Standard Test Method for Measurement of Thickness of Transparent or Opaque Coatings by Double-Beam Interference Microscope Technique[1]

This standard is issued under the fixed designation B 588; 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 the thickness of transparent metal oxide and metallic coatings by utilizing a double-beam interference microscope.[2]

1.2 The test method requires that the specimen surface or surfaces be sufficiently mirrorlike to form recognizable fringes.

1.3 This test method can be used nondestructively to measure 1 to 10μ m thick transparent coatings, such as anodic coatings on aluminum. The test method is used destructively for 0.1 to 10 μm thick opaque coatings by stripping a portion of the coating and measuring the step height between the coating and the exposed substrate. The stripping method can also be used to measure 0.2 to 10 μm thick anodic coatings on aluminum.

1.4 The test method is usable as a reference method for the measurement of the thickness of the anodic film on aluminum or of metallic coatings when the technique includes complete stripping of a portion of the coating without attack of the substrate. For anodic films on aluminum, the thickness must be greater than 0.4 μm; the uncertainty can be as great as 0.2 μm. For metallic coatings, the thickness must be greater than 0.25 μm; the uncertainty can be as great as 0.1 μm.

1.5 *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 504 Test Method for Measurement of Thickness of Metallic Coatings by the Coulometric Method[3]

3. Summary of Test Method

3.1 While observing the specimen surface through the interference microscope, the top surface of the coating and the substrate surface are located with white light interference fringe group(s). Then the elevation difference between the two surfaces is ascertained by counting the number of monochromatic fringes by which the white light fringes are displaced. The number of fringes, multiplied by one half of the light wavelength, is the film thickness.

3.2 When light is reflected, it undergoes a phase shift, the magnitude of which depends on the material and on its structure. The uncertainty of the thickness measurement due to this phenomenon is, theoretically, less than ⅛ the wavelength of the light for metals and ¼ wavelength for nonmetallic coatings on metal. Those uncertainties are included in those given in 1.4. They can be eliminated for measurements made in accordance with 1.3 and 7.1.2 by coating the specimen after the stripping operation with a thin but uniform reflective layer of a metal by evaporation. The two reflecting surfaces will then be of the same material and the phase shifts will be the same.

3.3 The aperture of the microscope objective contributes to the fringe displacement by an amount determined by the aperture size. Therefore, a correction[4] is added equal to $\alpha^2/4$ where α, expressed in radians, is the arc sine of the numerical aperture of the microscope objective.

NOTE 1—When the angle is given in radians and is less than 0.6, the angle is approximately equal to its sine.

3.4 With a reticle such as shown in the figures, the fringe count is likely to have an uncertainty of 1⁄10 wavelength (1⁄5 fringe interval). More precise measurements can be made with the aid of a filar micrometer eyepiece.

4. Significance and Use

4.1 The thickness of a coating is often critical to its performance.

4.2 For some coating-substrate combinations, the interference microscope method is a reliable method for measuring coating thickness.

4.3 This test method is suitable for specification acceptance.

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

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[2] Saur, R. L., "New Interference Microscope Techniques for Microtopographic Measurements in the Electroplating Laboratory," *Plating*, PLATA, Vol 52, July 1965, pp. 663–666.

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

[4] Bruce, C. F., and Thornton, B. S., *Journal of Scientific Instruments*, JSINA, Vol 34, 1957, p. 203.

5. Apparatus

5.1 *Interference Microscope* equipped with a reticle or filar micrometer eyepiece for linear measurements.

5.2 *Incandescent and Monochromatic Light Sources.*

6. Sample Preparation for Destructive Technique

6.1 *Anodic Coating on Aluminum*—After masking (Note 2), the coating is stripped by immersion in a solution containing 33 g/L chromic acid (CrO_3) and 0.5 cm^3/L phosphoric acid (H_3PO_4) (85%). Operating temperature is 85 to 95°C.

NOTE 2—Masking for both transparent and opaque coatings can be accomplished by applying an adhesive tape such as 3M #470 or equivalent with its edge at a location where the thickness measurement is desired. The tape must be sufficiently adherent and impervious to protect the coating beneath from subsequent stripping action.

NOTE 3—In certain cases, this method causes attack of the basis metal. The attack is usually accompanied by pitting, which is easily observable in the interference microscope by comparing the general contour exhibited by the fringes on the unstripped portion with the general contour on the stripped portion. If such attack occurs, the method is not valid.

6.2 *Metallic Coatings on Metallic Substrates*—After masking (Note 2), the coating is stripped without attack of the substrate (see Appendix X1).

7. Thickness Measurement

NOTE 4—Many surfaces have microscopical ridges or valleys produced by a previous operation (such as rolling or polishing). Measurements of film thickness are made best with the fringes oriented in a direction perpendicular to the directional surface roughness.

7.1 *Transparent Coatings*:

7.1.1 *Nondestructive Technique:*

7.1.1.1 As the surface of a specimen is viewed through the interference microscope using the incandescent illuminator (white light), adjust the microscope fine-focus knob and the reference mirror controls so that a group of strong fringes (arising from the coating-substrate interface) and a group of weak fringes (arising from the coating-air interface) are both in view as illustrated in Fig. 1A.

7.1.1.2 Determine the number of monochromatic fringes between the centers of the white light fringe groups. Appendix X2 indicates alternative ways of doing this.

7.1.1.3 Calculate thickness T as follows:

$$T = (n\lambda/2\mu)\ [1 + (\alpha^2/4)] \quad (1)$$

where:
n = number of fringes,
λ = wavelength of monochromatic light, µm,
μ = refractive index of coating for light of wave length, λ, and
α = arc sine (numerical aperture of objective) in radians.

Thus for the thickness of the anodic coating on aluminum represented in Fig. 1,

$$T = [(24 \times 0.546)/(2 \times 1.62)]\ [1 + (0.78^2/4)] = 4.66\ \mu m \quad (2)$$

where the monochromatic source is a mercury green light with a wavelength of 0.546 µm, where the refractive index of the anodic coating is 1.62, and where alpha is equal to 0.78.

7.1.2 *Destructive Technique*:

7.1.2.1 Position the boundary between the stripped and unstripped portion of the specimen in the field of view of the microscope.

7.1.2.2 As the surface of the specimen is viewed through the interference microscope using the white light, adjust the microscope fine-focus knob and the reference mirror controls so that the group of fringes arising from the bare substrate and the weak fringes arising from the coating-air interface are both in view, as illustrated in Fig. 2A.

7.1.2.3 Determine the number of monochromatic fringes between the centers of the white light fringe groups. Appendix X2 indicates alternative ways of performing this procedure.

7.1.2.4 Calculate thickness T as follows:

$$T = (n\lambda/2)\ [1 + (\alpha^2/4)] \quad (3)$$

where:
n = number of fringes,
λ = wavelength of monochromatic light, µm, and
α = arc sine (numerical aperture of objective) in radians.

7.2 *Opaque Coatings—Destructive Technique*:

7.2.1 Position the boundary between the stripped and unstripped portions of the specimen in the field of view of the microscope.

7.2.2 As the surface of the specimen is viewed through the interference microscope using the incandescent illuminator, adjust the microscope fine-focus knob and the tilt of the reference mirror so that the fringe group on both sides of the boundary is in the field of view, as illustrated in Fig. 3A.

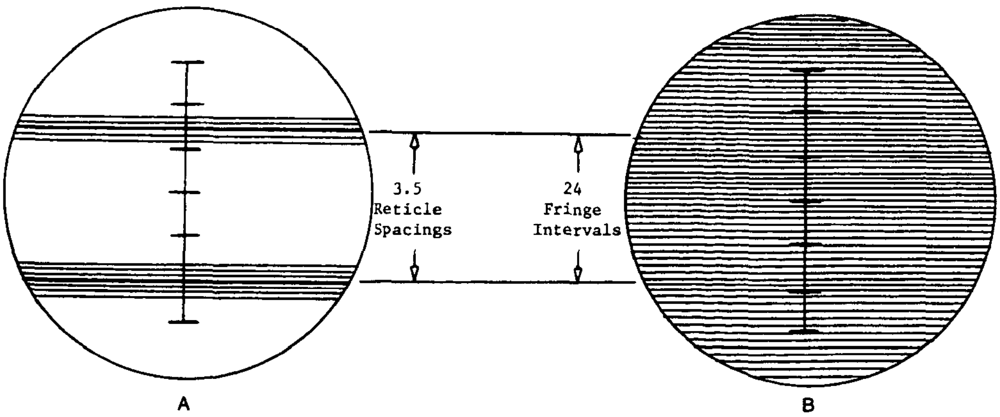

FIG. 1 Anodized Aluminum Surface as Seen Through Interference Microscope Using White (A) or Monochromatic (B) Light

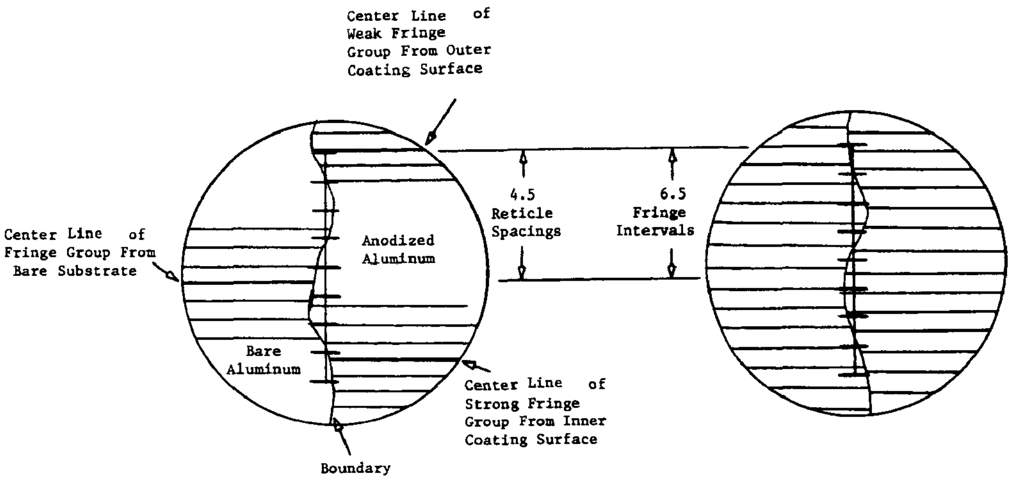

FIG. 2 Fringes Formed on Anodized Surface, on Which the Anodic Coating Has Been Completely Stripped from the Left Portion, as Seen Through an Interference Microscope Using White (A) or Monochromatic (B) Light

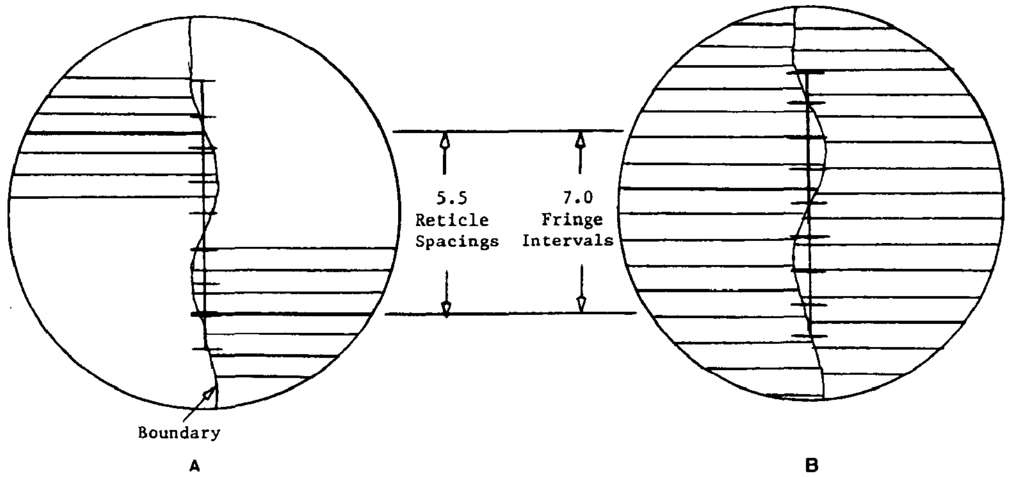

FIG. 3 Nickel-Chromium Boundary as Seen Through Interference Microscope Using White (A) or Monochromatic (B) Light

7.2.3 Determine the number of monochromatic fringes between the centers of the white light fringe groups. Appendix X2 indicates alternative ways of performing this procedure.

7.2.4 Calculate thickness T as follows:

$$T = (n\lambda/2)\,[1 + (\alpha^2/4)] \qquad (4)$$

where:

n = number of fringes,
λ = wavelength of monochromatic light, µm, and
α = arc sine (numerical aperture of objective) in radians.

8. Accuracy Requirement

8.1 *Transparent Coating on Metal Substrate*—The entire procedure shall be such that the coating thickness can be determined either within ±0.2 µm or within 5 % of the coating thickness, whichever is greater.

8.2 *Metal Coating on Metal Substrate*—The entire procedure shall be such that the coating thickness can be determined either within ±0.1 µm or within 5 % of the coating thickness, whichever is greater.

9. Precision and Bias

9.1 A satisfactory interlaboratory comparison of this test method has not yet been conducted.

APPENDIXES

(Nonmandatory Information)

X1. STRIPPING OF METALLIC COATINGS[5]

X1.1 The cell and electronic equipment used for the coulometric method of measuring coating thickness, Method B 504, provides a convenient way of masking and stripping a small area of coating. Chromium coatings may be stripped from nickel or steel by anodic disolution at 5 to 10V in a 5 g/L sodium carbonate (Na_2CO_3) solution using at least a full-wave rectifier filtered with 10 000 μF capacitance.

[5] Saur, R. L., and Basco, R. P., "Power Supply for Anodic Stripping of Chromium on Nickel Electrodeposits," *Plating*, PLATA, Vol 57, July 1970, p. 714.

X2. COUNTING MONOCHROMATIC FRINGES

X2.1 *White Light Fringes*—Chromatic aberrations impose a limit to the way a microscope can be used, and the extent of these aberrations should be determined. With white light and with a specimen and the microscope adjusted so that the central fringe of the color fringes crosses the center of the field, the central fringe usually has a different color near the edge of the field. For example, the central fringe may be black at the center off the field and composed of contiguous red, black, and green layers near the edge. If the fringe pattern is moved perpendicularly across the field, the black central line may become colored and an adjacent line becomes black so that the original central line loses its identity.

X2.1.1 The change is associated with chromatic aberrations that give rise to measurement errors. The operator is advised to scan the field with the central fringe and to note the extent of aberrations. The observations described in the following paragraphs should be confined to those parts of the field within which the central fringe of each fringe group does not change color.

X2.2 For alternative means of measuring the fringe displacement see X2.3, X2.4, X2.5, and X2.6. The methods of X2.4 and X2.5 can be used if chromatic aberrations interfere with the method of X2.3. The method of X2.6 completely avoids any chromatic aberrations, but is difficult to use if the fringe displacement is much more than about five fringes and cannot be used for the nondestructive technique (7.1.1).

X2.3 *Monochromatic Fringes with Stationery White Light Fringes*—The microscope is adjusted as described in 7.1.1.1, 7.1.2.2, or 7.2.2 so that the two groups of color fringes are in the field. The positions of the central fringes on the reticle and the reticle interval between them are noted (Fig. 1(*A*), Fig. 2(*A*), and Fig. 3(*A*)). Monochromatic light is then substituted for the white light without disturbing the specimen or microscope settings, and the monochromatic fringes within the same reticle interval are counted (Fig. 1(*B*), Fig. 2(*B*), and Fig. 3(*B*)).

X2.4 *Monochromatic Fringes with Resetting of White Light Fringes*:

X2.4.1 The microscope is adjusted using white light so that the eyepiece hairline is over the central fringe that locates the coating (or substrate surface), Fig. 4(*A*). Using the fine focus control on the "compensator" control (the compensator adjusts the relative path lengths of the two interferometer beams), the amount of adjustment required to bring the central fringe locating the substrate (or coating) surface to the original position of the first central fringe Fig. 4(*B*), is noted and is estimated (from previous experience) in terms of number of monochromatic fringes. The first position, Fig. 4(*A*), is reinstated, the monochromatic light is substituted for the white light. The monochromatic fringes are shifted with respect to the hairline by the previously estimated number of fringes. Then with white light, it is noted what additional adjustment is required to bring the second central fringe into position, and an

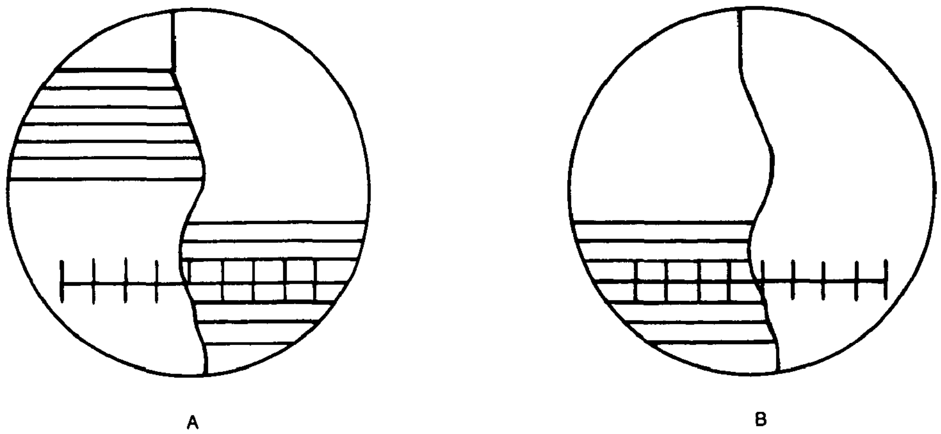

FIG. 4 Coating—Substrate Boundary with Parallel Reticle Using White Light At Beginning (*A*) and End (*B*) of Measurement (X2.3)

improved estimate is made of the total adjustment required in terms of the number of monochromatic fringes. Beginning with reinstatement of the first position, the process is repeated several times until the estimated number of fringes proves to be the adjustment needed to bring the second central fringe into position, Fig. 4(*B*).

X2.4.2 For accurate measurements, the procedure described in X2.4.1 is used to determine the whole number of fringe spacings. Additional displacement of less than one fringe spacing is estimated directly from the monochromatic fringes (Fig. 5).

X2.5 *Stage Elevation*—The microscope is equipped with a means of precisely moving the stage relative to the microscope objective with the movement being calibrated using monochromatic fringes. The movement can be controlled with a cantilever system and micrometer, with a piezoelectric device, or with the fine focus control. One measures the movement required to shift the position of one central fringe to that of the other, Fig. 4.

X2.6 *Beveled Boundary*—If a boundary is formed by stripping part of the coating and if the boundary is beveled so

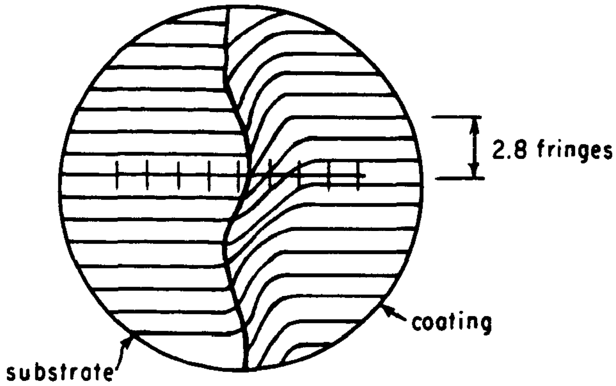

FIG. 5 Beveled Coating—Substrate Boundary With Monochromatic Fringes

that each monochromatic fringe can be followed across the boundary, white light need not be used. The eyepiece hairline is superimposed over one of the monochromatic fringes and one counts the number of fringes it traverses (Fig. 5); that is, the number of fringes by which the fringe pattern is displaced. The method is difficult to use if the displacement is much more than about five fringes.