Designation: F1801 – 20

Standard Practice for Corrosion Fatigue Testing of Metallic Implant Materials[1]

This standard is issued under the fixed designation F1801; 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 practice covers the procedure for performing corrosion fatigue tests to obtain *S-N* (3.2.1) fatigue curves or statistically derived fatigue strength values, or both, for metallic implant materials. This practice describes the testing of axially loaded fatigue specimens subjected to a constant amplitude, periodic forcing function in saline solution at 37°C and in air at room temperature. The environmental test method for implant materials may be adapted to other modes of fatigue loading such as bending or torsion. While this practice is not intended to apply to fatigue tests on implantable components or devices, it does provide guidelines for fatigue tests with standard specimens in an environment related to physiological conditions.

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, health, and environmental 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 Verification of Testing Machines

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

E739 Practice for Statistical Analysis of Linear or Linearized Stress-Life (*S-N*) and Strain-Life (ε-*N*) Fatigue Data

E1012 Practice for Verification of Testing Frame and Specimen Alignment Under Tensile and Compressive Axial Force Application

E1150 Definitions of Terms Relating to Fatigue (Withdrawn 1996)[3]

F86 Practice for Surface Preparation and Marking of Metallic Surgical Implants

F601 Practice for Fluorescent Penetrant Inspection of Metallic Surgical Implants

G15 Terminology Relating to Corrosion and Corrosion Testing (Withdrawn 2010)[3]

2.2 *ANSI Standard:*[4]

ANSI B46.1 Surface Texture

3. Terminology

3.1 *Definitions:*

3.1.1 The terminology used in conjunction with this practice complies with E1150 and Terminology G15.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *S-N curves—S-N* curves (also known as Wöhler-curves) show the correlation between the applied stress (*S*) and the counted number (*N*) of cycles to failure.

[1] This practice 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] The last approved version of this historical standard is referenced on www.astm.org.

[4] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

4. Significance and Use

4.1 Implants, particularly orthopedic devices, are usually exposed to dynamic forces. Thus, implant materials must have high fatigue resistance in the physiological environment.

4.1.1 This practice provides a procedure for fatigue testing in a simulated physiological environment. Axial tension-tension fatigue tests in an environmental test chamber are recommended as a standard procedure. The axial fatigue loading shall comply with Practices E466 and E467.

4.1.1.1 Bending and rotating bending beam fatigue tests or torsion tests may be performed in a similar environmental cell.

4.1.2 This practice is intended to assess the fatigue and corrosion fatigue properties of materials that are employed or projected to be employed for implants. This practice is suitable for studying the effects of different material treatments and surface conditions on the fatigue behavior of implant materials. The loading mode of the actual implants may be different from that of this practice. Determining the fatigue behavior of implants and implant components may require separate tests that consider the specific design and loading mode.

4.1.3 As a substitute for body fluid, 0.9 % saline solution is recommended as a standard environment. One of the various Ringer's solutions or another substitute for body fluid may also be suitable for particular tests. However, these various solutions may not give equal fatigue endurance results. The chloride ions are the most critical constituent in these solutions for initiating corrosion fatigue.

4.1.4 Because implants are manufactured from highly corrosion-resistant materials, no visible corrosion may be detectable when inspected by means of optical microscopy or scanning electron microscopy. Only a decrease of fatigue strength in the high cycle range may be noticeable. Therefore, *S-N* curves covering a broad fatigue loading range should be generated in the test solution and in air. Comparison of fatigue curves generated in air and saline solution may be the only way to assess the effect of the saline environment.

4.1.5 Where the fatigue behavior of a material system is already established, it may suffice to test modifications of the material properties or surface condition in only a selected stress range.

4.1.6 The recommended loading frequency of one hertz corresponds to the frequency of weight bearing during walking. For screening tests, higher test frequencies may be used; but it must be realized that higher frequencies may affect the results.

4.1.7 *Summary of Standard Conditions*—For interlaboratory comparisons the following conditions are considered as the standard test. Axial tension-tension tests with cylindrical specimens in 0.9 % saline solution at 37°C and air at room temperature under a loading frequency of 1 Hz.

5. Testing Equipment

5.1 The mechanics of the testing machine should be analyzed to ensure that the machine is capable of maintaining the desired form and magnitude of loading for the duration of the test (see Practices E4).

5.2 *Axial Fatigue Testing:*

5.2.1 Tension-tension fatigue tests may be performed on one of the following types of axial fatigue testing machines:

5.2.1.1 Mechanical,

5.2.1.2 Electromechanical or magnetically driven, and

5.2.1.3 Hydraulic or electrohydraulic.

5.2.2 The machine shall have a load-monitoring system, such as a transducer mounted in series with the specimen. The test loads shall be monitored continuously in the early stage of the test and periodically thereafter, to ensure that the desired load is maintained. The magnitude of the varying loads, measured dynamically as described in Practice E467, shall be maintained within an accuracy of less than or equal to 2 % of the extreme loads applied during testing.

5.3 *Non Axial Fatigue Testing*—Corrosion fatigue tests under loading conditions different from axial tension-tension may be requested. In such cases established experimental arrangements for bending, rotating bending beam, or torsional testing may replace the axial tension-tension mode. An environmental test chamber is attached to the equipment and the environmental tests are carried out under conditions as described in this standard. Except for the mechanical testing arrangements the conditions of this standard practice apply where possible. Reporting should follow Section 9 and should include all details where the testing deviates from the standard procedure.

5.4 *Environmental Chamber:*

5.4.1 For corrosion fatigue testing, the machine shall be fitted with an environmental test cell surrounding the specimen gauge section as shown in Fig. 1. A heated solution reservoir, a solution pump, and connecting lines for circulating the test solution to the specimen surface are required. The solution should be pumped from the reservoir through the system at a rate that will maintain the temperature at 37 ± 1°C in the test cell, but with flow rates low enough to avoid flow-dependent phenomena like erosion-corrosion. The reservoir should have a minimum capacity of 1000 mL per square centimeter of specimen surface exposed to the electrolyte. The reservoir shall be vented to the atmosphere. If the solution volume decreases, the reservoir shall be replenished with distilled water to maintain the saline concentration, or the solution should be exchanged. During long testing periods exchange of the solution is recommended. A typical environmental test cell for axial fatigue testing is shown in Fig. 1.

5.4.2 The test equipment should be manufactured of materials or should be protected in such a manner that corrosion is avoided. In particular galvanic corrosion in conjunction with the test specimen and loosening of the specimen grips due to corrosion must be avoided.

6. Test Solution

6.1 To prepare the saline solution, dissolve 9 g of reagent-grade sodium chloride in distilled water and make up to 1000 mL. If a substitute for 0.9 % saline solution is used, such as one of the various Ringer's solutions, note that in the report.

7. Test Specimen

7.1 *Specimen Design:*

7.1.1 *Axial Fatigue Testing:*

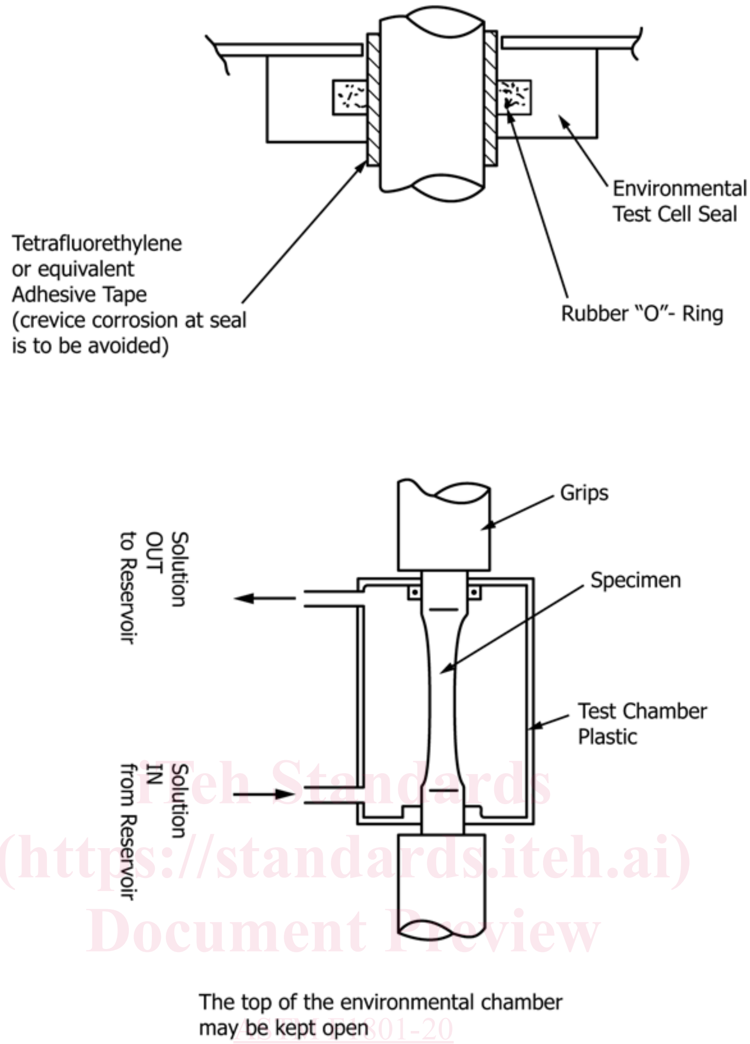

FIG. 1 Example for Environmental Chamber for Axial Corrosion Fatigue Testing

7.1.1.1 The design of the axial load fatigue test specimens should comply to Practice E466 (see Fig. 2, Fig. 3, Fig. 4, and Fig. 5). For the dimensional proportions of flat specimens refer to the drawing in Practice E468. The ratio of the test section area to end section area will depend on the specimen geometry and should comply to those standards. The test specimens specified in Practices E466 and E468 are designed so that fatigue failure should occur in the section with reduced diameter and not at the grip section.

7.1.1.2 For bending tests one may refer to the specimen configuration suggested in Practice E466.

7.1.1.3 To calculate the load necessary to obtain the required stress, the cross-sectional area of the specimen test-section must be measured accurately. The dimensions should

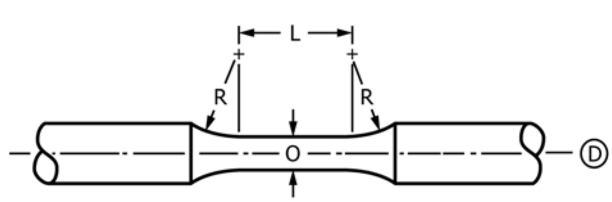

FIG. 2 Specimens With Tangentially Blending Fillets Between the Test Section and the Ends

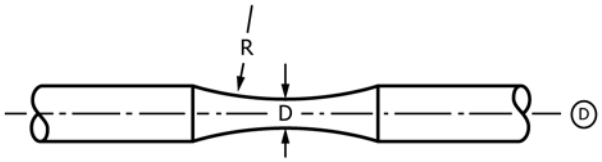

FIG. 3 Specimens With a Continuous Radius Between Ends

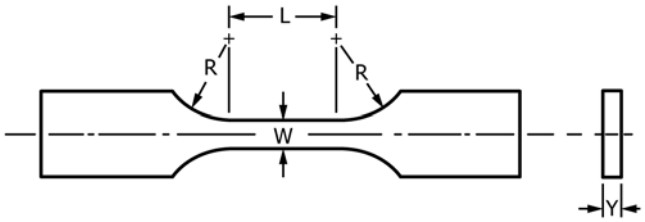

FIG. 4 Specimens With Tangentially Blending Fillets Between the Uniform Test Section and the Ends

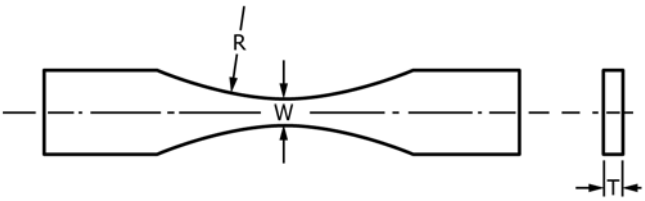

FIG. 5 Specimens With Continuous Radius Between Ends

be measured to the nearest 0.03 mm [0.001 in.] for specimens less than 5.00 mm thick [0.197 in.], and to the nearest 0.05 mm [0.002 in.] for specimens more than 5.00 mm thick [0.197 in.]. Surfaces intended to be parallel and straight should be carefully aligned.

7.2 *Specimen Dimensions*—Consult Practices E466 and E468 for the dimensions of fatigue specimens for axial tension-tension loading (Fig. 2, Fig. 3, Fig. 4, and Fig. 5). If bending specimens corresponding to the example of Practice E466 are used, observe the suggested dimensions.

7.3 *Specimen Preparation:*

7.3.1 The method of surface preparation and the resulting surface condition of the test specimens are of great importance because they influence the test results strongly. Standard preparation shall consist of machining, grinding, or polishing, or all of these. A final mechanical polish is suggested to give a finish of 16 Min RA or less in accordance with ANSI B46.1. Alternatively, a finish with 600 grit paper in the longitudinal direction may be used. However, specimens that are to be compared should be prepared the same way. Mechanically finished specimens shall then be degreased in acetone, flushed first with ethyl alcohol, then with distilled water, and finally blown dry with warm air.

7.3.1.1 Surface passivation may be carried out where appropriate (compare Practice F86).

7.3.1.2 The surface preparation may be also exactly as used or intended to be used for surgical implants. A full account of the surface preparation should be given in the test protocol.

7.3.2 All specimens used in any given series of experiments, including comparison between the air and liquid environments, should be prepared with the same geometry and by the same method to ensure comparable and reproducible results. Regardless of the machining, grinding, or polishing method used, the final mechanical working direction should be approximately parallel to the long axis of the specimen to avoid notch effects of surface grooves.

7.3.3 Fillet undercutting and the introduction of residual stresses into the specimen must be avoided. Both effects can be caused by poor machining practice. Fillet undercutting can be identified by visual inspection. The introduction of unwanted residual stresses can be avoided by careful control of the machining process.

7.3.4 Specimens that are subject to surface alterations under ambient conditions shall be protected appropriately, preferably in an inert medium or exsiccator, to prevent surface change until the beginning of the test.

7.3.5 Visual inspections at a magnification of approximately 20× shall be performed on all specimens. When such inspections reveal potential defects, nondestructive dye penetrant, ultrasonic methods, or other suitable tests may be employed. Dimensional inspection should be conducted without altering or damaging the specimen's surface. Specimens with surface defects should not be used for testing. Inspection should take place prior to final surface cleaning.

7.3.6 Immediately prior to testing, the specimens may be steam sterilized at a temperature of 120 ± 10°C and a pressure of 0.10 MPa [14.5 psi] to simulate the actual implant surface conditions. Specimens shall be allowed to cool to room temperature prior to testing. This sterilizing procedure is not mandatory. If it is used, it should be employed consistently in test series that are related and should be reported in the test protocol.