Designation: F1044 – 05 (Reapproved 2011)ε1

Standard Test Method for Shear Testing of Calcium Phosphate Coatings and Metallic Coatings[1]

This standard is issued under the fixed designation F1044; 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 shear testing of continuous calcium phosphate coatings and metallic coatings adhering to dense metal substrates at ambient temperatures. It assesses the degree of adhesion of coatings to substrates, or the internal cohesion of a coating in shear, parallel to the surface plane.

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 may involve hazardous materials, operations, and equipment. 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]

E4 Practices for Force Verification of Testing Machines

E6 Terminology Relating to Methods of Mechanical Testing

E8/E8M Test Methods for Tension Testing of Metallic Materials

3. Terminology

3.1 *Definitions*—Terminology E6 shall be considered as applying to the terms used in this test method.

4. Summary of Test Method

4.1 *Shear Method for Calcium Phosphate or Metallic Coatings:*

4.1.1 This test method consists of subjecting a specimen assembly composed of one coated and one uncoated component to a shear load. The components to be tested may be bonded together directly by thermomechanical means (for example, sintering or diffusion bonding) or may be bonded together by use of a polymeric adhesive. The adhesive may be in film form or bulk form, but it must have a minimum bulk shear strength of 34.5 MPa [5000 psi] or as great as the minimum required adhesion or cohesion strength of the coating, whichever is greater.

4.1.2 The shear load must be applied parallel to the plane of the coating utilizing a tensile machine, which is capable of determining the maximum strength of the coating or coating attachment to the substrate interface.

4.2 *Shear Method for Metallic Coatings Only*—The lap shear method consists of subjecting a porous coated area to single shear loading, generally utilizing suitable polymeric adhesive or bone cement adhesive and test jig in a tension machine, and determining the maximum shear stress required to obtain separation (that is, the shear strength of the coating/substrate bond or shear strength of the coating).

5. Significance and Use

5.1 The shear test method is recommended for shear testing of calcium phosphate and metallic/substrate combinations and can provide information on the adhesive or cohesive strength of coatings under a uniaxial shear stress.

5.2 The test method may be useful for comparative evaluation of adhesive or cohesive strengths of a variety of types of coatings. Information developed using this test method may be useful for certain quality control and design purposes.

5.3 The test method should not be considered to provide an intrinsic values for utilization directly in making calculations such as determining the ability of a coating to withstand specified environmental stresses.

5.4 Processing variables, such as substrate preparation prior to coating, surface texture, coating technique variables or

[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.

post-coating heat treatment, or heat may introduce a significant effect on the results of the shear test. The specimen being evaluated must be representative of the actual end-use coating.

6. Apparatus

6.1 *Testing Machines*—Machines used for testing shall conform to the requirements of Practices E4. The loads used in determining shear strength and yield strength shall be within the loading range of the testing machine as defined in Practices E4. See also Test Methods E8/E8M.

6.2 *Gripping Devices:*

6.2.1 *General*—Various types of grips may be used to transmit the load applied 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. 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 *Aligned Interface Method for Calcium Phosphate or Metallic Coatings:*

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

6.2.2.2 A drawing of the adapter to mate the shear fixture to the tensile machine is shown in Fig. 2.

6.2.2.3 A schematic of the test assembly is shown in Fig. 3.

6.2.3 *Lap Shear Method for Metallic Coatings Only:*

6.2.3.1 *Lap Shear Testing Bonding Fixture*—A bonding fixture of the type shown in Fig. 4 or equivalent shall be designed and machined with sufficient precision to minimize movement of the specimen during curing of the adhesive. Some coatings, such as porous fiber metal coatings, may be bonded by sintering without the use of this fixture.

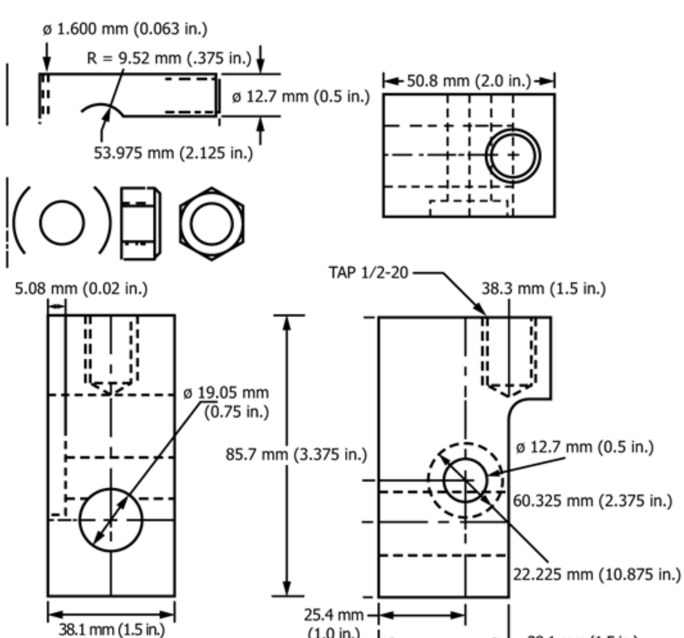

FIG. 1 Gripping Device for Shear Testing

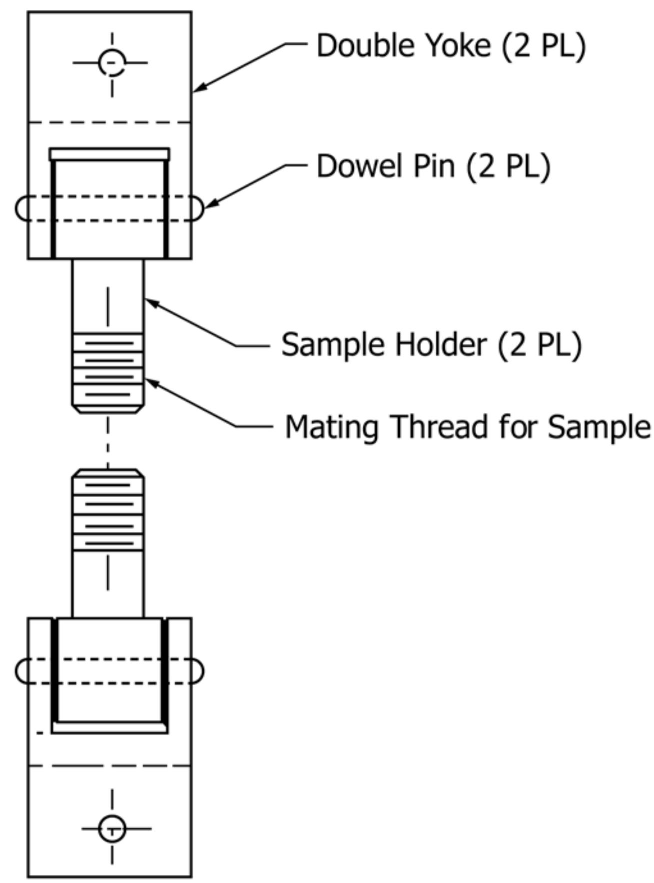

FIG. 2 Adapter to Mate the Gripping Device to the Tensile Machine

6.2.3.2 *Lap Shear Test Loading Grips*—A loading jig of the type shown in Fig. 5 or equivalent shall be used. It shall be made of hardened steel having a hardness of not less than Rockwell C60. To minimize the possible effect of distortion of the device under load, fitted and machined steel bolts may be used to hold the components together. The interfaces between the tongue and clevises shall be smooth.

7. Adhesive Bonding Materials

7.1 *Adhesive Bonding Agent*—A polymeric adhesive bonding agent in film form, or filled viscous adhesive cement, when used, 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.

7.1.2 In instances where porosity extends to the coating/substrate interface, the bonding agent shall be sufficiently viscous and application to the coating sufficiently careful to assure that it will not penetrate through the coating to the

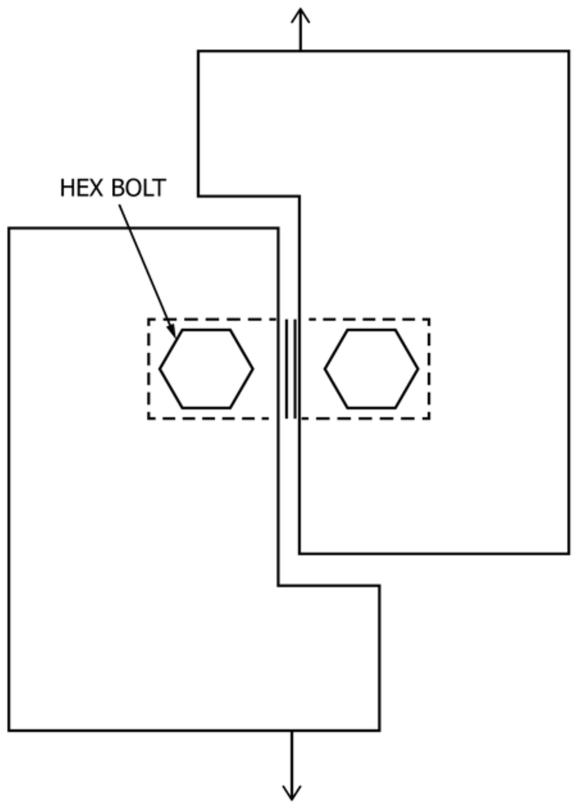

FIG. 3 Drawing of the Recommended Shear Test Specimen Assembly

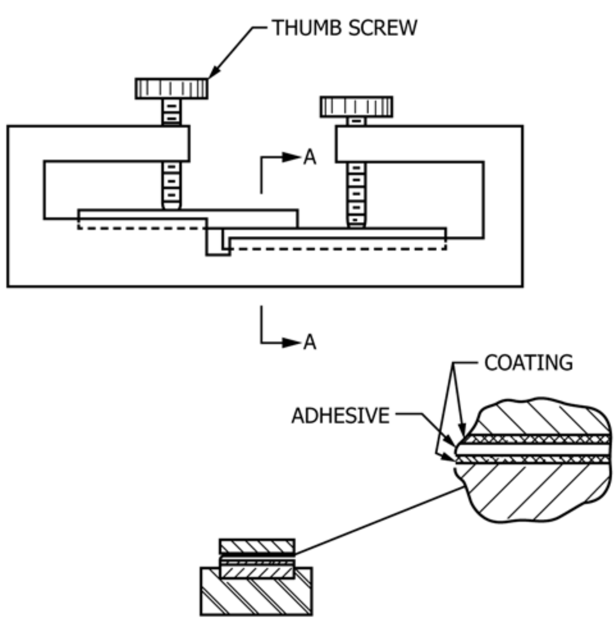

FIG. 4 Lap Shear Bonding Fixture

substrate. The FM 1000 Adhesive Film[3] with a thickness of 0.25 mm [0.01 in.] has proven satisfactory for this test.

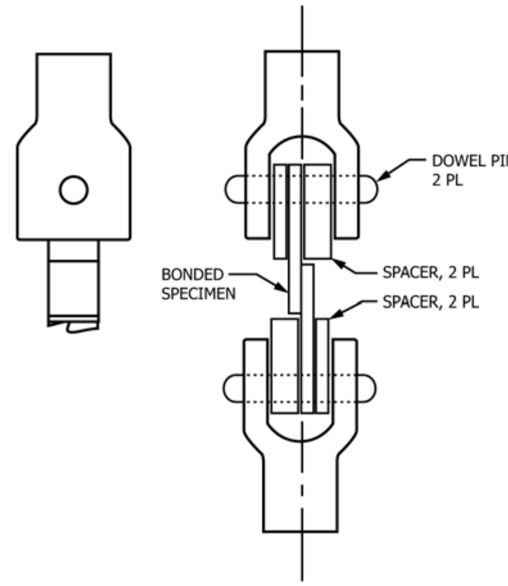

FIG. 5 Lap Shear Loading Grips

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 calcium phosphate coating to establish the performance of the adhesive.

8. Test Specimen

8.1 *General:*

8.1.1 In order to ensure precision and accuracy in test results, it is important that care be exercised in the preparation of specimens, both in machining and in the case of multi-part specimens, in the assembly. Specimen components must be properly aligned in order that generated stresses be purely axial, that is, parallel to the coated surface.

8.1.2 *Aligned Interface Method Specimen*—This shear test specimen is illustrated in Fig. 6. 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 bonding of the adhesive.

8.1.2.1 The cross-sectional area of the substrate upon which the coating is applied shall be nominal 2.84 cm^2 [0.44 $in.^2$]. When specimens of another cross-sectional area are used, the data must be demonstrated to be equivalent to 2.84 cm^2 standard cross-sectional area and the specimen size should be reported.

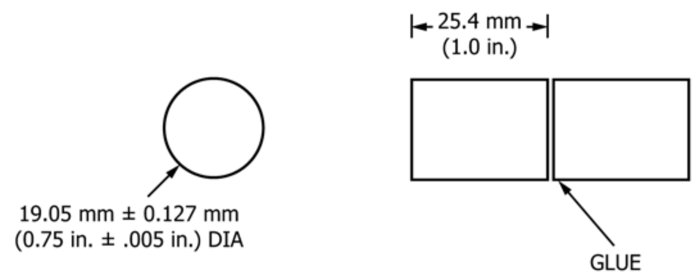

FIG. 6 Aligned Interface Specimen

[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.

8.1.3 *Lap Shear Specimen*—Lap shear specimens shall consist of substrate plates of the type shown in Fig. 7 or equivalent. The dimensions of test specimens differing from Fig. 7 shall be included in the report.

8.1.3.1 *Test Specimen Positioning*—The specimen shall be positioned in the loading grips so as to cause the load axis to be coincident with the plane of the adhesive. This may be accomplished by suitably shimming the specimen within the grips.

8.2 *Specimen Coating Preparation:*

8.2.1 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.

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

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

8.2.3.1 If the passivation and sterilization processes can be shown not to influence the shear strength, these steps may be eliminated.

8.2.4 *Inspection*—Before testing, visual inspections should be performed on 100 % of the test specimens. Lack of coating in highly stressed regions, as well as nonuniform coating appearance, shall be cause for specimen rejection.

8.2.4.1 *Coating Thickness Measurement*—The dimensions of the coating planar surface shall be measured to the nearest 0.80 mm [1⁄32 in.]. For lap shear specimens, the thickness should be measured at the center and all four corners of the coated area to 0.03 mm [0.001 in.]. Coating thickness may not vary by more than ±15 % of the thickness at the center.

9. Procedure

9.1 Prepare a minimum of five test specimens and apply the coating of interest to the face of each in the same manner as for actual production devices.

9.2 *FM–1000 Glue Curing*—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 having a thickness of 0.25 mm [0.01 in.]. This material has successfully been cured using the following cycle:

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

9.2.2 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.

9.2.2.1 Maintain alignment of the coating and the matching counterface during the curing of the adhesive.

9.2.3 Place the assembly in an oven and heat at 176°C [350°F] for 2 to 3 h.

9.2.3.1 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 initially the curing cycle be optimized without the coating present.

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

9.2.5 Remove all excess glue which has protruded from the coated surface without compromising the integrity of the sample.

9.3 *3M Scotch - Weld 2214 - NMF Structural Adhesive Gluing Procedure:*

9.3.1 Ensure that surface to be bonded is clean, degreased, and free of any loose particles and beads.

9.3.2 Apply a thin layer of the adhesive evenly to the coated surface of the test coupons. A spatula or tongue depressor may be used as an application device.

9.3.3 Squeeze excess adhesive out of the assembly using hand pressure while coated faces of the test coupons are butted together.

9.3.4 Place the specimen thus prepared in the fixture shown in Fig. 5. During curing, ensure that the fixture maintains a mild pressure at the joint, and force bonding faces to remain parallel and maintain the axial alignment.

9.4 *Direct Thermomechanical Fabrication*—Metallic coating specimens may be tested in either the aligned interface or single lap configuration. For sintered or diffusion bonded coating systems, the test specimen configurations indicated in Figs. 4-7 may be constructed in a single process with the coating application without the use of adhesive. The details of the specimen fabrication process will necessarily be unique to each coating system. Perform appropriate inspections to ensure

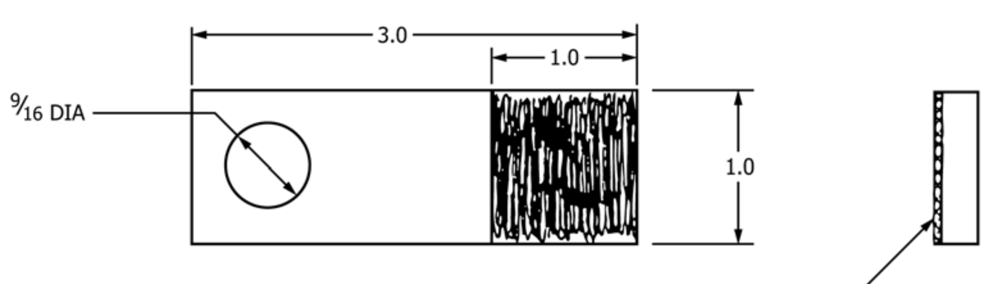

FIG. 7 Lap Shear Specimen (Metal Coatings Only)

that the microstructure and morphology of the coating in the test specimen configuration are representative of finish production parts (see 8.2).

9.5 *Aligned Interface Test:*

9.5.1 Place the specimen assembly in the grips so that the long axis of the specimen is perpendicular to the direction of applied shear load through the centerline of the grip assembly.

9.5.2 Apply a shear load to each test specimen at a constant rate of cross-head speed of 0.25 cm/min [0.1 in./min]. Continue the test until complete separation of the components has been achieved. Record the maximum load applied.

9.6 *Lap Shear Test:*

9.6.1 Following curing, place the specimen in the test assembly shown in Fig. 6.

9.6.2 Load the specimens at a constant rate of cross-head speed of 0.25 cm/min [0.1 in./min]. Continue the test until complete separation of the components has been achieved. Record the maximum load applied.

9.7 Discard specimens for which the adhesive has penetrated to the substrate and do not include the results in the analysis and report.

10. Stress Calculation

10.1 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 bond area, as follows:

$$S = F/A \quad (1)$$

where:

S = adhesion or cohesion strength,
F = maximum load to failure, and
A = cross-sectional area.

11. Report

11.1 Report the following information:

11.1.1 Identification of the materials used in the specimen, including bonding agent used,

11.1.2 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,

11.1.3 Dimensional data, including the bond cross-sectional area and the thickness of the coating,

11.1.4 Number of specimens tested,

11.1.5 All values for the failure load, including maximum, minimum, and mean, and

11.1.6 The mode of failure (for example, cohesive versus adhesive) for each test specimen.

12. Precision and Bias

12.1 *Precision*—The precision of this test method was established by an interlaboratory comparison among six laboratories. The specimens tested were of the lap shear design and were plasma-spray coatings of titanium-6aluminum-4vanadium on a titanium-6aluminum-4vanadium substrate. The population mean shear strength was 4292 lbf/in.^{2} [29.6 MPa] for all samples tested by all laboratories.

12.1.1 *Repeatability*—For replicate results obtained by the same operator on nominally identical test materials, the repeatability standard deviation (S_r) was 698 lbf/in.^{2} [4.81 MPa]. Any two such results would be expected to differ by more than 1954 lbf/in.^{2} [13.5 MPa] only 1 time in 20.

12.1.2 *Reproducibility*—For independent results obtained by different operators working in different laboratories on nominally identical test materials, the reproducibility standard deviation (S_R) was 729 lbf/in.^{2} [5.03 MPa]. Any two such results would be expected to differ by more than 2041 lbf/in.^{2} [14.1 MPa] only 1 time in 20.

12.1.3 *Bias*—Since the measurement of coating shear strength by this test method is a destructive test measurement unique to each individual test sample, no independent determination of a correct or reference value is possible. Thus, no statements regarding the deviation of values measured in accordance with these procedures, from accepted values, is possible.

13. Keywords

13.1 ceramic materials; hydroxylapatite; shear testing; tribasic calcium phosphate

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 coatings on the resulting shear 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: (*1*) the influence of the above processing steps, and (*2*) the integrity of the coating and the coating/substrate interface.