



Standard Test Method for Measurement of Thickness of Metallic Coatings by Measurement of Cross Section with a Scanning Electron Microscope¹

This standard is issued under the fixed designation B 748; 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.

1. Scope

1.1 This test method covers the measurement of metallic coating thicknesses by examination of a cross section with a scanning electron microscope (SEM).

1.2 *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 ASTM Standards:

E 3 Guide for Preparation of Metallographic Specimens²

E 766 Practice for Calibrating the Magnification of a Scanning Electron Microscope²

3. Summary of Test Method

3.1 A test specimen is cut, ground, and polished for metallographic examination by an SEM of a cross section of the coating. The measurement is made on a conventional micrograph or on a photograph of the video waveform signal for a single scan across the coating.

4. Significance and Use

4.1 This test method is useful for the direct measurement of the thicknesses of metallic coatings and of individual layers of composite coatings, particularly for layers thinner than normally measured with the light microscope.

4.2 This test method is suitable for acceptance testing.

4.3 This test method is for the measurement of the thickness of the coating over a very small area and not of the average or minimum thickness per se.

4.4 Accurate measurements by this test method generally require very careful sample preparation, especially at the greater magnifications.

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

5. Equipment

5.1 The scanning electron microscope shall have a resolution of at least 50 nm. Suitable instruments are available commercially.

6. Factors Affecting the Measurement Reliability

6.1 *Surface Roughness*—If the coating or its substrate is rough relative to the coating thickness, one or both of the interfaces bounding the coating cross section may be too irregular to permit accurate measurement of the average thickness in the field of view.

6.2 *Taper of Cross Section*—If the plane of the cross section is not perpendicular to the plane of the coating, the measured thickness will be greater than the true thickness. For example, an inclination of 10° to the perpendicular will contribute a 1.5 % error. True thickness, (t), equals measured thickness, (t_m), multiplied by the cosine of the angle of inclination (θ): $t = t_m \times \cos(\theta)$. (See X1.3.2.)

6.3 *Specimen Tilt*—Any tilt of the specimen (plane of the cross section) with respect to the SEM beam, may result in an erroneous measurement. The instrument should always be set for zero tilt.

6.4 *Oblique Measurement*—If the thickness measurement is not perpendicular to the plane of the coating, even when there is no taper (6.2) or tilt (6.3), the measured value will be greater than the true thickness. This consideration applies to the conventional micrograph (9.3.1) and to the direction of the single video waveform scans (9.3.2).

6.5 *Deformation of Coating*—Detrimental deformation of the coating can be caused by excessive temperature or pressure during the mounting and preparation of cross sections of soft coatings.

6.6 *Rounding of Edge of Coating*—If the edge of the coating cross section is rounded, that is, if the coating cross section is not completely flat up to its edges, the observed thickness may differ from the true thickness. Edge rounding can be caused by improper mounting, grinding, polishing, or etching.

6.7 *Overplating of Specimen*—Overplating of the test specimen serves to protect the coating edges during preparation of cross sections and thus to prevent an erroneous measurement. Removal of coating material during surface preparation for overplating can cause a low thickness measurement.

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.

Current edition approved Feb. 23, 1990. Published April 1990. Originally published as B 748 – 85. Last previous edition B 748 – 85.

² *Annual Book of ASTM Standards*, Vol 03.01.