

Designation: F1624 – 09

StandardTest Method for Measurement of Hydrogen Embrittlement Threshold in Steel by the Incremental Step Loading Technique¹

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.

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1. Scope 1.2 This test method is used to evaluate quantitatively:

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.

1.2.1 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

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.

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.

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, 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 Screws⁵

J81 Thread Rolling Screws⁵

J1237 Metric Thread Rolling Screws⁵

2.3 ANSI/ASME:

B18.18.2M Inspection and Quality Assurance for High-Volume Machine Assembly Fasteners, 1987⁶

- B18.18.3M Inspection and Quality Assurance for Special Purpose Fasteners, 1987⁶
- B18.18.4M Inspection and Quality Assurance for Fasteners for Highly Specialized Engineering Applications, 1987⁶
- 2.4 Related Publications:

ASTM STP 543, Hydrogen Embrittlement Testing, 1974⁷ ASTM STP 962, Hydrogen Embrittlement: Prevention and Control, 1985⁷

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-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.

 $3.2.2 \,\sigma_{net}$ = 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 $\sigma_{\text{th-IHE}}$ = IHE threshold stress — test conducted in air — geometry dependent.

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

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

3.2.8 K_{th-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.

 $^{^{\}rm 4}\,{\rm The}$ 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 σ_{th-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 $\sigma_{\text{th-EHE}}$ 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^{TM⁸}, for determining the onset of SCG with a step modified, incrementally increasing, slow strain rate test under displacement control. 1624-09

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.

⁸ Equipment specifically designed to conduct this test method is available at www.fdi.nu/

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, $P_{\rm th}$. Load one sample to rupture at a rate consistent with Test Methods E8 to establish the fast fracture strength (FFS) or load, $P_{\rm FFS}$, for a given specimen geometry, ($P_{\rm FFS} = P_{\rm c}$ 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 \geq 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 \geq 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 $P_{\text{th-n}}$ 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 $P_{\text{th-n}}$ 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 $P_{\rm th}$, is used to calculate KI_{EHE}, KI_{SCC}, or KI_{IHE}. The invariant threshold load is attained when the



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

difference between two subsequent threshold loads is less than 5 % of $P_{\rm FFS}$. The value of $P_{\rm th-EHE}$, $P_{\rm th-SCC}$, or $P_{\rm th-IHE}$ 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)

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

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

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



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 \geq 33 HRC to 45 HRC

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, $\Delta = (x/y)$ 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 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:

I

$$A_{\rm net}$$
 = net cross-sectional area,

M = the applied moment,

= the distance from the neutral axis to the stressed

= 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.



SCG => subcritical crack growth

FIG. 5 Extrapolation Method for Type A Threshold Load Determination

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

9.5.2 A further designation of σ_{th-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 ($\Delta \sigma / \Delta t$) 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 $(P_{\rm th})$ and any threshold stress or stress intensity parameter that has been calculated from the threshold load.

NOTE 2—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 3—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

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https://standards.iteh.ai/catalog/standards/sist/91ae9ANNEX4-4e40-8934-1062168c4ee0/astm-f1624-09

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,