



Designation: **F2660–12** **F2660 – 13**

Standard Test Method for Qualifying Coatings for Use on A490 Structural Bolts Relative to Environmental Hydrogen Embrittlement¹

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, 1/2 to 1-1/2 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

¹ This test method is under the jurisdiction of ASTM Committee **F16** on Fasteners and is the direct responsibility of Subcommittee **F16.01** on Test Methods. Current edition approved ~~Sept. 1, 2012~~ Nov. 1, 2013. Published ~~November 2012~~ December 2013. Originally approved in 2012 as F2660–12. DOI: 10.1520/F2660-12-10.1520/F2660-13.

² 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.

³ The last approved version of this historical standard is referenced on www.astm.org.

TABLE 1 Specimen Bolt Sizes and Wedge Tensile Load Values

Nominal Size	Minimum Length	Tensile Stress Area	Wedge Tensile Load	
		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

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:

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 Specimen bolts shall be from homogeneous lots traceable to given mill heats of steel alloy.~~

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 a 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 $\pm 0.5\%$, programmable to increase 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, $FFS(B)_{\text{coated}}$ must be within $\pm 5\%$ of the average fast fracture strength for uncoated bolts, $FFS(B)_{\text{uncoated}}$. If the coated bolts exhibit a fast fracture strength that is below 95% of the fast fracture strength for uncoated bolts, the coating is disqualified from this test.

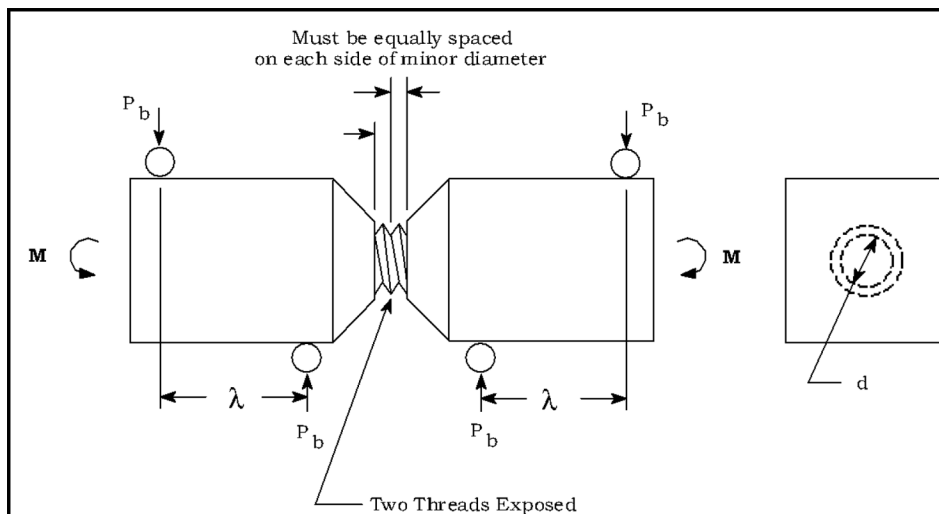


FIG. 1 Four-Point Bend Loading of Fasteners. Maximum Tensile Stress, $\sigma_t = 32P_b\lambda/\Pi d^3$

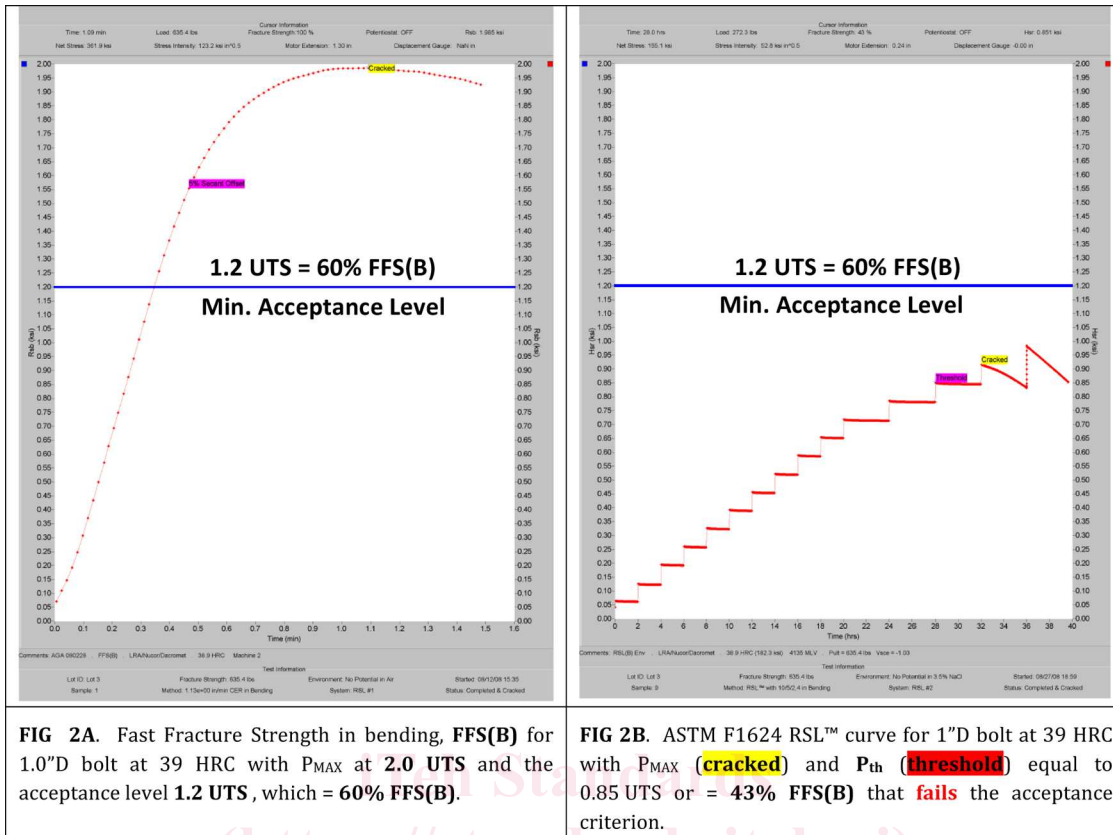


FIG. 2

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 Addendum) 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 count 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.