized count, or (*c*) the coating thickness expressed either in thickness or mass per unit area units.

7. Factors Affecting the Measuring Accuracy

7.1 *Counting Statistics:*

7.1.1 Radioactive disintegration takes place randomly. Thus, during a fixed time interval, the number of beta particles backscattered will not always be the same. This gives rise to statistical errors inherent to radiation counting. In consequence, an estimate of the counting rate based on a short counting interval (for example, 5 s) may be appreciably different from an estimate based on a longer counting interval, particularly if the counting rate is low. To reduce the statistical error to an acceptable level, it is necessary to use a counting interval long enough to accumulate a sufficient number of counts.

7.1.2 At large total counts, the standard deviation (σ) will closely approximate the square root of the total 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 ten times more precise than that obtained with a count of 1000. Whenever possible, a counting interval should be chosen that will provide a total count of at least 10 000, which corresponds to a statistical error of 1 % for the count rate. It should be noted, however, that a 1 % error in the count rate can correspond to a much larger percentage error in the thickness measurement, the relative error depending on the atomic number spread or ratio between coating and substrate materials.

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

NOTE 1—The accuracy of a thickness measurement by beta backscatter is generally poorer than the precision described in 6.1, inasmuch as it also depends on other factors that are described below. Methods to determine the random errors of thickness measurements before an actual measurement are available from some manufacturers.

7.2 *Coating and Substrate Materials*—Because the backscatter intensity depends on the atomic numbers of the substrate and the coating, the repeatability of the measurement will depend to a large degree on the difference between these atomic numbers; thus, with the same measuring parameters, the greater this difference, the more precise the measurement will be. As a rule of thumb, for most applications, the difference in atomic numbers should be at least 5. For materials with atomic numbers below 20, the difference may be reduced to 25 % of the higher atomic number; for materials with atomic numbers above 50, the difference should be at least 10 % of the higher atomic number. Most plastics and related organic materials (for example, photoresists) may be assumed to have an equivalent atomic number close to 6. (Appendix X3 gives atomic numbers of commonly used coating and substrate materials.)

7.3 *Aperture:*

7.3.1 Despite the collimated nature of the sources used in commercial backscatter instruments, 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 aperture plate(n). It is, therefore, desirable to use a material with a low atomic number for the construction of the platen and to select the largest aperture possible. Measuring errors will be increased if the edges of the aperture opening are worn or damaged, or if the test specimen does not properly contact these edges.

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

7.4 *Coating Thickness:*

7.4.1 In the logarithmic range, the *relative measuring error* is nearly constant and has its smallest value.

7.4.2 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 $x_n = 0.35$, the relative errors of the linear and logarithmic ranges are about the same. Thus, the relative error at this point may, for most practical purposes, be used to calculate the absolute error over the linear range.

7.4.3 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 coating thickness.

7.4.4 For instruments that indicate only backscatter count rate and not thickness directly, the count rate is normally converted to a thickness by means of an appropriate graph. Such graphs are generally valid only within a specific range of coating thicknesses so that extrapolation of a linear range calibration curve (straight line on rectangular coordinates) into the logarithmic thickness range will result in measurement errors. Similarly, extrapolation of a logarithmic range calibration into the linear thickness range will also produce significant errors. Many instruments that indicate coating thickness directly are limited to the combined linear and logarithmic coating thickness ranges but will be in error if measurements are attempted in the hyperbolic thickness range. The instrument manufacturer's instructions must be followed relative to the limiting coating thicknesses beyond which the particular instrument being used may give substantial errors.

7.5 *Resolving Time of the Detector*—Because of the dead time of Geiger-Müller tubes (see 3.1.8), the number of pulses displayed by the readout instrument is always less than the actual number of backscattered beta particles. Normally, this does not diminish the measuring accuracy significantly unless the count rate is so high as to saturate the detector.

7.6 *Source Geometry*— The greatest measurement precision 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 beta particles emitted by the source should be backscattered from the test specimen, and not from the aperture plate(n). The instructions furnished by the manufacturer of the instrument for mounting the source shall be followed exactly.

7.7 *Curvature*—This test method is sensitive to the curvature of the test specimen. However, the normalized backscatter curve remains nearly the same if the surface of the test specimen does not protrude into the aperture of the platen by more than about 50 µm. By the use of specially selected aperture platens or masks where the isotope is premounted in a fixed, optimum position, it is possible to obtain nearly identical readings on both flat and curved specimens. This permits the use of flat calibration standards for the measurement of curved specimens. The relationship between maximum aperture size and specimen surface curvature is peculiar, in most cases, to the individual instrument design. These details are therefore best obtained from the manufacturer's data.

7.8 *Substrate Thickness:*

7.8.1 *Test Specimens with Single-Layer Coatings:*

7.8.1.1 This test method is sensitive to the thickness of thin substrates, but for each isotope and material there is a critical thickness, called "saturation thickness," beyond which the measurement will no longer be affected by an increase of the substrate thickness. This thickness depends on the energy of the isotope and on the density of material. If the saturation thickness is not supplied by the manufacturer, it should be determined experimentally.

7.8.1.2 If the substrate thickness is less than the saturation thickness, effective saturation thickness can sometimes be obtained by backing up the substrate with more of the same material, but only if the substrate is not coated on both sides. If the substrate is of constant thickness, the instrument may be calibrated for that thickness of substrate. However, if the substrate thickness is less than the saturation thickness and also varies in thickness, this method will not yield a single value for the coating thickness, but a range of values with an upper and lower limit.

7.8.2 *Test Specimens with Multiple-Layer Coatings:*

7.8.2.1 If the intermediate layer adjacent to the coating is thicker than the saturation thickness, this test method will not be affected by any variations in the substrate thickness as long as the instrument is calibrated with standards having the intermediate coating material as the basis material.

7.8.2.2 If the thickness of the intermediate layer is less than saturation thickness, but constant in thickness, the instrument may be calibrated for that particular combination of materials. However, if the thickness of this intermediate layer is less than saturation thickness and varies in thickness, this method will not yield a single value for the coating thickness, but a range of values with an upper and lower limit.

7.9 *Surface Cleanliness*—Foreign material, such as dirt, grease, and corrosion products, will produce erroneous readings. Natural oxide coatings, which form on some metal coatings, also tend to produce low readings, especially if the measurement requires the use of an isotope having an energy of less than 0.25 MeV.

7.10 *Substrate Material*—To obtain accurate thickness readings, it is necessary that the backscatter produced by the substrate materials of the test specimen and that of the calibration standard be the same. If they are different, other calibration standards will have to be used, or appropriate corrections made. Beta backscatter instruments are available that can automatically make these corrections.

7.11 *Density of Coating Material*—The beta backscatter method is basically a method of comparing the mass per unit area of the coating of the test specimen to that of the calibration standard. If the instrument readout is in units of mass per unit area, the linear thickness is obtained by dividing by the coating density:

$$T = \frac{M \times 10}{D}$$

If the instrument readout is in linear units and if there is a difference between the coating densities of the calibration standards and of the test specimens, a density correction must be applied:

$$T = \frac{T^* \times D^*}{D}$$

where:

T = linear thickness of coating of test specimen, µm,
T^* = linear thickness readout of instrument, µm,
D = density of coating of test specimen, g/cm^3,
D^* = density of coating of calibration standard, g/cm^3, and
M = mass per unit area of coating of test specimen, mg/cm^2.

In addition to porosity, voids, and inclusions, codeposited materials can influence the density of the coating. For most metallic elements the effects are usually considered negligible for deposits obtained under normal conditions from properly maintained electroplating baths free of contamination. The only documented exception is gold, the density of which is dependent on the deposition process.

7.12 *Composition of Coating*—Because the composition of a coating affects the mass of coating per unit area, it will also affect the instrument response (amount of backscattered beta