Designation: B588 – 88 (Reapproved 2006)

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 B588; 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:*[3]

B504 Test Method for Measurement of Thickness of Metallic Coatings by the Coulometric Method

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.

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

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

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.

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.

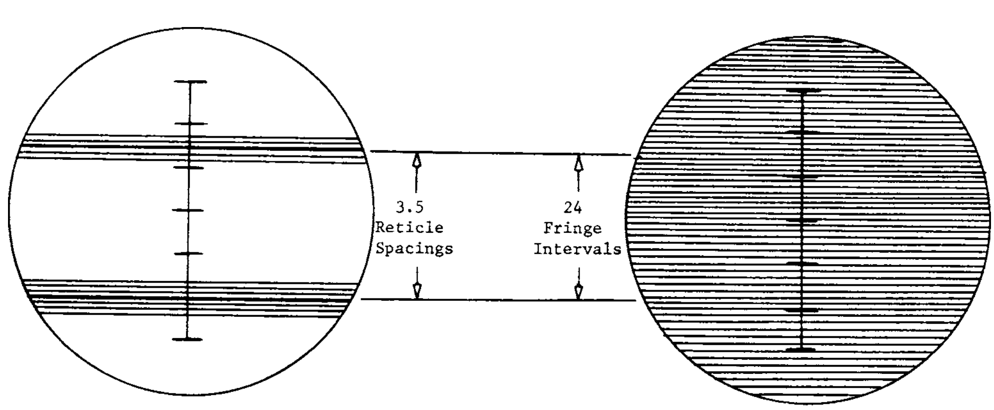

FIG. 1 Anodized Aluminum Surface as Seen Through Interference Microscope Using White (A) or Monochromatic (B) Light