Designation: ~~F1160 – 05 (Reapproved 2011)ε1~~ F1160 – 14

Standard Test Method for Shear and Bending Fatigue Testing of Calcium Phosphate and Metallic Medical and Composite Calcium Phosphate/Metallic Coatings[1]

This standard is issued under the fixed designation F1160; 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.

~~ε1 NOTE—Units information was editorially corrected in January 2012.~~

1. Scope

1.1 This test method covers the procedure for determining the shear and bending fatigue performance of calcium phosphate coatings and of porous and nonporous metallic coatings and for determining the bending fatigue performance of metallic coatings ~~oversprayed~~ over sprayed with calcium phosphate. This test method has been established based on plasma-sprayed titanium and plasma-sprayed hydroxylapatite coatings. The efficacy of this test method for other coatings has not been established. 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 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 which most closely replicates the actual loading configuration is preferred.

1.2 The values stated in either SI units or inch-pound units are to be regarded separately as standard. The values stated in each system may not be exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems may result in non-conformance with the standard.

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:*[2]

E6 Terminology Relating to Methods of Mechanical Testing
~~E206 Definitions of Terms Relating to Fatigue Testing and the Statistical Analysis of Fatigue Data; Replaced by E 1150 (Withdrawn 1988)[3]~~
E466 Practice for Conducting Force Controlled Constant Amplitude Axial Fatigue Tests of Metallic Materials
E467 Practice for Verification of Constant Amplitude Dynamic Forces in an Axial Fatigue Testing System
E468 Practice for Presentation of Constant Amplitude Fatigue Test Results for Metallic Materials
E1012 Practice for Verification of Testing Frame and Specimen Alignment Under Tensile and Compressive Axial Force Application
E1832 Practice for Describing and Specifying a Direct Current Plasma Atomic Emission Spectrometer

~~3. Definitions~~

~~3.1 The definitions of terms relating to shear and fatigue testing appearing in Terminology E6 shall be considered as applying to the terms used in this test method.~~

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

3. Terminology

3.1 The definitions of terms relating to shear and fatigue testing appearing in Terminology E6 shall be considered as applying to the terms used in this test method.

3.2 *loading points, n*—objects in contact with the test beam or bar used to apply force to the beam or bar, usually radiused to con concentrate the force to a point or a line.

4. Summary of Test Method

4.1 *Shear Fatigue Testing:*

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

4.1.2 This test method is designed to allow the coating to fail at either the coating/substrate interface, within the coating, or at the ~~glue/coating~~ interface between the coating and the adhesive bonding agent used to transmit the ~~load~~force to the coating.

4.2 *Bending Fatigue Testing:*

4.2.1 The primary intent of the bending fatigue test is to quantify the effect that the coating has on the substrate it is applied to. Secondarily, it may be used to provide a subjective evaluation of coating adhesion, (that is, spalling resistance, cracking resistance, and so forth).

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, and so forth, on the fatigue resistance of 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 coated materials for service under condition 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 Practice E466, Section 7. See also Practices E467 and E1012 and ~~Definitions~~Terminology ~~E206~~E1832.

6.2 *Shear Fatigue Test Grips:*

6.2.1 *General*—~~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 and that the coating test plane be parallel to the axial ~~load.~~force. 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.

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

6.2.4 Figs. 3 and 4 show ~~a~~ schematics of the test setup.

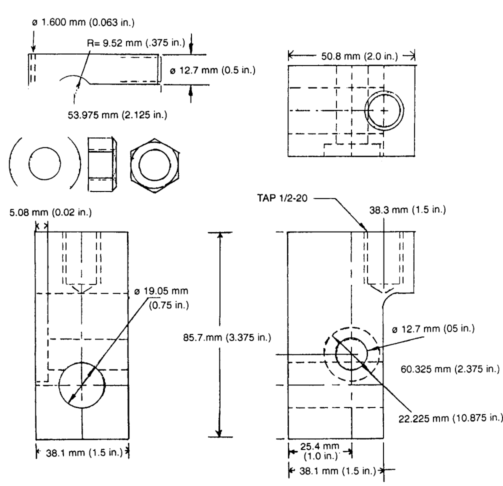
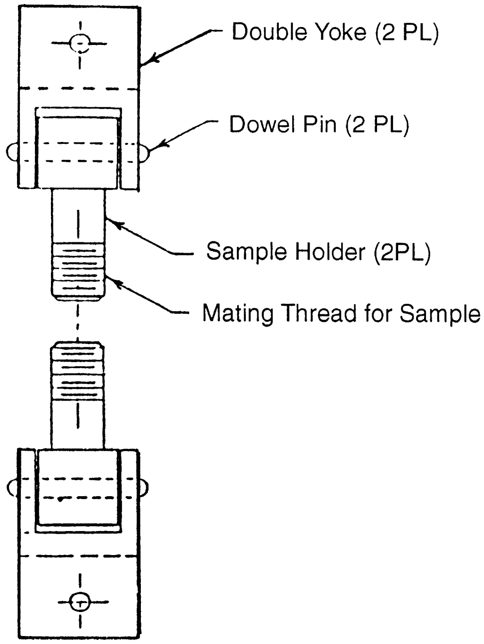


FIG. 1 Gripping Device for Shear Testing



NOTE 1—(2 PL) indicates the top and bottom adapters are identical.

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

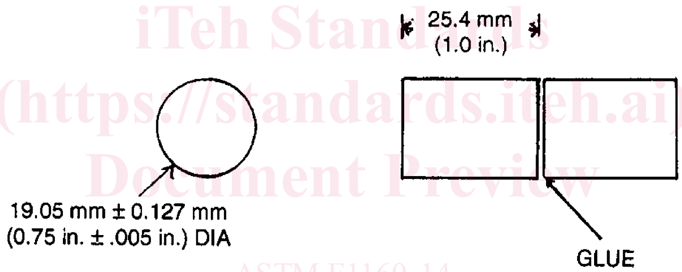


FIG. 3 Schematic of the Shear Test Setup

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.

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, whichever is greater. The 34.5 MPa bonding strength is the static strength of the adhesive. The fatigue strength of the adhesive is usually less than that value. In fatigue the coating under test is often stronger than the adhesive causing the fracture to occur at the adhesive interface. If it is desirable to continue a fatigue test after fracture through the adhesive, the test sample may be rebonded and testing.

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 ensure that it will not penetrate through the coating to the substrate. The FM 1000 Adhesive Film[3] with a thickness of 0.25 mm [0.01 in.] has proven satisfactory for this test method.

~~[3] The last approved version of this historical standard is referenced on www.astm.org.~~

[3] The sole source of supply of the apparatus known to the committee at this time is Cytec Engineered Materials, Inc., 1300 Revolution St., Havre de Grace, MD 21078. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

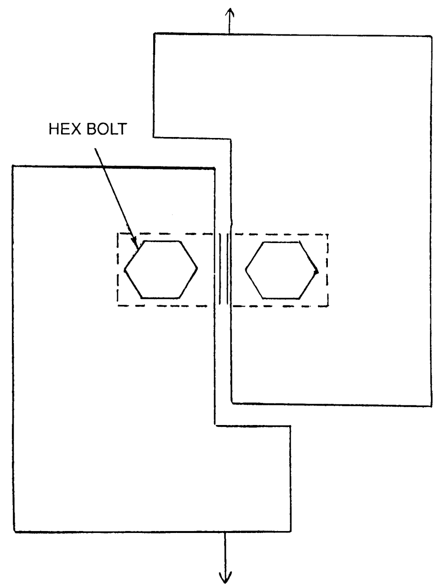


FIG. 4 Drawing of the Recommended Shear Test Specimen Assembly

7.1.3 If a material other than FM 1000 is used, or the condition of the FM 1000 is unknown, it must be tested to establish its equivalence to fresh FM 1000. Testing should be performed without the presence of the coating to establish the performance of the adhesive. Two alternative adhesives that have been used successfully are HYSOL 9514 and 3M 2214 non-metallic filled. Validation data on Hysol 9514 from Indolab GmbH is presented in Appendix X1. These adhesives may not be suitable for HA coatings because they could penetrate the HA.

8. Test Specimen

8.1 *Shear Fatigue Specimen for Calcium Phosphate and Metallic Coatings Only:*

8.1.1 The recommended shear test specimen and setup is illustrated in Figs. 3 and 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 $in.^2$]. 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 for Calcium Phosphate, Metallic, and Calcium Phosphate-Metallic Composite Coatings:*

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 *R* ratio for bending fatigue tests shall be 0.1 or less excluding rotating beam samples. For rotating beam samples the *R* ratio shall be -1.0. However, the design ~~must~~shall meet certain general criteria as follows:

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

8.2.1.2 Specimens using a flat tapered beam configuration should be designed such that a tapered gauge section with a constant surface stress exists ~~in the test section~~ when the specimen is constrained at one end and ~~point loaded perpendicular to~~ force applied through loading points perpendicular to and centered on the beam axis at the other end (that is, cantilever ~~loading).~~loading, usually a tapered cantilever beam as shown in Fig. 10).

8.2.1.3 Four-point bend specimens consisting of straight bars of constant, usually rectangular, cross section loaded in four-point bending also produce a region of constant surface stress in the center span between the two center loading points. The distance between the two external loading points shall always be identical (see Fig. 9).

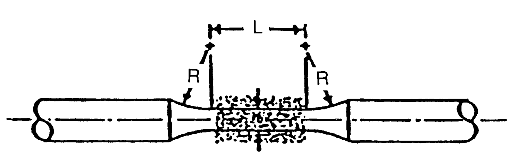


FIG. 5 Bending Fatigue Specimen With Tangentially Blending Fillets Between the Test Section and the Ends for Rotating Beam or Axial Loading

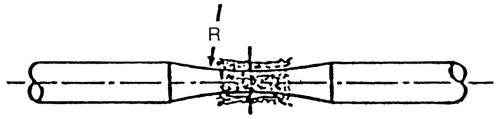


FIG. 6 Specimens With a Continuous Radius Between the Ends for Rotating Beam or Axial loading

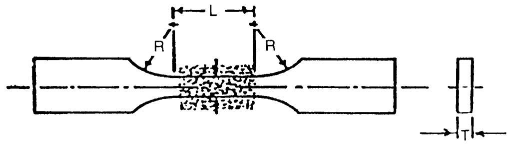


FIG. 7 Specimens With Tangentially Blending Fillets Between the Uniform Test Section and the Ends for Axial Loading

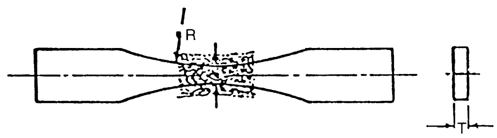


FIG. 8 Specimens With a Continuous Radius Between the Ends for Axial Loading

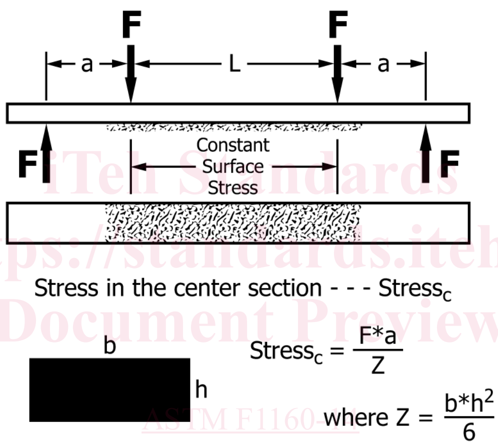


FIG. 9 Four-pointed Bend Test Configuration and Stress Calculation for a Rectangular Cross Section Test Piece

8.2.1.4 Rotating beam specimens may have unique dimensions, depending upon the type of machine used. Appropriate ~~manufacturers'~~manufacturers' specifications for these specimens should be used.

8.2.1.5 The tensile surface edges of the flat tapered cantilever beam specimen and the four point bend specimen may be broken to a small non zero radius to avoid stress concentrations at the edge.

8.3 *Specimen Coating Preparation:*

8.3.1 Coatings may be applied by any one of a number of techniques. All test specimens for coating characterization shall be prepared from indicative coating lots, using production feedstock lots and be coated on the same equipment used for actual implants. The coating should consist of a layer which is mechanically or chemically attached and covers the surface.

8.3.2 Coatings should be applied as follows:

8.3.2.1 For the shear fatigue specimens, the coating should be applied to the 19.05-mm [0.75-in.] diameter face only (see Fig. 3).

8.3.2.2 For the rotating beam and axial fatigue test specimens the coating shall be applied all around and extend slightly beyond the reduced sections (see Figs. 5-8). For the tension-tension bending fatigue specimens, the coating ~~should~~shall be applied to the ~~reduced section only, with the exception of the constant stress specimen which~~side that will be loaded in tension only. The coating shall extend well beyond the tapered gauge area to keep stress concentrations at the transition from coated surface to uncoated surface out of the high stress regions (Fig. 10 ~~should have coating in the entire region of constant stress (see~~) On the four-point bend test sample the coating shall be extended outside of the inner loading points to keep a possible stress concentration ~~Figs. 5-9).~~at the transition from coated surface to uncoated surface outside the maximum stress center region.).

8.3.3 All thermal treatments normally performed on the devices should be performed on the test specimens.

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

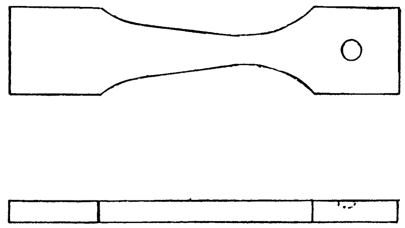


FIG. ~~9~~10 Tapered Beam Configuration for Bend Testing

8.3.5 *Inspection*—Before testing, visual inspections should be performed on 100 % of the test specimens. Non-uniform coating density shall be cause for specimen rejection. For the shear fatigue specimen, lack of coating on the coated face shall be cause for specimen rejection. For the bending fatigue specimen, lack of coating in highly stressed regions shall be cause for specimen rejection.

9. Procedure

9.1 The number of specimens required for testing, as well as the test methods in which the fatigue data may be interpreted, can vary. Several test methods are referenced in this test method.[4,5,6]

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 used shall not exceed ~~170 Hz.~~50 Hz for rotating beam tests and 30 Hz for bending fatigue tests. The test frequencies should be carefully selected to avoid inertial effects from the mass of the test fixtures.

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 is FM 1000 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 to 0.295 MPa [20 to 43 psi] between the coating and the opposing device that will test the coating.

~~9.4.1.2.1~~

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

(1) Care shall be taken to maintain alignment of the coating and the matching counterface during the test.

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

~~9.4.1.3.1~~

~~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 first without the coating present.~~

(1) The exact amount of time necessary to cure the adhesive shall 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 first without the coating present.

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

9.4.1.5 Remove all excess adhesive which has protruded from the coated surface. ~~This process must~~Demonstrate that this process does not compromise the integrity of the ~~sample.~~test result.

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).

9.4.3 Specimens for which the adhesive has penetrated to the substrate shall be discarded and the results not included in the analysis and report.

9.5 *Bending Fatigue Specimens:*

9.5.1 Appropriate testing of the uncoated substrate material, upon which the coating will be applied, should be performed 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, the surface roughness should be reported.

9.5.1.2 For composite calcium phosphate-metallic coatings, additional baseline testing of specimens with only the metallic coating should also be preformed to allow an assessment of the effects of each coating.

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

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

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