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Standard Test Methods for Thickness of Diffusion Coating¹

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1. Scope

- 1.1 These test methods cover two procedures for measuring the thickness of diffusion coatings.
- 1.2 Test Method A is the determination of the dimensionalchange thickness, defined as the difference in the thickness of the part before and after coating. (The terms micrometer thickness and part growth are considered synonymous with dimensional change thickness.)
- 1.3 Test Method B is the determination of total coating thickness, defined as the distance between the observably unaffected substrate and the exterior surface of the coating. This includes the total of all included phases, zones and layers. (The term case depth is considered to be synonymous with total coating thickness.) The total coating thickness is determined by cross-sectioning the coating, preparing a metallurgical mount and microscopically measuring the coating thickness.
- 1.4 The total coating thickness as determined microscopically from a cross-section will usually be greater than, or equal to, the dimensional change thickness determined by part growth. When the coating is produced primarily by reaction with the substrate, the substrate-coating interface recedes as the substrate is consumed in the reaction. In such cases the difference between the total coating thickness and the dimensional change thickness is the thickness of the substrate consumed.
- 1.5 Diffusion coatings are usually formed at elevated temperatures for service at elevated temperatures. This means that diffusion coatings are dynamic systems which are continually undergoing changes while in an elevated-temperature environment. It is necessary to know that certain phases are growing at the expense of others and to know the previous history of a coating to understand the significance of coating thickness
- 1.6 Values in SI units are to be regarded as the standard. Inch-pound units are provided for information only.
- 1.7 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:
- D 374 Test Methods for Thickness of Solid Electrical Insu-
- E 3 Methods of Preparation of Metallographic Specimens³

3. Significance and Use

3.1 A diffusion coating is one produced by causing an element or elements to react with or diffuse into, or both, the surface of a metallic substrate, thus chemically altering the substrate adjacent to the surface. To appreciate the significance of coating thickness measurements one must understand the contributions to a particular coating of solid-solution zones in the substrate and reaction products such as intermetallic compounds.

4. Test Method A

- 4.1 Apparatus—The instrument shall be a machinist's type micrometer without a locking device. If calibrated in inches, it shall be constructed with a vernier reading to 0.0001 in. (0.1) mil). If calibrated in metric units, it shall be capable of reading to 0.01 mm. It shall have a ratchet or similar mechanism, such as a friction thimble, for controlling measuring pressure and shall have anvil and spindle surfaces 6.00 ± 0.03 mm $(0.250 \pm$ 0.001 in.) in diameter. It shall meet all other requirements and calibration procedure for Method A of Test Method D 374.
 - 4.2 Procedure:
- 4.2.1 Clean the area selected for coating-thickness measurement of dust or other powdery materials prior to coating. Record the precise area to be measured, so that the same area can be remeasured after coating.
- 4.2.2 Take a minimum of two readings, using the machinist's micrometer, in each area selected. Use the same procedure for using the micrometer as that stipulated in Test Methods
- 4.2.3 After the part has been coated and the surface has been cleaned of superficial powder or dirt, repeat the measurement procedure. It is necessary to ensure that the same location is
- 4.2.4 The thickness of the part after coating minus the thickness of the part prior to coating divided by two, is the dimensional change thickness per coated surface.

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² Annual Book of ASTM Standards, Vol 10.02.

³ Annual Book of ASTM Standards, Vol 03.01.

5. Test Method B

- 5.1 Apparatus:
- 5.1.1 Standard Metallurgical Specimen-Preparation Equipment.
- 5.1.2 Metallographic or Optical Microscope.
- 5.1.3 The equipment used shall be consistent as to type and quality as specified in Methods E 3.
 - 5.2 Procedure:
- 5.2.1 Section with a fine grained cut-off wheel the area of the coated part in which the total coating thickness is to be measured. Control the clamping of the part, lubricant cooling fluid, and control the cutting rate to produce a square cut edge without chipping the coating. Exercise care to ensure that the cut is perpendicular to the surface of the coating.
- 5.2.2 After cleaning with an organic solvent, plate the section (electrolytically or electroless) with nickel, chromium, or other metallic plating which approximates the hardness of the coated substrate. As an alternative to plating, wrap the specimen in a metallic foil. The foil applied must give sufficient support to prevent chipping, and be in sufficient proximity to the specimen to prevent gaps and rounding of the edges during polishing.
- 5.2.3 Use the standard techniques detailed in Methods E 3 for mounting and polishing of the metallurgical specimen.
- 5.2.4 The etching procedure, if required, shall be in general accordance with Methods E 3. The specific etchant and etch time, if employed, will be by mutual agreement between the customer and vendor. The etching procedure will clearly demark the boundaries of the coating as well as properly differentiate included zones, if it is desirable to measure their thickness as well.
- 5.2.5 Measure the total coating thickness microscopically with the aid of a calibrated-filar eyepiece, or by direct measurement of a projected image on ground glass. Magnify the coating a minimum of $250\times$ diameters, although $500\times$ is preferable. When measuring from ground glass, measure the image of the coating to the closest 1.27 mm (0.05 in.). The exterior bounds of the coating is the average of the peaks and valleys. Visually average over a minimum length of 10 mils

(0.254 mm (0.010 in.)). Determine the interior bounds of the coating as the depth to which the substrate has been visually altered by the coating. (One must appreciate that this depth is highly dependent upon the etchant employed, and mutual agreement of concerned parties should be sought.) As was the case of the exterior bounds, visually average over a 10 mil (0.254 mm (0.010 in.)) length. Measure the specific zones or phases by a mutual agreement between the purchaser and the vendor. Measure the total coating thickness at a minimum of 10 locations around the entire metallographic specimen with the measurement points separated by at least 20 mils (0.508 mm (0.020 in.)). Average the individual total coating-thickness measurements.

6. Report

- 6.1 The report shall include the following:
- 6.1.1 Thickness as mils per surface. Maximum, minimum, and average of the individual measurements made on a specific part or specimen.
- 6.1.2 Type of coating thickness determined and the method employed,
 - 6.1.3 Specific area where the thickness was measured,
- 6.1.4 Type and condition of the coating such as "as deposited" or "after a specified term of service,"
- 6.1.5 If Test Method B is employed, the etching procedure. A photomicrograph showing the bounds of a typical area of the coating is suggested, and
- 6.1.6 If Test Method B is employed, the thickness of any phases or layers as agreed upon between the purchaser and the seller in mils per surface.

7. Precision and Bias

7.1 Test Method A (dimensional change thickness)— ± 0.2 mil (0.005 mm (0.0002 in.)). This test method assumes that both sides of surfaces of the coated piece have an equal coating thickness, if both surfaces are coated. The validity of this assumption will vary with different coatings and processes of application.

7.2 Test Method B (total coating thickness)— ± 0.1 mil (0.0001 in., 0.0025 mm).

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