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Standard Test Method for Measurement of Thickness of Metallic Coatings by Measurement of Cross Section with a Scanning Electron Microscope[1]

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 microsope (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 Specimens[2]

E 766 Practice for Calibrating the Magnification of a Scanning Electron Microscope[2]

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 directon 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.

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

6.8 *Etching*—Optimum etching will produce a clearly defined and narrow dark line at the interface of two metals. A wide or poorly defined line can result in an inaccurate measurement.

6.9 *Smearing*—Polishing may leave smeared metal that obscures the true boundary between the two metals and results in an inaccurate measurement. This may occur with soft metals like lead, indium, and gold. To help identify whether or not there is smearing, repeat the polishing, etching, and measurement several times. Any significant variations in readings indicates possible smearing.

6.10 *Poor Contrast*—The visual contrast between metals in the SEM is poor when their atomic numbers are close together. For example, bright and semibright nickel layers may not be discriminable unless their common boundary can be brought out sufficiently by appropriate etching and SEM techniques. For some metal combinations, energy dispersive X-ray techniques (see X1.4.5) or backscatter image techniques (see X1.4.6) may be helpful.

6.11 *Magnification*:

6.11.1 For any given coating thickness, measurement errors tend to increase with decreasing magnification. If practical, the magnification should be chosen so that the field of view is between 1.5 and 3× the coating thickness.

6.11.2 The magnification readout of an SEM is often poorer than the 5 % accuracy often quoted and the magnification has been found for some instruments to vary by 25 % across the field. Magnification errors are minimized by appropriate use of an SEM stage micrometer and appropriate experimental procedure. (See Practice E 766.)

6.12 *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. This can be very important.

6.13 *Stability of Magnification*:

6.13.1 The magnification of an SEM often changes or drifts with time. This effect is minimized by mounting the stage micrometer and test specimen side by side on the SEM stage so as to keep the transfer time short.

6.13.2 A change in magnification can occur when adjustments are made with the focusing and other electronic SEM controls. Such a change is prevented by not using the electronic focus controls or other electronic SEM controls after photographing the stage micrometer scale except to focus with the mechanical *X*, *Y*, and *Z* controls. Appropriate manipulation of the *X*, *Y*, and *Z* controls will bring the specimen surface to the focal point of the SEM beam.

6.14 *Stability of Micrographs*—Dimensional changes of micrographs can take place with time and with temperature and humidity changes. If the calibration micrograph of the stage micrometer scale and the micrograph of the test specimen are kept together and time is allowed for stabilization of the photographic paper, errors from this source will be minimized.

7. Preparations of Cross Sections

7.1 Prepare, mount, grind, polish, and etch the test specimen so that the following occurs:

7.1.1 The cross section is perpendicular to the plane of the coating,

7.1.2 The surface is flat and the entire width of the coating image is simultaneously in focus at the magnification to be used for the measurement,

7.1.3 All material deformed by cutting or cross sectioning is removed,

7.1.4 The boundaries of the coating cross section are sharply defined by contrasting appearance, or by a narrow, well-defined line, and

7.1.5 If the video waveform signal is to be measured, the signal trace is flat except across the two boundaries of the coating.

7.2 For further guidance see Appendix X1.

8. Calibration of Magnification

8.1 Calibrate the SEM with an SEM stage micrometer and determine the magnification factor, *M*, in accordance with Practice E 766 (see X1.4.2). Other calibration methods may be used if it can be demonstrated that they are sufficiently accurate for meeting the requirement of Section 12.

8.2 If practical, the stage micrometer and the test specimen shall be mounted side by side on the SEM stage.

9. Procedure

9.1 Operate the SEM in accordance with the manufacturer's instructions.

9.2 Take into account the factors listed in Sections 6 and 12.

9.3 Make a micrograph of the test specimen under the same conditions and instrument settings as used for the calibration and make an appropriate measurement of the micrograph image. Carry out this step in accordance with 9.3.1 or 9.3.2.

9.3.1 *Conventional Micrograph*:

9.3.1.1 With the boundaries of the coating clearly and sharply defined, make conventional micrographs of the SEM stage micrometer scale and of the test specimen.

9.3.1.2 Measure the micrographs to at least the nearest 0.1 mm using a diffraction plate reader or equivalent device. If this is not practical, it may be because poor sample preparation is causing the boundaries of the coating to be poorly defined.

9.3.2 *Video Waveform Signal*:

9.3.2.1 Photograph the video waveform signal for a single scan across the coating cross section and across the SEM stage micrometer scale.

9.3.2.2 To measure the coating, measure the horizontal distance between the inflection points of the vertical portions of the scan at the boundaries of the coating. Make the measurements to the nearest 0.1 mm using a diffraction plate reader or equivalent device.

9.3.3 For further guidance see Appendix X1.

10. Calculation and Expression of Results

10.1 Calculate the thickness according to the expression:

$$T = 1000 \times d/M \quad (1)$$

where:

T = coating thickness, in μm,
d = linear distance on micrograph, in mm, and
M = magnification factor as defined in Practice E 766.

11. Report

11.1 The report of the measurements shall give the following information:

11.1.1 Date measurements were made,

11.1.2 The title, number, and year of issue of this test method,

11.1.3 Identification of the test specimen(s),

11.1.4 Location of measurement on test specimen(s),

11.1.5 The measured values and their arithmetic mean,

11.1.6 The calibrated magnification as measured with an SEM micrometer scale immediately before the test specimen measurements,

11.1.7 Type of measurement: conventional micrograph or video waveform signal,

11.1.8 Any unusual feature of the measurement that might affect the results, and

11.1.9 Name of individual responsible for the measurements.

12. Precision and Bias

12.1 The instrument, its operation, and its calibration shall be such that the uncertainty of the measurements shall be less than 0.1 μm or 10 %, whichever is larger.

12.2 For a thin gold coating, one laboratory reported measurement uncertainty of 0.039 μm for the SEM stage micrometer scale, 0.02 μm for the measurement of the calibration micrographs, and 0.02 μm for measurement of the video waveform signal scan. Based on practical experience, a repeatability of 0.1 μm or better may be assumed.

APPENDIX

(Nonmandatory Information)

X1. TECHNIQUES OF SPECIMEN PREPARATION AND USE OF THE SEM

X1.1 Introduction

X1.1.1 The preparation of specimens and measurement of coating thickness are greatly dependent on individual techniques and there is a variety of suitable techniques available. (See Guide E 3.) 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.

X1.2 Mounting

X1.2.1 To prevent rounding of the edge of the coating 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 coating with a coating at least 10 μm thick of a metal of similar hardness to the coating. The overplate should also give an electron signal intensity different from that of the coating. The mounting material or sample surface must be electrically conducting and grounded to prevent a surface charge buildup in the SEM.

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 grind time and pressure to a minimum). If, before grinding, reference marks are inscribed on the sides of the mount, any inclination from horizontal is easily measurable. Grind the mounted specimens on suitable abrasive paper, using an acceptable lubricant, such as water, and apply minimum pressure to avoid bevelling the surface. Initial grinding should employ 100 or 180 grade abrasive to reveal the true specimen profile and to remove any 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. Then polish successively with 6 to 9, 1, and 0.5-μm diamond on microcloth. Some metallographers prefer the use of 0.3- and 0.05-μm alumina.

X1.3.2 A convenient way to check for tapering of the cross section is to mount a small diameter rod or wire with the specimen so that the perpendicular cross section of the rod is parallel to that of the coating. If a taper is present, the cross section of the rod will be elliptical.

X1.3.3 If the video waveform signal scan technique is used, it is important that scratches be completely removed and that overpolishing does not selectively remove one of the metals more than the other so that the signal scan is distorted. With careful polishing, it is often unnecessary to use chemical etches.

X1.4 Use of SEM

X1.4.1 If the image of the cross section, as revealed in a conventional micrograph, is measured; and if the boundaries of the coating cross section are revealed solely by the photographed contrast between the two materials; the apparent width of the coating cross section can vary, depending on the contrast and brightness settings. The variation can be as great as 10 % without any change in instrument magnification. To minimize the resulting uncertainty, adjust the contrast and brightness so that the image contains surface detail of the materials on either side of each boundary.

X1.4.2 Because the magnification of an SEM can change spontaneously with time and can change as a result of changing other instrument settings, it is advisable to calibrate the instrument immediately before or after measurement of the test

specimen. For critical measurements, the average of measurements made before and after measurement of the test specimen should be used. This assures that no change in the magnification occurred and it provides information about the precision of the calibration.

X1.4.3 If the video waveform trace is measured, the measurement is made of the horizontal distance between the inflection points at the boundaries. The inflection point is half way between the horizontal traces of the two materials.

X1.4.4 For a video-waveform trace, select a portion of the polished specimen that yields a flat, smooth signal.

X1.4.5 Many SEMs are equipped with energy dispersive X-ray spectroscopy (EDS) which can be helpful in identi-fying the metal-coating layers. At best the resolution of EDS is about 1 μm and often it is poorer.

X1.4.6 The use of backscatter images instead of secondary electron images can also be helpful in distinguishing metal layers with atomic numbers as close together as 1.0 and with a resolution of 0.1 μm.