



Designation: F1147 – 05 (Reapproved 2017)^{ε1}

Standard Test Method for Tension Testing of Calcium Phosphate and Metallic Coatings¹

This standard is issued under the fixed designation F1147; 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—Editorial corrections were made throughout in June 2017.

1. Scope

1.1 This test method covers tension testing of calcium phosphate and metallic porous 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 tension normal to the surface plane.

1.2 The values stated in inch-pound units are to be regarded as standard. The values given in parentheses are mathematical conversions to SI units that are provided for information only and are not considered 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.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*²

E4 Practices for Force Calibration and Verification of Testing Machines

E6 Terminology Relating to Methods of Mechanical Testing

E8/E8M Test Methods for Tension Testing of Metallic Materials

F1501 Test Method for Tension Testing of Calcium Phosphate Coatings (Withdrawn 2000)³

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.

phate Coatings (Withdrawn 2000)³

3. Terminology

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

4. Summary of Test Method

4.1 The tensile test method consists of subjecting a specimen assembly composed of one coated and one uncoated component to a tensile load. In the case of the calcium phosphate coatings, the components to be tested must be bonded together by use of a polymeric adhesive. In the case of the metallic coatings, the components may either be bonded with the adhesive, or sintered together. The adhesive may be in film form or bulk form, but it must have a minimum bulk tensile strength of 34.5 MPa (5000 psi) or as great as the minimum required adhesion or cohesion strength of the coating, whichever is greater.

4.2 The tensile load must be applied normal to the plane of the coating utilizing a tension machine which is capable of determining the maximum strength of the coating or coating attachment to the substrate interface.

5. Significance and Use

5.1 The tensile test method is recommended for tension testing of calcium phosphate/substrate or porous metal coating/substrate combinations and can provide information on the adhesive or cohesive strength of coatings under (uniaxial) tensile stress.

5.2 The test method may be useful for comparative evaluation of adhesive or cohesive strengths of a variety of types of coatings. Coatings may be applied using a variety of methods, including, but not limited to, plasma-spraying and sintering. Information developed using this test method may be useful for certain quality control and design purposes.

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

5.3 The test should not be considered to provide an intrinsic value 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 postcoating heat treatment variables may introduce a significant effect on the results of the tension 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 tensile 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 tensile 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 perpendicular 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 *Yoke and Dowel Pin Grips*—A schematic diagram of a typical gripping device for specimens with holes drilled for pin loading is illustrated in Fig. 1. There should be two perpendicular pins in order to maximize off-axis loading.

7. 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 a tensile 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 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 substrate. The FM 1000 Adhesive Film⁴ with a thickness of 0.25 mm (0.01 in.) has proven satisfactory for this test. 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 fresh FM 1000. Testing should be performed without the presence of the coating to establish the performance of the adhesive.

⁴ 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.

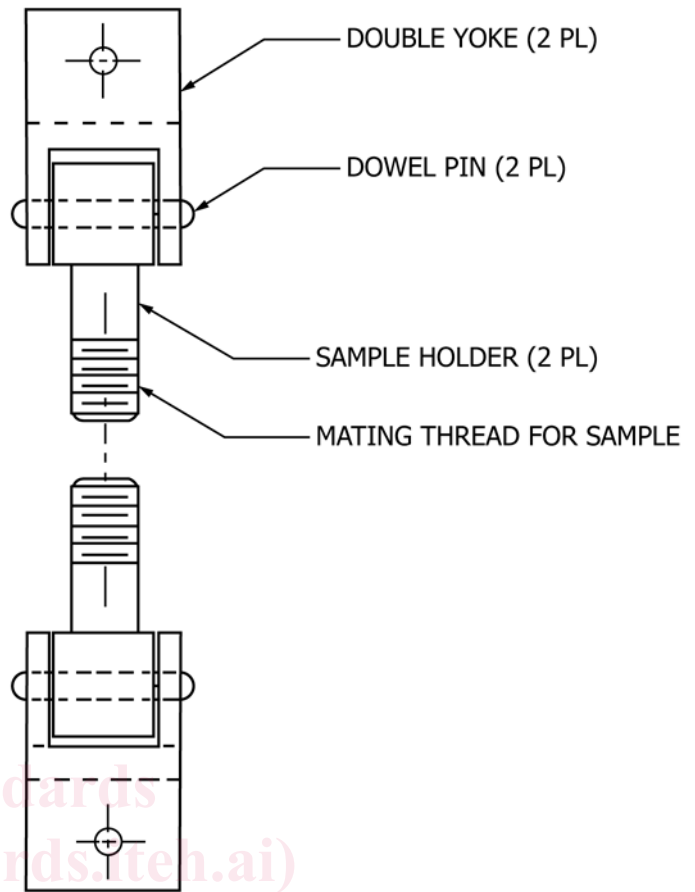


FIG. 1 Yoke and Dowel Pin Grips for Tensile Testing Porous Surfaces

8. Test Specimens

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, normal to the coated surface.

8.1.2 *Tensile-Type Specimens*—Three general types of test specimens are illustrated in Figs. 2 and 3. A complete, assembled test assembly consists either of two solid pieces; one with a coated surface and the other with an uncoated surface or three solid pieces; two with uncoated surfaces and one with a coating applied on one side. The uncoated surface may be roughened to aid in the bonding of the adhesive.

8.1.3 The cross-sectional area of the substrate upon which the coating is applied shall be nominal 5.07 cm² (0.78 in.²). When specimens of another cross-sectional area are used, the data must be demonstrated to be equivalent to a 5.07 cm² standard cross-sectional area, and the specimen size should be reported.

8.1.4 All test specimens for coating characterization shall be prepared from indicative coating lots, using production feed-stock lots and be coated on the same equipment used for actual implants.

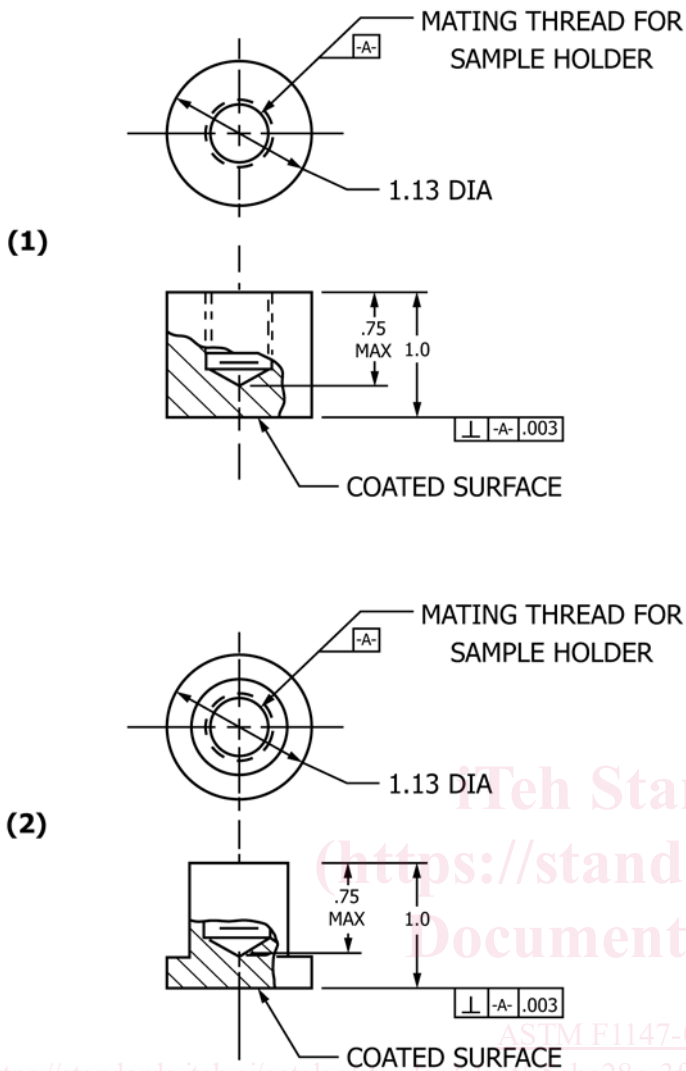


FIG. 2 Tensile Specimens for Testing Porous Surfaces

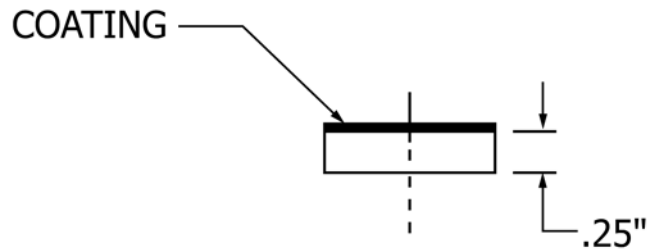
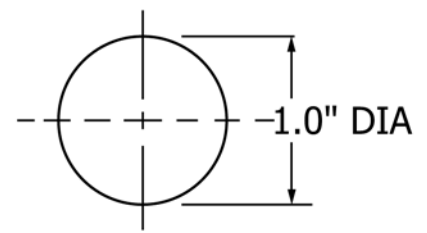


FIG. 3 Smooth Tensile Specimens

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: Where testing porous coatings, more than one layer of glue may need to be employed.

NOTE 1—In the case of porous coatings, care must be taken to guarantee that the adhesive does not bond to the substrate.

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

9.1.3 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.

9.1.3.1 Care must be taken to maintain alignment of the coating and the matching counterface during the curing of the adhesive.

9.1.4 Place the assembly in an oven and heat at 176°C (350°F) for 2–3 h.

9.1.4.1 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 without the coating present, first.

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

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

9.2 Place the specimen assembly in the grips so that the long axis of the specimen coincides with the direction of applied tensile load through the centerline of the grip assembly.

8.2 Specimen Coating Preparation:

8.2.1 Coatings may be applied by any one of a number of techniques. 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 tensile 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 non-uniform coating appearance, shall be cause for specimen rejection.

9. Procedure

9.1 Specimens tested with FM 1000 adhesive shall be prepared as follows:

9.1.1 *Curing the Adhesive*—The test results achieved are greatly dependent upon the adhesive used and the way in which

9.3 Apply a tensile load to each test specimen at a constant rate of cross-head speed of 0.25 cm/min (0.10 in./min). The test should be continued until complete separation of the components has been achieved. Record the maximum load applied.

10. Calculation

10.1 Calculate the substrate area upon which the coating is applied to the nearest 0.006 cm² (0.001 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 The report shall include the following information:

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

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

11.1.3 Dimensional data including the bond cross-sectional area and the thickness of the porous or other coated layer.

11.1.4 Number of specimens tested.

11.1.5 Report all values for the failure load, including maximum, minimum, and mean.

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

12. Precision and Bias

12.1 *Precision (beaded F75)*—The precision of this test method was established by an interlaboratory comparison among six (6) laboratories. The specimens tested were sintered-bead coatings of F75 (cobalt-chromium-molybdenum) alloy on F75 substrates. The population mean tensile strength was 4325 lbf/in.² (29.8 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 repeat-

ability standard deviation (S_p) was 613 lbf/in.² (4.23 MPa). Any two such results (would be expected to differ by more than) 1715 lbf/in.² (11.8 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 772 lbf/in.² (5.32 MPa). Any two such results (would be expected to differ by more than) 2160 lbf/in.² (14.8 MPa) only 1 time in 20.

NOTE 2—The precision study under 12.1 was conducted using coupons which had a one square inch area (1.13 in. diameter). The bead tensile strength was intentionally lowered for this round robin comparison to produce coating fractures rather than glue failures in the coupons.

12.2 *Precision (HA Coating)*—The precision of this test method was established by an interlaboratory comparison among seven (7) laboratories. The substrate of the specimens tested was titanium-6aluminum-4vanadium, which was hydroxylapatite-coated. The mean tensile strength was 8405 lbf/in.² (58.0) MPa for all samples tested by all laboratories.

12.2.1 *Repeatability*—For replicate results obtained by the same operator on nominally-identical test materials, the repeatability standard deviation S_r was 1016 lbf/in.² (7.0 MPa). Any two such results (would be expected to differ by more than) 2845 lbf/in.² (19.6 MPa) only one time in 20.

12.2.2 *Reproducibility*—For independent results obtained by different operators working in different laboratories on nominally-identical test materials, the reproducibility standard deviation (S_R) was 1758 lbf/in.² (12.1 MPa). Any two such results (would be expected to differ by more than) 4922 lbf/in.² (33.9 MPa) only one time in 20.

NOTE 3—The precision study under 12.2 was run using a cross-head speed of 0.05 in./min (0.12 cm/min).

12.3 *Bias*—Since the measurement of porous-coating tensile 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 adherence; ceramic materials; hydroxylapatite; interface; porous coating; tension testing; tribasic calcium phosphate