Designation: F1624 – 06

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.

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 acceptable inch-pound units shall be regarded as the standard. 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.

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.*

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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.

2. Referenced Documents

2.1 *ASTM Standards:*[3]

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[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, Direct Tension Indicators, and Rivets

F2078 Terminology Relating to Hydrogen Embrittlement Testing

G5 Reference Test Method for Making Potentiostatic and 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}—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 hydrogen-charging 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; 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_{IHE} = invariant value of the IHE threshold stress intensity — test conducted in air — not geometry dependent.

3.2.10 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}.

3.2.11 NFS = Notched Fracture Strength.

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.

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[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] Withdrawn. 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.

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_{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 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.

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.

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**).

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.

NOTE 1—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*:

8.1.1 Load one specimen to rupture at a rate consistent with Test Methods E8 to establish the maximum fracture load or NFS for a given specimen geometry, (NFS = P_c in Fig. 1).

8.1.2 Another sample (#1) is tested by applying an incremental or step loads under displacement control in tension or four-point bending, programmed to attain P_c.

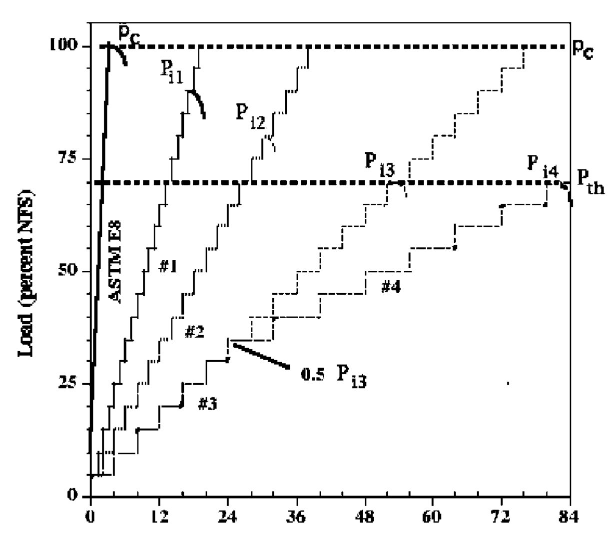

FIG. 1 Schematic of Suggested Protocol for a Step Loading Profile to Determine Threshold