Designation: F 1659 – 95

Standard Test Method for Bending and Shear Fatigue Testing of Calcium Phosphate Coatings on Solid Metallic Substrates[1]

This standard is issued under the fixed designation F 1659; 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 the procedure for the performance of calcium phosphate ceramic coatings in shear and bending fatigue modes. In the shear fatigue mode this test method evaluates the adhesive and cohesive properties of the coating on a metallic substrate. In the bending fatigue mode, this test method evaluates both the adhesion of the coating as well as the effects that the coating may have on the substrate material. These test methods are limited to testing in air at ambient temperature. These test methods are not intended for application in fatigue tests of components or devices; however, the test method that most closely replicates the actual loading configuration is preferred.

1.2 The values stated in SI units are to be regarded as the standard. The inch-pound units given in parentheses are for information only.

1.3 *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 6 Terminology Relating to Methods of Mechanical Testing[2]

E 206 Definitions of Terms Relating to Fatigue Testing and the Statistical Analysis of Fatigue Data[3]

E 466 Practice for Constant Amplitude Axial Fatigue Tests of Metallic Materials[2]

E 467 Practice for Verification of Constant Amplitude Dynamic Loads in an Axial Load Fatigue Testing Machine[2]

E 468 Practice for Presentation of Constant Amplitude Fatigue Test Results for Metallic Materials[2]

3. Terminology

3.1 *Definitions:*

3.1.1 The definitions of terms relating to shear and fatigue testing appearing in Terminology E 6 and Terminology E 206 shall be considered as applying to the terms used in this test method.

4. Summary of Test Methods

4.1 *Shear Fatigue Testing*:

4.1.1 The intent of the shear fatigue test is to determine the adhesive or cohesive strength of the coating, or both.

4.1.2 This test is designed to allow the coating to fail at either the coating/substrate interface, within the coating, or at the glue/coating interface.

4.2 *Bending Fatigue Testing*:

4.2.1 The intent of the bending fatigue test is to quantify the effect that the coating has on the substrate to which it is applied. It may also be used to provide a subjective evaluation of coating adhesion (that is, spalling resistance, cracking resistance, etc.).

4.2.2 This test method is designed to first provide a substrate fatigue strength to serve as a baseline to assess the effects of the coating on the resulting fatigue strength of the system.

5. Significance and Use

5.1 The shear and bending fatigue tests are used to determine the effect of variations in material, geometry, surface condition, stress, etc., on the fatigue resistance of calcium phosphate coated metallic materials subjected to direct stress for up to 10^7 cycles. These tests may be used as a relative guide to the selection of calcium phosphate coated materials for service under conditions of repeated stress.

5.2 In order that such basic fatigue data be comparable, reproducible, and can be correlated among laboratories, it is essential that uniform fatigue practices be established.

5.3 The results of the fatigue test may be used for basic material property design. Actual components should not be tested using these test methods.

6. Equipment Characteristics

6.1 Equipment characteristics shall be in accordance with Section 7 on Adhesive Bonding Materials of Practice E 466.

6.2 *Shear Fatigue Test Grips*:

6.2.1 *General*—Various types of grips may be used to transmit the load to the specimens by the testing machine. To ensure axial shear stress, it is important that the specimen axis coincide with the centerline of the heads of the testing machine

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[1] This test method is under the jurisdiction of ASTM Committee F-4 on Medical and Surgical Materials and Devices and is the direct responsibility of Subcommittee F04.13 on Ceramic Materials.

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

[3] Discontinued; see *1987 Annual Book of ASTM Standards*, Vol 03.01.

and that the coating test plane be parallel to the axial load. Any departure from this requirement (that is, any eccentric loading) will introduce bending stresses that are not included in the usual stress calculation (force/cross-sectional area).

6.2.2 A drawing of a typical gripping device for the test assembly is shown in Fig. 1.

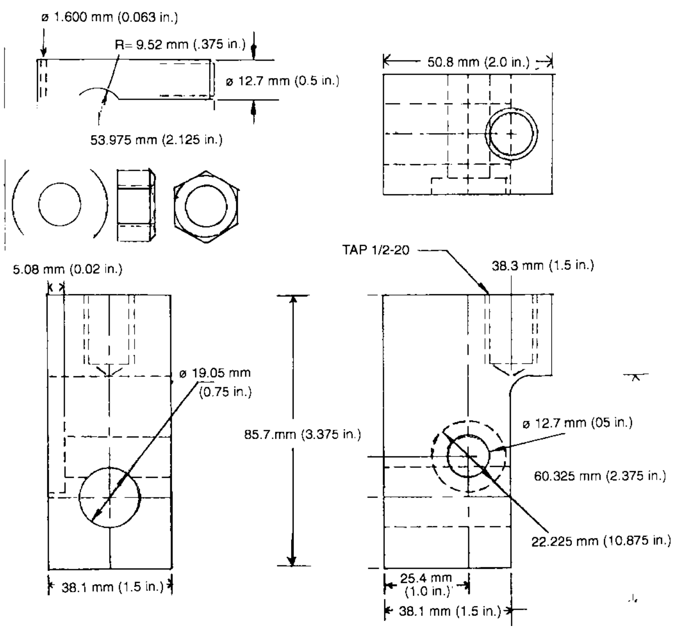

FIG. 1 Gripping Device for Shear Testing

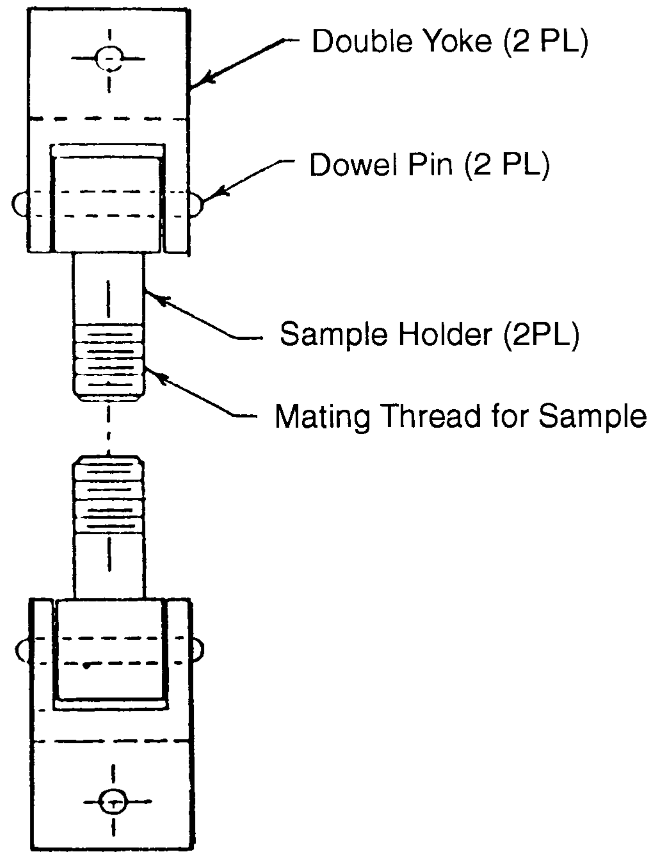

FIG. 2 Adaptor to Mate the Gripping Device to the Tensile Machine

6.2.3 Fig. 2 shows a drawing of the adaptor to mate the shear fixture to the tensile machine.

6.2.4 Fig. 3 shows a schematic of the test setup.

6.3 *Bending Fatigue Test Grips*—There are a variety of testing machines that may be employed for this test (that is, rotating beam fatigue machines and axial fatigue machines). The gripping method for each type of equipment shall be determined by either the manufacturer of that equipment (rotating beam machines) or the user.

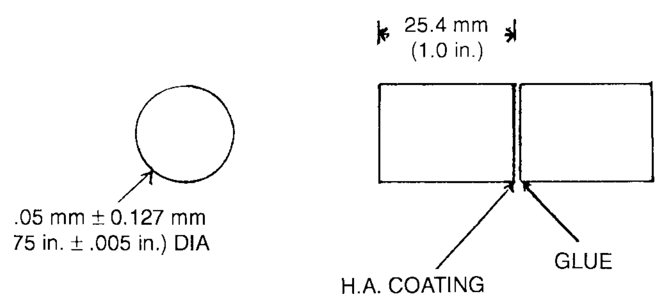

FIG. 3 Schematic of the Shear Test Set-up

7. Adhesive Bonding Materials

7.1 *Adhesive Bonding Agent*—A polymeric adhesive bonding agent in film form, or filled viscous adhesive cement, shall be identified and shall meet the following requirements.

7.1.1 The bonding agent shall be capable of bonding the coating on the test specimen components with an adhesive shear strength that is at least 34.5 MPa (5000 psi) or as great as the minimum required adhesion or cohesion strength of the coating.

7.1.2 In instances where coating porosity extends to the coating/substrate interface, the bonding agent shall be sufficiently viscous and application to the coating sufficiently detailed, to assure that it will not penetrate through the coating to the substrate. The FM 1000 Adhesive Film[4] with a thickness of 0.25 mm (0.01 in.) has proven satisfactory for this test.

7.1.3 If a material other than FM 1000[4] is used, or the condition of the FM 1000[4] is unknown, it must be tested to establish its equivalence fresh FM 1000.[4] Testing should be performed without the presence of the calcium phosphate coating to establish the performance of the adhesive.

8. Test Specimens

8.1 *Shear Fatigue Specimen*:

8.1.1 The recommended shear test specimen and setup is illustrated in Fig. 3 and Fig. 4, respectively. A complete, assembled test assembly, consists of two solid pieces: one with a coated surface and the other with an uncoated surface. The uncoated surface may be roughened to aid in the adhesion of the adhesive bonding agent.

8.1.2 The cross-sectional area of the substrate upon which the coating is applied shall be a nominal 2.85 cm^2 (0.44 $in.^2$).

[4] Available from American Cyanamid, Engineering Materials Division, Wayne, New Jersey.

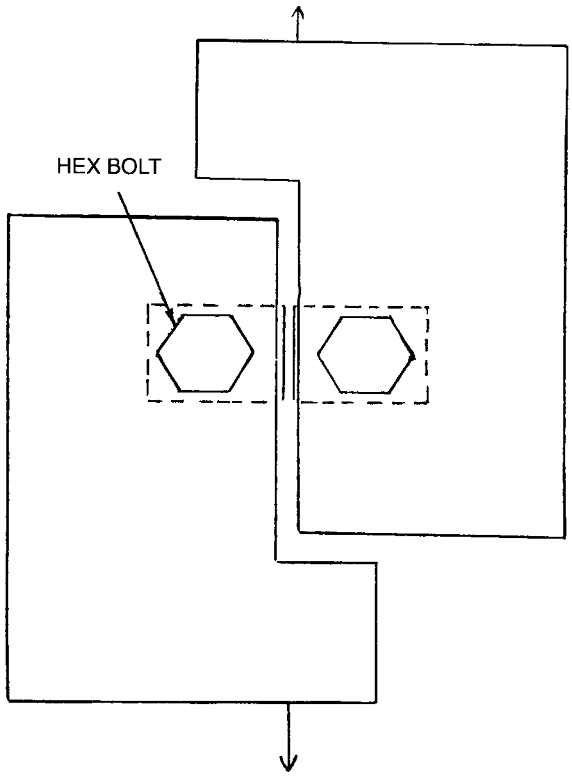

FIG. 4 Drawing of the Recommended Shear Test Specimen Assembly

When specimens of another cross-sectional area are used, the data must be demonstrated to be equivalent to the results produced using the 2.85 cm^2 standard cross-sectional area and the specimen size should be reported.

8.2 *Bending Fatigue Specimen*:

8.2.1 The type of specimen used will depend upon the objective of the test program, the type of equipment, the equipment capacity, and the form in which the material is available. The design, however, must meet certain general criteria as follows:

8.2.1.1 The design of the specimen should be such that if specimen failure should occur, it should occur in the test section (reduced area as shown in Figs. 5-8, and Fig. 9).

8.2.1.2 Specimens employing a flat tapered beam configuration should be designed such that a constant surface stress exists in the test section when the specimen is constrained at one end and point loaded perpendicular to the beam axis at the other end (that is, cantilever loading).

8.2.1.3 Rotating beam specimens may have unique dimensions, depending upon the type of machine used. Use appropriate manufacturers' specifications for these specimens.

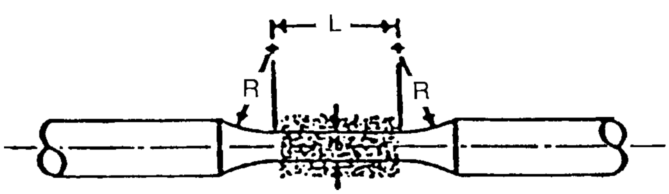

FIG. 5 Bending Fatigue Specimen With Tangentially Blending Fillets Between the Test Section and the Ends for Rotating Beam or Axial Loading

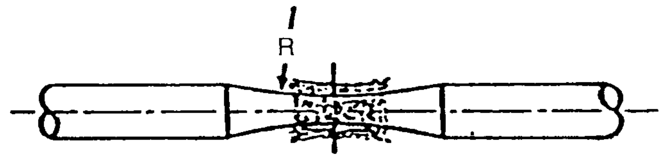

FIG. 6 Specimens With a Continuous Radius Between the Ends for Rotating Beam or Axial Loading

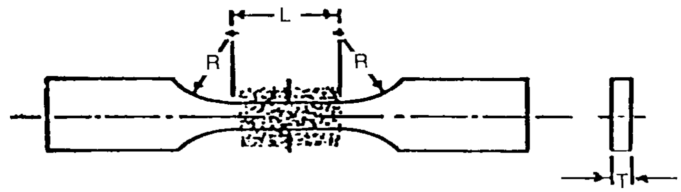

FIG. 7 Specimens With Tangentially Blending Fillets Between the Uniform Test Section and the Ends for Axial Loading

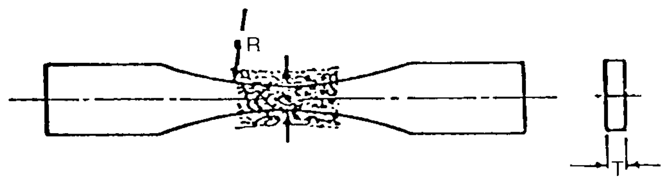

FIG. 8 Specimens With a Continuous Radius Between the Ends for Axial Loading

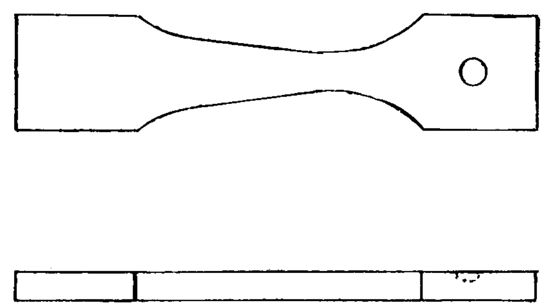

FIG. 9 A Tapered Beam Configuration for Bend Testing

8.3 *Specimen Coating Preparation*:

8.3.1 Calcium phosphate coatings may be applied by any one of a number of techniques. Apply the coating in the same manner as the actual device. The coating should consist of a layer that is mechanically or chemically attached and covers the surface.

8.3.2 Apply coatings as follows:

8.3.2.1 For the shear fatigue specimens apply the coating to the 19.05 mm (0.75 in.) diameter face only (see Fig. 4).

8.3.2.2 For the bending fatigue specimens, apply the coating to the reduced section only, with the exception of the constant stress specimen that should have coating in the entire region of constant stress (see Figs. 5-8, and Fig. 9).

8.3.3 Perform all thermal treatments normally performed on the devices on the test specimens.

8.3.4 If employed, passivation and sterilization techniques should be consistent with those used for actual devices.

8.3.5 *Inspection*—Before testing, perform visual inspections on 100 % of the test specimens. Lack of coating in highly stressed regions, as well as non-uniform coating density, shall be cause for specimen rejection.

9. Procedure

9.1 The number of specimens required for testing, as well as the methods in which the fatigue data may be interpreted, can vary. Several test methods are referenced in this test method.[5,6,7]

9.2 The type of specimen used will depend upon the objective of the test program, the type of equipment available, the equipment capacity, and the form in which the material is available. The specimen chosen should come as close to matching the intended application as possible.

9.3 The test frequency employed shall not exceed 170 Hz.

9.4 *Shear Fatigue Specimens*:

9.4.1 *Curing the Adhesive*—The test results achieved are greatly dependent upon the adhesive used and the way in which it is cured. One suggested adhesive commonly used with calcium phosphate coatings is FM-1000[4] having a thickness of 0.25 mm (0.01 in.). This material has successfully been cured using the following cycle:

9.4.1.1 Align the adhesive with the surface of the coating, taking precautions to align the adhesive in the center of the coating.

9.4.1.2 Apply a constant force using a calibrated high temperature spring, resulting in a stress of 0.138 MPa (20 psi) between the coating and the opposing device that will test the coating.

9.4.1.3 Care must be taken to maintain alignment of the coating and the matching counterface during the test.

9.4.1.4 Place the assembly in an oven and heat at 176°C for 2 to 3 h.

9.4.1.5 The exact amount of time necessary to cure the adhesive will need to be determined by each user, as oven temperature may vary with load size and oven type. It is suggested that the curing cycle be optimized without the coating present, first.

9.4.1.6 Remove the cured assembly from the oven and allow it to cool to room temperature.

9.4.1.7 Remove all excess glue that has protruded from the coated surface. This process must not compromise the integrity of the sample.

9.4.2 Place the specimen assembly in the grips so that the long axis of the specimen is perpendicular to the direction of the applied shear load through the centerline of the grip assembly (see Fig. 3).

9.5 *Bending Fatigue Specimens*:

9.5.1 Perform appropriate testing of the uncoated substrate material, upon which the coating will be applied, to establish a baseline from which to assess the effect of the coating.

9.5.1.1 The baseline test specimens may or may not be grit blasted depending upon the objective of the test. In either event, report the surface roughness.

9.5.2 When mounting the specimen, alignment is crucial. Factors such as poorly machined specimens and misalignment of machine parts might result in excessive vibration leading to erroneous results.

9.5.3 For the rotating beam test, do not apply the load until the machine is operating at the frequency desired for testing.

9.5.4 For the purpose of calculating the applied loads on the test specimen, to determine the applied stresses, measure the dimensions from which the substrate area is calculated to the nearest 0.03 mm (0.001 in.) for dimensions equal to or greater than 5.08 mm (0.200 in.) and to the nearest 0.013 mm (0.0005 in.) for dimensions less than 5.08 mm (0.002 in.).

9.5.4.1 For the coated specimens, use the uncoated substrate dimensions to calculate the applied stress.

10. Test Termination

10.1 Continue the testing until the specimen fails or until a predetermined number of cycles has been reached (typically 10^7 cycles). Failure may be defined as: complete separation of the coating; visible cracking at a specified magnification; a crack of certain dimensions; or by some other criterion.

11. Calculation

11.1 *Shear Fatigue Specimens*—Calculate the substrate area upon which the coating is applied to the nearest 0.06 cm^2 (0.01 $in.^2$). Record peak (failure) load and calculate failing stress in MPa (psi) of adhesive area as follows:

$$adhesion\ or\ cohesion\ strength = maximum\ load/cross-section\ area \quad (1)$$

11.2 *Bending Fatigue Specimens*—For the purpose of calculating the applied loads on the test specimen to determine the applied stresses, measure the dimensions from which the substrate adhesive area is calculated to the nearest 0.03 mm (0.001 in.) for dimensions equal to or greater than 5.08 mm (0.200 in.) and to the nearest 0.013 mm (0.0005 in.) for dimensions less than 5.08 mm (0.002 in.).

12. Report

12.1 The test report procedure and results shall be in accordance with Practice E 468, and include the following information:

12.1.1 Identification of the materials used in the specimen, including bonding agent used;

12.1.2 Identification of methods used to apply the coating including the coating method, heat-treatment, or other data, if available, including date, cycle number, and time and temperature of run;

12.1.3 Dimensional data including the bond cross-sectional area and the thickness of the coating;

12.1.4 Number of specimens tested;

12.1.5 All values for the applied stress and cycles to failure (or run-out);

12.1.6 The mode and location of failure (for example, cohesive versus adhesive) for each test specimen;

12.1.6.1 This may also be performed at various intervals during the test;

12.1.7 The criteria selected for failure, including the number of cycles chosen for run-out;

12.1.8 For the bending fatigue tests report the R ratio (minimum stress/maximum stress);

12.1.9 The test frequency;

[5] Collins, J. A., *Failure of Materials in Mechanical Design*, John Wiley & Sons, New York, 1981.

[6] *Handbook of Fatigue Testing*, *ASTM STP 566*, ASTM, 1974.

[7] Frost, N. C., Marsh, K. J., and Pook, C. P., *Metal Fatigue*, Oxford University Press, London, 1974.

12.1.10 The specimen size for the shear fatigue test if different than the standard size; and

12.1.11 The substrate surface roughness for the baseline bending fatigue test.

13. Precision and Bias

13.1 *Precision*—The precision of this test method is being established.[8]

13.2 *Bias*—The bias of this test method includes the quantitative estimates of the uncertainties of the dimensional measuring devices, the calibration of test equipment, and the skill of the operators. At this time, statements on bias should be limited to the documented performance of particular laboratories.

14. Keywords

14.1 ceramic materials; hydroxylapatite; fatigue testing; tribasic calcium phosphate

[8] Test results that might allow statistical evaluation for this statement are herewith solicited.

APPENDIX

(Nonmandatory Information)

X1. RATIONALE

X1.1 This test method is needed to aid in the development of a high quality material for use in load-bearing implant applications. The influence of calcium phosphate coatings on the resulting fatigue behavior of the system must be viewed as a combination of the surface roughening treatments required to apply the coating, the thermal effects of the coating process, and any other secondary treatments employed. The purpose of this test method is to provide the following information: the influence of the above processing steps and the integrity of the coating and the coating/substrate interface. Round robin testing of this test method is not available at this time.