Designation: F 1940 – 01

Standard Test Method for Process Control Verification to Prevent Hydrogen Embrittlement in Plated or Coated Fasteners[1]

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 (ϵ) 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 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 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 Test Method F 519 testing, see publications ASTM STP 543[2] and ASTM STP 962.[3] The stress concentration factor is at a K_t = 3.1 ± 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 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 (≤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[4]

E 4 Practices for Force Verifications of Testing Machines[5]

E 8 Test Methods for Tension Testing of Metallic Materials[5]

E 18 Test Methods of Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials[5]

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications[6]

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods[6]

---

[1] This test method is under the jurisdiction of ASTM Committee F16 on Fasteners and is the direct responsibility of Subcommittee F16.93 on Quality Assurance Provisions for Fasteners.

Current edition approved April 10, 2001. Published July 2001. Originally published as F 1940 – 98. Last previous edition F 1940 – 99.

[2] *Hydrogen Embrittlement Testing, ASTM STP 543,* American Society for Testing and Materials, 1974.

[3] *Hydrogen Embrittlement; Prevention and Control, ASTM STP 962,* American Society for Testing and Materials, 1985.

[4] *Annual Book of ASTM Standards*, Vol 11.01.

[5] *Annual Book of ASTM Standards*, Vol 03.01.

[6] *Annual Book of ASTM Standards*, Vol 14.02.

E 399 Test Method for Plane-Strain Fracture Toughness of Metallic Materials[5]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[6]

E 1823 Terminology Related to Fatigue and Fracture Testing[5]

F 519 Test Method for Mechanical Hydrogen Embrittlement Evaluation of Plating Processes and Service Environments[7]

F 1137 Specification for Phosphate/Oil and Phosphate/Organic Corrosion Protective Coatings for Fasteners[8]

F 1624 Test Method for Measurement of Hydrogen Embrittlement in Steel by the Incremental Loading Technique[7]

F 1941 Specification for Electrodeposited Coatings on Threaded Fasteners (Unified Inch Screw Threads (UN/UNR))[8]

G 5 Reference Test Method for Making Potentiostatic and Potentiodynamic Anodic Polarization Measurements[9]

2.2 *SAE Standards:*

AMS 2759 Hot Drawn, Normalized and Tempered Steel Bars. UNS G43406 (AISI E 4340)[10]

AMS 3078 Corrosion Preventive Compound, Solvent Cutback, Cold-Application[10]

AMS 6415 [10]

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 $NFS(B)_{F\ 1624}$—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 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 $NSF(B)_{F\ 1624}$ 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 ≥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 because of 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 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 ≥75 % of the incremental step load notched bend fracture stress, $NFS(B)_{F\ 1624}$, 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 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^{-1} is required to conduct these tests. Testing machines shall be

[7] *Annual Book of ASTM Standards*, Vol 15.03.

[8] *Annual Book of ASTM Standards*, Vol 01.08.

[9] *Annual Book of ASTM Standards*, Vol 03.02.

[10] Available from Society of Automotive Engineers (SAE), 400 Commonwealth Dr., Warrendale, PA 15096.

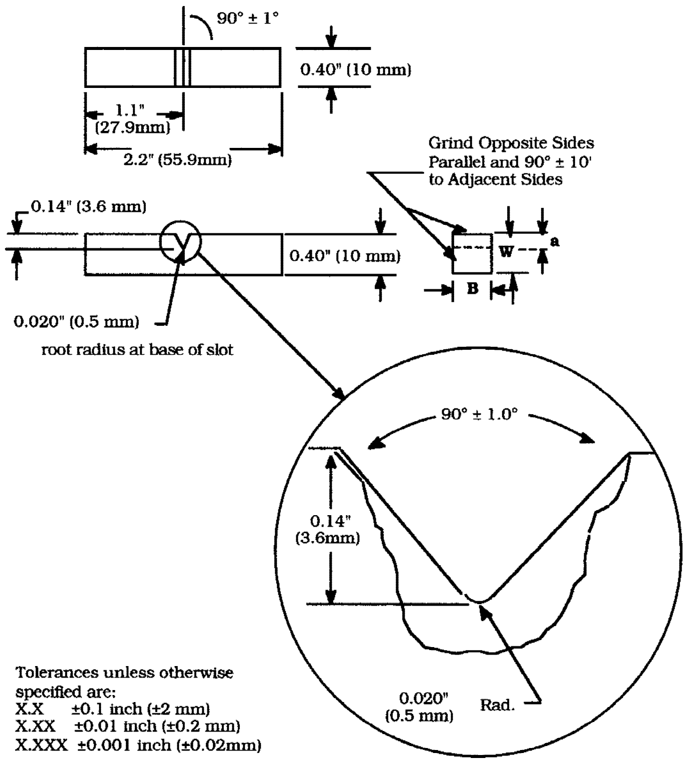


FIG. 1 Dimensional Requirements for a 0.4W-Notched Square Bar Bend Specimen

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 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.

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 $K_t = 3.1 \pm 0.2$.

NOTE 2—For the relationship between geometry and $K_{t,}$ see *Stress Concentration Factors*.[11]

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 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 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 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 before machining is prohibited.

[11] Peterson, R. E., *Stress Concentration Factors*, John Wiley and Sons, New York, 1974.

8.5 *Storage*—Before 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, 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/min (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 5 h at an imposed potential of −1.2 V versus SCE in a 3.5 % NaCl solution and no delayed fracture occurs in less than 14 h 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, $NFS(B)_{F\ 1624}$.

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

Item	Sampling of Each Lot	Requirement/Method
Hardness[A]	5 %	50 to 52 HRC in accordance with Test Method E 18. Round the average of three readings per specimen in accordance with Practice 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.

[A]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.

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 –1.2 V 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)

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.

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 before 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 reuse 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 $ISL_{th\text{-}air}$—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 $NFS(B)_{F\ 1624}$ 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 crack growth at that displacement and corresponding load. The load measured at the constant displacement recorded before the 5 % load drop is recorded as the threshold for that specimen. If the specimen fractures while attempting to reach a new displacement and corresponding higher load, the previous load is recorded. The test results are recorded as a threshold, which is a percentage of the notched fracture stress for that specimen configuration, and not as pass/fail as with the sustained load test, time-to-fracture criterion.

9.2.4 Time can be used as a criterion for achieving a threshold. As an example, specimens achieve a ≥76 % $NFS(B)_{F\ 1624}$ when no delayed fracture occurs in less than 12 h, in accordance with Table 4.

9.3 *Optional Limits*:

9.3.1 Since embrittlement related to hydrogen content varies with hardness, actual fasteners made of low-strength steel might have more tolerance for residual hydrogen because of the process and might not need the rigorous requirement set forth in this standard for threshold. Therefore, adjustments in threshold requirements can be made once a correlation is established. As an example, a threshold of less than 75 % of the fracture strength that is not necessarily hydrogen free can be considered adequate for many applications of lower strength steels.

9.3.2 To obtain a correlation between actual production fasteners and threshold levels in this standard, the threshold level or hydrogen tolerance level for the production fasteners can be measured using Test Method F 1624. An example of an adjustment to the threshold is shown in Appendix X1.

10. Interpretation of Results

10.1 When test specimens exceed 75 % of $NFS(B)_{F\ 1624}$ or ≥12 h, the plating bath is considered to be nonembrittling.

10.2 If any test results are marginal or suspect, the actual product lot can be tested in accordance with Test Method F 1624 to determine if the threshold of the actual fastener is ≥90 % of the bend ultimate strength of the fastener.

10.3 Rupture load and net tensile stress for the four-point bend specimens are correlated using the equation $\sigma_{net} = My/I$ for the specimen geometry provided in Fig. 1. The corresponding average rupture load is reported in units of X.XX lbs (Y.Y N) and corresponding net stress in units of X.XX ksi (Y.Y MPa).

10.4 *Statistical Process Control*:

10.4.1 The sampling or statistical process control plan used to evaluate the process for the prevention of hydrogen embrittlement (IHE) shall be agreed to between the manufacturer and the purchaser.

10.4.2 The >75 % $NFS(B)_{F\ 1624}$ threshold used to qualify the process is specified as a minimum value for individual data. If statistical limits are to be applied, they are to be established through agreement between the manufacturer and purchaser.

11. Report

11.1 A test report shall be produced upon completion of testing that bears the following minimum information:

11.1.1 A specimen lot acceptance and sensitivity certification report,

11.1.2 Identification of the process line,

11.1.3 A description of the plating or coating process,

11.1.4 The threshold load, or percent of notched fracture strength or notch bend strength of bare specimens, as appropriate,

11.1.5 The time under load, and

11.1.6 Disposition of the results.

12. Precision and Bias

12.1 *Precision*—An interlaboratory test program (ASTM Research Report F16–1000) was designed to estimate the precision of the ISL test as it applies to this test method. The experimental results were entirely generated using notched square bar standard test specimens. Two testing modes were used; testing in air (that is, no imposed potential) and testing under potential (for simulated hydrogen charging conditions).

12.1.1 *Within Laboratory Study*—In this part of the test program, a large number of specimens (minimum 30) were tested in air within 1 laboratory to estimate repeatability within a single laboratory. The time span for testing 30 specimens was approximately 8 weeks. This was due to the length of the test cycle, which can be as long as 24 h. Therefore, to detect any systematic shift in the values generated by the test apparatus, this test was repeated twice in the space of 1 year. The summary results of the study are presented in Table 5.

The term repeatability limit is used as specified in Practice E 177.

TABLE 4 Minimum Requirements for a Step-Loading Profile for Accelerated (≤24 h) Incremental Step Load Threshold Determination

$\%NFS_{F\ 1624}$	# h	Σh	$\%NFS_{F\ 1624}$	# h	Σh	$\%NFS_{F\ 1624}$	# h	Σh
10	1	1	65	1	8	85	1	15
20	1	2	70	1	9	90	1	16
30	1	3	72	1	10	95	1	17
40	1	4	74	1	11	100	1	18
50	1	5	76	1	12	105	1	19
55	1	6	78	1	13	110	1	20
60	1	7	80	1	14		. . .	

TABLE 5 Within Laboratory Notch Fracture Strength, $NFS_{(Baseline)}$ Summary of Results

	SQBs Tested *N*	Avg. *x*	Std. Dev s	Min.	Max.	95 % Repeatability Limit *r*
Study 1	37	219.5	6.52	204.4	232.1	18.26
Study 2	30	218.5	4.22	210.8	225.9	11.82
Average of study averages, $\bar{x}$= 219.0						
Average of study standard deviations, $\bar{s}$= 5.37						

TABLE 6 Precision Statistics

Imposed Potential	Fracture Strength Average $\bar{x}$	Repeatability Standard Deviation S_r	Reproducibility Standard Deviation S_R	95 % Repeatability Limit r	95 % Reproducibility Limit R
-1.2 V	71.22	9.88	9.88	27.66	27.66
-1.0 V	85.12	9.70	9.70	27.15	27.15
-0.9 V	102.97	10.02	10.02	28.06	28.06
-0.8 V	179.33	9.77	12.44	27.35	34.83
AIR	221.82	5.81	7.16	16.27	20.06

12.1.2 *Interlaboratory Study*—Four testing facilities[12], each using a single ISL loading frame, participated in the study. With the exception of the number of participating laboratories, four instead of a minimum of six, the study was modeled on Practice E 691.[13] The study consisted of testing square bar specimens at five different conditions, four at different applied potentials, –0.8, -0.9, -1.0, and -1.2 V and one in air. Each laboratory performed five replicate tests for each condition. The precision statistics are presented in Table 6.

The terms repeatability limit and reproducibility limit are used as specified in Practice E 177.

12.2 *Bias*:

12.2.1 To eliminate any bias of results as a result of variation in the conditions of specimen manufacture, all the specimens used for this study were E4340 notched square bar specimens, obtained from a single controlled production lot, manufactured with minimal variation. Therefore, note that variance within the specimen population, however minimal, was implicitly considered in the precision estimates.

12.2.2 All of the instruments were subject to normal calibration procedures by the equipment manufacturer. Any results obtained through obvious error in procedure or equipment malfunction were disqualified from the study.

12.2.3 This method has no bias because comparative measurement of hydrogen embrittlement is defined only in terms of this test method.

12.2.4 Random lot-to-lot bias in the properties of square bar specimens related to raw material or specimen manufacture may exist. This test method produces a quantitative fractional measure based on the baseline fracture strength of square bar specimens not exposed to hydrogen. Since there is no universally accepted reference or laboratory suitable for determining the bias for square bar specimens, no justifiable statement of bias can be made in relation to the baseline fracture strength of specimens. However, lot-to-lot bias for square bar specimens does not affect the test fractional results provided a baseline fracture strength is established for every lot of square bar specimens.

13. Keywords

13.1 coating; delayed failure; displacement control; EHE; fasteners; hydrogen embrittlement; IHE; incremental step load; loading rate; plating; steel; threshold

[12] Galvano Division of Ifastgroupe, Camcar-Textron, Elco-Textron, RSL Technology Center.

[13] This study was conducted in 1997–1998. At the time, there was a very limited number of facilities equipped to perform such testing. Further testing involving more facilities shall be conducted to make the study fully compliant with Practice E 691.

APPENDIXES

(Nonmandatory Information)

X1. ALTERNATE SQUARE BAR THRESHOLD DETERMINATION FOR SPECIFIC PRODUCT LOTS

X1.1 Scope

X1.1.1 Since embrittlement related to hydrogen content can vary with hardness, actual fasteners made of low-strength steel might have more tolerance for residual hydrogen because of the process and might not need the rigorous requirement set forth in this standard for threshold. Therefore, adjustments in threshold requirements can be made for a specific lot of fasteners once a correlation is established.

NOTE X1.1—Note that embrittlement related to hydrogen can also vary with other metallurgical and chemical characteristics of steel and that "low-strength steel" is not always a predictor of more tolerance for residual hydrogen.

X1.1.2 To obtain a correlation between actual production fasteners from singular lots and specimen threshold levels in this standard, the threshold level or hydrogen tolerance level for the production hardware can be measured using four-point bending in accordance with Test Method F 1624 as a function of an applied electrical potential verses a saturated calomel electrode, (SCE) in a 3.5 % sodium chloride solution. An example of four-point bend fixturing used for Test Method F 1624 testing is shown in Fig. X1.1 in which the tensile stress in bending, σ_b, at the root of the thread can be computed using the following formula:

$$\sigma_b = (32\,M/\pi D_t^3) \quad \text{(X1.1)}$$

where:

D_t = minimum thread diameter (inch) and
M = applied moment (inch-pounds) which = $P_b * \lambda$.

X1.1.3 Once the threshold for the product has been determined as a function of the applied potential, the percent fracture strength for the measured thresholds at each potential are plotted as shown in Fig. X1.2. A statistical response in the data must be expected, and therefore judgment in defining a region bounded by upper and lower limits is required. Using

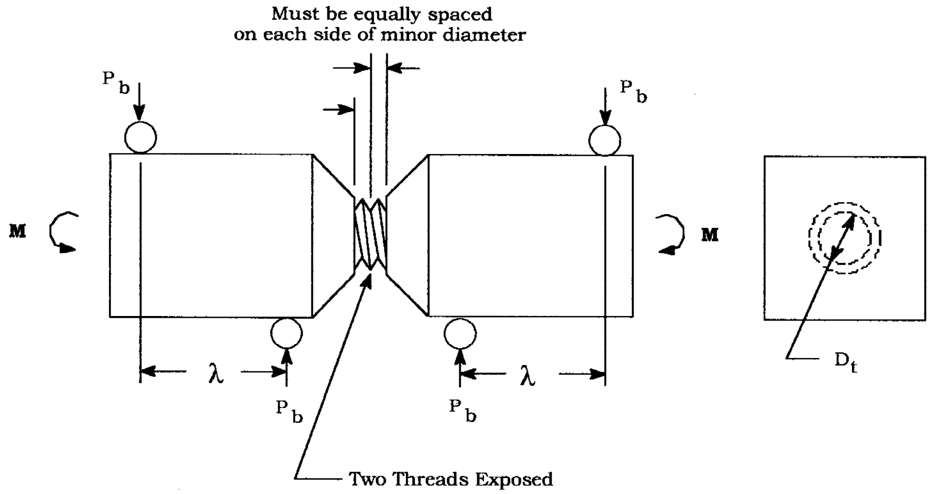
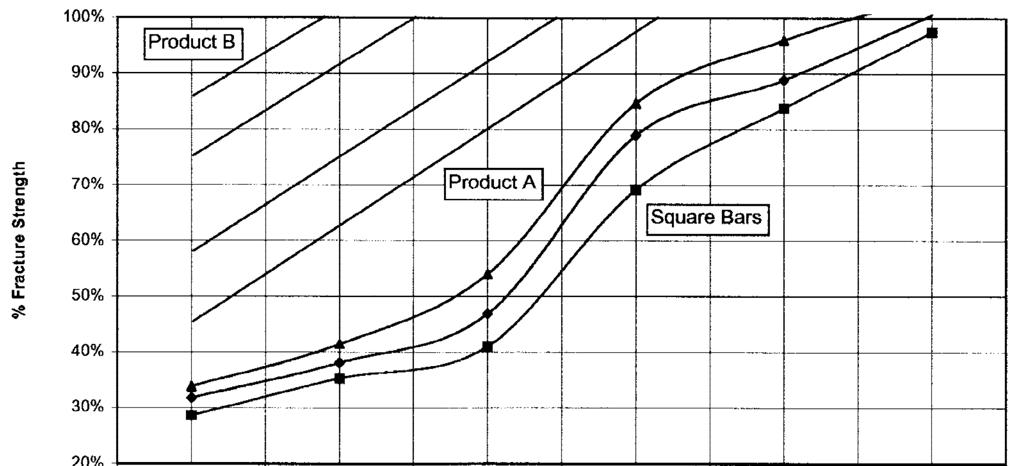


FIG. X1.1 Example of Test Method F 1624 Four-Point Bend Test Fixtures



	-1.2 V	-1.0 V	-0.9 V	-0.8 V	-0.7 V	AIR
Avg	31.8%	38.1%	46.8%	78.9%	88.9%	100.6%
Min	28.6%	35.3%	41.0%	69.2%	83.8%	97.5%
Max	33.9%	41.4%	54.0%	84.7%	96.0%	104.1%

Electrical Potential

FIG. X1.2 Threshold Determination Product Versus Sq(B)s

actual square bar data generated at the same potentials and this data, the alternate threshold can then be determined.

X1.2 Alternate Threshold Determination

X1.2.1 For a specific lot of product, find the potential at which the lower limit of the threshold region intersects the 100 % fracture strength line.

X1.2.2 With this potential find, the corresponding percent fracture strength for the square bar at its upper limit. This fracture strength is the alternate threshold that can be used for this product.

X1.2.3 *Example 1*—For Product Lot A on Fig. X1.2, the potential at which the lower limit of the threshold region intersects the 100 % line is −0.80 V versus SCE. The upper limit for square bar percent fracture strength at this potential is 75 %. This is the threshold specified in this document.

X1.2.4 *Example 2*—For Product Lot B on Fig. X1.2, the potential at which the lower limit of the threshold region intersects the 100 % line is −0.95 V versus SCE. The upper limit for square bar percent fracture strength at this potential is 40 %. For this product, an alternate threshold could be used since this steel is considerably less sensitive to residual hydrogen.

X2. APPLICATION GUIDELINE

X2.1 Scope

X2.1.1 This application guideline is targeted to the general fastener plating and coating industry. It is a tested and viable model, designed to be used as a template for the application of Test Method F 1940. As such, it does not specify any mandatory requirements; however, it should serve as a checklist for anyone who wishes to use the Incremental Step Load (ISL) test method for process verification to prevent hydrogen embrittlement in plated or coated fasteners. Specific testing procedures, sampling schedules, and acceptance criteria should be established based upon the individual characteristics of each process and upon agreement between the purchaser and the supplier.

X2.2 Testing Criteria

X2.2.1 Each individual plating process shall be tested and qualified independently.

X2.2.2 The supplier shall require that the purchaser provide certification of chemical and mechanical properties of the fasteners to be coated. This will allow the supplier to gage the relative susceptibility of the fasteners to internal hydrogen embrittlement (IHE).

X2.2.2.1 Increasing hardness, tensile strength, and carbon content in martensitic steel are the most obvious characteristics that will increase the susceptibility of fasteners to IHE. Consequently, the most susceptible products should be processed on the best-qualified line(s).

X2.2.3 Testing shall be conducted at the highest specified pickling acid concentration and the longest pickling duration for a given line. In the case of an electroplating line, testing shall also be conducted at the highest operational current density in the electroplating cell.

X2.2.4 Statistical process control methodology and criteria can be applied to the test procedure upon agreement between the supplier and the purchaser. Process control or statistical process control must be well documented to establish the stability of the process and the ability to control process parameters and characteristics. The results of this control shall be used in conjunction with the ISL test results as justification for a decrease in testing frequency.

X2.2.5 A minimum of three square bar specimens shall be placed in a single processing unit. A processing unit can be a barrel, a rack, a drum, or a basket depending on the nature of the process being tested. For the sake of simplicity, the processing unit will be referred to as a unit.

X2.2.5.1 The average of the three results within a unit shall represent a single data point for statistical evaluation. Variation within each unit must be within ±10 % of the measured average threshold for the group of three specimens. This is a benchmark for the validity of the results within a single unit.

X2.2.6 Variation of results from one unit to the next must be within ±10 % of the measured average threshold for the population of units to meet process control objectives.

X2.2.7 If the measured average threshold for any unit is less than 75 % of the certified average notched fracture strength $NFS(B)_{F\ 1624}$, it is recommended that an agreement be reached between the supplier and the purchaser as to the minimum acceptable ISL threshold for processed specimens. The basis for such an agreement should be established through threshold testing of the product. (See 9.3 and Appendix X1.)

X2.3 Sampling Schedule

X2.3.1 *Stage 1*—Test three specimens in one unit daily for a minimum of one operational week. If variation of the test results remains within the acceptable range, go to Stage 2. If not, testing must continue to determine and eliminate the cause of variation.

X2.3.2 *Stage 2*—Test three specimens in one unit weekly for a minimum of four weeks. If variation of the test results remains within the acceptable range, go to Stage 3. If not, testing must continue to determine and eliminate the cause of variation. It might be necessary to return to Stage 1.

X2.3.3 *Stage 3*:

X2.3.3.1 Test three specimens in one unit monthly for as long as process stability has been established by achieving and maintaining acceptable variation of results. In case of unacceptable variation, testing must continue to determine and eliminate the cause of variation. It might be necessary to return to Stage 1 or Stage 2.

X2.3.3.2 It is possible to reduce the testing frequency further through the establishment of operating limits for the process control variables. For this to be accomplished, multilevel experimentation must be conducted to determine the impact of each variable on process performance.

ADDITIONAL REFERENCES

(**1**) Raymond, L., "The Susceptibility of Fasteners to Hydrogen Embrittlement and Stress Corrosion Cracking," *Handbook of Bolts and Bolted Joints*, Marcel Decker, Inc., New York, 1998, Chapter 39, p.723.

(**2**) Interrante, C.G., Raymond, L., "Hydrogen Damage," *Corrosion Tests and Standards*, ASTM Manual Series: MNL 20, 1995, Chapter 27, p.272.

(**3**) Tyler, P.S., Levy, M., Raymond, L., "Investigation of the Conditions for Crack Propagation and Arrest Under Cathodic Polarization by Rising Step Load Bend Testing," *Corrosion*, NACE, Feb. 1991, V.47, No. 2, pp. 82-86.

(**4**) Raymond, L. and Crumly, W./R., "Accelerated, Low-Cost Test Method for Measuring the Susceptibility of HY-Steels to Hydrogen Embrittlement," *Current Solutions to Hydrogen Embrittlement in Steels*, Proceedings of the First International Conference, ASM, Metals Park, OH, 1982, p. 477.

(**5**) National Materials Advisory Board, "Rapid Inexpensive Tests for Determining Fracture Toughness," NMAB 328, National Academy of Sciences, Washington, DC, 1976.