INTERNATIONAL

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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 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]

IEEE/ASTM SI 10 Standard for Use of the International System of Units (SI): The Modern Metric System[7]

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:*

3.3.1 x_t—thickness of the surface layer.

3.3.2 R—radius of the machined groove.

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

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

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

3.3.6 n—number of fields measured

3.3.7 s—standard deviation

3.3.8 *95 % CI*—confidence interval

3.3.9 t—a multiplier related to the number of fields examined and used in conjunction with the standard deviation of the measurements to determine the 95% CI.

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[1] This test method is under the jurisdiction of ASTM Committee E04 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.

3.3.10 *% RA*—relative accuracy

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 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.

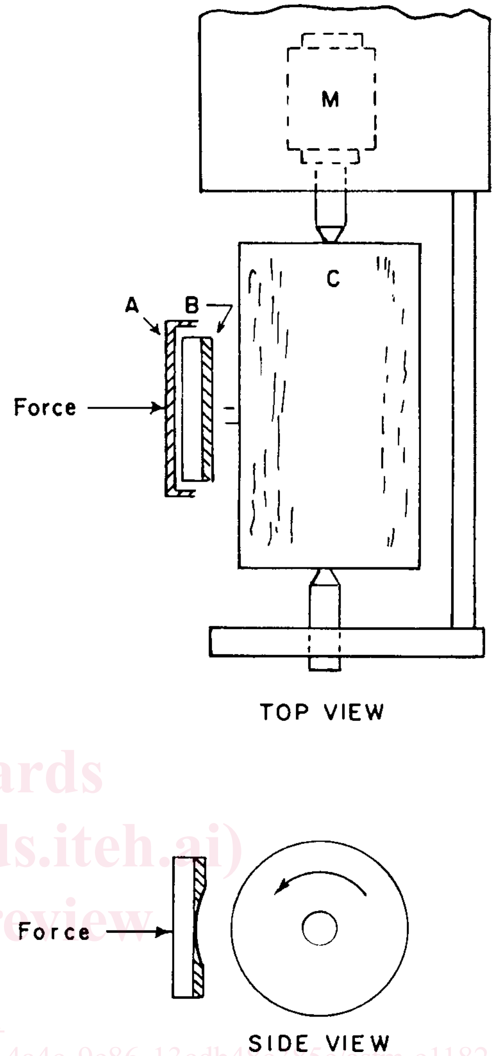


FIG. 1 Schematic Showing the Rotating Spindle (*C*), Drive Motor (*M*), Specimen Holder (*A*) and Specimen (*B*) for Producing Radial Sections in Coated Specimens

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.

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.

9. Calibration and Standardization

9.1 The micrometer eyepiece or recticle scale shall be calibrated with a certified stage micrometer at the same magnification, by the same operator, using the same optics and lighting as used for the measurements. Filtered or monochromatic light shall be used for best precision. The calibration interval on the stage micrometer shall be centered in the field of view and shall be restricted to the center portion of the image.

9.2 The distance between the two lines of the stage micrometer used for the calibration shall be known within 0.2 µm or 0.1 %, whichever is greater.

9.3 Repeated calibrations of the micrometer eyepiece should reveal a spread of measurements of less than 1 %.

9.4 Filar micrometer eyepieces are calibrated in the same manner using a certified stage micrometer.

9.5 To verify that the correlation factor, C, is correct, perform radial section measurements on several specimens. Then, section the test specimens in the grooved regions and measure the coating thickness in the traditional manner with vertical sections, taking care to avoid specimen edge rounding. Compare the test results between the two methods. If there is a consistent bias in the test results, recompute C using (Eq 1) to eliminate the bias.

10. Procedure

10.1 Select the desired line or spot spindle for the desired cutting rate and surface finish.

10.2 Clamp the specimen in a specimen holder compatible with the specimen size and shape.

10.3 Place the specimen holder in the holder support bracket against the rotating abrasive-coated spindle.

10.4 Activate the coolant flow to the spindle.

10.5 Select the appropriate load force.

10.6 Place the specimen against the rotating abrasive-coated spindle for a time sufficient to produce a radial groove of a depth sufficient to penetrate to the substrate.

10.7 Clean the specimen with a suitable solvent to remove all traces of the abrasive compound, grinding swarf, or other contamination.

10.8 Place the specimen under a low-power microscope or stereomicroscope and adjust the illumination to examine the radial groove.

10.9 Select an etchant or staining solution appropriate to the materials being evaluated.

10.9.1 Coated metal or alloy specimens should be etched even if the contrast between the surface layer and the substrate appears to be adequate. Etching will remove any trace of soft metal which may be smeared over a harder metal during radial sectioning and improve definition of the interface boundary.

10.9.2 Etchants must be selected based on the nature of the surface layer, or layers, and the substrate material. Recommended etchants are listed in Test Methods B 487 and F 110.

10.9.3 Etching is usually not required to observe the interface boundary between oxide, carbide and nitride coatings and the substrate.

10.10 Apply a small quantity of the etchant or staining solution to the groove and observe the resultant delineation of the layer interface or interfaces.

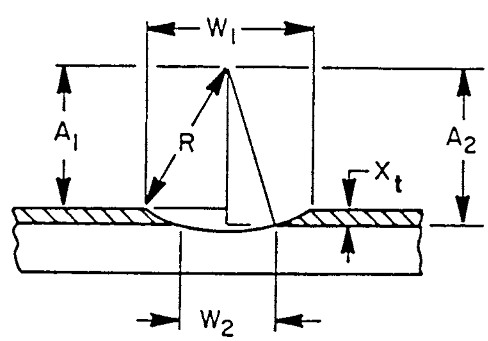
10.11 When the interface or interfaces are clearly defined, remove the specimen and halt the etching or staining action by rinsing the specimen with flowing water. Rinse the specimen with alcohol and dry it with a blast of hot air or compressed air. Reinspect the specimen to ensure that the interface or interfaces are clearly and distinctly revealed. Repeat the etching or staining operation if necessary.

10.12 Place the specimen on the stage of a high quality reflected-light microscope or metallograph, or toolmaker's microscope, fitted with a calibrated measuring eyepiece reticle or filar micrometer eyepiece and adjust the magnification, illumination, and focus. Measure the width of the groove at the top surface of the coated specimen, W_1, and at the coating-substrate interface, W_2, as shown in Fig. 2. For best accuracy, measure W_1 and W_2 at that location in the groove where W_2 is approximately one third as large as W_1.

10.13 Specimens with more than one surface layer can be measured in the same manner to determine the thickness of each layer. For such specimen W_1 is the width of the groove at the top of each layer and W_2 is the width of the groove at the bottom of each layer.

10.14 Using the known radius, R, of the sectioning spindle employed, and the widths, W_1 and W_2, calculate the layer thickness, x_t, using (Eq 1) (see 4.4).

10.15 The widths, W_1 and W_2, will vary somewhat depending on the surface roughness at W_1 and the smoothness of the original substrate at W_2. Several measurements (at least three) of W_1 and W_2 shall be made at different locations along the groove to assess the thickness variability at the test location.



NOTE 1—The thickness x_t is $A_2 - A_1$, that is, the difference in heights of two triangles where the groove radius R is a common hypotenuse. The base of one triangle is $W_1/2$ while the base of the other triangle is $W_2/2$.

FIG. 2 Schematic of the Geometric Principle of the Arcuic Trigonometric Method Used to Determine Layer Thickness Based on Measurements of Radially Machined Grooves

10.16 Repeat the radial sectioning and measuring process at other locations on the specimen and on other specimens from the sample to further document the variability of the layer thickness.

10.17 If the surface of the coated sample is not smooth, measurement of W_1 and W_2 shall be made at locations judged to be representative of the surface, that is, halfway between hills and valleys. If the maximum or minimum thickness of the surface layer is desired, measurements shall be made where the surface layer appears to be thickest or thinnest.

10.18 The number of test locations at which measurements are made, and the number of radial sections measured at each test location, shall be sufficient to ensure statistical confidence that the thickness measurement definition meets the required needs.

11. Calculation

11.1 The mean surface layer thickness at each test location shall be computed based on the measurements of either the average, minimum, or maximum thickness as required for the specific application. The specific $x_{t\,i}$ values made at each radial section at each test location are summed and divided by the number of measurements, n, as follows:

$$\bar{x}_t = \frac{\sum_{i=1}^{n} x_{ti}}{n} \tag{2}$$

where the bar above x_t indicates that the quantity is the average for the specimen. The range of the test values is given by subtracting the smallest x_{ti} value from the largest x_{ti} value.

11.2 The standard deviations of the measurement values x_{ti} for each test location is calculated by:

$$s = \left[\frac{1}{n-1}\sum_{i=1}^{n}(\bar{x}_{ti} - x_t)^2\right]^{1/2} \tag{3}$$

11.3 The 95 % confidence interval, *CI*, is calculated as follows:

$$95\ \%\ CI = \pm\frac{t \cdot s}{\sqrt{n}} \tag{4}$$

where values of t are listed in Table 1 as a function of $n - 1$. The thickness value is expressed as $\bar{x}_t \pm CI$ at each location.

11.4 An estimate of the percentage of error associated with the thickness measurement is obtained as:

$$\%\ \mathrm{RA} = \frac{CI}{\bar{x}_t} \times 100 \tag{5}$$

11.4 If the percentage of error is excessive, more measurements shall be made. To decrease the percentage of error by 50 %, approximately four times the original number of measurements, n, should be measured.

11.5 The calculations described in 11.1 to 11.4 are repeated for each location sampled. The results should be tabulated listing the specimen locations, $\bar{x}_t$, range (optional), *s, 95 % CI*, and % RA (or $\bar{x}_t$ ± *95 % CI* in one column). A grand mean for the sample may be computed based on the $\bar{x}_t$ values at each test location.

TABLE 1 *t* Values for Calculating 95 % Confidence Intervals

$n^A - 1$	t	$n - 1$	t
2	4.303	13	2.160
3	3.182	14	2.145
4	2.776	15	2.131
5	2.571	16	2.120
6	2.447	17	2.110
7	2.365	18	2.101
8	2.306	19	2.093
9	2.262	20	2.086

[A] *n* is the number of measurements.

12. Precision and Bias

12.1 The radial sectioning procedure for measuring the thickness of surface layers is best suited for measurement of thin layers, that is, thicknesses from 0.05 to 200 µm.

12.2 The variability of the surface layer thickness and surface layer roughness will influence the precision and bias of the thickness measurement.

12.3 The sharpness of the interface between the surface layer and the substrate and the contrast difference between the surface layer and substrate will influence the precision and bias of the thickness measurement.

12.4 The microscope variables (nature and quality of the illumination, NA of the objective, magnification, calibration of the measuring device, and the like) will influence the precision and bias of the thickness measurement.

12.5 The spindle radius must be known. Wear of the spindle will change its radius. Periodic inspection of the spindles and measurement of their diameters is required.

12.6 The geometrical magnification increases as the spindle diameter increases. The practical upper limit of the spindle diameter is reached when the effective angle in the groove is so small that rounding of the groove edges is excessive. A spindle diameter of 38.1 ± 0.0254 mm (1.500 ± 0.001 in.) is a good choice for most applications and was used for the study reported in 12.11.

12.7 The number of locations measured on each radial groove and the number of radial grooves made on each test piece will influence the precision and bias of the thickness measurement.

12.8 The difference between W_1 and W_2 is important for accurate measurements. When W_2 is very small, the highest magnification of the layer thickness results but accurate measurement of W_2 is difficult. At the other extreme, a very deep groove produces little layer thickness magnification and measurement accuracy again suffers. The recommended approach is to make measurements at that location in the groove where W_2 is about one-third W_1.

12.9 If the widths W_1 and W_2 vary substantially within the radial groove, the operator's selection of average, minimum or maximum thickness locations for measurement may bias test results due to subjective nature of the choice of W_1 and W_2.

12.10 An interlaboratory round robin was conducted where a number of laboratories measured the same radial grooves on four specimens to determine the plating thicknesses. The results of this study were analyzed in accordance with Practice E 691 and are summarized in Appendix X1.[9]

[9] Supporting data have been filed at ASTM Headquarters. Request RR:E-04-1000.