Designation: F 1624 – 00

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

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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 (finite life) or below which fracture will never occur (infinite 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 four to 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 (>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 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. 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 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.*

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[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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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.

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

[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.