



## 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 F 1940; 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 ( $\epsilon$ ) 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 F 1137 and F 1941. 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 utilized 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 applies to externally threaded tensile fasteners that can also be loaded in bending during installation.

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 F 1624).

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 ferrous fasteners that are susceptible to time-delayed fracture caused by the diffusion of hydrogen under stress.

1.6 This test method uses a notched square bar specimen that conforms to Test Method F 519, Type 1e, except that the radius is increased to accommodate the deposition of a larger range of platings and coatings. For the background on F 519 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 50–52 HRC is used to produce a “worst case” condition and maximize sensitivity to IHE.

1.8 A notched four point bend 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 F 1624. The Test is an accelerated ( $\leq 24$ h) incrementally increasing step load test method that measures the threshold for hydrogen stress cracking that is used to quantify the amount of residual hydrogen in the specimen.

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 E 8. 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:

- D 1193 Specification for Reagent Water<sup>4</sup>
- E 4 Practices for Force Verifications of Testing Machines<sup>5</sup>
- E 8 Test Methods for Tension Testing of Metallic Materials<sup>5</sup>
- E 18 Test Methods of Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials<sup>5</sup>
- E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications<sup>6</sup>
- E 399 Test Method for Plane-Strain Fracture Toughness of Metallic Materials<sup>5</sup>

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee F-16 on Fasteners and is the direct responsibility of Subcommittee F16.93 on Quality Assurance Provisions for Fasteners.

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<sup>2</sup> *Hydrogen Embrittlement Testing*, ASTM STP 543, ASTM, 1974.

<sup>3</sup> *Hydrogen Embrittlement; Prevention and Control*, ASTM STP 962, ASTM, 1985.

<sup>4</sup> *Annual Book of ASTM Standards*, Vol 11.01.

<sup>5</sup> *Annual Book of ASTM Standards*, Vol 03.01.

<sup>6</sup> *Annual Book of ASTM Standards*, Vol 14.02.

E 1823 Terminology Related to Fatigue and Fracture Testing<sup>5</sup>

F 519 Test Method for Mechanical Hydrogen Embrittlement Evaluation of Plating Processes and Service Environments<sup>7</sup>

F 1137 Specification for Phosphate/Oil and Phosphate/Organic Corrosion Protective Coatings for Fasteners<sup>8</sup>

F 1941 Specification for Electrodeposited Coatings on Threaded Fasteners (Unified Inch Screw Threads (UN/UNR))<sup>8</sup>

F 1624 Test Method for Measurement of Hydrogen Embrittlement in Steel by the Incremental Loading Technique<sup>7</sup>

G 5 Reference Test Method for Making Potentiostatic and Potentiodynamic Anodic Polarization Measurements<sup>9</sup>

## 2.2 SAE Standards:

AMS 2759 Hot Drawn, Normalized and Tempered Steel Bars. UNS G43406 (AISI E 4340)<sup>10</sup>

AMS 3078 Corrosion Preventive Compound, Solvent Cut-back, Cold-Application<sup>10</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 due to hydrogen introduced into steel from external sources.

3.1.2 *internal hydrogen embrittlement (IHE)*—test conducted in air – embrittlement due to 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  $NFS(B)_{F 1624}$ —notched fracture strength in air of a bare specimen in bending at Test Method F 1624 step loading rates.

3.1.6 *process*—a defined event or sequence of events which may include pretreatments, plating or coating, and post treatments 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  $NSF(B)_{F 1624}$  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 F 1624.

4.3 If the threshold in air of the specimen is  $\geq 75\%$   $NFS(B)_{F 1624}$ , 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  $< 75\%$   $NFS(B)_{F 1624}$ , 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 due to the process. Therefore, threshold requirements must be adjusted based upon the correlation between the specimen fracture strength  $NBS(B)_{F 1624}$  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 due to 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,  $NBS(B)_{F 1624}$ , 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 holding 0.5 % of the  $NFS(B)$  and is programmed to incrementally increase in steps of load and time to vary the effective strain rate at the root of the notch between  $10^{-5}s^{-1}$  and  $10^{-8}s^{-1}$  is required to conduct these tests. Testing machines shall be within the guidelines of calibration, force range, resolution, and verification of Practice E 4.

6.2 *Gripping Devices*—Pin loading devices consistent with Test Method E 399 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

<sup>7</sup> Annual Book of ASTM Standards, Vol 15.03.

<sup>8</sup> Annual Book of ASTM Standards, Vol 01.08.

<sup>9</sup> Annual Book of ASTM Standards, Vol 03.02.

<sup>10</sup> Available from Society of Automotive Engineers (SAE), 400 Commonwealth Drive, Warrendale, PA 15096.

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 G 5.

NOTE 1—A loading device, that meets the displacement control step load test requirements and the potentiostatic control requirements of Test Method F 1624 and Test Method F 519 is available.<sup>11</sup>

7. Materials and Reagents

7.1 Materials—UNS G43406 (AISI E 4340) 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 D 1193 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 E 1823.

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

<sup>11</sup> The sole source of supply of the apparatus known to the committee at this time is available from Fracture Diagnostics, Inc., P.O. Box 6401, Denver, CO, 80206. If you are aware of alternative suppliers, please provide this information to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,<sup>1</sup> which you may attend.

NOTE 2—For the relationship between geometry and  $K_t$ , see Peterson, R. E., *Stress Concentration Factors*, John Wiley and Sons, 1974.

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 HRC - 52 HRC in accordance with E 18. Rounding in accordance with Practice E 29 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 mid-way 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. (10mm) dimension shall be produced by low stress grinding. The notch shall be rough machined by wire EDM to within 0.020 in. (0.5 mm) of the final notch depth and low stress ground to the final depth. No chemical or mechanical cleaning shall be allowed after final machining.

8.4.4 Straightening after final heat treatment prior to machining is prohibited.

8.5 Storage—Prior to plating or coating, all specimens shall be protected during storage to prevent corrosion. A suitable means of protection is to coat the specimen with a corrosion preventive compound meeting the requirements of ASM 3078.

8.6 Inspection:

8.6.1 A lot shall consist of only those specimens cut from the same heat of steel in the same orientation, heat treated together in the same furnace, quenched and tempered together,

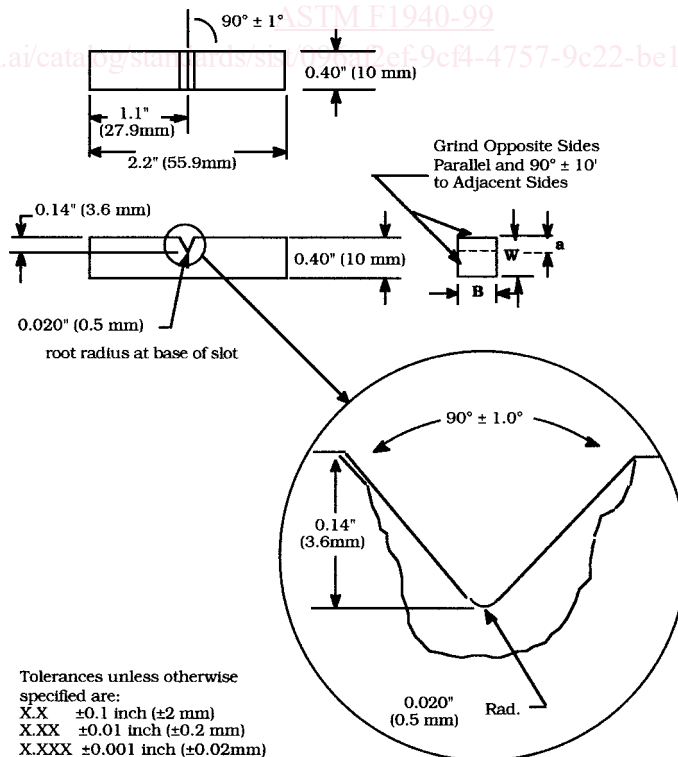


FIG. 1 Dimensional Requirements for a 0.4W-Notched Square Bar Bend Specimen

and subjected to the same manufacturing processes.

8.6.2 One transverse section shall be microstructurally examined to ensure that if any orientation effects exist, the notch will be in the LS-orientation in accordance with Terminology E 1823.

8.6.3 All notched square bar bend specimens shall be considered suitable for test purposes if the sampling and inspection results conform to the requirements of Table 1.

8.6.4 The notched bend fracture strength, NFS(B), of bare specimens is measured in air in four point bending under displacement control at loading rates of 50 to 250 ksi per minute (350 to 1700 MPa/min). The rupture load is used as a measure of strength.

8.7 Sensitivity Test:

8.7.1 The sensitivity to IHE must be demonstrated for each lot of specimens by exposing three trial specimens in air and three trial specimens in an embrittling environmental after manufacture and inspection in accordance with 8.4 through 8.6. The specimens tested shall be representative of the lot.

8.7.2 Sensitivity Specimen Preparation:

8.7.2.1 Ultrasonically clean in acetone for 5 to 10 min to remove the corrosion preventive compound and oils/dirt.

8.7.2.2 Do not acid clean.

8.7.3 Based on the loading profile schedule in Table 2, the requirements for sensitivity of the heat treated lot of specimens shall be demonstrated if bare specimens fracture in less than 5h at an imposed potential of -1.2V vs SCE in a 3.5 % NaCl solution and no delayed fracture occurs in less than 14h, or ≥ 85 % NFS(B) on bare specimens tested in air (see Table 3).

8.7.4 The average of the results of the three bare specimens tested in air shall be used as the baseline notched fracture strength, NFS(B) F 1624.

8.8 Certification:

8.8.1 Each lot of specimens manufactured shall be certified to indicate that it meets the conditions found in this section, including the following information:

8.8.1.1 Manufacturer of specimen lot.

8.8.1.2 Traceability to raw material, heat treatment, manufacturing and inspection.

8.8.1.3 Test results for requirements in Table 1 and Table 3.

TABLE 2 Minimum Step Loading Profile Requirements for Accelerated (< 24h) Incremental Step Load Sensitivity Tests

%NFS(B)	# h	Σh	%NFS(B)	# h	Σh	%NFS(B)	# h	Σh
10	1	1	65	1	8	88	1	15
20	1	2	70	1	9	90	1	16
30	1	3	75	1	10	92	1	17
40	1	4	80	1	11	94	1	18
50	1	5	82	1	12	96	1	19
55	1	6	84	1	13	98	1	20
60	1	7	86	1	14	100	1	21

TABLE 3 Sensitivity Test Requirements of Specimens

Bare in air	Each specimen tested shall have threshold ≥ 85 % of the average notched bend fracture strength, NFS(B) (Table 1)
Bare at potential of -1.2V versus SCE in 3.5% NaCl Solution in Specification D 1193 Type IV Reagent Water	Each specimen tested shall have threshold ≤ 50 % of the average notched bend fracture strength NFS(B) (Table 1)

9. Process Control Testing

9.1 Testing Protocol:

9.1.1 Specimen Preparation—The specimens, as received, shall be processed and qualified with the fasteners. It is important that the specimens be exposed to the same process as the fasteners, for the test to be a valid. Even if the fasteners do not require a degreasing and cleaning process prior to plating or coating, the specimens shall be degreased to remove the corrosion preventive compound and cleaned in acetone and then placed in the process with the fasteners. An application guideline, to be used as a template for the use of this test method, is provided in Appendix X2.

9.1.2 Number—One or more specimens per process per inspection period shall be used.

9.1.3 Test specimens shall be processed once. Stripping and re-use of specimens is prohibited.

9.1.4 The nominal print dimensions from Fig. 1 of the bare metal specimens shall be used in all calculations.

9.1.5 The specimens shall be in the LS-orientation with the notch loaded in tension.

9.2 Load—Incremental step loads and hold times under displacement control shall be used to detect the onset of subcritical crack growth, or threshold that is used to quantify the amount of residual hydrogen in a specimen.

9.2.1 A specific incremental step load and holding time protocol in accordance with Test Method F 1624 is prescribed. Instrumented testing equipment with adjustable constant displacement loading is required as described in Test Method F 1624.

9.2.2 ISL<sub>th-air</sub>—To measure the threshold of a plated 0.4W-SqB(B) specimen, the plated notched bend specimens are tested in air in four point bending under displacement control at Test Method F 1624 loading rates (see Table 4). The threshold is the maximum load at the onset of cracking. The onset of cracking is defined as a 5 % drop in load with respect to NFS(B)<sub>F 1624</sub> under displacement control.

9.2.3 While a specimen is being held at constant displacement, a load drop of 5 % will constitute the onset of subcritical

TABLE 1 Lot Acceptance Criteria for 0.4W-Notched Square Bar Bend Specimens

Item	Sampling of Each Lot	Requirement/Method
Hardness <sup>A</sup>	5 %	50HRC - 52HRC in accordance with E 18. Round the average of three readings per specimen in accordance with E 29.
Dimensions	100 %	Meet tolerances of corresponding drawings. Notch dimension verified with shadow graphic projection at 50 to 100 %.
Notched Fracture Strength in Bending, NFS(B)	10 ea	NFS(B) of each specimen must be within ±5 % of the average.

<sup>A</sup> If the hardness requirements of any of the sampled specimens are not satisfied, only those specimens of the lot that are individually inspected for conformance to these requirements shall be used for testing.