

# Standard Test Method for Process Control Verification to Prevent Hydrogen Embrittlement in Plated or Coated Fasteners<sup>1</sup>

This standard is issued under the fixed designation F1940; 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 ( $\varepsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This test method covers a procedure to prevent, to the extent possible, internal hydrogen embrittlement (IHE) of fasteners by monitoring the plating or coating process, such as those described in Specifications F1137 and F1941. The process is quantitatively monitored on a periodic basis with a minimum number of specimens as compared to qualifying each lot of fasteners being plated or coated. Trend analysis is used to ensure quality as compared to statistical sampling analysis of each lot of fasteners. This test method consists of a mechanical test for the evaluation and control of the potential for IHE that may arise from various sources of hydrogen in a plating or coating process.

1.2 This test method consists of a mechanical test, conducted on a standard specimen used as a witness, for the evaluation and control of the potential for IHE that may arise from various sources of hydrogen in a plating or coating process.

1.3 This test method is limited to evaluating hydrogen induced embrittlement due only to processing (IHE) and not due to environmental exposure (EHE, see Test Method F1624).

1.4 This test method is not intended to measure the relative susceptibility of steels to either IHE or EHE.

1.5 This test method is limited to evaluating processes used for plating or coating ferrous fasteners.

1.6 This test method uses a notched square bar specimen that conforms to Test Method F519, Type 1e, except that the radius is increased to accommodate the deposition of a larger range of platings and coatings. For the background on Test Method F519 testing, see publications ASTM STP 543<sup>2</sup> and ASTM STP 962.<sup>3</sup> The stress concentration factor is at a  $K_t$  =

 $3.1 \pm 0.2$ . The sensitivity is demonstrated with a constant imposed cathodic potential to control the amount of hydrogen. Both the sensitivity and the baseline for residual hydrogen will be established with tests on bare metal specimens in air.

1.7 The sensitivity of each lot of specimens to IHE shall be demonstrated. A specimen made of AISI E4340 steel heat treated to a hardness range of 50 to 52 HRC is used to produce a "worst case" condition and maximize sensitivity to IHE.

1.8 The test is an accelerated ( $\leq$ 24 h) test method to measure the threshold for hydrogen stress cracking, and is used to quantify the amount of residual hydrogen in the specimen. The specimen undergoes sustained load and slow strain rate testing by using incremental loads and hold times under displacement control to measure a threshold stress in an accelerated manner in accordance with Test Method F1624.

1.9 In this test method, bending is used instead of tension because it produces the maximum local limit load tensile stress in a notched bar of up to 2.3 times the yield strength as measured in accordance with Test Method E8/E8M. A fastener that is unintentionally exposed to bending on installation may attain this maximum local tensile stress.

1.10 The values stated in inch-pound units are to be regarded as standard. The values given in parentheses are mathematical conversions to SI units that are provided for information only and are not considered standard.

1.11 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:<sup>4</sup>

D1193 Specification for Reagent Water

E4 Practices for Force Verification of Testing Machines

E8/E8M Test Methods for Tension Testing of Metallic Materials

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<sup>&</sup>lt;sup>2</sup> Hydrogen Embrittlement Testing, ASTM STP 543, American Society for Testing and Materials, 1974.

<sup>&</sup>lt;sup>3</sup> Hydrogen Embrittlement; Prevention and Control, ASTM STP 962, American Society for Testing and Materials, 1985.

<sup>&</sup>lt;sup>4</sup> 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.

- E18 Test Methods for Rockwell Hardness of Metallic Materials
- E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications
- E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods
- E399 Test Method for Linear-Elastic Plane-Strain Fracture Toughness  $K_{Ic}$  of Metallic Materials
- E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method
- E1823 Terminology Relating to Fatigue and Fracture Testing
- F519 Test Method for Mechanical Hydrogen Embrittlement Evaluation of Plating/Coating Processes and Service Environments
- F1137 Specification for Phosphate/Oil Corrosion Protective Coatings for Fasteners
- F1624 Test Method for Measurement of Hydrogen Embrittlement Threshold in Steel by the Incremental Step Loading Technique
- F1941 Specification for Electrodeposited Coatings on Threaded Fasteners (Unified Inch Screw Threads (UN/ UNR))
- G5 Reference Test Method for Making Potentiodynamic Anodic Polarization Measurements

2.2 SAE Standards:

- AMS 2759 Hot Drawn, Normalized and Tempered Steel Bars. UNS G43406 (AISI E4340)<sup>5</sup>
- AMS 3078 Corrosion Preventive Compound, Solvent Cutback, Cold-Application<sup>5</sup> AMS 6415<sup>5</sup>

## 3. Terminology

3.1 Terms and Symbols Specific to This Standard:

3.1.1 *environmental hydrogen embrittlement (EHE)*—test conducted in a specified environment—embrittlement caused by hydrogen introduced into steel from external sources.

3.1.2 *internal hydrogen embrittlement (IHE)*—test conducted in air—embrittlement caused by residual hydrogen from processing

3.1.3  $ISL_{th}$ —threshold from an incremental step load test on a plated or processed specimen.

3.1.4 NFS(B)—notched fracture strength in air of a bare specimen in bending at loading rates of 50 to 250 ksi/min (350 to 1700 MPa/min).

3.1.5  $NFS(B)_{F1624}$ —notched fracture strength in air of a bare specimen in bending at Test Method F1624 step loading rates.

3.1.6 *process*—a defined event or sequence of events that may include pretreatments, plating, or coating and posttreatments that are being evaluated or qualified.

3.1.7 *threshold*—the maximum load at the onset of cracking that is identified by a 5 % drop in load of NSF(B)<sub>F1624</sub> under displacement control.

## 4. Summary of Test Method

4.1 Specimens of fixed geometry, certified to have been heat treated to a hardness range of 50 to 52 HRC, and which have been certified to exhibit sensitivity to embrittlement from trace amounts of residual hydrogen in steel, are processed with actual parts.

4.2 An unstressed test specimen is processed in accordance with the plating or coating process being qualified. The specimen is then tested under incremental step load to measure the threshold stress. The loading rate must be slow enough to ensure that the threshold stress will be detected if deleterious amounts of hydrogen are present in "worst case" sensitized specimens. Loading rate protocols are defined in 9.2 and Test Method F1624.

4.3 If the threshold in air of the specimen is  $\geq$ 75 % NFS(B)<sub>F1624</sub>, then the process is considered as to not produce sufficient hydrogen to induce time delayed IHE failures in the plated or coated fasteners. See 9.3 for optional limits.

4.4 If the threshold in air of the specimen is <75 % NFS(B)<sub>F1624</sub>, then the process is considered potentially embrittling. Actual fasteners made with steel having a hardness lower than that of the square bar specimen have more tolerance for residual hydrogen because of the process. Therefore, threshold requirements must be adjusted based upon the correlation between the specimen fracture strength NBS(B)<sub>F1624</sub> and actual fastener hardness. An example of this adjustment is presented in Appendix X1.

#### 5. Significance and Use

5.1 This test method establishes a means to verify the prevention, to the extent possible, of IHE in steel fasteners during manufacture by maintaining strict controls during production operations such as surface preparation, pretreatments, and plating or coating. It is intended to be used as a qualification test for new or revised plating or coating processes and as a periodic inspection audit for the control of a plating or coating process.

5.2 Passing this test allows fasteners to be stressed in tension to the minimum specified tensile load in air with almost no possibility of time delayed fracture in air as a result of IHE from processing. If the amount of residual hydrogen is not sufficient to induce cracking or fracture in the specimen under worst case conditions, then it can be concluded that all of the lots of fasteners processed during that period will not have sufficient residual hydrogen from processing to induce hydrogen embrittlement of the fasteners under stress in air if the process remains in control, unchanged and stable.

5.3 If certified specimens with demonstrated sensitivity to IHE, processed with the fasteners, have a threshold  $\geq$ 75 % of the incremental step load notched bend fracture stress, NFS(B)<sub>F1624</sub>, it is assumed that all fasteners processed the same way during the period will also pass any sustained load IHE test.

## 6. Apparatus

6.1 *Testing Machine*—A computerized, four-point bend, digital displacement controlled loading frame that is capable of

<sup>&</sup>lt;sup>5</sup> Available from Society of Automotive Engineers (SAE), 400 Commonwealth Dr., Warrendale, PA 15096-0001, http://www.sae.org.

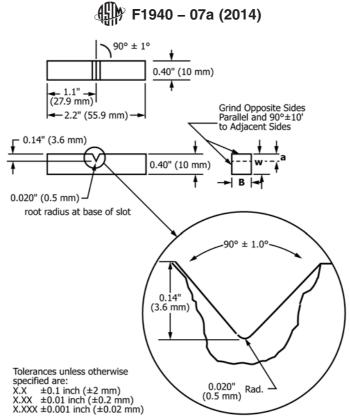


FIG. 1 Dimensional Requirements for a 0.4W-Notched Square Bar Bend Specimen

holding 0.5 % of the NFS(B) and is programmed to increase incrementally in steps of load and time to vary the effective strain rate at the root of the notch between  $10^{-5}$  and  $10^{-8}$  s<sup>-1</sup> is required to conduct these tests. Testing machines shall be within the guidelines of calibration, force range, resolution, and verification of Practice E4.

6.2 *Gripping Devices*—Pin-loading devices consistent with Test Method E399 four-point bend fixtures shall be used to transmit the measured load applied by the testing machine to the test specimen.

6.3 *Potentiostatic Control*—For verification testing of the sensitivity of the specimens to residual hydrogen from processing, an inert container and potentiostat shall be used to impose a cathodic potential on the specimen. The cathodic charging potential of the specimen can be controlled with a reference saturated calomel electrode (SCE) or equivalent reference electrode such as with A/AgCl in accordance with Practice G5.

Note 1—A loading device that meets the displacement control step load test requirements and the potentiostatic control requirements of Test Method F1624 and Test Method F519 is available.

#### 7. Materials and Reagents

7.1 *Materials*—UNS G43406 (AISI E4340) in accordance with AMS 6415.

#### 7.2 *Reagents:*

7.2.1 Corrosion preventive compound, meeting requirements of AMS 3078.

7.2.2 Solution of reagent water in accordance with Specification D1193 Type IV, and 3.5 % reagent grade NaCl.

#### 8. Test Specimen

8.1 The test specimen shall be a 0.4W-notched square bar bend specimen: 0.4W-SqB(B), as shown in Fig. 1.

8.2 The notch shall be in the LS orientation in accordance with Terminology E1823.

8.3 The stress concentration factor for the specimen is  $K_t$  = 3.1  $\pm$  0.2.

Note 2—For the relationship between geometry and  $K_{t_{\star}}$  see Stress Concentration Factors.  $^{6}$ 

#### 8.4 Manufacture:

8.4.1 The test specimen blanks shall be heat treated in accordance with AMS 2759 to meet the hardness requirement of 50 to 52 HRC in accordance with Test Methods E18. Rounding in accordance with Practice E29 permits an absolute hardness range of 49.6 to 52.5 HRC. The hardness shall be determined by the average of three measurements made approximately midway between the notch and the end of the specimen.

8.4.2 The surface finish of all notches shall be finished with a tool capable of attaining a surface roughness of 16 RMS or better. The other surfaces shall have a finish of 32 RMS or better.

8.4.3 All dimensions except for the length shall be produced after quenching and tempering to final hardness. The 0.40-in. (10-mm) dimension shall be produced by low stress grinding. The notch shall be rough machined by wire EDM to within

<sup>&</sup>lt;sup>6</sup> Peterson, R. E., *Stress Concentration Factors*, John Wiley and Sons, New York, 1974.