Designation: B487 – 85 (Reapproved 2013)

Standard Test Method for Measurement of Metal and Oxide Coating Thickness by Microscopical Examination of Cross Section[1]

This standard is issued under the fixed designation B487; 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 measurement of the local thickness of metal and oxide coatings by the microscopical examination of cross sections using an optical microscope.

1.2 Under good conditions, when using an optical microscope, the method is capable of giving an absolute measuring accuracy of 0.8 µm. This will determine the suitability of the method for measuring the thickness of thin coatings.

1.3 *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.* (This is especially applicable to the chemicals cited in Table X2.1.)

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E3 Guide for Preparation of Metallographic Specimens

3. Summary of Test Method

3.1 This test method consists of cutting out a portion of the test specimen, mounting it, and preparing the mounted cross section by suitable techniques of grinding, polishing, and etching. The thickness of the cross section is measured with an optical microscope.

NOTE 1—These techniques will be familiar to experienced metallographers but some guidance is given in Section 5 and in Appendix X1 for less experienced operators.

4. Significance and Use

4.1 Coating thickness is an important factor in the performance of a coating in service and is usually specified in a coating specification.

4.2 This method is suitable for acceptance testing.

5. Factors Influencing the Measurement Result

5.1 *Surface Roughness*—If the coating or its substrate has a rough surface, one or both of the interfaces bounding the coating cross section may be too irregular to permit accurate measurement. (See X1.4)

5.2 *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.

5.3 *Deformation of the Coating*—Detrimental deformation of the coating can be caused by excessive temperature or pressure during mounting and preparation of cross sections of soft coatings or coatings melting at low temperatures, and also by excessive abrasion of brittle materials during preparation of cross sections.

5.4 *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 true thickness cannot be observed microscopically. Edge rounding can be caused by improper mounting, grinding, polishing, or etching. It is usually minimized by overplating the test specimen before mounting. (See X1.2.)

5.5 *Overplating*—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.

5.6 *Etching*—Optimum etching will produce a clearly defined and narrow dark line at the interface of two metals. Excessive etching produces a poorly defined or wide line which may result in an erroneous measurement.

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

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[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.

5.7 *Smearing*—Improper polishing may leave one metal smeared over the other metal so as to obscure the true boundary between the two metals. The apparent boundary may be poorly defined or very irregular instead of straight and well defined. To verify the absence of smearing, the coating thickness should be measured and the polishing, etching, and thickness measurement repeated. A significant change in apparent thickness indicates that smearing was probably present during one of the measurements.

5.8 *Magnification*—For any given coating thickness, measurement errors generally increase with decreasing magnification. If possible, the magnification should be chosen so that the field of view is between 1.5 and 3 × the coating thickness.

5.9 *Calibration of Stage Micrometer*— Any error in calibration of the stage micrometer will be reflected in the measurement of the specimen. Errors of several percent are not unrealistic unless the scale has been calibrated or has been certified by a responsible supplier. The distance between two lines of a stage micrometer used for the calibration shall be known to within 0.2 µm or 0.1 %, whichever is the greater. If a stage micrometer is not certified for accuracy, it should be calibrated. A generally satisfactory means of calibration is to assume that the stated length of the full scale is correct, to measure each subdivision with a filar micrometer, and to calculate the length of each subdivision by simple proportion.

5.10 *Calibration of Micrometer Eyepiece :*

5.10.1 A filar micrometer eyepiece generally provides the most satisfactory means of making the measurement of the specimen. The measurement will be no more accurate than the calibration of the eyepiece. As calibration is operator dependent, the eyepiece shall be calibrated by the person making the measurement.

5.10.2 Repeated calibrations of the micrometer eyepiece can be reasonably expected to have a spread of less than 1 %.

5.10.3 Some image-splitting micrometer eyepieces have a nonlinearity that introduces an error of up to 1 % for short measurement distances.

5.11 *Alignment*—Errors can be introduced by backlash in the movement of the micrometer eyepiece. If the final motion during alignment of the hairline is always made in the same direction, this error will be eliminated.

5.12 *Uniformity of Magnification*—Because the magnification may not be uniform over the entire field, errors can occur if both the calibration and the measurement are not made over the same portion of the field with the measured boundaries centered about the optical axis.

5.13 *Lens Quality*—Lack of sharpness of the image contributes to the uncertainty of the measurement. Poor quality lenses could preclude accurate measurements. Sometimes image sharpness can be improved by using monochromatic light.

5.14 *Orientation of Eyepiece*—The movement of the hairline of the eyepiece for alignment has to be perpendicular to the boundaries of the coating cross section. For example, 10° misalignment will contribute a 1.5 % error.

5.15 *Tube Length*—A change in the tube length of the microscope causes a change in magnification and if this change occurs between the time of calibration and the time of measurement, the measurement will be in error. A change in tube length may occur when the eyepiece is repositioned within the tube, when the focus of the eyepiece tube is changed, and, for some microscopes, when the fine focus is adjusted or the interpupillary distance for binoculars is changed.

6. Preparation of Cross Sections

6.1 Prepare, mount, polish, and etch the specimen so that:

6.1.1 The cross section is perpendicular to the coating;

6.1.2 The surface is flat and the entire width of the coating image is simultaneously in focus at the magnification used for the measurement;

6.1.3 All material deformed by cutting or cross sectioning is removed.

6.1.4 The boundaries of the coating cross section are sharply defined by no more than contrasting appearance or by a narrow, well-defined line.

NOTE 2—Further guidance is given in Appendix X1. Some typical etchants are described in Appendix X2.

7. Procedure

7.1 Give appropriate attention to the factors listed in Section 5 and Appendix X1.

7.2 Calibrate the microscope and its measuring device with a certified or calibrated stage micrometer.

7.3 Measure the width of the image of the coating cross section at no less than five points distributed along a length of the microsection, and calculate the arithmetic mean of the measurements (see 8.1.5 and 8.1.6).

8. Test Report

8.1 The test report shall include the following information:

8.1.1 The date of test;

8.1.2 The number and title of this test method;

8.1.3 The identification of the test specimens;

8.1.4 The location on the coated item at which the cross section was made;

8.1.5 The measured thickness, in micrometres (millimetres if greater than 1 mm) at each point (7.3), and the length of section over which the measurements were distributed;

8.1.6 The local thickness, that is, the arithmetic mean of the measured thicknesses;

8.1.7 Any deviations from this test method;

8.1.8 Any factors that might influence interpretation of the reported results; and

8.1.9 The name of the operator and testing laboratory.

9. Precision and Bias

9.1 The microscope and associated equipment, its use, its calibration, and the method of preparation of the cross section shall be chosen so as to allow the coating thickness to be determined to within 1 µm or 10 %, whichever is the greater, of the actual coating thickness. Under good conditions, when using an optical microscope, the method is capable of giving an absolute measuring accuracy of 0.8 µm and for thicknesses greater than 25 µm a reasonable error is of the order of 5 % or better.