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Standard Test Method for Measurement of Coating Thickness by X-Ray Spectrometry¹

This standard is issued under the fixed designation B568; 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 U.S. Department of Defense.

1. Scope

1.1 This test method covers the use of X-ray spectrometry to determine thickness of metallic and some nonmetallic coatings.

1.2 The maximum measurable thickness for a given coating is that thickness beyond which the intensity of the characteristic secondary X radiation from the coating or the substrate is no longer sensitive to small changes in thickness.

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

1.4 Problems of personnel protection against radiation generated in an X-ray tube or emanating from a radioisotope source are not covered by this test method. For information on this important aspect, reference should be made to current documents of the National Committee on Radiation Protection and Measurement, Federal Register, Nuclear Regulatory Commission, National Institute of Standards and Technology (formerly the National Bureau of Standards), and to state and local codes if such exist.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

¹ This test method is under the jurisdiction of ASTM Committee B08 on Metallic and Inorganic Coatings and is the direct responsibility of Subcommittee B08.10 on Test Methods.

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2. Referenced Documents

2.1 *ASTM Standards*:²

E135 [Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials](#)

2.2 *International Standard*:

ISO 3497 [Metallic Coatings—Measurement of Coating Thickness—X-ray Spectrometric Methods](#)

3. Terminology

3.1 Definitions of technical terms used in this test method may be found in Terminology E135.

4. Summary of Test Method

4.1 *Excitation*—The measurement of the thickness of coatings by X-ray spectrometric methods is based on the combined interaction of the coating and substrate with incident radiation of sufficient energy to cause the emission of secondary radiations characteristic of the elements composing the coating and substrate. The exciting radiation may be generated by an X-ray tube or by certain radioisotopes.

4.1.1 *Excitation by an X-Ray Tube*—Suitable exciting radiation will be produced by an X-ray tube if sufficient potential is applied to the tube. This is on the order of 35 to 50 kV for most thickness-measurement applications. The chief advantage of X-ray tube excitation is the high intensity provided.

4.1.2 *Excitation by a Radioisotope*—Of the many available radioisotopes, only a few emit gamma radiations in the energy range 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 excitation include more compact instrumentation essentially monochromatic radiation, and very low background intensity. The major disadvantage of radioisotope excitation is the much lower intensities available as compared

² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

with X-ray tube sources. X-ray tubes typically have intensities that are several orders of magnitude greater than radioisotope sources. Due to the low intensity of radioisotopes, they are unsuitable for measurements on small areas (less than 0.3 mm in diameter). Other disadvantages include the limited number of suitable radioisotopes, their rather short useful lifetimes, and the personnel protection problems associated with high-intensity radioactive sources.

4.2 Dispersion—The secondary radiation resulting from the exposure of an electroplated surface to X radiation usually contains many components in addition to those characteristic of the coating metal(s) and the substrate. It is necessary, therefore, to have a means of separating the desired components so that their intensities can be measured. This can be done either by diffraction (wavelength dispersion) or by electronic discrimination (energy dispersion).

4.2.1 Wavelength Dispersion—By means of a single-crystal spectrogoniometer, wavelengths characteristic of either the coating or the substrate may be selected for measurement. Published data in tabular form are available that relate spectrogoniometer settings to the characteristic emissions of elements for each of the commonly used analyzing crystals.

4.2.2 Energy Dispersion—X-ray quanta are usually specified in terms of their wavelengths, in angstroms (Å), or their equivalent energies in kiloelectron volts (keV). The relationship between these units is as follows:

$$(\text{keV})(\text{Å}) = 12.396$$

where:

keV = the quantum energy in thousands of electron volts, and
Å = the equivalent wavelength in angstroms (10^{-10} m).

In a suitable detector (see 4.3.2), X rays of different energies will produce output pulses of different amplitudes. After suitable amplification, these pulses can be sorted on the basis of their amplitudes and stored in certain designated channels of a multichannel analyzer, each adjacent channel representing an increment of energy. Typically, a channel may represent a span of 20 eV for a lithium-drifted silicon detector or 150 to 200 eV for a proportional counter. From six to sixty adjacent channels can be used to store the pulses representing a selected characteristic emission of one element, the number of channels depending on the width of the emission peak (usually displayed on the face of a cathode ray tube). The adjacent channels used to store the pulses from the material under analysis are called the “region of interest” or ROI.

4.3 Detection:

4.3.1 Wavelength Dispersive Systems—The intensity of a wavelength is measured by means of an appropriate radiation detector in conjunction with electronic pulse-counting circuitry, that is, a scaler. With wavelength dispersive systems, the types of detectors commonly used as the gas-filled types and the scintillation detector coupled to a photomultiplier tube.

4.3.2 Energy-Dispersive Systems—For the highest energy resolution with energy dispersive systems, a solid-state device such as the lithium-drifted silicon detector must be used. This type of detector is maintained at a very low temperature in a

liquid-nitrogen cryostat (77K). Acceptable energy resolution for most thickness measurement requirements can be realized with proportional counters, and these detectors are being used on most of the commercially available thickness gages based on X-ray spectrometry. In setting up a procedure for coating-thickness measurement using an energy-dispersive system, consideration should be given to the fact that the detector “sees” and must process not only those pulses of interest but also those emanating from the substrate and from supporting and masking materials in the excitation enclosure. Therefore, consideration should be given to restricting the radiation to the area of interest by masking or collimation at the radiation source. Similarly, the detector may also be masked so that it will see only that area of the specimen on which the coating thickness is to be determined.

4.4 Basic Principle—A relationship exists between coating thickness and secondary radiation intensity up to the limiting thickness mentioned in 1.2. Both of the techniques described below are based on the use of primary standards of known coating thicknesses which serve to correlate quantitatively the radiation intensity and thickness.

4.5 Thickness Measurement by X-Ray Emission—In this technique, the spectrogoniometer is positioned to record the intensity of a prominent wavelength characteristic of the coating metal or, in the case of an energy-dispersive system, the multichannel analyzer is set to accept the range of energies comprising the desired characteristic emission. The intensity of the coating’s X-ray emission (coating ROI) will be at a minimum for a sample of the bare substrate where it will consist of that portion of the substrate fluorescence which may overlap the ROI of the coating and a contribution due to background radiation. This background radiation is due to the portion of the X-ray tube’s output which is the same energy as the coating’s X-ray emission. The sample will always scatter some of these X rays into the detector. If the characteristic emission energies of the coating and substrate are sufficiently different, the only contribution of the substrate will be due to background. For a thick sample of the solid coating metal or for an electroplated specimen having an “infinitely thick” coating, the intensity will have its maximum value for a given set of conditions. For a sample having a coating of less than “infinite” thickness, the intensity will have an intermediate value. The intensity of the emitted secondary X radiation depends, in general, upon the excitation energy, the atomic numbers of the coating and substrate, the area of the specimen exposed to the primary radiation, the power of the X-ray tube, and the thickness of the coating. If all of the other variables are fixed, the intensity of the characteristic secondary radiation is a function of the thickness or mass per unit area of the coating. The exact relationship between the measured intensity and the coating thickness must be established by the use of standards having the same coating and substrate compositions as the samples to be measured. The maximum thickness that can be measured by this method is somewhat less than what is, effectively, infinite thickness. This limiting thickness depends, in general, upon the energy of the characteristic X-ray and the density and absorption properties of the material under analysis. The typical relationship between a coating thickness and

the intensity of a characteristic emission from the coating metal is illustrated by the curve in the Appendix, Fig. X1.1.

4.6 Thickness Measurements by X-Ray Absorption—In this technique the spectrometer, in the case of a wavelength-dispersive system, is set to record the intensity of a selected emission characteristic of the basis metal. In an energy-dispersive system, the multichannel analyzer is set to accumulate the pulses comprising the same energy peak. The intensity will be a maximum for a sample of the uncoated basis metal and will decrease with increasing coating thickness. This is because both the exciting and secondary characteristic radiations undergo attenuation in passing through the coating. Depending upon the atomic number of the coating, when the coating thickness is increased to a certain value, the characteristic radiation from the substrate will disappear, although a certain amount of scattered radiation will still be detected. The measurement of a coating thickness by X-ray absorption is not applicable if an intermediate coating is present because of the indeterminate absorption effect of intermediate layer. The typical relationship between coating thickness and the intensity of a characteristic emission from the substrate is shown in the Appendix, see Fig. X1.2.

4.7 Thickness and Composition Measurement by Simultaneous X-ray Emission and Absorption (Ratio Method)—It is possible to combine the X-ray absorption and emission techniques when coating thicknesses and alloy composition are determined from the 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.

4.8 Multilayer Measurements—Many products have multilayer coatings in which it is possible to measure each of the coating layers by using the multiple-energy-region capability of the multichannel analyzer of an energy-dispersive system. The measuring methods permit the simultaneous measurement of coating systems with up to three layers. Or the simultaneous measurement of thickness and compositions of layers with up to three components. Such measurements require unique data processing for each multilayer combination to separate the various characteristic emissions involved, to account for the absorption by intermediate layers, and to allow for any secondary excitation which may occur between layers. Typical examples of such combinations are gold on nickel on copper and nickel on copper on steel.

4.9 Mathematical Deconvolution—When using a multichannel analyzer a mathematical deconvolution of the secondary radiation spectra can be used to extract the intensities of the characteristic radiation. This method can be used when the energies of the detected characteristic radiations do not differ sufficiently (for example, characteristic radiation from Au and Br). This method sometimes is described as *numerical filtering* in order to distinguish from the technique of setting fixed Region of Interest (ROI) channel limits in the multichannel analyzer.

5. Significance and Use

5.1 This is a sensitive, noncontact, and nondestructive method for measuring the coating thickness (and in some

cases, coating composition) of metallic and some nonmetallic coatings over a range of thicknesses from as little as 0.01 μm to as much as 75 μm depending on the coating and substrate materials. It can be used to measure coating and base combinations that are not readily measured by other techniques.

5.2 The coating thickness is an important factor in the performance of a coating in service.

6. Factors Affecting Accuracy

6.1 Counting Statistics—The production of X-ray quanta is random with respect to time. This means that during a fixed time interval, the number of quanta emitted will not always be the same. This gives rise to the statistical error which is inherent in all radiation measurements. In consequence, an estimate of the counting rate based on a short counting interval (for example, 1 or 2 s) may be appreciably different from an estimate based on a longer counting period, particularly if the counting rate is low. This error is independent of other sources of error such as those arising from mistakes on the part of the operator or from the use of inaccurate standards. 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. When an energy-dispersive system is being used it should be recognized that a significant portion of an intended counting period may be consumed as dead time, that is, time during which the count-rate capacity of the system is exceeded. It is possible to correct for dead-time losses. The manufacturer's instructions for accomplishing this with his particular instrumentation should be followed.

6.1.1 The standard deviation, s , of this random error will closely approximate the square root of the total count; that is, $s = \sqrt{N}$. The "true" count will lie within $N \pm 2s$ 95 % of the time. To understand the significance of the precision, it is helpful to express the standard deviation as a percent of the count, $100 \sqrt{N}/N = 100/\sqrt{N}$. Thus, 100 000 would give a standard deviation indicating 10 times the precision (one-tenth the standard deviation) obtained from 1000 counts. This is because $(100/\sqrt{1000})/(100/\sqrt{100000}) = 10$. This does not mean that the result would necessarily be ten times as accurate (see 7.2).

6.1.2 A counting interval should be chosen that will provide a net count of at least 10 000. This would correspond to a statistical error in the count rate of 1 %. The corresponding standard deviation in the thickness measurement is a function of the slope of the calibration curve at the point of measurement. Most commercially available instruments display the standard deviation directly in units of thickness.

6.2 Coating Thickness—The precision of the measurement will be affected by the thickness range being measured. In the curve shown in the Appendix, see Fig. X1.1, the precision will be best in the portion of the curve from approximately 0.25 to 7.5 μm . The precision rapidly becomes poorer in the portion of the curve above approximately 10 μm . The situation is similar for the absorption curve shown in the Appendix, see Fig. X1.2. At coating thicknesses greater than approximately 10 μm , the intensity changes very little with the coating thickness and, therefore, the precision in that region is poor. These limiting thicknesses are, in general, different for each coating material.