Designation: F 1854 – 01

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.

1.2 Porous coatings with significant gradients in the porosity from the substrate to the surface will require use of the alternate sample orientation method outlined in 8.2.

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

1.4 *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:*

E 3 Practice for Preparation of Metallographic Specimens[2]

E 883 Guide for Reflected Light Photomicrography[2]

3. Terminology

3.1 *Definitions:*

3.1.1 *field*—the image of a portion 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.

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.

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.

5. Significance and Use

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

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

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

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 unless the void is a regular geometric shape that can be defined mathematically.

6. Apparatus

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

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

[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.

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

should be uniformly spaced and parallel with at least five parallel lines.

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. Good metallographic preparation techniques, in accordance with Practice E 3 and Guide E 883 must be used to prevent deformation of the surface of the section or creation of any other artifacts that will alter the stereological properties 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 the solid substrate. In the case of the alternate mounting method shown in 8.4, extreme care must be taken to keep the substrate interface parallel to the final working surface.

8. Sample Selection

8.1 *Normal Section Orientation*:

8.1.1 A 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.

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

8.2 *Alternative Orientation Method*:

8.2.1 An 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

8.2.2 At 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.

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

8.2.4 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 assure that the metallographic sections that are used are free of artifacts.

9. Procedure

9.1 *Mean Coating Thickness*:

9.1.1 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.

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

9.1.3 The 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.

9.1.4 If 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.

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

9.1.6 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:

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

where:
t_i = the individual magnified thickness line length,
M = the magnification, and
T = the mean coating thickness.

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

where:
S = the standard deviation estimator.

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

where:
CI = the confidence interval.

9.2 *Volume Percent Void*:

9.2.1 X2.1 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).

9.2.2 If 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.

9.2.3 The 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.

9.2.4 Grid 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.

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

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

9.2.7 The 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.

$$P_v = \frac{P_\alpha}{P_T} \times 100 \tag{4}$$

where:
$P(\alpha)$ = the total number of counted points,
$P(T)$ = the total number of grid points, and
$P(v)$ = the volume percent void.

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

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

9.2.10 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 (P_v) for that working surface, the standard deviation estimator (S) and the 95 % confidence interval (CI) should be calculated for each working surface. The equations for calculating these values are as follows:

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

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

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

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

$$V_v = P_v \tag{8}$$

9.3 *Mean Intercept Length*

9.3.1 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.

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

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

9.3.4 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.

9.3.4.1 The first method counts the number of intersections along the grid lines. Each time the grid line goes from either solid to void or void to solid is counted as one intersection. The number of intersections (n_i) is twice the number of intercepts (N_v).

$$N_v = \frac{n_1}{2} \tag{9}$$

9.3.4.2 In the second method the crossing direction of any intersection on any line determines if it is counted as an intercept. If the beginning of the line starts on a void, count the transitions from void to solid. If that same line ends on a void, count that as a one more intercept. If the beginning of a line starts on solid, count the transitions from solid to void. If the same line ends on a solid, there is no additional count.

9.3.5 At least ten different fields should be counted on each working surface.

9.3.6 For the alternative method, the orientation of the grid should be random.

9.3.7 The mean intercept length (L_v) can be calculated from the total length of lines (L_T), the number of intercepts (N_v), the magnification M, and the previously calculated volume percent void (V_v). The calculation is as follows:

$$L_v = \frac{V_v \times \frac{L_t}{M}}{N_v} \tag{10}$$

10. Report

10.1 Report the chemical composition of the substrate structure and the coated material.

10.2 The original morphological form of the coated material (that is, powder, wire, and so forth) and the method of application will be reported.

10.3 The number of fields of view for each measurement, the magnifications used, and the grids and lines used shall be reported.

10.4 The mean coating thickness and confidence interval, the volume percent void and confidence interval, and the mean linear intercept will be reported.

10.5 If curved substrates are used, that information must be included in the report, along with an estimate of the local radius of curvature.

10.6 If the alternative orientation method is used, that information must be reported together with the distance from the substrate for each set of measurements.

10.7 For image analysis systems that measure on digitized video images, the minimum measurement value, that is, the dimension of a single pixel, must be reported at all magnifications used in the measurements.

11. Precision and Bias

11.1 The following factors could significantly affect the results obtained using this method:

11.1.1 The presence of structural gradients or inhomogeneities in the section can influence the precision and accuracy of the measurements. If the amount of void in the porous coating changes with thickness, the alternative mounting method and serial grinding may be used to characterize the gradient.

11.1.2 The quality of sample preparation can influence precision and bias.

11.1.3 The counting of grid points and line ends at void boundaries presents an opportunity for bias in the measurements. The method outlined in 9.3.4.1 and 9.3.4.2 must be used.

11.1.4 The number of fields measured, the method of field selection, and their spacing can influence the precision and accuracy. Random selection of fields and, except where noted, random orientation of grids within the field can help eliminate bias.

11.2 The results of an interlaboratory comparison, although not comprehensive enough to support statistical statements about the precision of this method, are presented in Appendix X4 for information only.

12. Keywords

12.1 metallography; porosity; porous coatings; stereology; thickness

APPENDIXES

(Nonmandatory Information)

X1. RATIONALE

X1.1 Porous coatings are applied to the surface of medical implants. Standardized techniques should be available to characterize the structure of the porous coatings. These techniques can be used to estimate the uniformity and repeatability of these porous structures.

X1.2 Porous coatings with significant gradients in the porosity from the substrate to the surface may not be accurately measurable using these test methods.

X2. SAMPLE ARRAYS OF LINES FOR LINE INTERCEPT WORK

X2.1 See Fig. X2.1.

X3. ARRAYS OF POINTS FOR POINT COUNTING METHODS

X3.1 See Fig. X3.1.

X4. PRECISION AND BIAS

X4.1 *Review of the Round Robin*—Ten laboratories were involved with the round robin. Each laboratory was provided with a copy of six photomicrographs of a sintered titanium bead coating on a Ti-6Al-4V solid substrate. The photomicrographs were taken, at random, at an angle of 90° to the substrate, from six, pocketed disk-shaped samples from a powder-coating vendor. The side walls of the pockets were never included in the micrographs. Each participant was instructed to evaluate three fields from each photomicrograph. One of the laboratory participants only provided thickness measurement data.

The participants reported only the average result ($N = 18$) for each parameter measured, so that within-laboratory repeatability cannot be determined. The following Table X4.1 may provided some guidance as to the between-laboratory reproducibility.

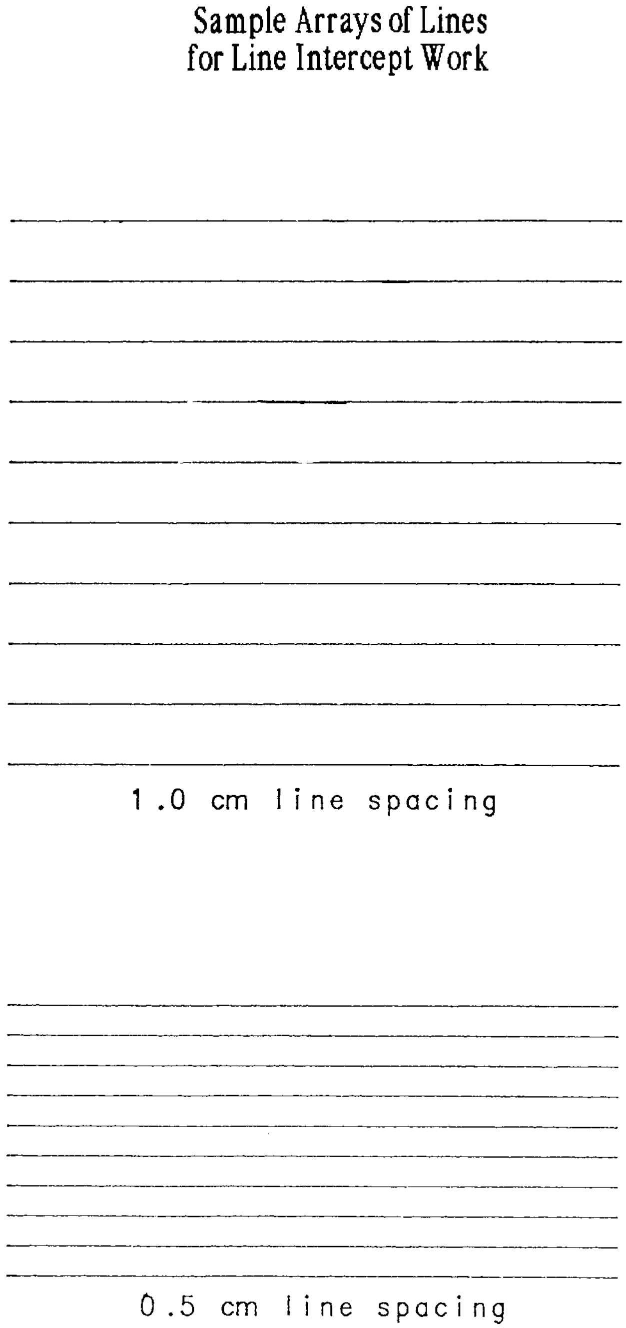


FIG. X2.1 Sample Arrays of Lines for Line Intercept Work

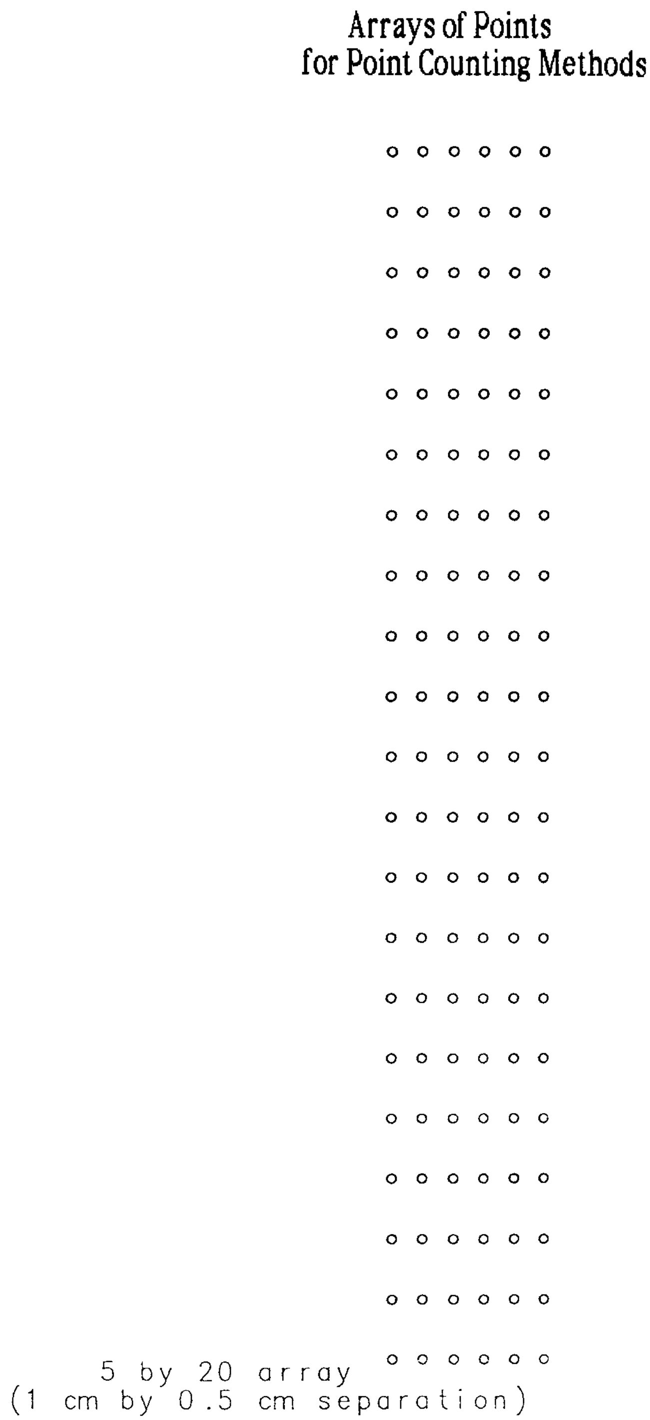
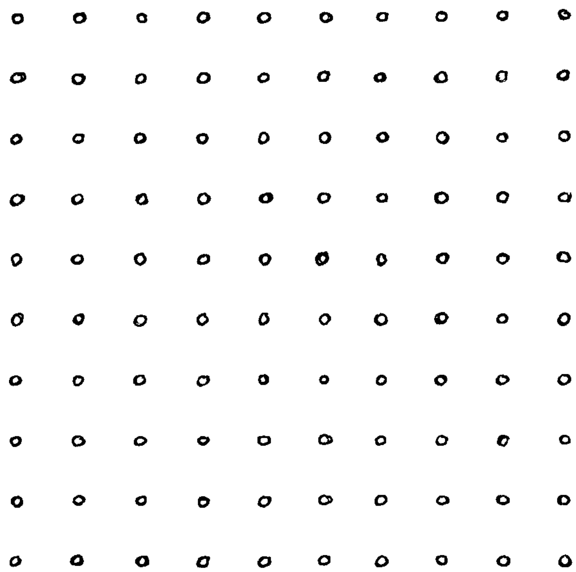


FIG. X3.1 Array of Points for Point Counting Methods

Arrays of Points
for Point Counting Methods



10 by 10 array (1 cm separation)

10 by 10 array
(1 cm by 0.5 cm separation)

FIG. X3.1 Array of Points for Point Counting Methods *(continued)*

TABLE X4.1 Results from ASTM Stereology Round Robin

Participant	Thickness	Volume % Void	Mean Intercept Length
A	55.32	36.66	6.50
B	55.28	35.55	6.28
C	54.72	35.30	6.83
D	55.86	33.50	5.31
E	55.50	34.70	7.05
F	56.26	36.25	6.46
G	55.94	34.98	7.92
H	56.40	36.91	7.60
I	54.91	34.49	6.80
J	54.56		
Mean	55.537	35.371	6.929
Standard deviation	0.641	1.102	0.574

BIBLIOGRAPHY

(**1**) *Practical Applications of Quantitative Metallography, ASTM STP 839*, 1984.
(**2**) *Stereology and Quantitative Metallography, ASTM STP 504*, 1972.
(**3**) Underwood, E. E., *Quantitative Stereology*, Addison-Wesley Publishing Company, 1970.
(**4**) Vandervoort, G., *Metallography, Principles and Practice*, McGraw-Hill, New York, 1984.