Designation: F1801 – 97 (Reapproved 2014)

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* 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 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

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:*

ANSI B46.1 Surface Texture[4]

3. Terminology

3.1 *Definitions:*

3.1.1 The terminology used in conjunction with this practice complies to Terminology 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.

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 Practice E466 and Practice 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

[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.

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 by optical or electron-optical (SEM) means. Only a decrease of fatigue strength in the high cyclic life range may be noticeable. Therefore, *S-N* curves covering a broad fatigue loading range should be generated in 0.9 % saline solution (Ringer's solutions) and 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 37°C 0.9 % saline solution and air 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 other typical Ringer's solutions are used, note the solution in the report.

7. Test Specimen

7.1 *Specimen Design:*

7.1.1 *Axial 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 Practice E466 and Practice 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

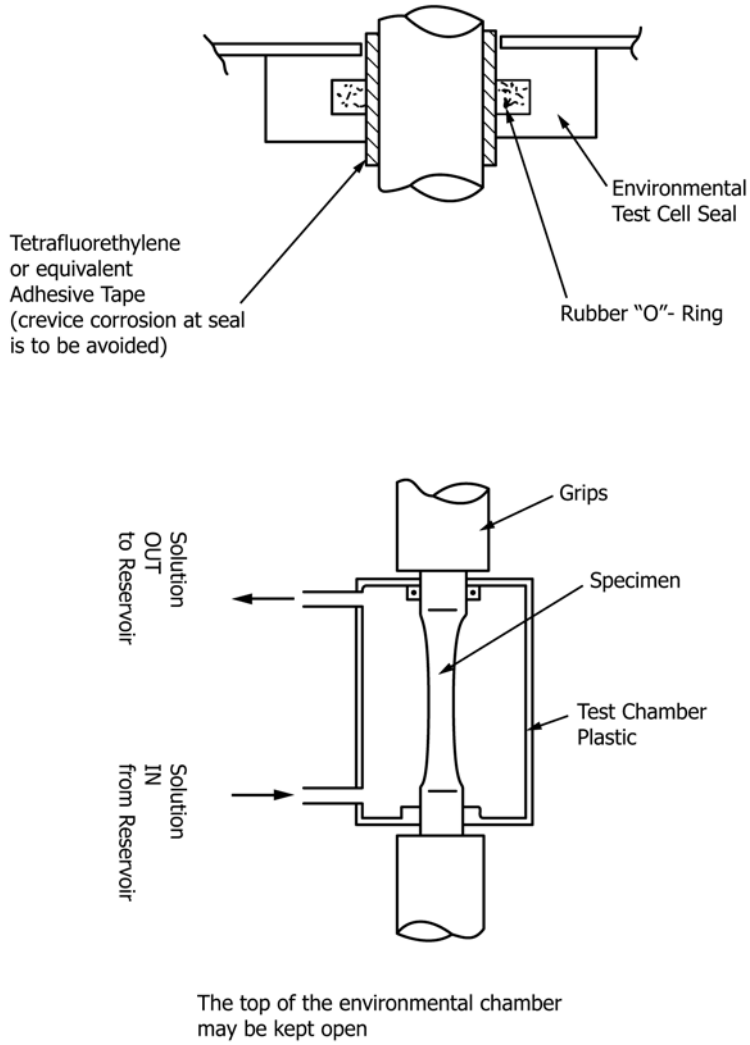


FIG. 1 Example for Environmental Chamber for Axial Corrosion Fatigue Testing

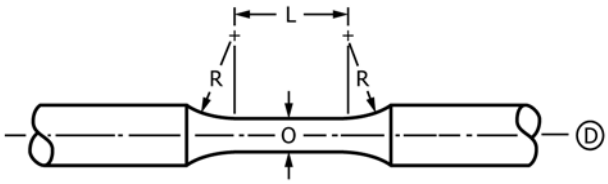


FIG. 2 Specimens With Tangentially Blending Fillets Between the Test Section and the Ends

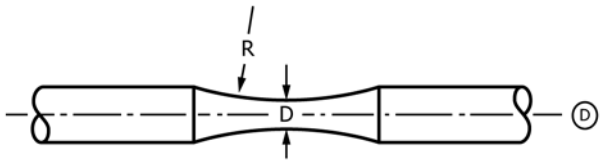


FIG. 3 Specimens With a Continuous Radius Between Ends

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 Practice E466 and Practice 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 F466 are used, observe the suggested dimensions.

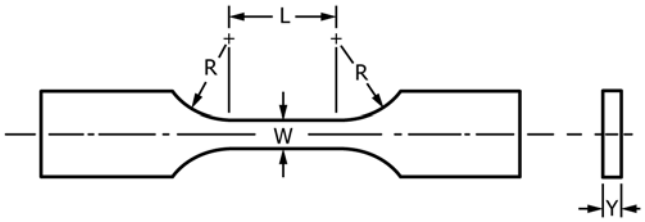


FIG. 4 Specimens With Tangentially Blending Fillets Between the Uniform Test Section and the Ends

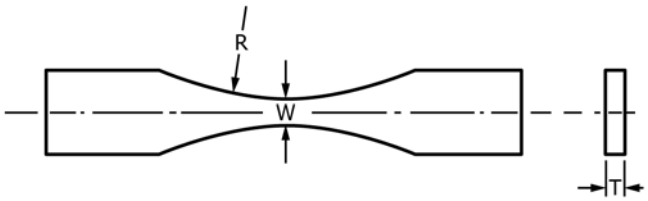


FIG. 5 Specimens With Continuous Radius Between Ends

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.

7.3.7 In the liquid environmental testing, the time elapsed between surface preparation and testing can influence the results due to the growth of a passive film. The elapsed time should thus be reported.

8. Procedure

8.1 *Test Set-Up:*

8.1.1 Specimen grips shall be designed so that alignment is consistently good from one specimen to the next. Every effort should be made to prevent misalignment, due either to twisting (rotation of the grips) or to displacement in their axes of symmetry.

8.1.2 For axial fatigue testing, alignment should be verified according to Practice E4, Practice E467, and Practice E1012.

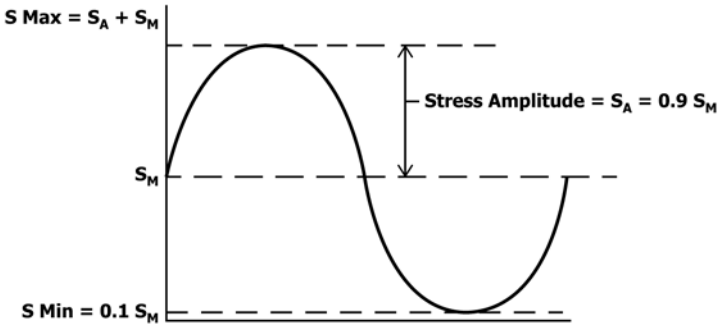
8.2 *Test Conditions:*

8.2.1 The environment shall be air at room temperature or 0.9 weight % NaCl solution at 37 ± 1°C. The pH should be measured before and after the test is begun and should be monitored at 24 h intervals, and at the end of the test.

8.2.1.1 The specimens should be exposed to the liquid environment 2 h prior to the start of the cyclic loading.

8.2.2 Mechanical test conditions for tension-tension, constant amplitude loading are shown in Fig. 6, with an "*A*" ratio equal to 0.9 or an "*R*" value equal to 0.053. Other values for S_{max} and the *A* and *R* ratios may be used, but must be reported.

8.2.2.1 The fatigue test should be carried out at a frequency of 1 Hz. Preliminary screening may be performed at a



"A" Ratio = $\frac{S_A}{S_M} = 0.90$

"R" Ratio = $\frac{S\ Min}{S\ Max} = 0.053$

DEFINITIONS

SYMBOL	DESCRIPTION	FORMULA
S Min	Minimum Stress	$S\ Min = 0.1\ S_M$
S Max	Maximum Stress	$S\ Max = S_A + S_M$
S_M	Mean Stress	$S_M = \frac{S\ Max + S\ Min}{2}$
S_R	Stress Range	$S_R = S\ Max - S\ Min$
S_A	Stress Amplitude	$S_A = \frac{S_R}{2} = \frac{S\ Max - S\ Min}{2}$
"R"	Stress Ratio	$R = \frac{S\ Min}{S\ Max}$
"A"	"A" Ratio	$A = \frac{S_A}{S_M}$

FIG. 6 Loading Conditions

frequency of 30 Hz. While this is a relatively high frequency for implant applications, it allows rapid elimination of those candidate materials that have particularly poor fatigue or corrosion fatigue properties. Materials that appear satisfactory when tested at 30 Hz shall be retested at 1 Hz.

8.2.3 A minimum of three specimens at each chosen stress level shall be tested to yield an *S/N* curve that covers at least the range of 10^4 to 10^6 cycles, in case of uncertainties more specimens must be tested. Specimens shall be loaded to stress levels that allow the development of an *S/N* curve both within and outside of this life cycle range. Thus, specimens should be tested at a minimum of five different stress levels. It is recommended that specimens of materials intended to be used for prostheses be loaded up to 10^7 cycles. When statistical methods of fatigue testing are used,[5,6] a minimum of six samples per stress level must be tested.

8.2.4 Each test shall be continued until the specimen fails, unless it appears that the stress is below the fatigue endurance limit. Failure is defined as complete separation. If this definition does not apply in cases where the axial tension-tension mode is not chosen, the failure criteria need to be reported.

9. Report

9.1 Specimen characteristics and preparation, fatigue test procedures, and results shall be reported in accordance with Practice E468. The following minimum information and data shall be reported for each combination of environment and loading frequency:

9.1.1 *Material Indentification:*

9.1.1.1 Chemical composition,

9.1.1.2 Production process (casting, forging, extruded bar etc.),

[5] *Manual on Statistical Planning and Analysis of Fatigue Experiments, ASTM STP 588*, Little and Tebe, eds.

[6] *Statistical Analysis of Fatigue Data, ASTM STP 744* , Little and Ekvall, eds.

9.1.1.3 Mechanical/thermal processing (cold worked, annealed, etc.),
9.1.1.4 Microstructure, and
9.1.1.5 Specification data (if appropriate).
9.1.2 *Material Properties:*
9.1.2.1 Ultimate tensile strength,
9.1.2.2 Yield strength,
9.1.2.3 Elongation at failure, and
9.1.2.4 Hardness.
9.1.3 *Type of Specimen:*
9.1.3.1 Shape of specimen and dimensions,
9.1.3.2 Machining method,
9.1.3.3 Surface condition and preparation, and
9.1.3.4 Sterilization (if used).
9.1.4 *Fatigue Test Program:*
9.1.4.1 Type of fatigue test,
9.1.4.2 Statistical approach and analysis,
9.1.4.3 Significant variations,
9.1.4.4 Type of machine,
9.1.4.5 Failure criterion, and
9.1.4.6 Wave form and frequency.
9.1.5 *Environmental Conditions:*
9.1.5.1 Ambient laboratory air temperature and humidity.
9.1.5.2 Time elapsed between specimen preparation and exposure to the test solution.
9.1.5.3 Dimensions of the environmental chamber, composition of test solution, reservoir volume, flow rate, solution temperature, pH values, and timing of pH measurements.

9.2 The fatigue test results shall be presented graphically as *S/N* curves for each combination of environment and loading frequency; the curves shall show the failure points of each specimen, and the criteria for curve development as shown in Fig. 1 of Practice E468. The following data should be obtainable from each *S/N* curve:

9.2.1 The fatigue strength at 10 000 and 100 000 cycles,
9.2.2 The fatigue strength at 1 000 000 cycles,
9.2.3 Indication of fatigue limit if possible, and
9.2.4 The report of fatigue strength at 10 000 000 cycles (suggested in cases where the material is intended to be used for prostheses).

9.3 If special statistical test methods are employed, the data shall be presented in correspondence to that method.

10. Precision and Bias

10.1 *Precision:*

10.1.1 Precision can be assessed only after interlaboratory tests have been carried out and the results are tabulated.

10.1.2 For verification of specimen alignment and loading of testing machines see Practice E1012 and Practice E467, respectively.

10.2 *Bias*—No statement can be made as to bias of this practice since no acceptable reference values are available, nor can they be obtained because of the destructive nature of the tests.

11. Keywords

11.1 corrosion fatigue; metallic implant materials; physiological environment

APPENDIX

(Nonmandatory Information)

X1. RATIONALE

X1.1 This practice provides a practice for the assessment of the corrosion fatigue behavior of metallic materials intended to be used in the body environment.

X1.2 To evaluate the effect of the environment, fatigue tests must be performed in air and in the environment under otherwise exactly the same conditions. This may be achieved by testing in parallel in units with identical loading arrangements, or consecutively on the same testing unit.

X1.3 The physiological environment is simulated by 0.9 % saline solution at 37 ± 1°C. Of significance in this test solution is the chloride ion concentration. Regarding metal corrosion, this is the most aggressive species which is contained in the body fluid in about the same concentration. Furthermore, 0.9 % isotonic saline solution is used in surgery for irrigation.

X1.4 Other species of the physiological environment, such as proteins, can have inhibitory effects that counteract the chloride ion activity.

X1.5 The effect of the environment on the fatigue resistance may be very mild and without any morphological signs of corrosion. The environment may only influence the fatigue life by some effects on the growth or deterioration of the passive film on the metal surface.

X1.6 Environmental effects may be only observed in certain sections of the Wöhler curve.