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Standard Test Method for Measurement of Hydrogen Embrittlement Threshold in Steel by the Incremental Step Loading Technique[1]

This standard is issued under the fixed designation F 1624; 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.

~~ϵ[1] NOTE—Footnote 10 has been added editorially to Note 1.~~

INTRODUCTION

Hydrogen embrittlement is caused by the introduction of hydrogen into steel that can initiate fracture as a result of residual stress or in service when external stress is applied **(1)**. The hydrogen can be generated during cleaning or plating processes~~,~~ or the exposure of cathodically protected steel parts to a service environment including fluids, cleaning treatments, or maintenance chemicals that may contact the surface of steel componets.

The combined residual and applied stress above which time-delayed fracture will occur (~~F~~finite~~ L~~ life) or below which fracture will never occur (~~I~~infinite~~ L~~ life) is called the threshold stress. Historically, time-to-failure sustained load tests have been conducted to determine the threshold stress. This technique may require 12 to 14 specimens and several high-load capacity machines to measure the threshold stress in high-strength steel (>175 ksi), taking as long as 3 to 4 months. The run-out time can be as long as ~~4~~ four to ~~5~~ five years per U.S. Navy requirements for low-strength steels (<175 ksi) at 33 to 35 HRC. In Test Method E 1681, more than 10 000 h ~~(>1~~ (>one year) are specified for run-out for precracked specimens.

This standard provides an accelerated method to measure the threshold stress or threshold stress intensity for the onset of hydrogen stress cracking in steel within one week on only one machine.

This method can be used to ~~rapidly~~ determine rapidly the effects of residual hydrogen in a part caused by processing or quantify the relative susceptibility of a material under a fixed set of hydrogen-charging conditions. For precracked specimens, the threshold stress intensity as defined in Test Method E 1681 can be measured, only in a much shorter time.

[1] This test method is under the jurisdiction of ASTM Committee F-7 on Aerospace and Aircraft and is the direct responsibility of Subcommittee F07.04 on Hydrogen Embrittlement.

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1. Scope

1.1 This test method establishes a procedure to measure the susceptibility of steel to a time-delayed failure such as that caused by hydrogen. It does so by measuring the threshold for the onset of subcritical crack growth using standard fracture mechanics specimens, irregular-shaped specimens such as notched round bars, or actual product such as fasteners **(2)** (threaded or unthreaded) springs or components as identified in SAE J78, J81, and J1237.

1.2 This test method is used to evaluate quantitatively:

1.2.1 The relative susceptibility of steels of different composition or a steel with different heat treatments;

1.2.2 The effect of residual hydrogen in the steel as a result of processing, such as melting, thermal mechanical working, surface treatments, coatings, and electroplating;

1.2.3 The effect of hydrogen introduced into the steel caused by external environmental sources of hydrogen, such as fluids and cleaners maintenance chemicals, petrochemical products, and galvanic coupling in an aqueous environment.

1.3 The test is performed either in air, to measure the effect if residual hydrogen is in the steel because of the processing (IHE), or in a controlled environment, to measure the effect of hydrogen introduced into the steel as a result of the external sources of hydrogen (EHE) as detailed in ASTM STP ~~4~~ 543.

1.4 The values stated in acceptable U.S. units shall be regarded as the standard. The values stated in metric units may not be exact equivalents. Conversion of the U.S. units by appropriate conversion factors is required to obtain exact equivalence.

1.5 *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:*

B 602 Test Method for Attribute Sampling of Metallic and Inorganic Coatings[2]

E 4 Practices for Force Verification of Testing Machines[3]

E 6 Terminology Relating to Methods of Mechanical Testing[3]

E 8 Test Methods for Tension Testing of Metallic Materials[3]

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications[4]

E 399 Test Method for Plane-Strain Fracture Toughness of Metallic Materials[3]

E 1681 Test Method for Determining Threshold Stress Intensity Factor for Environment-Assisted Cracking of Metallic Materials[3]

F 519 Test Method for Mechanical Hydrogen Embrittlement Testing of Plating Processes and Aircraft Maintenance Chemicals[5]

G 5 Reference Test Method for Making Potentiostatic and Potentiodynamic Anodic Polarization Measurements[6]

2.2 *SAE Standards:*

J78 Self-Drilling Tapping Screws[7]

J81 Thread Rolling Screws[7]

J1237 Metric Thread Rolling Screws[7]

2.3 *ANSI/ASME:*

B18.18.2M Inspection and Quality Assurance for High-Volume Machine Assembly Fasteners, 1987[8]

B18.18.3M Inspection and Quality Assurance for Special Purpose Fasteners, 1987[8]

B18.18.4M Inspection and Quality Assurance for Fasteners for Highly Specialized Engineering Applications, 1987[8]

2.4 *Related Publications:*

ASTM STP 543, Hydrogen Embrittlement Testing, 1974[9]

ASTM STP 962, Hydrogen Embrittlement: Prevention and Control, 1985[9]

3. Terminology

3.1 *Symbols*—Terms not defined in this section can be found in Terminology E 6 and shall be considered as applicable to the terms used in this test method.

3.1.1 P—applied load.

3.1.2 P_c—critical load required to rupture a specimen using a continuous loading rate.

3.1.3 P_i—crack initiation load for a given loading and environmental condition using an incrementally increasing load under displacement control.

3.1.4 P_{th}—threshold load where P_i is invariant with respect to loading rate. P_{th} is the basis for calculating the threshold stress or the threshold stress intensity.

3.1.5 IHE—Internal Hydrogen Embrittlement—test conducted in air.

3.1.6 EHE—Environmental Hydrogen Embrittlement—test conducted in a specified environment.

3.1.7 th—threshold—the lowest load at which subcritical cracking can be detected.

3.2 *Irregular Geometry-Type Specimens*—test sample other than a fracture mechanics-type specimen such as a notched round bar or fastener.

3.2.1 σ = applied stress.

3.2.2 σ_{net} = net stress based on area at minimum diameter of notched round bar.

3.2.3 σ_i = stress at crack initiation.

3.2.4 σ_{th} = threshold stress.

3.2.5 $\sigma_{th\text{-}IHE}$ = threshold stress—test conducted in air—geometry dependent.

3.2.6 $\sigma_{th\text{-}EHE}$ = threshold stress—test conducted in a specified environment—geometry dependent.

4. Summary of Test Method

4.1 The test method is based on determining the onset of subcritical crack growth with a modified, incrementally increasing, slow strain rate step-load test under displacement control **(3), (4), (5)**.

4.2 This test method measures the load necessary to initiate a subcritical crack in the steel at various, incremental loading rates, for specimens of different geometry and different environmental conditions.

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[2] *Annual Book of ASTM Standards*, Vol 02.05.

[3] *Annual Book of ASTM Standards*, Vol 03.01.

[4] *Annual Book of ASTM Standards*, Vol 14.02.

[5] *Annual Book of ASTM Standards*, Vol 15.03.

[6] *Annual Book of ASTM Standards*, Vol 03.02

[7] Available from Society of Automotive Engineers, 400 Commonwealth Dr., Warrendale, PA 15096–0001.

[8] Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

[9] Available from ASTM, 100 Barr Harbor Dr., PO Box C700, West Conshohocken, PA 19428.

4.2.1 By varying the incremental loading rate, the threshold stress can be determined.

4.3 Four-point bending is used to maintain a constant moment along the specimen. This condition is used to simplify the calculation of stress or stress intensity for an irregular cross section.

4.4 The minimum or invariant value of the stress with regard to the loading rate is the threshold stress for the onset of crack growth as a result of hydrogen embrittlement for a given geometry.

4.5 In tension and bending, the onset of subcritical crack growth as a result of hydrogen in steel is identified by decrease in load while holding the displacement constant.

4.6 The displacement is incrementally increased in tension or four-point bending and the resulting load is monitored. While the displacement is held constant, the onset of subcritical crack growth is detected when the load decreases by a predetermined amount.

4.7 The loading rate must be sufficiently slow to permit hydrogen to diffuse and induce cracking that manifests itself as a degradation in strength.

5. Significance and Use

5.1 This test method is used for research, design, service evaluation, manufacturing control, and development. This test method quantitatively measures stress parameters that are used in a design or failure analysis that takes into account the effects of environmental exposure including that which occurs during processing, such as plating (see ASTM **(6)** (ASTM STP 962).

5.2 For plating processes, the value of $\sigma_{th\text{-}IHE}$ is used to specify quantitatively the maximum operating stress for a given structure or product.

5.3 For quality control purposes, an accelerated test is devised that uses a specified loading rate, which is equal to or lower than the loading rate necessary to determine the threshold stress (see 8.1).

5.4 For fasteners, the value of $\sigma_{th\text{-}IHE}$ is used to specify quantitatively the maximum stress during installation and in service to avoid premature failure caused by residual hydrogen in the steel as a result of processing.

5.5 For fasteners, the value of $\sigma_{th\text{-}EHE}$ is used to specify quantitatively the maximum stress during installation and in service to avoid failure from hydrogen absorbed during exposure to a specific environment.

5.6 To measure the relative susceptibility of steels to hydrogen pickup from various fabrication processes, a single, selected, discriminating rate is used to rank the resistance of various materials to hydrogen embrittlement.

6. Apparatus

6.1 *Testing Machine*—Testing machines shall be within the guidelines of calibration, force range, resolution, and verification of Practices E 4.

6.2 *Gripping Devices*—Various types of gripping devices shall be used in either tension or four-point bending to transmit the measured load applied by the testing machine to the test specimen.

6.3 *Test Environment*—The test shall be conducted in air or any other suitable controlled environment using an appropriate inert container.

6.3.1 *Potentiostatic Control*—The corrosion potential of the specimen can be controlled with a reference S saturated C calomel E electrode (SCE) or equivalent reference electrode such as Ag/AgCl in accordance with Test Method G 5. The imposed potential is typically cathodic, ranging from 0.0 to −1.2 V versus SCE in a 3.5 weight percent NaCl solution **(7)**.

NOTE 1—Apparatus, grips, load adapters, and environmental chambers that have been found to meet the standard are available from Fracture Diagnostics, Inc., P.O. Box 6401, Denver, Co 80206.[10]

7. Sampling and Test Specimens

7.1 *Sampling*—For research, design, and service evaluation and development, the sampling size depends on the specific requirements of the investigator. For manufacturing control, loading rates shall be fixed, but statistically significant sampling sizes are used such as ANSI/ASME B18.18.2M, B18.18.3M, or B18.18.4M and Test Method B 602 for fasteners. For other quality assurance tests, the sampling size shall be in compliance with the requirements of the specification.

7.2 *Test Specimens*—The test specimen should be classified as either fracture mechanics-type specimens or irregular-shaped specimens **(8)**.

7.2.1 Fracture mechanics-type specimens are defined in standards such as Test Method E 399.

NOTE 2—The maximum stress used during fatigue precracking must be less than 60 % of any measured value of load for crack initiation for the data to be valid.

7.2.2 Irregular geometry-type specimens shall be either specimens as defined in standards such as Test Method F 519 or specimens from product. The product shall be tested either substantially full size or as a machined specimen.

8. Procedure

8.1 *Determination of Threshold Load (a Suggested Test Protocol)*:

[10] The apparatus is covered by a patent. Interested parties are invited to submit information regarding the identification of an alternative(s) to this patented item to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend.

8.1.1 Load one specimen to rupture at a rate consistent with Test Methods E 8 to establish the maximum fracture load for a given specimen geometry, P_c (see Fig. 1).

8.1.2 Another sample (#No. 1) is tested by applying an incremental or step loads under displacement control in tension or four-point bending, programmed to attain P_c.

8.1.3 Another sample (#No. 2) is tested using load increments programmed to attain P_{i1}. In no case should the loading rate be faster than that of the previous sample. This load sequence will continue until a significant drop in load is detected that will establish the value designated as P_{i2}.

8.1.4 Subsequent specimens are tested in load increments programmed to attain P_{i2}, and so forth. It is suggested that the duration be doubled, at least at values in excess of 0.5 P_{ii}. The procedure shall be continued until an invariant value (P_{th}) is obtained.

8.1.5 At a minimum, equal load increments for 1 h each shall be used in step loading to attain P_c. For an initial 8-h test, each load step shall be $P_c/8$. For an initial 20-h test, each load step shall be $P_c/20$. The load at which subcritical crack growth occurs is detected by a significant drop in load, P_{i1}. Testing shall be continued until crack growth is detected or rupture occurs. For steels with a hardness of 48 HRC or greater, an 8-h step-loading profile is suggested in Table 1. For steels less than 48 HRC, a 20-h step-loading profile is suggested in Table 2.

8.1.6 Crack growth shall be considered to have occurred if the measured load on a sample drops by more than the established accuracy of the test apparatus, while the displacement is held constant, with the exception identified in 8.1.6.1.

8.1.6.1 Any load drop depicted as a decreasing rate shall be attributed to plasticity or creep in the specimen and test apparatus is not considered crack growth and is not defined as the crack initiation load, P_{ii} (see Fig. 2, Type A).

8.1.6.2 Any load drop depicted as an increasing rate shall be attributed to subcritical crack growth in the specimen. The load is defined as the crack initiation load, P_{ii} (see Fig. 2, Type B).

8.1.6.3 The load at the transition from a constant or decreasing rate to an increasing rate is defined as the crack initiation load, P_{ii} (see Fig. 2, Type C).

8.1.7 Verification of crack growth is obtained by loading the tested specimen to failure. Methods such as Test Methods E 8 or Test Method E 399 shall be used. Fractographic analysis may be used to verify the existence of subcritical cracking.

9. Calculations

9.1 Stress parameters are calculated from the load measurements in Section 8.1.

9.2 The relationship between load and net stress (σ_{net}) is given as P/A_{net} for tensile specimens and My/I for bend specimens,

where:

A_{net} = net cross-sectional area,
M = the applied moment,
y = the distance to the stressed ligament, and
I = the cross-sectional moment of inertia.

9.3 The ultimate tensile strength (UTS) per Test Methods E 8 is given as P_c/A_{net}.

9.4 The threshold stress (σ_{th}) is calculated from the same mathematical relationship as UTS except that the threshold load (P_{th}) is used instead of P_c.

9.5 The threshold stress (σ_{th}) is measured in an aqueous environment under a cathodic or hydrogen-producing environment or in air for electroplated parts. These values are not necessarily the same.

9.5.1 A further designation of $\sigma_{th\text{-}EHE}$ is used if the test is conducted in a specified environment.

9.5.2 A further designation of $\sigma_{th\text{-}IHE}$ is used if the test is conducted in air.

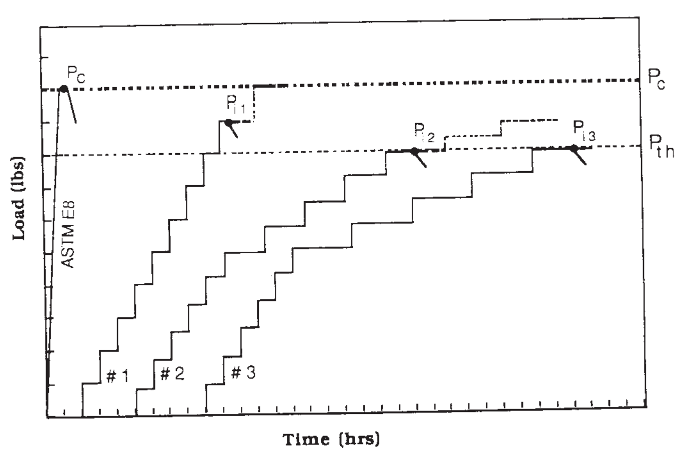

FIG. 1 Suggested Protocol for a Loading Profile to Determine Threshold

TABLE 1 Suggested Protocol for Eight-Step Test

8-h Test Load (% P_{ii})[A]	Step Duration in Hours Load Profile			
	#1	#2	#3	#4
12.5	1	1	1	1
25.0	1	1	1	1
37.5	1	1	1	1
50.0	1	1	1	1
62.5	1	2	4	8
75.0	1	2	4	8
87.5	1	2	4	8
100.0	1	2	4	8
Σ hours =	8	12	20	36

[A] P_{ii}

TABLE 2 Suggested Protocol for 20-Step Test

20-h Test Load (% P_{ii})[A]	Step Duration in Hours Load Profile			
	#1	#2	#3	#4
5	1	1	1	1
10	1	1	1	1
15	1	1	1	1
20	1	1	1	1
25	1	1	1	1
30	1	1	1	1
35	1	1	1	1
40	1	1	1	1
45	1	1	1	1
50	1	1	1	1
55	1	2	4	8
60	1	2	4	8
65	1	2	4	8
70	1	2	4	8
75	1	2	4	8
80	1	2	4	8
85	1	2	4	8
90	1	2	4	8
95	1	2	4	8
100	1	2	4	8
Σ hours =	20	30	50	90

[A] P_{ii}

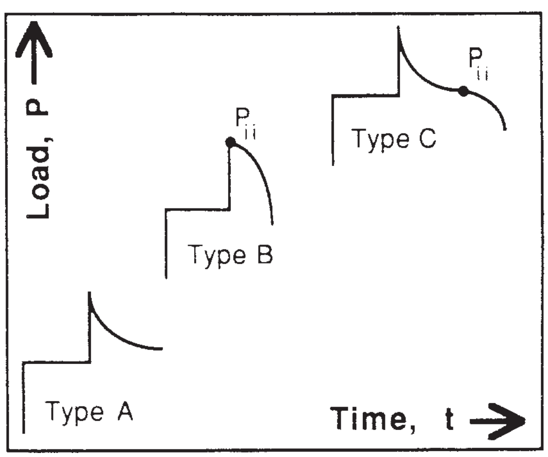

FIG. 2 Definition of Crack Initiation Load, P_{ii}

10. Report

10.1 Test information on materials not covered by a product specification shall be reported in accordance with 10.2 or both 10.2 and 10.3.

10.2 Test information to be reported shall include the following when applicable:

10.2.1 Material and sample identification.

10.2.2 Specimen type of either fracture mechanics or irregular geometry. Fracture mechanics-type specimens with specified geometry shall be reported as described in Test Method E 399. Irregular geometry type specimens are classified according to their respective standard or specification.

10.2.3 Report the fracture load and any maximum fracture stress or stress intensity parameter that has been calculated from the rupture load.

10.2.4 Report the threshold load (P_{th}) and any threshold stress or stress intensity parameter that has been calculated from the threshold load.

NOTE 3—When testing irregular geometry type specimens, note that the test results are geometric specific and deviations will occur from one type of sample to another of the same material if similar geometric test samples are not used.

NOTE 4—Use the loading code of "B" for four-point bending and "T" for tension.

10.2.5 Loading and duration of each increment.

10.2.6 Method used to determine loading rate.

10.2.7 Environmental conditions.

10.3 Test information to be available on request shall include:

10.3.1 Table identifying time at each step of loading profile similar to Table 1 or Table 2.

10.3.2 Equations used to calculate fracture mechanics properties and estimate stresses on irregularly shaped geometry.

10.3.3 Fixture dimensions pertaining to how irregular test specimens were loaded and what specific geometry was tested.

10.3.4 Use Practice E 29 for rounding of test results.

11. Precision and Bias

11.1 *Precision*—The precision of the procedure in this test method for measuring the susceptibility to hydrogen embrittlement in steel is being determined.

11.2 *Bias*—There is no known bias in this test method.

12. Keywords

12.1 constant extension rate; CERT delayed brittle failure; decreasing loading rate; displacement control; hydrogen embrittlement threshold; hydrogen stress cracking; rising step load; RSL; slow strain rate

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