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Standard Test Method for Measurement of Metal and Oxide Coating Thickness by Microscopical Examination of a Cross Section¹

This standard is issued under the fixed designation B 487; 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 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:*

E 3 Methods of Preparation of Metallographic Specimens²

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.

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 \times the coating thickness.

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

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.

APPENDIXES

(Nonmandatory Information)

X1. GUIDANCE ON THE PREPARATION AND MEASUREMENT OF CROSS SECTIONS

X1.1 *Introduction*—The preparation of test specimens and measurement of coating thickness are greatly dependent on individual techniques and there is a variety of suitable techniques available. It is not reasonable to specify only one set of techniques, and it is impractical to include all suitable techniques. The techniques described in this appendix are intended as guidance for metallographers not experienced in measurements of coating thickness. For additional guidance see Methods E 3.

X1.2 *Mounting*:

X1.2.1 To prevent rounding of the edge of the cross section, the free surface of the coating should be supported so that there is no space between the coating and its support. This is usually achieved by overplating the specimen with a coating at least 10- μm thick of a metal of similar hardness to the coating. For hard, brittle coatings (for example oxide or chromium coatings) tightly wrapping the specimen in soft aluminum foil before mounting has proved successful.

X1.2.2 If the coating is soft, overplating with a metal which is softer will make polishing more difficult, because the softer metal tends to be polished away more rapidly.

X1.2.3 Overplating of zinc or cadmium coatings with copper may cause difficulty because of the tendency, during subsequent etching, of dissolved copper to deposit on the coatings. It is better to overplate zinc with cadmium and vice versa.

X1.3 *Grinding and Polishing*:

X1.3.1 It is essential to keep the cross-section surface of the mount perpendicular to the coating. This is facilitated by incorporating additional pieces of a similar metal in the plastic mounting, near the outer edges, by periodically changing the direction of grinding (rotating through 90°) and by keeping the grinding time and pressure to a minimum. If, before grinding, reference marks are inscribed on the side of the mount, any inclination from horizontal is easily measurable.

X1.3.2 Grind the mounted test specimens on suitable abrasive paper, using an acceptable lubricant, such as water or mineral spirits, and apply minimum pressure to avoid bevelling of the surface. Initial grinding should employ 100 or 180 grade abrasive to reveal the true specimen profile and to remove deformed metal. Subsequently, use Grades 240, 320, 500, and 600 without exceeding grinding times of 30 to 40 s on each paper; alter the direction of scratches by 90° for each change of paper. A final polish of 2 to 3 min on a rotating wheel charged with 4 to 8- μm diamond paste particles and lubricated with mineral spirits should suffice to remove scratches for final

examination. If an especially high degree of surface finish is required, a further treatment, using diamond paste of approximately 1- μm particles, may be employed.

X1.3.3 If very soft materials are being prepared, abrasive particles may become embedded during grinding. This may be minimized by totally immersing abrasive papers in a lubricant during grinding or by using a copious flow of lubricant. If abrasive particles do become embedded, they may be removed by applying a short, light hand polish with metal polish after grinding and before diamond finishing or by one or more cycles of alternate etching and polishing.

X1.4 *Etching*—Etching is usually advisable to promote contrast between the metal layers, to remove traces of smeared metal, and to develop a fine line at the boundary of the coating. Some typical etchants are given in Appendix X2.

X1.5 *Measurement*:

X1.5.1 The measuring device may be a filar micrometer or a micrometer eyepiece. The latter has a lower precision. An image-splitting eyepiece is advantageous for thin coatings on rough substrate surfaces. Measurement of the image projected on to a ground-glass plate is usually less satisfactory because of the lack of sharpness of the image and poor legibility of the ruler when the projected image is visible.

X1.5.2 The measuring device should be calibrated at least once before and once after a measurement, unless repeated experience indicates otherwise.

X1.5.3 When making calibration and coating measurements, both should be made by the same operator, the stage micrometer and the coating should be centered in the field, and each measurement at a point should be made at least twice and averaged.

X1.5.4 For critical and referee measurements, all steps for the preparation of cross sections and measurement of coating thickness, from grinding with 600 grade or coarser abrasive, up to and including the determination, should be performed at least twice. With good techniques and equipment, and smooth coating and substrate surfaces, repeatability within 2 % or 0.5 μm , whichever is the greater, is reasonable.

X1.5.5 Some microscopes are subject to a spontaneous movement of the stage relative to the objective, possibly due to nonuniform thermal effects from the light source. Such a movement during the measurement can cause an erroneous measurement at moderate and high magnifications. This can be minimized by completing the measurement quickly and by measuring each interval twice, once from left to right and once from right to left.

X2. SOME TYPICAL ETCHANTS FOR USE AT ROOM TEMPERATURE

TABLE X2.1 Etchants

Etchant	Use and Remarks
Nitric acid (sp gr 1.42): 5 mL Ethanol (95 %): 95 mL Caution —This mixture can be explosively unstable, particularly if heated.	For nickel or chromium coatings on steel Etches steel This etchant should be freshly prepared.
Iron(III)chloride hexahydrate (FeCl ₃ ·6H ₂ O): 10 g Hydrochloric acid (sp gr 1.16): 2 mL Ethanol (95 %): 98 mL	For gold, lead, silver, nickel and copper coatings on steel, copper, and copper alloys. Etches steel, copper, and copper alloys.
Nitric acid (sp gr 1.42): 50 mL Glacial acetic acid: 50 mL	For determination of thickness of individual layers of multilayer coatings of nickel on steel and copper alloys; distinguishes each layer of nickel by identifying structures. Etches nickel; excessive attack on steel and copper alloys.
Ammonium persulfate: 10 g Ammonium hydroxide (sp gr 0.88): 2 mL Distilled water: 90 mL	For tin and tin alloy coatings on copper and copper alloys. Etches copper and copper alloys. This etchant should be freshly prepared.
Nitric acid (sp gr 1.42): 5 mL Hydrofluoric acid (sp gr 1.14): 2 mL Distilled water: 93 mL	For nickel and copper coatings on aluminum and its alloys. Etches aluminum and its alloys.
Chromium(VI) oxide (CrO ₃): 20 g Sodium sulfate: 1.5 g Distilled water: 100 mL	For nickel and copper on zinc-based alloys. Also suitable for zinc and cadmium on steel. Etches zinc, zinc-based alloys and cadmium.
Hydrofluoric acid (sp gr 1.14): 2 mL Distilled water: 98 mL	For anodized aluminum alloys. Etches aluminum and its alloys.
Ammonium hydroxide (sp gr 0.90): 1 part by volume Hydrogen peroxide (3 % solution): 1 part by volume	For nickel on copper and its alloys. Swab with a fresh solution. Etches the copper.
Sodium or potassium cyanide (10 % solution): 1 part Ammonium persulfate (10 % solution): 1 part Make up each solution fresh each time.	For silver and gold on copper and nickel alloys and steel.

X3. LIMIT OF RESOLUTION

X3.1 Resolution may be expressed as the minimum distance by which two points must be separated before they can be revealed as separate points in the image. For a microscope there is a theoretical limit of resolution determined by the numerical aperture (NA) of the objective. This theoretical limit is approached by good quality microscopes. For practical purposes better resolution cannot be obtained regardless of the quality of the optics or of the total magnification.

X3.2 Generally, the maximum useful magnification is about $1000 \times$ the NA of an objective. That is, for practical purposes, greater magnification will not reveal additional information nor impart better definition. Such additional magnification is often referred to as “empty magnification.”

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