Designation: F1624 – 12

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 F1624; 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.

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**).[2] 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 components. This method can be used to rapidly determine 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.

The combined residual and applied stress above which time-delayed fracture will occur (finite life) or below which fracture will never occur (infinite life) is called the threshold stress or threshold stress intensity (K) for precracked specimens. Historically, sustained load time-to-failure tests have been conducted on notched bars to determine the threshold stress for the onset of hydrogen stress cracking. This technique may require 12 to 14 specimens and several high-load capacity machines. For precracked specimens, the run-out time can be as long as four to five years per U.S. Navy requirements for low-strength steels at 33 to 35 HRC. In Test Method E1681, more than 10 000 h (> one year) are specified for low-strength steel (< 175 ksi) and 5000 h for high-strength steel (> 175 ksi).

This standard provides an accelerated method to measure the threshold stress or threshold stress intensity as defined in Test Method E1681 for the onset of hydrogen stress cracking in steel within one week on only one machine. The specific application of this standard to hydrogen embrittlement testing of fasteners is described in Annex A1.

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 543.

1.4 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.

NOTE 1—The values stated in metric units may not be exact equivalents. Conversion of the inch-pound units by appropriate conversion factors is required to obtain exact equivalence.

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

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[2] The boldface numbers in parentheses refer to the list of references at the end of this standard.

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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:*[3]

A574 Specification for Alloy Steel Socket-Head Cap Screws
A490 Specification for Structural Bolts, Alloy Steel, Heat Treated, 150 ksi Minimum Tensile Strength
B602 Test Method for Attribute Sampling of Metallic and Inorganic Coatings
E4 Practices for Force Verification of Testing Machines
E6 Terminology Relating to Methods of Mechanical Testing
E8 Test Methods for Tension Testing of Metallic Materials
E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications
E399 Test Method for Linear-Elastic Plane-Strain Fracture Toughness K_{Ic} of Metallic Materials
E812 Test Method for Crack Strength of Slow-Bend Precracked Charpy Specimens of High-Strength Metallic Materials (Withdrawn 2005)[4]
E1681 Test Method for Determining Threshold Stress Intensity Factor for Environment-Assisted Cracking of Metallic Materials
F519 Test Method for Mechanical Hydrogen Embrittlement Evaluation of Plating/Coating Processes and Service Environments
F606 Test Methods for Determining the Mechanical Properties of Externally and Internally Threaded Fasteners, Washers, and Rivets (Metric) F0606_F0606M
F2078 Terminology Relating to Hydrogen Embrittlement Testing
G5 Reference Test Method for Making Potentiodynamic Anodic Polarization Measurements
G129 Practice for Slow Strain Rate Testing to Evaluate the Susceptibility of Metallic Materials to Environmentally Assisted Cracking

2.2 *SAE Standards:*

J78 Self-Drilling Tapping Screws[5]
J81 Thread Rolling Screws[5]
J1237 Metric Thread Rolling Screws[5]

2.3 *ANSI/ASME:*

B18.18.2M Inspection and Quality Assurance for High-Volume Machine Assembly Fasteners, 1987[6]
B18.18.3M Inspection and Quality Assurance for Special Purpose Fasteners, 1987[6]
B18.18.4M Inspection and Quality Assurance for Fasteners for Highly Specialized Engineering Applications, 1987[6]

2.4 *Related Publications:*

ASTM STP 543, Hydrogen Embrittlement Testing, 1974[7]
ASTM STP 962, Hydrogen Embrittlement: Prevention and Control, 1985[7]

3. Terminology

3.1 *Symbols*—Terms not defined in this section can be found in Terminologies F2078 and E6 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}—the invariant threshold load. P_{th} is the basis for calculating the threshold stress or the threshold stress intensity.

3.1.5 $P_{th\text{-}n}$—the threshold load at a specified loading rate.

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

3.1.7 EHE—Environmental Hydrogen Embrittlement — test conducted in a specified hydrogen-charging environment.

3.1.8 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; examples include 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 or per Test Method E812 for bend specimens.

3.2.3 σ_i = stress at crack initiation.

3.2.4 σ_{th} = threshold stress.

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

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

3.2.7 $K_{th\text{-}IHE}$ = IHE threshold stress intensity at a specified loading rate — test conducted in air — not geometry dependent.

3.2.8 $K_{th\text{-}EHE}$ = EHE threshold stress intensity at a specified loading rate — test conducted in a specified hydrogen charging environment — not geometry dependent.

3.2.9 KI_{SCC} = invariant value of the threshold stress intensity for stress corrosion cracking—test conducted under open circuit corrosion potential or freely corroding conditions—not geometry dependent.

3.2.10 KI_{IHE} = invariant value of the IHE threshold stress intensity — test conducted in air — not geometry dependent.

[3] 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.

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

[5] Available from Society of Automotive Engineers (SAE), 400 Commonwealth Dr., Warrendale, PA 15096-0001.

[6] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036.

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

3.2.11 KI_{EHE} = invariant value of the EHE threshold stress intensity — test conducted in a specified hydrogen charging environment — not geometry dependent — equivalent to KI_{EAC}.

3.2.12 FFS = Fast Fracture Strength.

3.2.13 SCG = Subcritical Crack Growth.

4. Summary of Test Method

4.1 The test method is based on determining the onset of subcritical crack growth with a step modified, incrementally increasing, slow strain rate test (Practice G129) under displacement control **(3), (4), (5)**.

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

4.2.1 By progressively decreasing the 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 intensity (KI_{SCC}, KI_{IHE}, or KI_{EHE}) or stress for a given geometry with regard to the loading rate, is the threshold for the onset of crack growth due to hydrogen embrittlement.

4.5 In tension (T) and bending (B), the onset of SCG as a result of hydrogen in steel is identified by a concave decrease in load while holding the displacement constant. At net section yielding or above, a convex load drop is also observed.

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.

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 (see Pollock **(6)** and **(7)**).

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 **(8)** (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.

5.7 Annex A1 describes the application of this standard test method to hydrogen embrittlement testing of fasteners.

6. Apparatus

6.1 *Testing Machine*—Testing machines shall be within the guidelines of calibration, force range, resolution, and verification of Practices E4.

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 saturated calomel electrode (SCE) or equivalent reference electrode such as Ag/AgCl in accordance with Test Method G5. The imposed potential is typically cathodic, ranging from 0.0 to −1.2 V versus SCE (V_{SCE}) in a 3.5 weight percent NaCl solution **(9)**.

6.4 Equipment, such as RSL (trademarked),[8] for determining the onset of SCG with a step modified, incrementally increasing, slow strain rate test under displacement control.

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 Test Methods F606, ANSI/ASME B18.18.2M, B18.18.3M, or B18.18.4M and Test Method B602 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 **(10)**.

7.2.1 Fracture mechanics-type specimens are defined in standards such as Test Method E399.

[8] The sole source of supply of the apparatus known to the committee at this time is Fracture Diagnostics International, 20261 SW Acacia St., Newport Beach, CA 92660, http://www.fracturediagnostics.net. 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.

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 F519 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 (P_{th}):*

8.1.1 This test protocol requires that a minimum of three samples be tested to establish the threshold load, P_{th}. Load one sample to rupture at a rate consistent with Test Methods E8 to establish the fast fracture strength (FFS) or load, P_{FFS}, for a given specimen geometry, ($P_{FFS} = P_c$ in Fig. 1). This test provides the baseline reference data.

8.1.2 The specific load profile depends on the hardness of the samples within the ranges of ≥33 HRC to 45 HRC; >45 HRC to 54 HRC; and >54 HRC. The notation used for the incremental step load profile is (# / %P_{MAX}/hrs) where # is the number of steps, % P_{MAX} is the percent of the maximum anticipated load at each step, and hrs is the hold time for each step. For the hardness range of ≥33 HRC to 45 HRC, the loading profile is (10/5/2,4) or an initial loading profile of 10 steps at 5 % of P_{FFS} at each step for a hold time of 2 h, followed by 10 steps at 5 % of P_{FFS} at each step for a hold time of 4 h. Correspondingly, for hardness range of >45 HRC to 54 HRC, the loading profile is (10/5/1,2) and for > 54 HRC, the loading profile is (20/5/1).

8.1.3 In addition to the specific load profile, the subsequent P_{MAX} for each load profile is set to 1.1 times the $P_{th\text{-}n}$ of the previous test. The purpose of changing the maximum profile load is to reduce the loading rate and increase the resolution because each subsequent test sample results in a smaller step load.

8.1.4 The load $P_{th\text{-}n}$ is the threshold load, which is the load corresponding to the step before the onset of crack growth for a specific loading rate.

8.1.5 The invariant threshold load for the onset of hydrogen induced stress cracking P_{th}, is used to calculate KI_{EHE}, KI_{SCC}, or KI_{IHE}. The invariant threshold load is attained when the difference between two subsequent threshold loads is less than 5 % of P_{FFS}. The value of $P_{th\text{-}EHE}$, $P_{th\text{-}SCC}$, or $P_{th\text{-}IHE}$ is the lowest measured threshold value.

8.1.6 Referencing Fig. 1, the step load testing protocol can be summarized as follows:

SN(1)–Baseline: fast fracture test of specimen after plating to measure $P_{MAX} = P_{FFS}$. (This ensures that no cracks initiated or softening occurred during the plating process)

For the hardness range of >54 HRC (see Fig. 1)
SN(2)–(20/5/1) @ $P_{MAX} = P_{FFS}$; → $P_{th\text{-}1}$
SN(3)–(20/5/1) @ $P_{MAX} = 1.1 \times P_{th\text{-}1}$; → $P_{th\text{-}2}$
SN(4)–(20/5/1) @ $P_{MAX} = 1.1 \times P_{th\text{-}2}$; → $P_{th\text{-}3}$
and if necessary;
SN(5)–(20/5/1) @$P_{MAX} = 1.1 \times P_{th\text{-}3}$; → $P_{th\text{-}4}$
measures $P_{th\text{-}EHE}$, $P_{th\text{-}SCC}$, or $P_{th\text{-}IHE}$ when $\Delta P_{th} \leq 5\ \%\ P_{FFS}$

or,
For the hardness range of >45 HRC to 54 HRC (see Fig. 2)
SN(2)–(10/5/1,2) @ $P_{MAX} = P_{FFS}$; → $P_{th\text{-}1}$
SN(3)–(10/5/1,2) @ $P_{MAX} = 1.1 \times P_{th\text{-}1}$; → $P_{th\text{-}2}$
SN(4)–(10/5/1,2) @ $P_{MAX} = 1.1 \times P_{th\text{-}2}$; → $P_{th\text{-}3}$
and if necessary;
SN(5)–(10/5/1,2) @ $P_{MAX} = 1.1 \times P_{th\text{-}3}$; → $P_{th\text{-}4}$
measures $P_{th\text{-}EHE}$, $P_{th\text{-}SCC}$, or $P_{th\text{-}IHE}$ when $\Delta P_{th} \leq 5\ \%\ P_{FFS}$

or,
For the hardness range of ≥33 HRC to 45 HRC (see Fig. 3)
SN(2)–(10/5/2,4) @ $P_{MAX} = P_{FFS}$; → $P_{th\text{-}1}$
SN(3)–(10/5/2,4) @ $P_{MAX} = 1.1 \times P_{th\text{-}1}$; → $P_{th\text{-}2}$
SN(4)–(10/5/2,4) @ $P_{MAX} = 1.1 \times P_{th\text{-}2}$; → $P_{th\text{-}3}$
and if necessary;
SN(5)–(10/5/2,4) @ $P_{MAX} = 1.1 \times P_{th\text{-}3}$; → $P_{th\text{-}4}$
measures $P_{th\text{-}EHE}$, $P_{th\text{-}SCC}$, or $P_{th\text{-}IHE}$ when $\Delta P_{th} \leq 5\ \%\ P_{FFS}$

8.1.7 Crack growth shall be considered to have occurred if the measured load on a sample drops by more than the

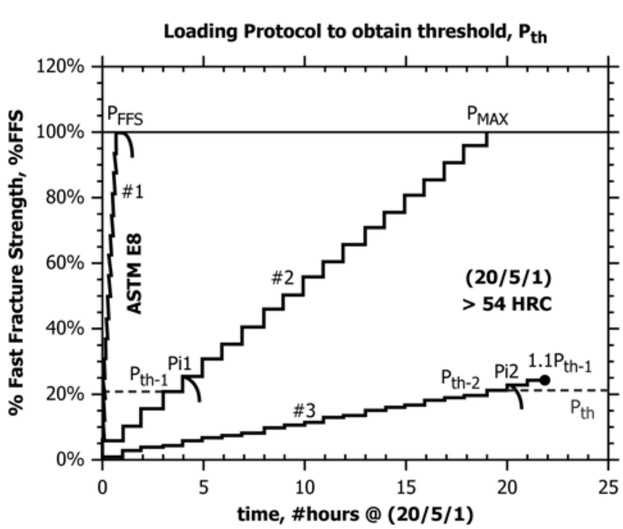


FIG. 1 Schematic of a (20/5/1) Step Loading Profile to Determine Threshold for the Hardness of Steel >54 HRC

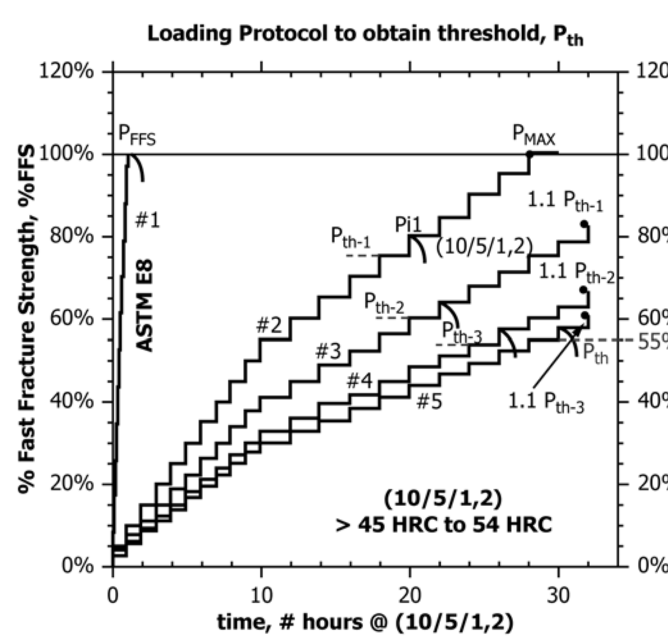


FIG. 2 Schematic of a (10/5/1,2) Step Loading Profile to Determine Threshold for the Hardness of Steel >45 HRC to 54 HRC

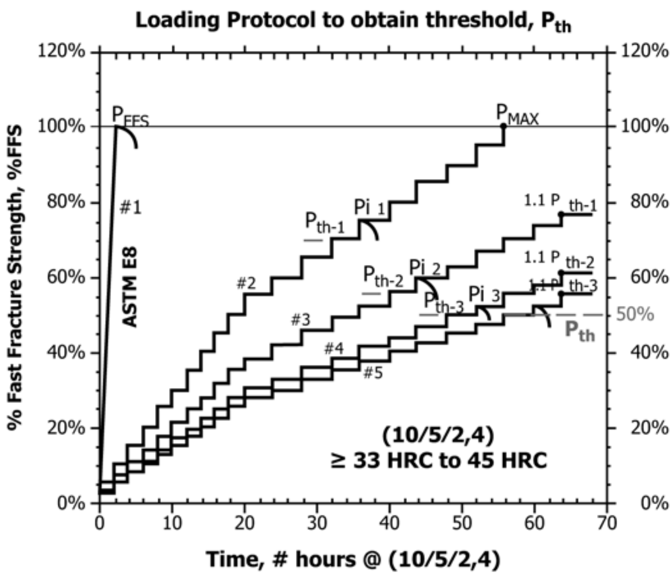
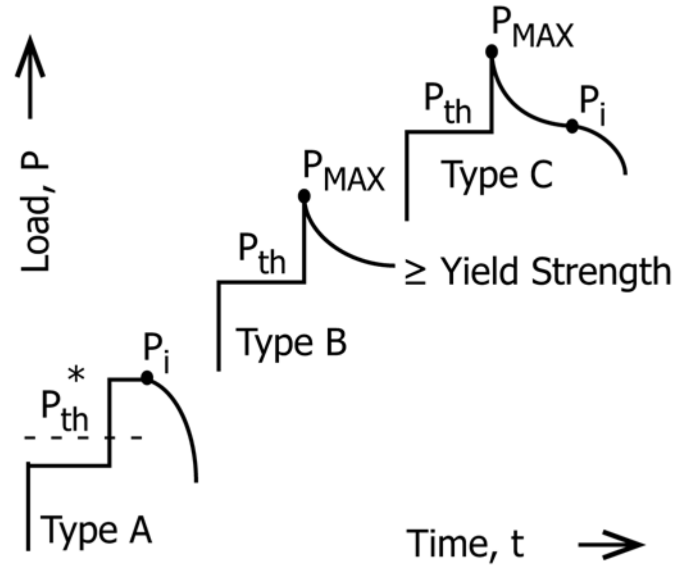


FIG. 3 Schematic of a (10/5/2,4) Step Loading Profile to Determine Threshold for the Hardness of Steel ≥ 33 HRC to 45 HRC



NOTE 1—*See Fig. 5 for calculation of additional increment.

FIG. 4 Definition of Crack Initiation Load, P_i Load and Threshold Load, P_{th}

established accuracy of the test apparatus, while the displacement is held constant, with the exception identified in 8.1.7.1.

8.1.7.1 The threshold is calculated from the load at the last step to maintain the load for the duration of the step. The threshold is defined as the stress or stress intensity calculated from the load at the onset of crack growth. A 5 % NFS load drop is used as an arbitrary guideline for the measurement of the onset of crack growth and is appropriate for materials with a rapid crack growth rate. For materials with extremely slow crack growth rates, a lesser value of load drop should be utilized that is more consistent with the visual detection of a load drop.

8.1.7.2 Any load drop depicted as an increasing rate (convex) shall be attributed to SCG in the specimen. The load is defined as the crack initiation load, P_i(see Fig. 4, Type A). The threshold load, P_{th}, is the step before initiation of crack growth.

8.1.8 If the load is maintained for only a fraction of the duration of the step (x), prior to SCG, the threshold can be estimated to be an additional increment above the last complete step (y) by a corresponding fractional amount of the step; that is, Δ = (x/y) of 5 % P_{max} used in the example in Fig. 5. If cracking begins immediately on reaching the next step (x = 0, Fig. 5), then use the previous load as the threshold, P_{th}.

8.1.9 Any load drop depicted as a decreasing rate (concave) shall be attributed to plasticity or creep in the specimen. This is not considered crack growth and is not defined as the crack initiation load, P_i (see Fig. 4, Type B). This behavior only occurs when the stress at the crack tip attains or exceeds the yield strength of the material. This is not a threshold value.

8.1.10 The load at the transition from a constant or decreasing rate to an increasing rate (concave to convex) is defined as the crack initiation load, P_i (see Fig. 4, Type C). The threshold load, P_{th}, is the step before initiation of crack growth.

8.1.11 Verification of crack growth is obtained by loading the tested specimen to fracture. Methods such as Test Methods

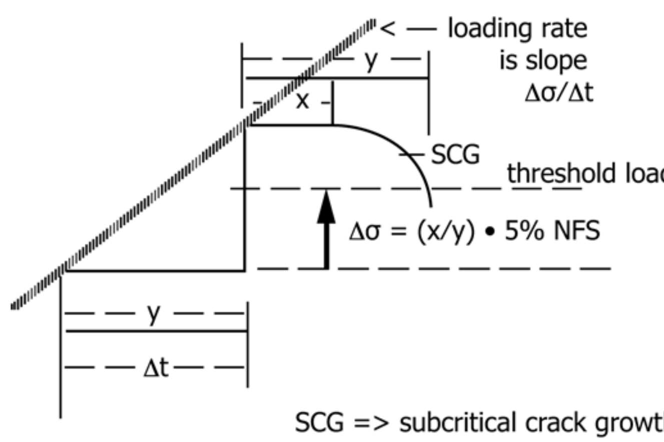


FIG. 5 Extrapolation Method for Type A Threshold Load Determination

E8 or Test Method E399 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 per Test Method E812 for bend specimens,

where:

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

9.3 The ultimate tensile strength (UTS) per Test Methods E8 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.

9.6 Stress intensity parameters per Test Method E399 are calculated from the load measurements in section 8.1.

9.7 The strain rate in units of in./in./s can be calculated by dividing the slope (see Fig. 5) of the loading rate ($\Delta\sigma$ / Δt) in units of ksi/second by the elastic modulus in units of ksi. In accordance with Practice G129, the loading rate should range from 10^{-5} s^{-1} to 10^{-8} s^{-1}.

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 types can be either fracture mechanics or irregular geometry. Fracture mechanics-type specimens with specified geometry shall be reported as described in Test Method E399. 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 and orientation specific and deviations will occur from one type of sample to another of the same material if identical 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 the loading profile similar to section 8.1.2.

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 E29 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 decreasing loading rate; delayed brittle failure; displacement control; fasteners; hydrogen embrittlement threshold; hydrogen induced stress cracking; rising step load; slow strain rate

ANNEX

A1. APPLICATION TO HYDROGEN EMBRITTLEMENT TESTING OF FASTENERS

INTRODUCTION

This annex addresses the specific use of this standard to determine the threshold stress for the onset of hydrogen embrittlement of fasteners. 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 defined in 1.3. Alloy/Coating systems should be specified. The Open Circuit Potential (OCP) or Corrosion Potential (E_{CORR}) should be measured in a 3.5 % NaCl solution to characterize the galvanic corrosion behavior of the coating relative to the specific grade of steel. A scribe mark should be inserted in the coating at the root of a thread to simulate a damaged coating or "holiday" in the coating. As a baseline, fasteners are tested in bending in air at Test Methods E8 loading rates to measure the Fast Fracture Strength, FFS(B) to obtain P_{MAX}. To measure the hydrogen embrittlement susceptibility (EHE), fasteners are tested in a salt-water environment using the step load procedure of Section 8 to measure P_{th}, except as modified herein. A minimum of three tests is required.

A1.1 Load Requirements

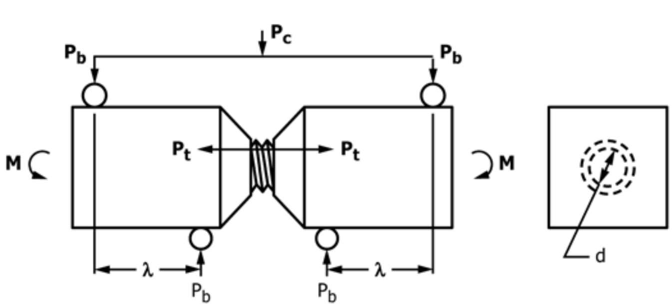
A1.1.1 Tensile fasteners can range from very small screws to 4-in. diameter (4"D) bolts per ANSI/ASME B18 or Specification A490, requiring a large load range for tensile testing from pounds to 1000 tons; therefore, it is wise to use the mechanical advantage of bending to reduce the testing loads. It is also more representative of the actual installation, wherein there is always some component of bending. For 4-point bending, such as a Test Method F519 Type 1c specimen with self-loading frame, the tensile loads are reduced by d/8L, where d = the minimum diameter of the fastener and L = length of them moment arm; or as an example, with an 8-in. long moment arm, the load in bending required to reach the tensile strength for a 1"D bolt would be 1/64 the load required in tension. Compression loading a bolt in 4-point bending is shown in Fig. A1.1 and a 4-point bend loading frame for a Type 1c specimen that can be adapted to fasteners is shown in Fig. A1.2.

A1.2 Bare Metal Surfaces

A1.2.1 During processing, hydrogen can be introduced into the fastener during melting of the steel, as a result of processing, such as pickling, or during any plating or coating operation. Subsequent thermal baking treatments are used to remove the hydrogen. Guidelines of time and temperature are provided under assumed process controls, but to quantitatively evaluate the level of hydrogen contamination or effectiveness of the baking treatment, mechanical testing must be conducted because any quantitative measurement of hydrogen is insufficient information, since the amount is alloy and microstructurally dependent. Testing is conducted in air and the parameter that must be measured is the threshold stress for the onset of hydrogen-induced stress cracking, which is calculated from P_{th}.

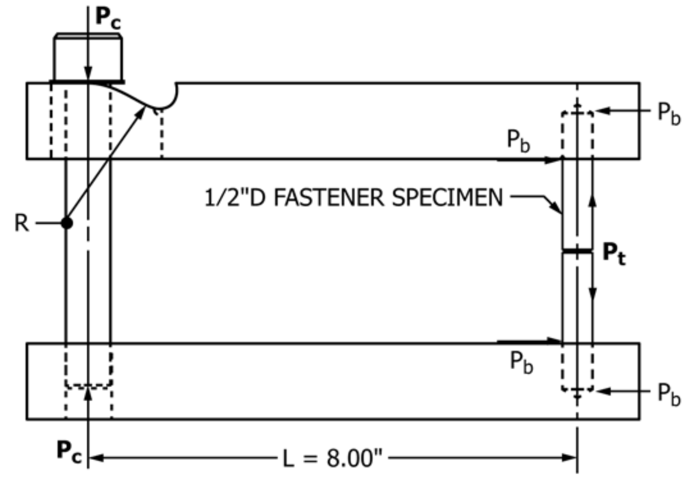
A1.3 Environmental Effects

A1.3.1 The environment and primarily aqueous environments are in situ sources of introducing hydrogen into the fastener while in service. This further contamination with hydrogen can occur on both bare metal surfaces, and even more aggressively on coated metal surfaces due to galvanic corrosion effects. Although platings or coatings are used to protect against general corrosion, they can be an aggressive source of hydrogen contamination in service. The fastener must always be maintained cathodic to the coating and the cathode is where the hydrogen is generated in an aqueous solution. When a cathodic coating is used, or the fastener is anodic, a crack in the coating can cause aggressive pitting corrosion into the substrate.



NOTE 1—For Net Tensile Stress (σ_t) in bending = σ_b , P_t = 8M/d ≥ 1.2$P_{UTS\text{-}E8}$, where M = $P_{b\lambda}$, and P_c = 2P_b.

FIG. A1.1 Compression Four-Point Bend Loading for Tensile Fasteners with Adapters



NOTE 1—For Net Tensile Stress (σ_t) in bending = σ_b , P_t = 8M/d ≥ 1.2$P_{UTS\text{-}E8}$, where M = LP_c.

FIG. A1.2 Four-Point Bend Loading Fixture for Fasteners per Test Method F519 Type 1c

A1.3.2 The metals involved in making a fastener can vary from zinc used in making the coating at -1.2 V_{SCE}, (Volts versus Saturated Calomel Electrode) to zero-V_{SCE} for stainless steels under freely corroding, E_{CORR} or OCP conditions. For plain carbon or low alloy steels that have an OCP of about -0.7 V_{SCE}, the cathodic or hydrogen charging conditions can be as much as 0.5 V with a zinc coating. For stainless steel fasteners that are noble or at 0 V_{SCE} when passivated, the cathodic charging potential with zinc or aluminum can be as much as 1.0 V. Any damage in the coating, immediately causes a "worst case" situation of a very local hydrogen attack. Without any damage, the hydrogen infusion is controlled by the permeability of the coating. As a result, to evaluate environmentally induced hydrogen effects, the actual fastener material should be tested with the coating in the actual environment and again, the parameter that must be measured is the threshold stress for the onset of hydrogen-induced stress cracking, which is calculated from the threshold load, P_{th}.

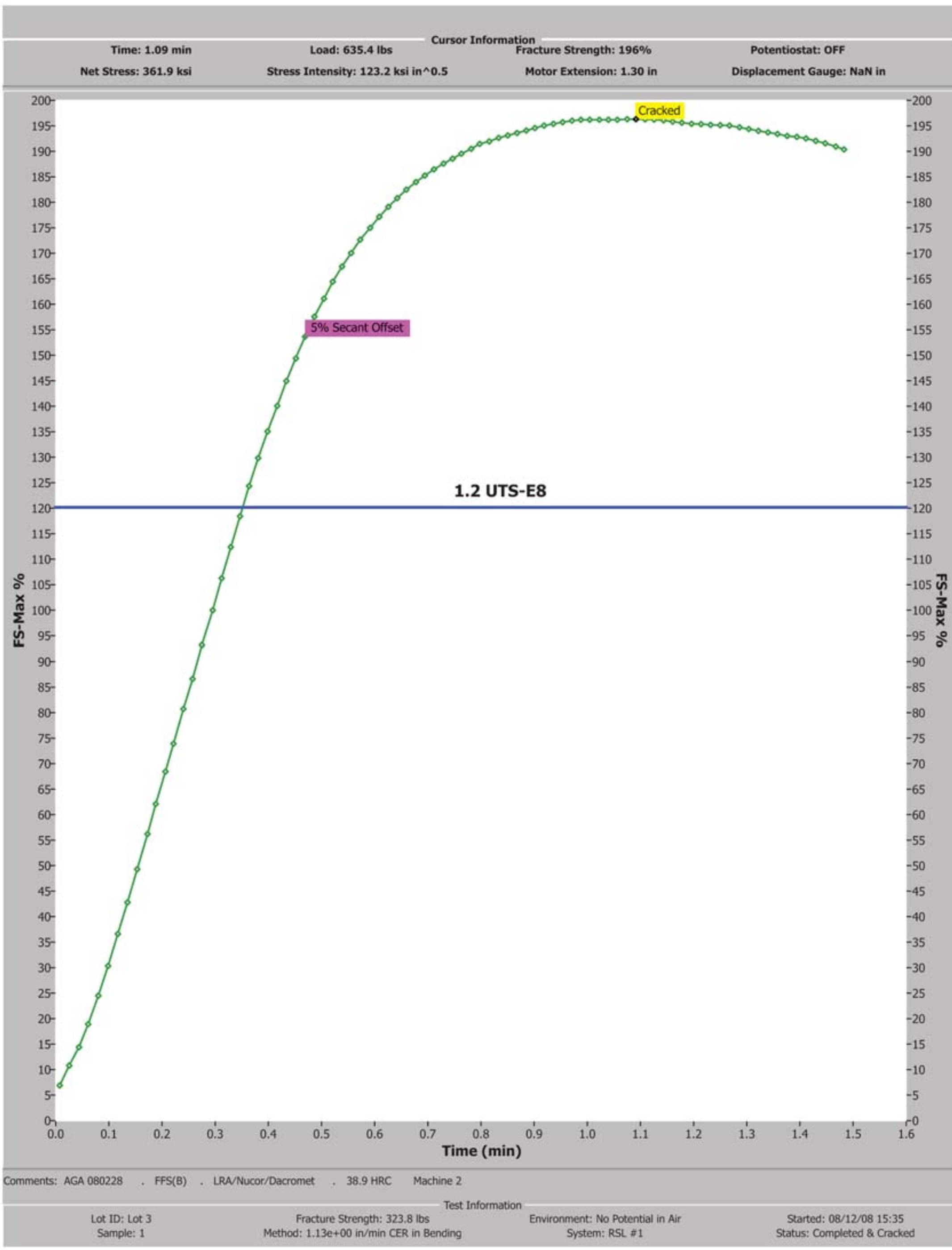
A1.4 Imposed Galvanic Potential

A1.4.1 To evaluate the general effects of coatings on fasteners and the influence of microstructure, bare bolts can be tested in an aqueous environment with an imposed cathodic potential to simulate the effects of a coating. These conditions would represent the "worst case" conditions of a damaged coating. The testing range of the imposed cathodic potentials could vary from that of zinc (–1.2 V_{SCE}) to the OCP of the fastener material. Again, the parameter that must be measured is the threshold stress for the onset of hydrogen-induced stress cracking, which is calculated from P_{th}.

A1.5 Bolt Diameter, Maximum Load in Bending

A1.5.1 The larger the diameter, the higher the maximum load. In bending, the ultimate or limiting bend stress is referenced as the Modulus of Rupture (MoR) or Rsb, the specimen strength ratio in bending per Test Method E812 or as defined in Terminology F2078, have a maximum value of about 2 x UTS_{E8}. Therefore, from the hardness conversion to tensile strength, the maximum anticipated or ultimate load (PULT) for a given bolt diameter in bending can be calculated.

A1.5.2 As a baseline, fasteners are tested in bending in air at Test Methods E8 loading rates to measure the Fast Fracture Strength, FFS(B), to obtain $P_{MAX} \leq P_{ULT}$ (Fig. A1.3). The initial value of P_{MAX} is taken as the 5 % secant offset. The minimum value should be ≥ 1.2 UTS, which corresponds to 75



NOTE 1—FFS(B) for 1.0"D bolt at 39 HRC with P_{MAX} at 2.0 UTS-E8 and the 1.2 UTS-E8 acceptance level and the 5 % secant offset identified.

FIG. A1.3 Fast Fracture Strength in Bending

% NTS (NTS = 1.6 UTS) in a notched round bar tension test per Test Method F519, Type 1a. P_{MAX} is used as the initial load in the step load test.

A1.5.3 Testing is to be carried out using tension bolts. The bolts shall be of 5D in length. Since all tension bolts are designed to fail in the threads, even in a wedge tensile test, the head may be removed. The test program requires a minimum of three samples to measure the threshold.

A1.6 Hardness

A1.6.1 The most significant variable for susceptibility to hydrogen induced cracking in applying this standard to steel fasteners is the hardness. Therefore, for the most efficient selection of specific loading rates the recommended loading profiles are grouped according to the hardness, measured per Test Methods F606.

HRC > 54; (20/5/1) profile: Fig. 1
HRC > 45 to 54; (10/5/1,2) profile: Fig. 2
HRC ≥ 33 to 45; (10/5/2,4) profile: Fig. 3

Each hardness range has an optimum loading sequence that minimizes the total test time for that hardness range. Obviously, the lower the hardness, the slower the loading rate or the longer the total test time to measure the threshold load. An actual (10/5/2,4) test profile is shown in Fig. A1.4.

A1.7 Coating

A1.7.1 The coating that is to be evaluated shall be applied to all fastener components used under normal production conditions.

A1.7.2 Alloy/Coating system should be specified. The OCP should be measured in a 3.5 % NaCl solution to characterize the galvanic corrosion behavior of the coating relative to the specific grade of steel. Insert a scribe mark in the coating at the root of a thread to simulate a damaged coating or "holiday" in the coating. The hardness of the scribe should be less than the hardness of the specimen in order to avoid scarring the surface of the notch.

A1.8 Test Procedures

A1.8.1 *Hydrogen Embrittlement Tests*—The threshold load, P_{th}, is obtained on completion of a minimum of three tests. The threshold is the lowest value of two consecutive tests, when the difference between them is within 5 % of FFS(B). Once an invariant value is obtained, no further tests are required. Otherwise, additional tests shall be performed following the protocol of 8.1.6 until an invariant value within 5 % of FFS(B) is obtained from two consecutive tests. Test results shall be autographically recorded in terms of load versus time and included as part of the report.

A1.8.2 *Testing Equipment*—A computerized, four-point bend, digital displacement controlled loading frame that is capable of stepping in 0.5 % load steps and is programmed to increase incrementally in steps of load and time to vary the effective strain rate at the root of the notch between 10^{-5} and $10^{-8}s^{-1}$ is required to conduct these tests. Testing machines shall be within the guidelines of calibration, force range, resolution, and verification of Practice E4.

A1.8.3 *Fixtures*—Various types of adapters may be used for the four-point bending to transmit the measured load applied by the testing equipment to the test specimen. Fig. A1.1 illustrates an example of a four-point bend-loading fixture that could be adapted to a Test Method F519, Type 1c loading frame (See Fig. A1.2).

A1.8.4 *Test Environment*—The test shall be conducted in a hydrogen producing environment by immersion of the fastener into a 3.5 % NaCl solution under freely corroding or OCP conditions or under potentiostatic control by imposing a galvanic cathodic potential in salt water contained in an appropriate inert container.

A1.8.5 *Potentiostatic Control*—For testing of the sensitivity of the fastener to EHE, an inert container and potentiostat can be used to impose a cathodic potential slightly more negative than the OCP of the coated fastener. The cathodic charging potential of the fastener is measure relative to a reference Saturated Calomel Electrode (SCE) or equivalent reference electrode such as with Ag/AgCl in accordance with Test Method G5.

A1.8.6 *OCP Testing*—If the fastener can be isolated from all other metal contacts, the test can be conducted under freely corroding or OCP conditions. Such testing most accurately represents the combined effects of general corrosion on the surface of the coating and stress corrosion cracking.

A1.9 Acceptance Criterion

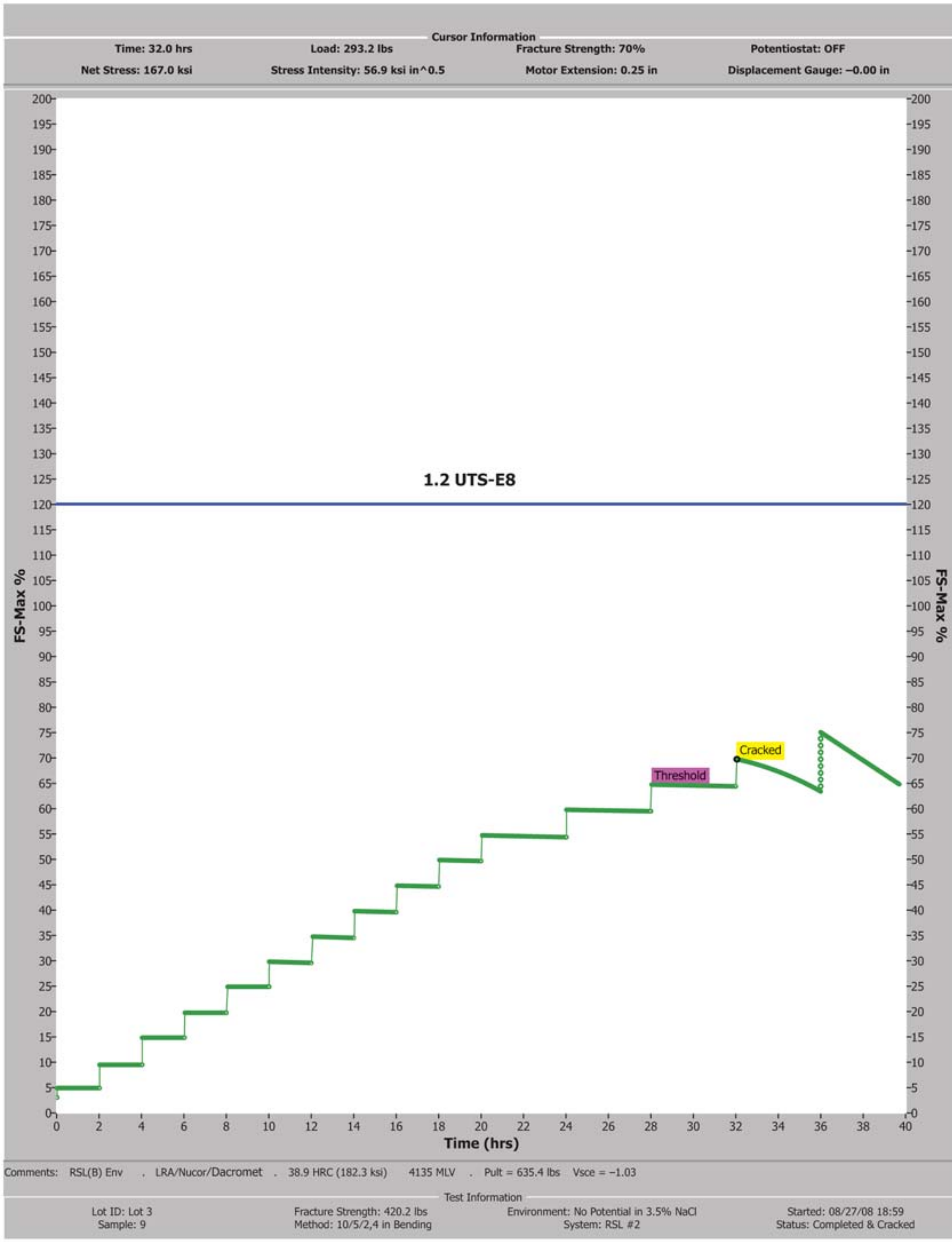
A1.9.1 Fasteners shall be tested to the same hydrogen embrittlement acceptance criterion as with Test Method F519, Type 1a specimens, which also require a "worst case" scenario and a threshold stress greater or equal than 75 % of the notch tensile strength (NTS) of the Type 1a specimen.

A1.9.1.1 The worst-case scenario for a coated fastener consists of Wedge Tension Testing the coated fastener per Test Methods F606 to verify that the fastener still meets the specified minimum ultimate tensile load after it has been coated. The coating shall have a Holiday (a scribe mark through the coating at the root of the thread). For the Type 1a specimen with *d*/D = 0.7, the threshold tensile stress is 75 % NTS (where NTS = 1.6 UTS_{E8} and 75 % NTS = 1.2 UTS_{E8}). In terms of load, the equivalent acceptance criterion in tension to be used on the coated bolts is $P_{t\text{-}th} \geq 1.2\ P_{t\text{-}UTS\ E8}$.

A1.9.1.2 Bending must be used to attain the tensile load to produce the equivalent stress, since *d*/D for fasteners is always greater than 0.8. Pure bending stresses (4-pt) using a rising step load under displacement control shall be used to determine a hydrogen embrittlement threshold stress level. Pure bending (4-pt) shall be used so that the stress may be calculated anywhere along the length of the fastener.

A1.9.1.3 Equivalence between pure tension and pure bending is based on the stress at the root of the thread. A fastener adapter loaded in four-point bending is shown in Fig. A1.1. The fastener may be cut to isolate a segment with threads. Four point bend loading may be obtained by applying an axial compression load (Fig. A1.1) or by using leverage in bending with a Test Method F519, Type 1c load fixture, Fig. A1.2.

A1.9.1.4 To be consistent with a notched round tensile coupon per Test Method F519, Type 1a, the load at threshold in



NOTE 1—For 1”D bolt at 39 HRC with P_{MAX}(cracked) and P_{th} identified at 65 % UTS-E8 that failed the acceptance criterion.

FIG. A1.4 ASTM F1624 Fig. 1C (10/5/2,4) RSL Curve

bending should exceed 1.2 $P_{t\text{-}UTS}$ as a measure of sufficient resistance to environmentally induced hydrogen stress cracking.

The tensile stress in bending (σ_b) and in tension (σ_t) at the root of the thread can be computed using the formulas:

$$\sigma_b = (32M/\pi d^3);\ and\ \sigma_t = (4P_t/\pi d^2) \qquad (A1.1)$$

where:

d = minimum thread diameter (in.), and
M = applied moment (in-lb).

For equivalent stresses, $\sigma_b = \sigma_t$, the load in tension (P_t) is given by:

$$P_t = 8M/d \qquad (A1.2)$$

A1.9.2 When applying the four-point load (P_b in Fig. A1.1) in an axial load compression fixture, the applied moment, $M = P_b\lambda$ and $P_c = 2P_b$ (Fig. A1.1). Therefore solving P_t in terms of P_c:

$$P_t = (4\lambda/d)P_c \geq 1.2\, P_{t-UTS\ E8} \quad (A1.3)$$

or

$$Pc \geq (d/4\lambda)1.2P_{t-UTS\ E8} \text{ Acceptance level, axial compression} \quad (A1.4)$$

A1.9.3 When applying the four-point load (P_b in Fig. A1.1) in a bend fixture such as the Type F519, Type 1c fixture with L = 8 in. and solving P_t in terms of P_c, where $M = 8L$ (Fig. A1.2):

$$P_t = (8L/d)P_c \geq 1.2\, P_{t-UTS\ E8} \quad (A1.5)$$

or

$$P_c \geq (d/8L)1.2P_{t-UTS\ E8} \text{ Acceptance level, compression bend} \quad (A1.6)$$

A1.9.4 *Example*—Per Specification A574, for a nominal 1/2 -1/3 in. alloy steel socket head cap screws, the hardness range is 39–45 HRC, the specified minimum ultimate tensile load = $P_{t\text{-UTS}} \geq$ 25,500 lb (25.5 kips) and, d_t = 0.400 in. The "worst case" or maximum hardness is 45 HRC, which equals 215 ksi UTS_{E8} or 27 kips. Therefore, the threshold tensile load $P_{t\text{-th}}$ = 1.2 $P_{t\text{-UTS}}$ = 32.4 kips.

A1.9.4.1 Using an axial load compression fixture (A1.9.2) for d_{min} = 0.4 in., λ = 1 in., and 1.2 $P_{t\text{-UTS}}$ = 32.4 kips, the acceptable minimum equivalent compression threshold load is:

$$P_{c-th} \geq 3.2\ kips \quad (A1.7)$$

A1.9.4.2 Using a bend fixture such as the Type F519, Type 1c fixture (A1.9.3) with L = 8 in., d_{min} = 0.4in., and 1.2 $P_{t\text{-UTS}}$ = 32.4 kips, the acceptable minimum equivalent compressive bend threshold load (P_c in Fig. A1.2) is:

$$P_{c-th} \geq 200\ lb \quad (A1.8)$$

A1.9.5 A typical 4-pont bend curve of a coated 1"D A490 bolt is shown in Fig. A1.3, with the 5 % secant offset load shown to be equal to 1.2 UTS_{E8} and the max load to be equal to 2.0 UTS_{E8}. Fig. A1.4 is an example of the EHE threshold load measured per F1624 for the bolt, equal to 0.9 UTS_{E8}. Per the criterion set forth in this standard, this coating would have failed to meet the minimum threshold requirements of equal to ≥ 1.2 UTS_{E8}.

A1.10 Report

A1.10.1 A test report shall be produced in accordance with Section 10. See Fig. A1.3 and Fig. A1.4. The report shall include:

A1.10.1.1 Executive summary,

A1.10.1.2 Introduction,

A1.10.1.3 Detailed coating process description and coating characteristics (including product name and coating applicator),

A1.10.1.4 Test methodology,

A1.10.1.5 Detailed results,

A1.10.1.6 Discussion of results, and

A1.10.1.7 Conclusion.

A1.10.1.8 All third party certifications and test reports shall be included in the appendixes.

REFERENCES

(1) Interrante, C. G., Raymond, L., "Hydrogen Damage," in *Corrosion Tests and Standards, MNL 20*, Baboian, R., Ed., American Society for Testing and Materials, West Conshohocken, PA, 1995, p. 272.

(2) Raymond, L., "The Susceptibility of Fasteners to Hydrogen Embrittlement and Stress Corrosion Cracking," in *Handbook of Bolts and Bolted Joints*, Marcel Decker, Inc., New York, 1998, p. 723.

(3) Raymond, L., "Application of a Small Specimen Test Method to Measure the Subcritical Cracking Resistance in HY-130 Steel Weldments," under Contract No. DTNSRDC-SME-CR-09–82, "High-Strength Steel Weldment Subcritical Cracking Program," under Navy Contract N00167–81–C-0100, LRA Labs (formerly named METTEK), Final Report No. 210123, March 1982.

(4) Raymond, L. and Crumly, W. R., "Accelerated, Low-Cost Test Method for Measuring the Susceptibility of HY-Steels to Hydrogen Embrittlement," in *Proceedings of the First International Conference on Current Solutions to Hydrogen Problems in Steel*, American Society for Metals, Metals Park, OH, Nov. 1982, pp. 477–480.

(5) Raymond, L., "Accelerated Stress Corrosion Cracking Screening Test Method for HY-130 Steel," under SBIR Phase I and Phase II NAVSEA Contract No. N00024–89–C-3833, LRA Labs, Final Report No. NAVSEA 80058, Dec. 1989 and Dec. 1993, respectively.

(6) Pollock, W. J., "Assessment of the Degree of Hydrogen Embrittlement Produced in High-Strength 4340 Steel by Plating and Baking Process Using Slow Strain Rate Testing," ASTM STP 962, p. 68.

(7) Pollock, W. J., "Assessment of the Degree of Hydrogen Embrittlement Produced in High-Strength 4340 Steel by Paint Strippers Using Slow Strain Rate Testing," ASTM STP 962, p. 372.

(8) Raymond, L., "Plating or Coating Processes Can Be Monitored for Control of Hydrogen Embrittlement," in American Fastener Journal, March/April 1998, p. 26.

(9) Tyler, P. S., Levy, M., Raymond, L., "Investigation of the Conditions for Crack Propagation and Arrest Under Cathodic Polarization by Rising Step Load Bend Testing," *Corrosion*, NACE, Vol. 47, No. 2, Feb. 1991, pp. 82–86.

(10) National Materials Advisory Board, "Rapid Inexpensive Tests for Determining Fracture Toughness," NMAB 328, National Academy of Sciences, Washington, DC, 1976 .

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