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Standard Test Method for Measurement of Surface Layer Thickness by Radial Sectioning[1]

This standard is issued under the fixed designation E 1182; 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.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This test method covers the radial sectioning technique[2,3,4] for measurement of the thickness of thin surface layers, made by a wide variety of processes, on metals, alloys, carbides, and oxides.

1.2 This test method is applicable to measurement of a wide variety of surface layer types where the interface between the layer and substrate is discernible by natural color or reflectivity differences or by means of color or reflectivity differences due to etching or staining.

1.3 This test method does not pertain to layer thickness measurements made by analysis of compositional variations.

1.4 This test method deals only with the recommended test method and nothing in it should be construed as defining or establishing limits of acceptability for any coating method.

1.5 The measurement values stated are in the metric system, as defined in Practice E 380.

1.6 *This standard does not purport to address all of the safety concerns 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.* For specific precautionary statements, see Section 7.

2. Referenced Documents

2.1 *ASTM Standards:*

B 487 Test Method for Measurement of Metal and Oxide Coating Thickness by Microscopical Examination of a Cross Section[5]

E 7 Terminology Relating to Metallography[6]

E 380 Practice for Use of the International System of Units (SI) (the Modernized Metric System)[7]

E 407 Practice for Microetching Metals and Alloys[6]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[7]

F 110 Test Method for Thickness of Epitaxial or Diffused Layers in Silicon by the Angle Lapping and Staining Technique[8]

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in this test method, see Terminology E 7.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *arcuic trigonometric measurement*—method for measuring the thickness of a surface layer using a radial cut of radius *R* through the layer into the substrate and measurement of the widths of the cut at the top of the layer and at the layer-substrate interface.

3.2.2 *radial sectioning*—a machining procedure for producing a precise groove on the surface of a sample to a depth below the layer interface, that is, through a surface layer into the substrate, using a line or spot spindle of known radius.

3.3 *Symbols:Symbols:*

3.3.1 *R*—radius of the machined groove.

3.3.2 W_1— width of the groove at the top surface.

3.3.3 W_2— width of the groove at the layer-substrate interface.

3.3.4 x_t—thickness of the surface layer.

3.3.5 *C*—correlation factor to correct for the deflection of the spindle when the spindle contacts the specimen.

4. Summary of Test Method

4.1 Radial sectioning, using either a line or spot sectioning spindle with a known, constant diameter, is used to cut tangentially into the surface of a coated specimen to a depth below the interface between the surface layer and the substrate.

4.2 The interface between the layer and substrate is revealed

[1] This test method is under the jurisdiction of ASTM Committee E-4 on Metallography and is the direct responsibility of Subcommittee E04.14 on Quantitative Metallography.

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[2] Happ, W. W., and Shockley, W., "Diffusion Depths in Silicon Measured by Using Cylindrical Grooves," *Bulletin of the American Physical Society*, Series II, Vol 1, 1956, p. 382.

[3] McDonald, B., and Goetzuberger, A., "Measurement of the Depth of Diffused Layers in Silicon by the Grooving Method," *Journal of the Electrochemical Society*, Vol 109, February 1962, pp. 141–144.

[4] Whitelam, F. E., "Using Radial Sectioning to Measure Thin Layers," *Metal Progress*, Vol 127, March 1985, pp. 45, 46, 49, and 50.

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

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

[7] *Annual Book of ASTM Standards*, Vol 14.02.

[8] *Annual Book of ASTM Standards*, Vol 10.05.

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by appropriate etching or staining techniques. For certain materials, such as oxide, carbide, or nitride layers, the interface will be clearly visible after radial sectioning.

4.3 The groove is examined using a metallurgical microscope and the widths, W_1 and W_2, are measured using a reticle scale or filar micrometer eyepiece.

4.4 The layer thickness, x_t, is calculated using the following equation:

$$x_t = \left[R^2 - \left(\frac{W_2}{2} - C\right)^2\right]^{1/2} - \left[R^2 - \left(\frac{W_1}{2} - C\right)^2\right]^{1/2} \quad (1)$$

The terms are defined in 3.3.

5. Significance and Use

5.1 Many processes are used to produce a specific type of surface layer on a substrate to produce desired surface properties, such as corrosion resistance, wear resistance, and so forth. Measurement of the thickness of these layers is an important quality control procedure.

5.2 The radial sectioning method is suitable for process control, research, development, and materials acceptance purposes.

5.3 The radial sectioning method and arcuic trigonometric measurement procedure are suited for measurement of surface layers with thicknesses in the range of 0.05 to 200 µm. Thicker layers should be measured by other procedures, such as standard cross sections, as described in Test Method B 487.

5.4 This test method shall not be used as a referee method for layers thinner than 0.5 µm if a more suitable method is available.

5.5 Measurement of the thickness of surface layers is influenced by the smoothness of the substrate and by the uniformity of the layer thickness.

6. Apparatus

6.1 *Line or Spot Spindle*, uniformly coated with a thin layer of abrasive, motor driven to rotate concentrically about its axis within ±0.0127 mm (±0.0005 in.). The abrasive particle size, spindle–binder type, lubricant–coolant type, spindle rpm, and section force are selected to provide the maximum cutting rate and optimum surface finish consistent with the characteristics of the coating and substrate. Typical abrasive particle sizes range from 0.25 to 15.0 µm with a size uniformity of ±33 % for abrasives with a nominal size greater than 1 µm and ±100 % for abrasives smaller than 1 µm. Fig. 1 shows a schematic of the device and the relationship of the specimen to the device.

6.2 *Specimen Holder*, to firmly hold the specimen against the rotating spindle. Holder may be a frame device designed to accommodate a variety of sample shapes and sizes while holding the specimen rigidly.

6.3 *Metallurgical Microscope*, equipped with a measuring reticle or filar micrometer eyepiece, or a toolmaker's microscope.

7. Safety Precautions

7.1 Safety precautions for handling etchants are provided in Test Methods B 487, F 110, and Practice E 407.

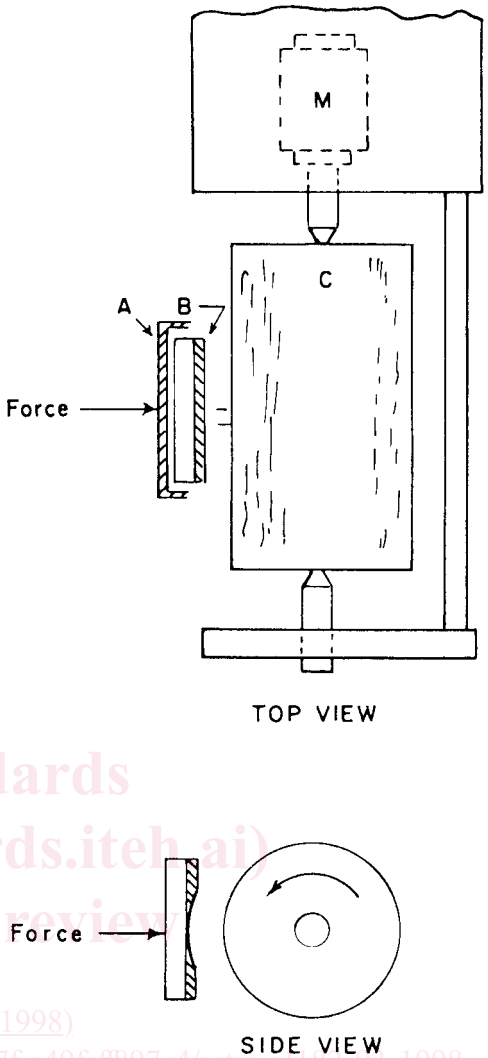

FIG. 1 Schematic Showing the Rotating Spindle (*C*), Drive Motor (*M*), Specimen Holder (*A*) and Specimen (*B*) for Producing Radial Sections in Coated Specimens

8. Sampling, Test Specimens, and Test Units

8.1 The thickness of surface layers and coatings will vary across the specimen. The thickness variability will depend on the coating process and parameters, size and shape of the coated specimen, etc.

8.2 Specimens shall be taken from one or more locations to assess the thickness and its variability. If cutting or shearing is required to obtain the required test specimens, such processes should not alter the surface layer of interest.

8.3 Specimens should be selected from areas that are representative of the bulk sample, are in critical areas, or are at locations where coating uniformity is difficult to obtain, depending on the purpose of the examination.

8.4 The extent of sampling must be guided by good engineering practice so that enough locations are tested to define the thickness without incurring excessive testing costs.

8.5 Specimen surfaces to be tested by radial sectioning shall be cleaned before testing. The cleaning solvents shall not alter the coated surface.

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