Standard Test Method for
Qualifying Coatings for Use on A490 Structural Bolts Relative to Environmental Hydrogen Embrittlement[1]

This standard is issued under the fixed designation F2660; 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.

1. Scope

1.1 This test method defines the procedures and tests to evaluate the effect of a coating system on the susceptibility to environmental hydrogen embrittlement (EHE) of an ASTM A490 high strength structural bolt.

1.2 This test method shall qualify a coating system for use with any size of A490 bolts (that is, ½ to 1-½ in.) high strength structural bolts, relative to EHE.

1.3 The characteristic to be evaluated by this test method is the susceptibility to EHE caused by hydrogen generated from corrosion protection of the steel bolt by sacrificial galvanic corrosion of the coating. Testing shall be performed on coated, specimen ASTM A490 bolts manufactured to the maximum susceptible tensile strength values (see Table 1) of the bolt (see Section 5 Specimen Bolt Requirements). The internal hydrogen embrittlement (IHE) susceptibility will also be inherently evaluated when the EHE is tested through this test method. There is no need for a separate IHE susceptibility test.

1.4 The values stated in inch-pound units are to be regarded as standard. No other units of measurement are included in this standard.

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:*[2]

A490 Specification for Structural Bolts, Alloy Steel, Heat Treated, 150 ksi Minimum Tensile Strength

E4 Practices for Force Verification of Testing Machines

E8/E8M Test Methods for Tension Testing 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

F1624 Test Method for Measurement of Hydrogen Embrittlement Threshold in Steel by the Incremental Step Loading Technique

F1789 Terminology for F16 Mechanical Fasteners

F2078 Terminology Relating to Hydrogen Embrittlement Testing

G3 Practice for Conventions Applicable to Electrochemical Measurements in Corrosion Testing

G15 Terminology Relating to Corrosion and Corrosion Testing (Withdrawn 2010)[3]

G44 Practice for Exposure of Metals and Alloys by Alternate Immersion in Neutral 3.5 % Sodium Chloride Solution

G82 Guide for Development and Use of a Galvanic Series for Predicting Galvanic Corrosion Performance

2.2 *Research Council on Structural Connections:*

Specification for Structural Joints Using High Strength Bolts (LRFD) Load and Resistance Factor Design

Specification for Structural Joints Using High Strength Bolts (ASD) Allowable Stress Design

2.3 *Other References:*

Townsend Jr., H. E., Met Trans, V6A, April, 1976

Raymond, L., The Susceptibility of Fasteners to Hydrogen Embrittlement and Stress Corrosion Cracking: Fastener System Design. In J. H. Bickford, & S. Nassar, Handbook of Bolts and Bolted Joints, New York, USA: Marcel Dekker, Inc., 1998, pp. 723-756

2.4 *International Standards Organization (ISO):*

ISO 17025 General Requirements for the Competence of Testing and Calibration Laboratories

3. Terminology

3.1 *Definitions:*

TABLE 1 Specimen Bolt Sizes and Wedge Tensile Load Values

Nominal Size	Minimum Length	Tensile Stress Area	Wedge Tensile Load	
(D)	(in.)	As (in.²)	Min. (lb)	Max. (lb)
1/2-13	1-1/2	0.1419	25,600	27,600
3/4-10	2-1/2	0.3345	59,000	63,000
1-8	5	0.6057	103,000	107,000

3.1.1 Terminology for this test method shall be used in accordance with Terminology F1789, Terminology F2078, and Terminology G15 except as described below.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *specimen lot, n*—at least 100 ASTM A490 specimen bolts manufactured in the same process from the same lot of a steel alloy.

3.2.2 *specimen bolt, n*—is a bolt with dimensions that conform to the requirements of A490 bolt dimensions but has mechanical properties, which exceed those of A490 requirements (see 5.1.1 and Table 1).

4. Significance and Use

4.1 This test method describes the testing procedure that shall be used to qualify a coating system that is under consideration for use on ASTM A490 high strength structural bolts made of any steel composition permitted by the A490 specification. The test method measures the susceptibility of coated specimen bolts to the influence of an externally applied potential (see 7.2.3.3) by testing for the threshold of embrittlement in a salt solution environment.

5. Specimen Bolt Requirements

5.1 *Coated Bolts:*

5.1.1 Testing shall be carried out using specially processed ASTM A490, Type 1 specimen bolts specially heat treated to achieve maximum strength values per Table 1 in order to qualify the Standard A490 bolts. Specimen bolts are designed to simulate a worst case material condition with respect to susceptibility to environmental hydrogen embrittlement (EHE). For this reason, wedge tensile values for specimen bolts exceed the maximum limits for A490 bolts; therefore, the specimen bolts shall not have standard A490 markings so they will not be identified as standard A490 bolts.

5.1.2 Specimen bolts shall be one of two nominal inch sizes: 3/4-10 UNC or 1-8 UNC. Alternatively, small specimen size bolts, 1/2-13 UNC may be used, but are subject to more severe strength requirements shown in 8.4.

5.1.3 Specimen bolts shall for a lot that displays the maximum wedge tensile load condition permitted by Specification A490, when tested in accordance with Test Methods F606. Table 1 gives the range of acceptable wedge tensile loads for each specimen bolt size.

5.1.4 Test results shall be provided by an ISO 17025 accredited laboratory. Wedge tensile strengths for the specimen bolts must be within the range specified in Table 1 and dimensional and compositional conformance to Specification A490 shall be provided by the supplier of each specimen lot.

5.2 *Coatings:*

5.2.1 The coating to be evaluated shall be applied to all specimen bolts under normal production conditions.

5.2.2 The process of coating specimen bolts shall include all post-coating processing under normal production conditions.

5.3 *Uncoated Bolts:*

5.3.1 Testing shall be carried out using ASTM A490 Type 1 bolts produced at the same time with the same processes and from the same lot of steel alloy as coated bolts.

6. Sample Quantities Required

6.1 A minimum of fifteen (15) bolts from any specimen lot shall be used for evaluation and qualification. Ten (10) bolts shall be coated and the remaining five (5) bolts shall remain uncoated. Additional samples may be required for repeat test and shall be held in contingency.

7. Test Procedures

7.1 *Open Circuit Potential (OCP):*

7.1.1 The freely corroding or Open Circuit Potential (OCP) shall be measured in 3.5% NaCl solution produced in accordance with Practice G44 to characterize the galvanic corrosion behavior of the coating relative to the steel bolt. The OCP measurement shall be made on a coated specimen bolt in accordance with Practice G3. The OCP measurement shall be taken using a potentiostat capable of making measurements with a resolution no less than ± 5 mV.

NOTE 1—If the coating is a known material then the measured OCP can be compared to the values described in Guide G82.

7.1.2 A second OCP test shall be performed and the two tests shall be compared for consistency. If the OCP test is not ± 5 mV with a known value for a known coating or with the other OCP test for an unknown coating, then a known material other than the coating shall be used to test the accuracy of the reference electrode. If the electrode is accurate, then another bolt sample shall be tested to obtain consistency. Some reasons for inconsistency include dissimilar materials in the test setup or coating voids that can change the OCP value.

7.2 *Environmental Hydrogen Embrittlement Testing:*

7.2.1 *Mechanical Test Set-up:*

7.2.1.1 The test shall be conducted on bolts that have been truncated by removal of the bolt head. Cut off the bolt head using a water cooled cut off saw or other device that does not cause excessive heating of the bolt. The length of the specimen bolt for testing shall be a minimum length of 1.5 inches and a maximum length of 4.0 inches. The truncated bolt specimen shall be adjusted to achieve the placement of a minimum of two threads between the gripping devices. The exposed threads shall be equally spaced on each side of the minor diameter of the threads. This placement of the bolt specimen in the gripping device is shown in Fig. 1.

7.2.1.2 The loading method required for this test is four-point (4 pt) bend, which produces constant moment along the gage section so that the stress may be calculated anywhere along the length of the fastener. The test is conducted under displacement control. The loading method shall have a specified load accuracy of ± 0.5%, programmable to increase
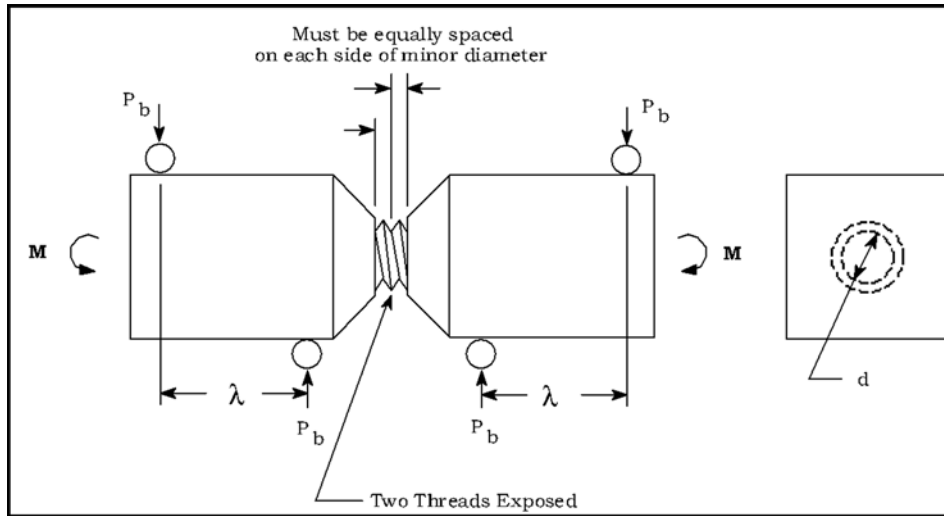
FIG. 1 Four-Point Bend Loading of Fasteners. Maximum Tensile Stress, $\sigma_t = 32P_b\lambda/\Pi d^3$

incrementally in steps of load and time. The loading method shall be within the guidelines of calibration, force range, resolution, and verification of Practices E4.

7.2.2 *Fast Fracture Testing:*

7.2.2.1 The first step in the testing sequence shall be a measurement of the fast fracture load of the specimen bolts in bending. Determine this value by performing a test in accordance with Test Method F1624, Section 8, as shown in Fig. 2A,

using a fast fracture protocol. Test a minimum of five uncoated specimen bolts and a minimum of five coated specimen bolts. The average of these five test results shall determine the fast fracture strength of each condition.

7.2.2.2 The average fast fracture strength in bending of coated bolts, FFS(B)$_{coated}$ must be within ± 5% of the average fast fracture strength for uncoated bolts, FFS(B)$_{uncoated}$. If the coated bolts exhibit a fast fracture strength that is below 95%

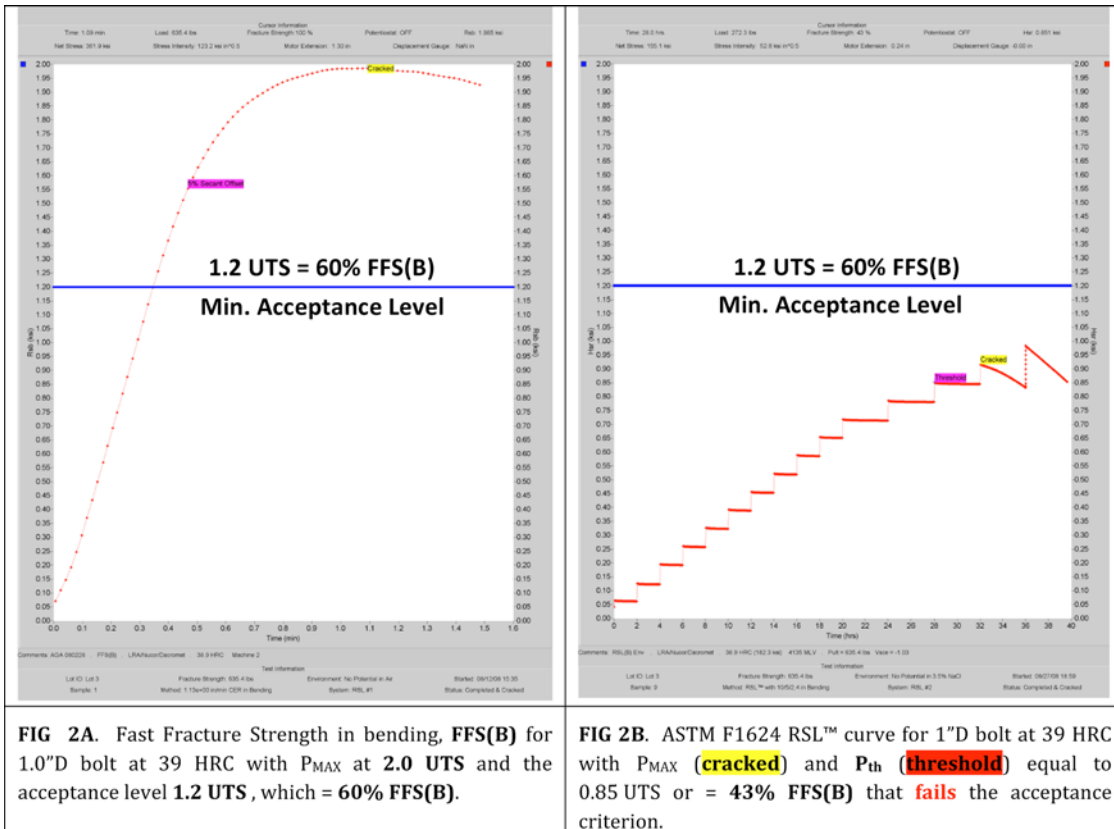FIG 2A. Fast Fracture Strength in bending, **FFS(B)** for 1.0"D bolt at 39 HRC with P$_{MAX}$ at **2.0 UTS** and the acceptance level **1.2 UTS**, which = **60% FFS(B)**.

FIG 2B. ASTM F1624 RSL™ curve for 1"D bolt at 39 HRC with P$_{MAX}$ (**cracked**) and P$_{th}$ (**threshold**) equal to 0.85 UTS or = **43% FFS(B)** that **fails** the acceptance criterion.

FIG. 2

of the fast fracture strength for uncoated bolts, the coating is disqualified from this test.

NOTE 2—Lower than 95% fast fracture strength for the coated samples is an indication that the coating process may have affected the strength of the specimen bolts.

7.2.3 *EHE Sample Testing:*

7.2.3.1 To measure the EHE susceptibility of the fastener/coating system, bolts are tested in the environment/setup described in section 7.1.1 and 7.2.3.3 using the step load methodology described in Test Method F1624, Section 8, to measure P_{th}.

7.2.3.2 A galvanic condition (see Annex A1) is created by inscribing a mark in the coating at the root of a bolt thread to expose the steel substrate. This condition simulates a damaged coating, also referred to as "coating holiday." The scribe mark in the coating shall be located between the exposed threads (see Fig. 1) between the gripping devices and shall have a length of at least one diameter and a width that exposes the thread root. Care must be taken not to cause any damage in the form of a notch to the bolt itself. To this end an abrasive medium, lower in hardness than the bolt material is recommended.

NOTE 3—For example, a wire can be placed at the base of the exposed thread in scribing the coating circumferentially to a length of one diameter.

7.2.3.3 To test at the OCP of the coating, the environmental chamber shall be partially filled with 3.5% NaCl solution produced in accordance with Practice G44 with the level of the solution being maintained below the threshold section of the bolt specimen. The reference electrode shall be placed in close vicinity to the scribe mark. An electrochemical potential equal to the measured OCP from section 7.1 shall be imposed on the specimen during the test to negate the influence of any dissimilar metals in the environmental chamber, such as the loading pins and gripping devices. When the potential reading is stable, the remaining salt solution is added to the chamber to fully immerse the bolt sample.

(1) As an alternative to imposing an electrochemical potential, the test can be performed under freely corroding conditions, provided the coated specimen is isolated from all metal contacts. It should be verified that there is no conductivity between the specimen and the adapters. The freely corroding potential, or OCP (section 7.1), shall be measured and recorded.

7.2.3.4 The loading profile of the first coated sample bolt shall be (10/5/2,4), or ten (10) steps tested with a hold time of 2-hours followed by ten (10) steps at a hold time of 4-hours in 5% increments of FFS(B)$_{coated}$, taken as the initial value of P_{max}. Loading is not released during the transition from the 2-hour to 4-hour hold requirement. The test proceeds until the sample experiences a load drop of more than 5% during any single step in the load rate. An example of the step loading profile is shown in Fig. 2B.

7.2.3.5 Subsequent tests shall be at progressively decreasing loading rates by using the same (10/5/2,4) profile and lowering P_{max} to the value of the previous threshold load thereby lowering the ΔP for each loading step. As P_{max} is decreased, the resolution is increased and the loading rate is decreased. The

test proceeds until the sample experiences a load drop of more than 5% during any single step in the load rate.

7.2.3.6 The threshold load for the coated bolts, P_{th}, is obtained when the threshold load for the subsequent test is within a value of 5% of P_{max} of the threshold load measured on the previous test at the higher loading rate. The minimum value of the threshold load obtained through these measurements shall be used in the calculation described in Section 8. The lowest threshold value established by consecutive tests shall be considered the threshold load for the coated bolts, P_{th}. The minimum number of samples to accomplish the establishment of the threshold load shall be five (5) coated specimen bolts.

8. EHE Acceptance Criterion for Coated ASTM A490 Bolts

8.1 Perform tests on fasteners to the same hydrogen embrittlement acceptance criterion as the Test Method F519, Type 1a specimens that require a threshold stress equal in value to that of 75% of the notch tensile strength (NTS = 1.6 UTS) of the Type 1a specimen, which is equal to or greater than 1.2 UTS.

8.2 To obtain the equivalent acceptance criterion for the coated ASTM A490 bolt, the net tensile stress at the root of the thread should also be equal to or greater than 1.2 UTS. Since d/D for fasteners is always greater than 0.8, that is greater than 0.7 used with Test Method F519 notched tensile specimen, the fastener must be tested in bending to attain the same stress level.

8.3 The acceptance criterion for EHE shall be a threshold load in bending, σ_b, to produce the same stress or greater than the load in tension that produces a stress of 1.2 UTS or $\sigma_b \geq 1.2$ UTS. Since the limit load of a bolt in bending equals $2.3YS \approx 2.0$ UTS; the acceptance criterion for EHE is equivalent to $\geq 60\%$ of the fast fracture load in bending, FFS(B)$_{coated}$.

8.3.1 Therefore for acceptance of a coating system for A490 bolts, the following condition must be met:

$$P_{th} \geq 0.6FFS(B)_{coated} \qquad (1)$$

8.4 *Bolt Size Equivalence*—Based on the Fastener Analysis Diagram (see Note 4), a smaller diameter bolt size can be tested to satisfy the acceptance criterion for a larger bolt size diameter if it attains a threshold load with a higher value than the minimum value of 60% of the fracture load in bending. This ratio is defined as the hydrogen susceptibility ratio, Hsr, in Terminology F2078. The acceptance criteria is based on Hsr being ≥ 1.2 or 60% FFS(B).

8.4.1 From the references in Note 4, the relationship between two diameters is defined by the following equation:

$$Hsr_0*f(d_0 / D_0)\sqrt{D_0} = Hsr_1*f(d_1 / D_1)\sqrt{D_1} \qquad (2)$$

8.4.2 Using the dimensional parameters for d_1 and D_1 for a larger size bolt and inputting a value of $Hsr_1 = 1.2$, the equivalent Hsr_0 value for the smaller sized tested bolt with d_0 and D_0 can be calculated. These are the minimum values for a smaller diameter bolt size to satisfy the acceptance criterion for a larger bolt size. The f(d/D) function is found in Tada, H., Paris, P. and Irwin, G., Stress Analysis of Cracks Handbook, S 27.2.

8.4.3 This equivalence among the different sizes is summarized in Table 2.

Note 4—For more detail see reference: Raymond, L., Handbook of Bolts and Bolted Joints, pp. 745-747. Calculations are based on dimensions given in ASME B1.1-2003, Table 6. Basic Dimensions for Coarse Thread Series (UNC/UNRC).

8.4.4 For example, using a 1/2"D bolt, which needs $\geq 60\%$ FFS(B) to satisfy its basic acceptance criterion requirement for EHE, would need to attain a threshold of $\geq 72\%$ FFS(B) to satisfy the acceptance criterion for a 3/4"D bolt, $\geq 82.2\%$ FFS(B) to satisfy the acceptance criterion for a 1.0"D bolt, or $\geq 98.3\%$ FFS(B) to satisfy the acceptance criterion for a 1.5"D bolt.

TABLE 2 Equivalent Acceptance Criteria @ Hsr $\geq$ 1.2 or 60% FFS(B)

Bolt Diameter	1/2"D	3/4"D	1.0"D	1.5"D
1/2"D	$\geq 60\%$ FFS(B)	$\geq 72\%$ FFS(B)	$\geq 82.2\%$ FFS(B)	$\geq 98.3\%$ FFS(B)
3/4"D	PASS	$\geq 60\%$ FFS(B)	$\geq 68.5\%$ FFS(B)	≥ 81.9 FFS(B)
1.0"D	PASS	PASS	$\geq 60\%$ FFS(B)	$\geq 71.8\%$ FFS(B)
1.5"D	PASS	PASS	PASS	$\geq 60\%$ FFS(B)

8.4.5 Using a 3/4"D bolt, which needs $\geq 60\%$ FFS(B) to satisfy its basic acceptance criterion requirement for EHE, would need to attain a threshold of $\geq 68.5\%$ FFS(B) to satisfy the acceptance criterion for a 1.0"D bolt or $\geq 81.9\%$ FFS(B) to satisfy the acceptance criterion for a 1.5"D bolt.

8.4.6 Using a 1.0"D bolt, which needs $\geq 60\%$ FFS(B) to satisfy its basic acceptance criterion requirement for EHE, would need to attain a threshold of $\geq 71.8\%$ FFS(B) to satisfy the acceptance criterion for a 1.5"D bolt.

8.4.7 Using a 1.5"D bolt, only $\geq 60\%$ FFS(B) needs to be attained to certify the coating.

8.5 Qualification of a large bolt size diameter, inherently qualifies any smaller diameter bolt.

9. Precision and Bias

9.1 The precision and bias of this test method has not yet been determined.

10. Keywords

10.1 environmental hydrogen embrittlement; galvanic potential; high strength bolts; hydrogen embrittlement; open circuit potential; salt solution; threshold stress

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ANNEX

(Mandatory Information)

A1. ENVIRONMENTALLY INDUCED HYDROGEN CAUSED BY GALVANIC CORROSION OF STRUCTURAL STEEL BOLT WITH COATING

A1.1 *Galvanic Corrosion*—Coatings on steel bolts are always anodic to the structural steel bolt; therefore, the coating sacrificially corrodes to protect the bolt. Conversely, the structural steel bolt is always cathodic to the coating. As a result, hydrogen is generated on the cathodic steel surface when the bare steel surface is exposed by wear or damage to the coating. Hydrogen is then absorbed with time under the influence of stress, resulting in a hydrogen assisted failure.

A1.2 *Threshold Load*—The difference in the corrosion potential between the coating and the steel bolt creates a hydrogen charging potential on the bolt. The less difference in potential, the smaller the amount of hydrogen being generated, resulting in a much higher threshold load to induce hydrogen stress cracking. For the coating system to be acceptable, the hydrogen being generated should not reduce the threshold load below the levels specified for each diameter bolt in Table 2.

APPENDIXES

(Nonmandatory Information)

X1. EXAMPLES OF LOAD REQUIREMENT CALCULATIONS

X1.1 The following approach may be used to calculate specific Load Requirements in bending for a given size of specimen bolts.

X1.1.1 The maximum tensile stress at the surface (σ_b) and in tension (σ_t) at the root of the thread can be computed using the formulas:

$$\sigma_b = (32\ M\ /\ \pi\ d^3); \text{ and } \sigma_b = (4\ P_t\ /\ \pi\ d^2)$$

where, d = minimum thread diameter (inch) and M = applied moment (in-lbs), where in four point bending, (λ and P_b as in Fig. 1) M=$P_b\lambda$.

X1.1.2 The threshold load determination depends on the bolt size and the specific equipment being used for testing to produce the bending load. For the threshold stress $\sigma_b \geq 1.2$ UTS, assuming that the strength of an ASTM A490 bolt equals the UTS that corresponds to the hardness, or $P_{t\text{-bolt}} \approx P_{t\text{-UTS}}$; the relationship of the acceptance threshold load ($P_{b\text{-th}}$) to the tensile strength of the bolt ($P_{t\text{-bolt}}$) or the load value measured in 5.1.4 of coated ASTM A490 bolts, is given by:

$$P_{b-th} \geq (d\ /\ 8\ \lambda) \times 1.2 P_{t-bolt} = \text{Acceptable Level Bending}$$

X1.1.3 Pure bending stresses (4-pt) using incremental step loading under displacement control per Test Method F1624 shall also be used to determine the hydrogen embrittlement threshold load, $P_{b\text{-th}}$. An example of results for a 1-inch bolt are shown in Fig. 2.

X1.2 *Examples of Calculations*—The threshold load determination depends on the bolt size and the specific equipment being used for testing. The following calculations are based on

particular bending fixture geometry where λ = 8 inches:[4]

X1.2.1 *For a 1"D Specimen Bolt:*

X1.2.1.1 The range of wedge tensile load for a 1"D specimen bolt (d = 0.8376") is 103,000 lbs. to 107,000 lbs. (See Table 1.)

$$P_{b-th} \geq (d\ /\ 8\ \lambda) \times 1.2 P_{t-bolt} \text{ or}$$
$$P_{b-th} \geq (0.8376\ /\ 64) \times 1.2 \times 107$$
$$P_{b-th} \geq 1,680\ \text{lbs}$$

X1.2.1.2 The minimum acceptable EHE threshold load in bending is 1,680 lbs. for a 1"D specimen bolt for all bolt alloy materials.

X1.2.2 *For a 3/4"D Specimen Bolt:*

X1.2.2.1 The range of wedge tensile load of a 3/4"D specimen bolt (d = 0.6201") is 59,000 lbs. to 63,000 lbs.

$$P_{b-th} \geq (d\ /\ 8\ \lambda) \times 1.2 P_{t-bolt} \text{ or}$$
$$P_{b-th} \geq (0.6201\ /\ 64) \times 1.2 \times 63$$
$$P_{b-th} \geq 730\ \text{lbs}$$

X1.2.2.2 The minimum acceptable EHE threshold load in bending is 730 lbs for a 3/4"D specimen bolt for all bolt alloy materials.

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[4] The sole source of supply of the apparatus known to the committee at this time is Fracture Diagnostics International, LLC (www.fracturediagnostics.net) in Newport Beach, CA. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend.

X2. OTHER RELATED TESTS

X2.1 This standard only addresses test methods relative to hydrogen assisted failures of structural bolts. Other related tests that must be performed to fulfill the requirements of IFI 144 include:

X2.1.1 Rotational Capacity Test

X2.1.2 Salt Spray Test

X2.1.3 Adhesion Test

X2.1.4 Paintability

X2.1.5 Torque/Tension/Angle study

X2.1.6 Coating thickness and thread fit and function study

X2.1.7 Cyclic Tests:

X2.1.7.1 Cyclic Test Under Load

X2.1.7.2 Cyclic Test No Load