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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Test Method for Measurement of Thickness of Anodic Coatings on Aluminum and of Other Transparent Coatings on Opaque Surfaces Using the Light-Section Microscope[1]

This standard is issued under the fixed designation B 681; 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 a procedure for the nondestructive measurement of the thickness of transparent anodic coatings on aluminum articles by means of the light-section microscope. This method may also be used to measure the thickness of any transparent coating on an opaque reflective surface.

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:*

B 244 Test Method for Measurement of Thickness of Anodic Coatings on Aluminum and of Other Nonconductive Coatings on Nonmagnetic Basis Metals with Eddy-Current Instruments[2]

B 487 Test Method for Measurement of Metal and Oxide Coating Thicknesses by Microscopical Examination of a Cross Section[2]

B 588 Test Method for Measurement of Thickness of Transparent or Opaque Coatings by Double-Beam Interference Microscope Technique[2]

2.2 *International Standard:*

ISO 2128 Anodizing of Aluminum and Its Alloys—Determination of Thickness of Anodic Oxide Coatings—Nondestructive Measurement by Split-Beam Microscope[3]

3. Summary of Test Method

3.1 The thickness of a transparent anodic coating or other transparent coating is determined by a microscopical method in which a thin beam of light is projected on to the specimen surface at an angle of 45° to the normal. The displacement between the rays reflected from the coating surface and from the coating-substrate interface is measured and is directly related to the coating thickness.

4. Significance and Use

4.1 The test method describes a rapid and nondestructive procedure for measuring the coating thickness of anodic oxides on aluminum and of other transparent coatings on opaque reflecting surfaces.

4.2 This test method is suitable for quality control purposes within manufacturing operations and for determining whether coated parts meet coating thickness requirements provided in applicable specifications.

4.3 The test method is limited by the following restrictions:

4.3.1 The coating must be sufficiently transparent to allow the light from the instrument to reflect off the coating-substrate interface and be visible as a distinct line.

4.3.2 Both the coating and substrate must be sufficiently smooth to allow the beams of light to reflect from the coating surface and the coating-substrate interface without significant distortion or aberration and to be clearly visible.

4.3.3 The index of refraction of the coating material must be known.

4.3.4 The coating must be in the thickness range of 2 to 40 µm.

4.4 This test method is suitable for most clear anodic coatings on aluminum articles used in interior or exterior applications for decorative or protective purposes. It is applicable to coatings that have been dyed if the depth of color is not so great as to obscure the coating-substrate interface. It is also applicable to some transparent organic coatings such as clear or dyed lacquers, provided that their thickness and uniformity fall within the guidelines given above.

4.5 This test method is not suitable for barrier layer anodic coatings, integral color coatings except in the lightest colors, hard anodic coatings, or some specialty types of coatings that are too thin, thick, or rough. Other methods such as the eddy-current technique, Test Method B 244, cross-section technique, Test Method B 487, and interference microscope technique, Test Method B 588, may be applicable to coatings on which the light-section microscope cannot be used.

5. Lacquered Anodic Coatings

5.1 This test method will not give accurate anodic coating

[1] This test method is under the jurisdiction of ASTM Committee B-8 on Metallic and Inorganic Coatings and is the direct responsibility of Subcommittee B08.10 on General Test Methods.

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[2] *Annual Book of ASTM Standards,* Vol 02.05.

[3] Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

thickness measurements on samples that have been lacquered after anodizing.

5.2 In the case of anodized and lacquered articles it is necessary to remove the lacquer overcoating with appropriate solvents in order to obtain an accurate anodic coating thickness.

6. Apparatus

6.1 *Light-Section Microscope,*[4] consisting of a light source and microscope with measuring capabilities, mounted on a suitable stand to allow observation of the specimen.

6.2 The light source shall be suitably bright to provide a sharp collimated beam of light, preferably either filtered or monochromatic, at an angle of incidence of 45° to the specimen surface. The beam shall be about 1 µm in width by 1 mm in length.

6.3 The microscope body shall be mounted such that it can focus on the specimen surface and with its axis in the plane of incident and reflected rays of light. The microscope body shall be normal to the incident beam and shall be at an angle of 45° to the specimen surface.

6.4 The magnification of the microscope shall be in the range from 100 to 500× with a higher magnification, for example, 400× for thinner coatings (2 to 10 µm), and lower magnification, for example, 200× for thicker coatings.

6.5 The microscope shall be equipped with a filar micrometer eyepiece capable of measuring object distances in the range from 1 to 40 µm with an accuracy of ± 0.2 µm.

6.6 The magnification of the microscope shall be verified. The procedure of 7.2 and 7.3 may be used for this purpose.

7. Calibration

7.1 The following calibration procedure may be used to verify the accuracy of the instrument. It should be used in critical applications or in cases where questionable results have been obtained.

7.2 Prepare a calibration standard or set of standards covering the range of coating thicknesses of interest by anodizing and sealing parts or panels similar or identical to the ones to be measured. Cut pieces of these samples and measure the coating thickness in accordance with Method B 487. The remaining part of the sample is the calibration standard.

7.3 Each operator of the instrument shall measure the calibration standards with the light-section microscope. Agreement between the two methods shall be within ± 1 µm or ± 10 % of the coating thickness, whichever is greater. If this agreement is not met, contact the manufacturer of the microscope and have it repaired.

8. Procedure

8.1 Make the thickness measurements on the significant surfaces of the test specimens: Take care to avoid biases from edge effects on the coating thickness.

8.2 Position the test specimens on the stage of the microscope such that the area to be measured is directly under the objective. Be sure to have the test area as level (parallel to the stage) as practical.

8.3 Bring the light-section microscope into focus so that the images of the reflected beams are in sharpest focus. Refer to the manufacturer's instructions to obtain the best focus.

8.4 Adjust the orientation of the light bands so that they are parallel to the cross hair. This will account for any minor deviations from level of the specimen. Refocus the instrument.

8.5 Select the light bands for measurement. On mat or dyed surfaces usually only two bands are visible, and in these cases there is no choice. In these cases, the light bands are reflected from the coating surface and from the coating-substrate interface. On bright or specular surfaces more bands are frequently observed (see Fig. 1). In this case the best accuracy is usually obtained by measuring the distance between the band reflected from the coating surface and the second coating-substrate

[4] The light-section microscope manufactured by Carl Zeiss, Inc., 444 Fifth Ave., New York, NY 10018, has been found satisfactory.

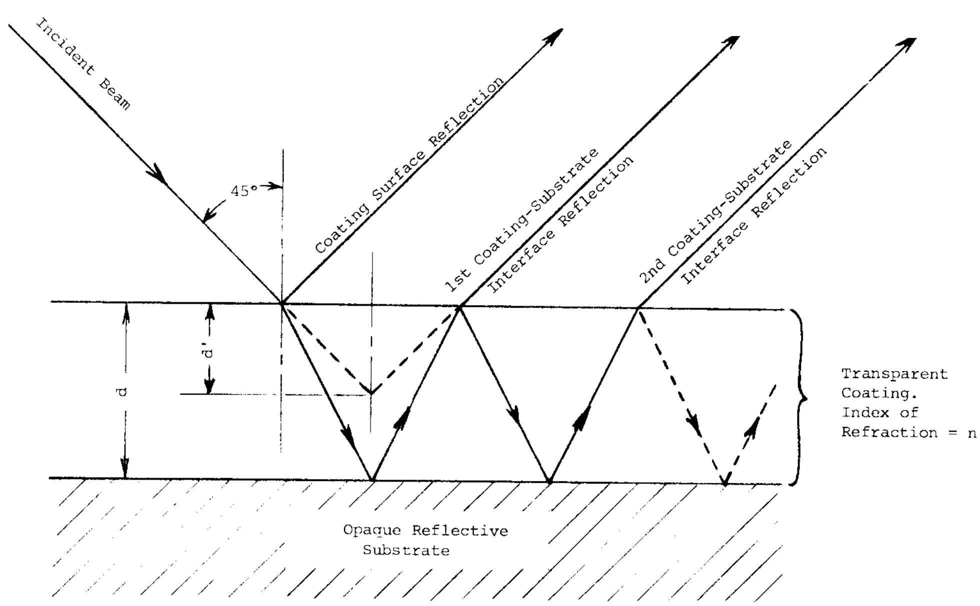

FIG. 1 Diagram Showing Light-Beam Paths

interface reflection. The reason for selecting the second reflection band is that the intensity of the first coating-substrate interface reflection is so great that the band appears quite broad, making accurate positioning of the cross hair in the center of the band difficult.

8.6 Position the cross hair in the center of the chosen band of light reflected from the coating-substrate interface, and then either adjust the micrometer scale to zero or read and record the micrometer scale.

NOTE 1—Some instruments have a filar micrometer eyepiece that has a double knob. One portion of the knob moves the cross hairs without moving the scale while the other moves both the scale and the cross hairs together. With this type instrument the scale should be set to zero and then the cross hair should be positioned in the center of the coating-substrate band of light using the portion of the knob that moves the cross hair without moving the scale. After this adjustment is made, the scale should be checked to verify that it is still set at zero.

8.7 Move the cross hair to the center of the coating surface band of light and read the micrometer scale. Approach the final position of the cross hair in both cases from the same direction to eliminate backlash errors in the micrometer. Record the reading.

8.8 Repeat the measurement for a total of five readings on each specimen. After each measurement move the specimen so that a different area is viewed.

9. Calculations

9.1 Multiply each reading by the magnification factor provided by the instrument manufacturer to obtain the apparent thickness, d'.

NOTE 2—The apparent thickness is directed proportional to the distance between the two light beams and with most instruments the read out multiplied by the magnification factor is the apparent thickness and not the distance between the two light beams.

9.2 The actual coating thickness is given by:

$$d = d' \, (2n^2 - 1)^{1/2} \quad (1)$$

where

d = actual coating thickness,
d' = apparent measured coating thickness, and
n = index of refraction of the transparent coating, which for most porous anodic coatings on aluminum ranges from 1.59 to 1.62.

9.3 When the second reflection from the coating-substrate interface is used, the value obtained for d in the above equation must be divided by two.

9.4 In general, sufficient accuracy is obtained if the equation $d = 2d'$ is used for anodic coatings on aluminum alloys. When the second reflection from the coating-substrate interface is used, the anodic coating thickness is given by $d = d'$.

9.5 Calculate the coating thickness from the measured beam spacing by the magnification factor provided by manufacturer.

9.6 Calculate the mean of the five thickness values obtained for each specimen.

10. Report

10.1 The report shall include the following information:

10.1.1 Designation, title, and issue of this test method,

10.1.2 Type of instrument used,

10.1.3 Index of refraction used for the coating material,

10.1.4 Whether the spacing measured was from the coating surface reflection to the first or second coating-substrate reflection, and

10.1.5 Mean, range, and number of thickness values measured or a list of the actual measurements.

11. Precision and Bias

11.1 The repeatability of measurements by a single experienced operator is usually better than ±0.2 µm (±2 standard deviation) for bright, anodized surfaces on smooth, flat specimens. The repeatability is however dependent upon the type of finish, the skill of the operator, and the adjustment of the instrument.

11.2 The reproducibility of measurements by different operators using different instruments is normally better than ±0.5 µm (±2 standard deviation) for bright, anodized surfaces on smooth, flat specimens. The reproducibility is however dependent on the skill of the operators and the type of finish being measured.

11.3 Several factors can contribute bias to the results obtained, including: the accuracy of the index of refraction value, the accuracy of the 45° angle of incidence of the beam of light, and the accuracy of the filar eyepiece micrometer. The use of the calibration procedure in Section 7 ensures accuracies of ±1 µm or 10 % of the coating thickness, whichever is greater.