~~Designation:F1854–01~~ Designation: F 1854 – 09

Standard Test Method for Stereological Evaluation of Porous Coatings on Medical Implants[1]

This standard is issued under the fixed designation F 1854; 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 stereological test methods for characterizing the coating thickness, void content, and mean intercept length of various porous coatings adhering to nonporous substrates.

~~1.2Porous coatings with significant gradients in the porosity from the substrate to the surface will require use of the alternate sample orientation method outlined in~~

1.2 A method to measure void content and intercept length at distinct levels ("tissue interface gradients") through the porous coating thickness is outlined in 9.4.

1.3 The alternate sample orientation method in 8.2 is not suitable for the tissue interface gradients method in 9.4.

~~1.3The values stated in SI units are to be regarded as the standard.~~

~~1.4~~

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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:*[2]

E 3 ~~Practice~~Guide for Preparation of Metallographic Specimens

E 883 Guide for ReflectedLight Photomicrography

3. Terminology

3.1 *Definitions:*

3.1.1 *field*—the portion of image of a ~~portion~~part of the working surface upon which measurements are performed.

3.1.2 *intercept*—the point on a measurement grid line projected on a field where the line crosses from solid to void or vice versa.

3.1.3 *measurement grid lines*—a evenly spaced grid of parallel lines all of the same length.

3.1.4 *porous coating*—coating on an implant deliberately applied to contain void regions with the intent of enhancing the fixation of the implant.

3.1.5 *substrate*—the solid material to which the porous coating is attached.

3.1.6 *substrate interface*—the region where the porous coating is attached to the substrate.

3.1.7 *working surface*—the ground and polished face of the metallographic mount where the measurements are made.

3.1.8 *tissue interface*—the surface of the coating that shall have first contact with biological tissue (that is, the top of the coating).

4. Summary of Test Method

4.1 *Mean Coating Thickness*—~~Measurement grid lines are oriented perpendicular to the substrate interface. On each of the lines in the group, the distance from the substrate interface to the last contact with the porous coating material is measured. The average of the line lengths over the group of lines is the local mean coating thickness.~~ —Evenly spaced parallel grid lines are oriented perpendicular to the coating-substrate interface. For each gridline, the distance from the coating-substrate interface to the last

[1] This test method is under the jurisdiction of ASTM Committee F04 on Medical and Surgical Materials and Devices and is the direct responsibility of Subcommittee F04.15 on Material Test Methods.

~~Current edition approved October 10, 2001. Published February 2002.~~

Current edition approved June 15, 2009. Published August 2009. Originally approved in 1998. Last previous edition approved in 2001 as F 1854 – 01.

[2] 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* ~~, Vol 03.01.~~volume information, refer to the standard's Document Summary page on the ASTM website.

contact with the porous coating material is measured as the coating thickness. The average of all of the coating thickness measurements obtained on a working surface is the mean coating thickness for that working surface.

4.2 *Volume Percent Void*—A regular grid of points is superimposed on a field from the working surface. The percentage of points that are in contact with void areas in the coating correlates with the volume percent of void present.

4.3 *Mean Void Intercept Length*—Measurement grid lines are oriented parallel to the substrate interface. The number of times the lines intercept voids is used with the volume percent void to calculate the mean void intercept length. —Measurement grid lines are oriented parallel to the substrate interface. The average length of the line segments overlaying the void space is the mean void intercept length. This is a representative measure of the scale, or size, of the pores in a porous structure.

4.4 *Tissue Interface Gradients*—The volume percent void and the mean void intercept length are characterized in three 200-µm-thick zones below the tissue interface.

5. Significance and Use

5.1These test methods are recommended for elementary quantification of the stereological properties of porous coatings and the solid substrates to which they are bonded.

5.1 All of these test methods are recommended for elementary quantification of the morphological properties of porous coatings bonded to solid substrates.

5.2 These test methods may be useful for comparative evaluations of different coatings or different lots of the same coating.

5.3All three methods should be performed on the same working surface.

5.4A statistical estimate can be made of the distributions of the mean coating thickness and the volume percent void. No estimate can be made of the distribution of intercept lengths unless the void is a regular geometric shape that can be defined mathematically.

5.3 With the exception of using the alternate mounting method, all the methods should be performed on the same working surfaces. The alternate mounting method can only be used for 9.2 and 9.3.

5.4 A statistical estimate can be made of the distributions of the mean coating thickness and the volume percent void. No estimate can be made of the distribution of intercept lengths.

5.5 There are limits to the accurate characterization of porosity, depending on spacing between the lines in the line grid (or points in the point grid) and the individual and cumulative fields used for the measurements. Increasing the size of the fields, increasing the number of fields, or decreasing the grid spacing will increase the accuracy of the measurements obtained.

5.6 This method is not suitable for ceramic coatings for which accurate coating cross sections cannot be produced using metallographic techniques.

5.7 This test method does not address characterization of coatings having a thickness of less than 300 µm.

6. Apparatus

6.1The procedures outlined in this test method can be performed manually or by an automated procedure. The following apparatus are necessary for manual calculations.

6.1 The procedures outlined in this test method can be performed manually or using digital image analysis techniques.

6.2 *Microscope*, or other suitable device with a viewing screen or photomicrographic capability of at least 125 by 175 mm should be used to image the working surface.

6.3*Transparent Sheet*, with measurement grid lines or points is superimposed on the viewing screen or photomicrograph for the measurements. The grid lines grids of points should be uniformly spaced and parallel with at least five parallel lines. , or other suitable device with a viewing screen, photomicrographic capability, or digital image capture capability should be used to image the sample fields of interest for these test methods.

6.3 For manual measurement, a transparent sheet, with measurement grid lines or points is superimposed on the viewing screen or photomicrograph for the measurements. The line grid (or point grid) and should consist of at least five uniformly spaced, parallel lines (or rows).

7. Metallography

7.1 The procedures outlined in this test method for characterizing porous coatings require the preparation of metallographic sections through the entire coating thickness into the solid substrate.sections. Good metallographic preparation techniques, in accordance with Practice E 3 and Guide E 883must , shall be used to prevent deformation of the surface of the section or creation of any other artifacts that will alter the stereological properties morphology of the metallographic section. An example of an unacceptable artifact would be the absence of a portion of the porous coating, caused by its removal, thereby creating an artificial void area.

7.2 Care must be taken to ensure that the working surface is perpendicular to the interface between the porous coating and substrate interface. When using the solid substrate. In the case of the alternate alternative mounting method shown in 8.4,8.1.2, extreme care must be taken to keep the substrate interface parallel to the final working surface.

8. Sample Selection Sample Working Surfaces and Fields

8.1 *Sample Orientation*:

8.1.1 *Normal Section Orientation* :

~~8.1.1A description of type of sample from which the sample metallographic sections are removed and the location of those sample metallographic sections should be included as part of the results.~~

~~8.1.2For accurate coating thickness measurements, the orientation of sample working surfaces should be approximately perpendicular to the substrate.~~

~~8.2~~

8.1.1.1 For accurate coating thickness measurements, the orientation of sample working surfaces should be approximately perpendicular to the plane of the substrate.

8.1.1.2 If the angle between the tangent to the coating-substrate interface at one edge of a field and the tangent to the substrate interface at the opposite edge of the field is greater than 2º, the substrate curvature is too large.

8.1.1.3 There is a practical limit to the magnification that can be used for measurement of the void content and mean intercept length. As magnification is increased, the number of fields should be increased to obtain a representative sample. If there are too few intercepts in the individual fields, the accuracy of the measurement could decrease.

8.1.2 *Alternative Orientation Method*:

~~8.2.1An alternate orientation is required for the volume percent void and mean intercept length measurements of coatings with a varying amount of porosity in the coating. The porous material and substrate should be mounted with the substrate interface parallel to the metallographic mount surface. The mount is ground parallel to the substrate interface and the measurements are taken at a fixed distance from the substrate interface. It is recommended that the measurements be made at 50% of the mean coating thickness~~

~~8.2.2At least one surface must also be ground on the same mount at 90° to the measurement surface. These will confirm that the interface surface is parallel to the working surface and allow measurement of the distance from the working surface to the substrate. The working surface must be parallel within ±5° of the substrate.~~

~~8.2.3This test method is not suitable for substrate interfaces with a radius of curvature less than 1 in. (25 mm).~~

~~8.2.4Since this test method also requires more aggressive porous surface removal to reach 50% of the mean coating thickness, it may be more susceptible to creation of metallographic artifacts. Care should be exercised to assure that the metallographic sections that are used are free of artifacts.~~

8.1.2.1 An alternate orientation may be used for the volume percent void and mean intercept length measurements. The section should be prepared such that the working surface is parallel to the substrate interface and the measurements should be taken at a fixed distance from the substrate interface. It is recommended that the measurements be made at about 50 % of the mean coating thickness.

8.1.2.2 At least one additional section immediately adjacent to the fields used on the working surface shall also be prepared perpendicular to the working surface. This shall confirm that the substrate interface is parallel to the working surface and allow measurement of the distance from the working surface to the substrate interface.

8.1.2.3 This test method is not suitable for substrate interfaces with a radius of curvature less than 25 mm.

8.1.2.4 Since this test method also requires more aggressive porous surface removal to reach 50 % of the mean coating thickness, it may be more susceptible to creation of metallographic artifacts. Care should be exercised to ensure that the metallographic sections that are used are free of artifacts.

8.2 *Field Parameters*:

8.2.1 *Resolution*:

8.2.1.1 The magnification used for the field should be high enough to resolve all the features that need to be measured.

8.2.1.2 For most porous coatings, the magnification should be high enough that features as small as 5 µm can be easily distinguished. If digital imaging is used, the pixel size should be less than or equal to 5 µm.

8.2.2 *Field Dimensions*:

8.2.2.1 The field height must include the full thickness of the porous coating for mean coating thickness (9.1).

8.2.2.2 A good rule of thumb for an accurate measurement of mean void intercept length is that the minimum field width should be greater than or equal to 5× the resulting mean void intercept length. For example, a mean void intercept length value of 200 µm should have a measurement field width of at least 1000 µm.

8.2.2.3 It is possible to measure the mean void intercept length in a field using a series of shorter non-overlapping grid lines. This does not change the requirement for the number of fields required for the calculation. Care should be exercised using multiple short lines in a single field, because it may be possible to make the grid lines so short that the accuracy of the result is affected.

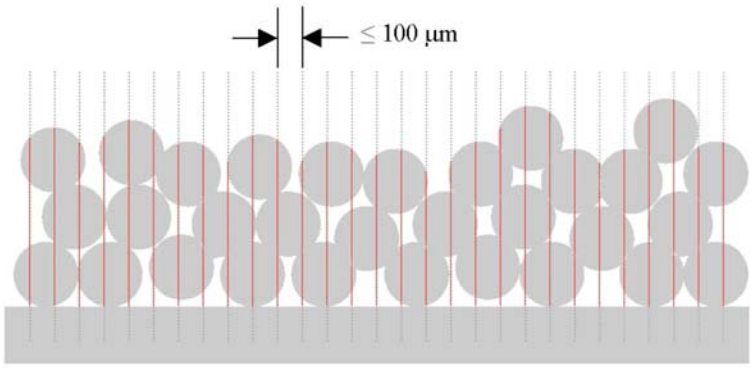
8.2.2.4 If the magnification used produces an image with a height or width smaller than that which is required, multiple images may be carefully stitched together to produce a field of sufficient height and width.

9. Procedure

9.1 *Mean Coating Thickness*:

~~9.1.1~~

9.1.1 An array of equally spaced parallel gridlines should be superimposed on the field perpendicular to the substrate interface, as shown in Fig. 1. The gridlines should be spaced no more than 100 µm apart. Appendix X2 ~~includes two sets of typical measurement grids each with ten measurement lines, one set separated by a distance of 1 cm and one set separated by a distance of 0.5 cm.~~



NOTE—The solid line is the measured distance.

FIG. 1 Illustration of Coating Thickness Measurement

~~9.1.2The magnification should be chosen such that the thickness of the coating in the image is at least 4 cm.~~

~~9.1.3The transparency of the measurement grid lines is superimposed on the field in an orientation approximately perpendicular to the substrate interface. The length of each line between the substrate interface to the last contact with the porous coating is measured on each line.~~

~~9.1.4If the angle between the substrate interface and any individual line of the group of parallel lines is not between 80 and 100°, the individual measurement is invalid.~~

~~9.1.5The procedure is repeated on a minimum of ten unique fields on each working surface.~~

~~9.1.6The average of all the measurements is the mean coating thickness for that working surface. The standard deviation estimator and the 95% confidence interval should be calculated for each working surface. The equations for calculating these values are as follows:~~ includes two typical sets of gridlines each with ten equally spaced parallel lines.

9.1.2 At each gridline, the distance from the substrate interface to the last contact with a solid coating feature is measured. A measurement is only valid if the gridline is oriented 90 ± 2° to the substrate interface.

9.1.3 Coating thickness measurements should be obtained over a continuous linear distance of at least 10 mm of porous surface with no overlap between measurement sites.

9.1.4 The average of all the measurements is the mean coating thickness for that working surface. The standard deviation estimator and the 95 % confidence interval should be calculated for each working surface. The equations for calculating these values are as follows:

$$\bar{T} = \frac{1}{Mxn}\sum_{i=1}^{n} t_i \tag{1}$$

$$\bar{T} = \frac{1}{M \times n}\sum_{i=1}^{n} t_i \tag{1}$$

where:

t_i = the individual magnified thickness line length,

n = the number of thickness measurements,

M = the magnification, and

$\bar{T}$ = the mean coating thickness.

$$\hat{S} = \sqrt{\frac{1}{n-1}\sum_{i=1}^{n}\left[\frac{t_i}{M_i} - \bar{T}\right]^2} \tag{2}$$

$$\hat{S} = \sqrt{\frac{1}{n-1} \times \sum_{i=1}^{n}\left[\frac{t_i}{M} - \bar{T}\right]} \tag{2}$$

~~S~~

where:

~~S~~$\hat{S}$ = ~~the standard deviation estimator.~~

$$CI = 2\times \frac{\hat{S}}{\sqrt{n}} \tag{3}$$

~~where:~~ the standard deviation estimator, and
CI = the confidence interval.

~~9.2~~

$$CI = 2 \times \frac{\hat{S}}{\sqrt{n}} \quad (3)$$

9.2 *Volume Percent Void*:

~~9.2.1X2.1 includes three different typical sets of 100-point grids. Two of the grids are spaced 10 by 10 (1.0 by 1.0 and 0.5- by 1.0-cm separation) and the other is a 20 by 5 grid (0.5- by 1.0-cm separation).~~

~~9.2.2If the void areas form a regular or periodic pattern on the working surface, the use of a grid having a similar pattern should be avoided.~~

~~9.2.3The transparency is superimposed randomly on a fields. The magnification of the field is chosen such that one of the available grids can fit completely within the porous coating in the working surface.~~

~~9.2.4Grid height must be greater than 50% of the mean coating thickness. For coatings consisting of regularly shaped particles, or those having porosity gradients, care must be taken not to influence results by nonrandom grid placement.~~

~~9.2.5The number of points in contact with void areas in the working surface will be counted and recorded.~~

~~9.2.6Count any points falling on a boundary between void area and solid area as one half. Any doubtful point should be counted as one half.~~

~~9.2.7The number of contact points, divided by the total number of points on the grid times 100 gives the percentage of grid points on the void for that field. This should be calculated for each grid application.~~

9.2.1 For this measurement, the field should be entirely contained between the tissue interface (see 9.4.1) and the substrate interface.

9.2.2 An array containing at least 100 regularly spaced points should be superimposed on the field, as shown in Fig. 2. The points should be spaced no more than 50 µm apart. If the void areas form a regular or periodic pattern, the use of a grid having a similar pattern should be avoided. The height of the array should be at least half the distance from the tissue interface to the substrate interface, thereby producing a value representative of an average for the entire coating thickness. Appendix X2 includes two typical arrays each with at least 100 regularly spaced points.

9.2.3 The number of points overlying void areas (Pα) on the working surface shall be counted and recorded. When using the manual method, any points falling on a boundary between a void area and solid features should be counted as one half. Any questionable points should be counted as one half.

9.2.4 The number of contact points in void area (P_α), divided by the total number of points on the grid (P_T) times 100 gives the percentage of grid points on the void for that field. This should be calculated for each grid application.

$$P_v = \frac{P_\alpha}{P_T} \times 100 \quad (4)$$

Pα

where:

~~*P(α)*~~ P_α = the total number of counted points,
~~*P(T)*~~ P_T = the total number of grid points, and
~~*P(v)*~~ P_v = the volume percent void.

~~9.2.8A minimum of 30 fields should be measured per working surface.~~

~~9.2.9Fields to be analyzed should include as much of the coating thickness as possible. When there are porosity gradients are~~

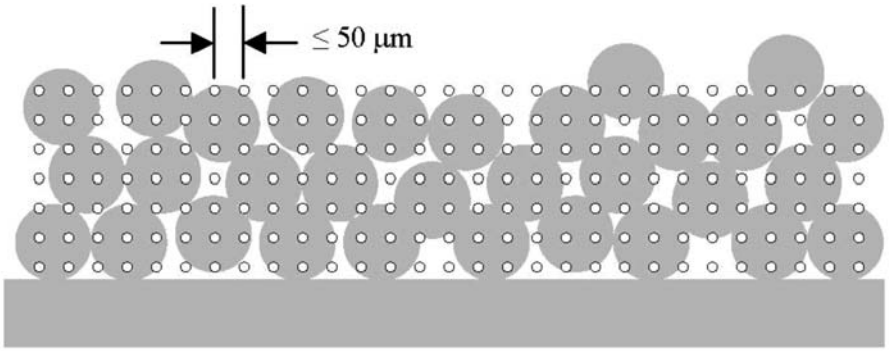


FIG. 2 Illustration of Volume Percent Void Measurement

~~present, the accuracy of the measurement is related to the amount and repeatability of gradient in the portion of the coating section analyzed in each field.~~

~~9.2.10The average percentage of the grid points on the voids provides an unbiased statistical estimator for the void volume percentage in the three dimensional structure. The mean void percentage (P~~

9.2.5 Volume percent voids should be measured over an area of the working surface totaling at least 15 mm^2 with no part of that area being measured more than once. If the coating thickness is below 500 µm, at least 3 cm of coating length must be used for the void measurement.

9.2.6 Fields to be analyzed should include as much of the coating thickness as possible.

9.2.7 These measurements may also be made with an appropriate digital image analysis system. This can be done by considering each pixel as a regularly spaced point in the array. The volume percent void for each field should be the ratio of the number of pixels representing void space to the total number of pixels in the image of the field.

9.2.8 The average percentage of the grid points on the voids provides an unbiased statistical estimator for the void volume percentage in the three dimensional structure. The mean void percentage ($\bar{P}_v$) for that working surface, the standard deviation estimator (~~*S*~~$\hat{S}$) and the 95 % confidence interval (*CI*) should be calculated for each working surface. The equations for calculating these values are as follows:

$$\bar{P}_v = \frac{1}{n}\sum_{i=1}^{n} P_{v_i} \tag{5}$$

$$\hat{S} = \sqrt{\frac{1}{n-1}\sum_{i=1}^{n}[P_{v_i} - \bar{P}_v]^2} \tag{6}$$

$$\hat{S} = \sqrt{\frac{1}{n-1}\times\sum_{i=1}^{n}[P_{v_i} - \bar{P}_v]} \tag{6}$$

$$CI = 2\times\frac{\hat{S}}{\sqrt{n}} \tag{7}$$

~~9.2.11The~~

9.2.9 The volume percent void estimate is given by the following relationship:

(8) $V v = Pv$

9.3 *Mean Intercept Length*

9.3.1 F1854-09_8

9.3 *Mean Void Intercept Length*:

9.3.1 For this measurement, the field should be entirely contained between the tissue interface (see 9.4.1) and the substrate interface.

9.3.2 An array of equally spaced parallel gridlines should be superimposed on the field parallel to the substrate interface, as shown in Fig. 3. The height of the array should be at least half the distance from the tissue interface to the substrate interface, thereby producing a value representative of an average for the entire coating thickness. The gridlines should be spaced no more than 100 µm apart. Appendix X2 ~~includes two sets of ten measurement lines, one set separated by a distance of 1 cm and one set separated by a distance of 0.5 cm. A transparency can be made of a group for manual work.~~

~~9.3.2The transparency is superimposed on each of the fields. The measurement line grid should fit completely within the porous coating cross section.~~

~~9.3.3The magnification will be chosen such that the number of counted intercepts per line should average five or more.~~

~~9.3.4The number of times that a void region is intercepted by the test lines (~~*includes two typical sets of gridlines each with ten equally spaced parallel lines.*

9.3.3 The number of times that a void region is intercepted by the test lines (N_v) is counted and recorded. There are two methods that can be used for counting.

~~9.3.4.1The~~9.3.3.1 The first method counts the number of intersections along the grid lines. Each time the grid line goes from

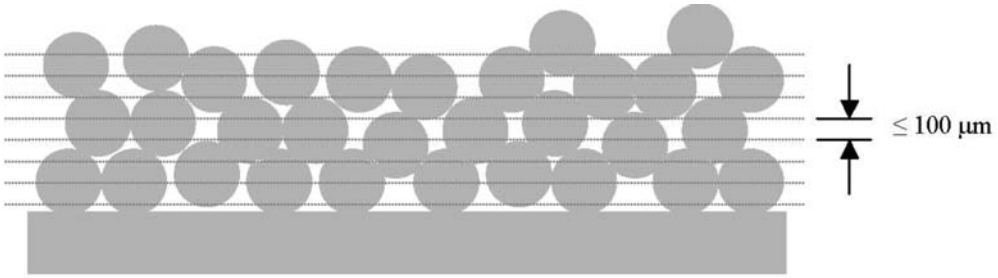


FIG. 3 Illustration of Mean Intercept Length Measurement Field