Designation: F 519 – 97$^{\epsilon 2}$

Standard Test Method for Mechanical Hydrogen Embrittlement Evaluation of Plating Processes and Service Environments[1]

This standard is issued under the fixed designation F 519; 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.

ϵ^1 NOTE—Fig. A3.2 was editorially changed to Fig. A4.2 in December 1997.
ϵ^2 NOTE—Note 3 was deleted in Fig. A2.1 and Footnote 11 has been added to Note A3.1 in July 2000.

1. Scope

1.1 This test method covers mechanical tests for the evaluation and control of the potential for hydrogen embrittlement that may arise from various sources of hydrogen in plating processes, or from subsequent service environments that include fluids, cleaning treatments, maintenance chemicals or gaseous environments that may contact the surface of steels.

1.2 This test method is intended for process control of hydrogen produced by different plating processes and exposure to different chemicals encountered in a service environment and not to measure the relative susceptibility of different steels.

1.3 This test method assumes that air melted AISI 4340 steel per MIL-S-5000 at 51 to 53 HRC is the worst case; that is, all other heat-treated, high-hardness steels are less susceptible to hydrogen embrittlement. The sensitivity to hydrogen embrittlement shall be demonstrated for each heat of AISI 4340 steel used in the manufacture of test specimens.

1.4 Test procedures and acceptance requirements are specified for seven specimens of different sizes, geometries, and loading configurations. For plating processes, notched specimens must exceed 200 h at a sustained load of 75 % of the fracture stress or exceed a threshold of 75 % of the fracture stress for a quantitative, accelerated (≤24 h) incrementally increasing or rising step load (RSL) test. For service environments, equivalent loading conditions and pass/fail requirements for each specimen are specified.

1.5 This test method is divided into two main parts. The first part gives general information concerning requirements for hydrogen embrittlement testing. The second part is composed of annexes that give specific requirements for the seven specimens covered by this test method.

1.5.1 Annex A1 details notched tensile specimens under constant load; Annex A2 details notched round bars tensile, bend, and C-ring specimens using a self-loading fixture; Annex A3 details a notched four point bend specimen that combines sustained load and slow strain rate testing for hydrogen embrittlement by using incremental loads and hold times under displacement control to measure a threshold stress in an accelerated manner. The test is described as an accelerated (≤24 h) incrementally increasing or rising 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. Annex A4 details a smooth O-ring under displacement control; Annex A5 details testing in a service environment.

1.6 Specific requirements for the two types of specimen and the seven specific loading and geometrical configurations are as listed:

1.6.1 Type 1—Notched Specimens
1.6.2 Type 1a: Round Bar Tension—Constant Load
1.6.3 Type 1a.1—Standard Size Annex A1
1.6.4 Type 1a.2—Oversized Annex A1
1.6.5 Type 1b: Round Bar Tension—Self Loading Fixture-Annex A2
1.6.6 Type 1c: Round Bar Bend—Self Loading Fixture-Annex A2
1.6.7 Type 1d: C-ring Bend—Self Loading Fixture-Annex A2
1.6.8 Type 1e: Square Bar Bend—Displacement Annex A3
1.6.9 Type 2—Smooth Specimens
1.6.10 Type 2a: O-ring—Displacement Control Annex A4

1.7 The values stated are in American Standard units.

1.8 *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:*

B 851 Specification for Automated Controlled Shot Peening of Metallic Articles Prior to Nickel, Autocatalytic Nickel, or Chromium Plating, or Final Finish[2]

[1] This specification is under the jurisdiction of ASTM Committee F07 on Aerospace and Aircraft and is the direct responsibility of Subcommittee F07.04 on Hydrogen Embrittlement.

Current edition approved May 10, 1997. Published August 1997. Originally published as F 519 – 77. Last previous edition F 519 – 93.

[2] *Annual Book of ASTM Standards*, Vol 02.05.

D 1193 Specification for Reagent Water[3]
E 4 Practices for Force Verification of Testing Machines[4]
E 8 Test Methods for Tension Testing of Metallic Materials[4]
E 18 Test Methods for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials[4]
E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications[5]
E 616 Terminology Relating to Fracture Testing[4]
E 709 Guide for Magnetic Particle Examination[6]
F 1624 Test Method for Measurement of Hydrogen Embrittlement Threshold in Steel by the Incremental Step Loading Technique[7]
G 5 Reference Test Method for Making Potentiodynamic Anodic Polarization Measurements[8]

2.2 *AMS Standards:*
AMS 2430 Shot Peening[9]

2.3 *Military and Federal Standards:*
MIL-S-5000 Steel, Chrome-Nickel-Molybdenum (E 4340) Bars and Reforging Stock[10]
MIL-H-6875 Heat Treatment of Steels, Process for[10]
MIL-C-16173 Corrosion Preventive Compound, Solvent Cutback, Cold-Application[10]
Federal Specification QQ-P-416 Plating, Cadmium (Electrodeposited)[10]

3. Summary of Test Methods

3.1 *Plating Processes*—Unstressed test specimens are cleaned, plated, and baked in accordance with the plating specification to which the process is to be qualified. Specimens are then maintained under a sustained load in air to measure the time to rupture or under a rising step load to measure the threshold stress.

3.2 *Service Environments*—Plated test specimens must first be certified by the requirements stated herein for the plating process. They are then tested under load in the service environment. The sequence of exposure to the environment and loading shall be as defined in Annex A5.

4. Significance and Use

4.1 *Plating Processes*—This test method establishes a means to prevent hydrogen embrittlement of steel parts during manufacture by maintaining strict controls during production operations such as surface preparation, pretreatments, and plating. It is intended to be used as a qualification test for new plating processes and as a periodic inspection audit for the control of a plating process.

4.2 *Service Environment*—This test method establishes a means to determine the hydrogen embrittlement potential of chemicals that may contact plated steel parts during manufacturing, overhaul, and service life. The details of testing in a service environment are found in Annex A5.

5. Apparatus

5.1 *Testing Machine*—Testing machines shall be within the guidelines of calibration, force range, resolution, and verification of Practices E 4.

5.2 *Gripping Devices*—Various types of gripping devices shall be used in either tension or bending to transmit the measured load applied by the testing machine or self-loading frame to the test specimen.

5.3 *Environmental Testing*—For testing in service environments, an inert container and fixture arrangement that is suitably electrically isolated from the specimen or compensated to prevent galvanic coupling shall be use for testing in aqueous environments. The corrosion potential of the specimen can be controlled with a reference Saturated Calomel Electrode (SCE) or equivalent reference electrode such as with Ag/AgCl in accordance with Test Method G 5.

6. Materials and Reagents

6.1 *Materials*:

6.1.1 AISI E 4340 per MIL-S-5000.

6.1.2 Aluminum oxide (150 grit or finer), and 180-grit silicon carbide paper.

6.1.3 Clean shot, size in accordance with Specification B 851.

6.2 *Reagents*:

6.2.1 *Corrosion*—Preventive compound, meeting requirements of MIL-C-16173, Grade 2.

6.2.2 *Cadmium*—Cyanide electroplating bath (Federal Specification QQ-P-416). (Table 1).

6.2.3 Maintenance chemicals, cleaners, paint strippers, and aqueous environments.

6.2.4 Chromic acid.

6.2.5 *Water*—Specification D 1193 Type IV.

7. Test Specimens

7.1 *Configuration*:

TABLE 1 Electroplating Bath Compositions and Operating Conditions for Sensitivity Test of Heats of AISI 4340 Steel

Item	Treatment A	Treatment B
Bath composition:		
Cadmium (as CdO)	4.5 oz/gal (33.7 g/L)	4.5 oz/gal (33.7 g/L)
Sodium cyanide (NaCN)	14 oz/gal (104 g/L)	14 oz/gal (104 g/L)
Ratio NaCN to CdO	3	3
pH	12	12
Temperature	70–90°F (21–32°C)	70–90°F (21–32°C)
Sodium hydroxide (NaOH)	2.5 oz/gal (18.7 g/L)	2.5 oz/gal (18.7 g/L)
Brightener such as Rohco 20 × L or equivalent	2.0 oz/gal (15.0 g/L)	...
Electroplating current	10 A/ft^2(108 A/m^2)	60 A/ft^2(645 A/m^2)
Electroplating time	30 min	6 min
Baking:		
Baking temperature	375 ± 25°F (190 ± 14°C)	375 ± 25°F (190 ± 14°C)
Baking time: Type 1 Specimens	Do Not Bake	23 h
Baking time: Type 2a Specimen	8 h	23 h

[3] *Annual Book of ASTM Standards*, Vol 11.01.
[4] *Annual Book of ASTM Standards*, Vol 03.01.
[5] *Annual Book of ASTM Standards*, Vol 14.02.
[6] *Annual Book of ASTM Standards*, Vol 03.03.
[7] *Annual Book of ASTM Standards*, Vol 15.03.
[8] *Annual Book of ASTM Standards*, Vol 03.02.
[9] Available from AMS, 400 Commonwealth Dr., Warrendale, PA 15096.
[10] Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.

7.1.1 Seven specific dimensional drawings with tolerances are given for the two types of specimens in the following annexes:

7.1.1.1 Type 1—Notched Specimens

7.1.1.2 Type 1a: Round Bar Tension—Constant Load

7.1.1.3 Type 1a.1—Standard Size—per Fig. A1.1 Annex A1

7.1.1.4 Type 1a.2—Oversized—per Fig. A1.2 Annex A1

7.1.1.5 Type 1b: Round Bar Tension—per Fig. A2.1, Fig. A2.2, Fig. A2.3 Annex A2

7.1.1.6 Type 1c: Round Bar Bend—per Fig. A2.4, Fig. A2.5 Annex A2

7.1.1.7 Type 1d: C-ring Bend—per Fig. A2.6, Fig. A2.7 Annex A2

7.1.1.8 Type 1e: Square Bar Bend—per Fig. A3.1 Annex A3

7.1.1.9 Type 2—Smooth Specimens

7.1.1.10 Type 2a: O-ring—per Fig. A4.1, Fig. A4.2 Annex A4

NOTE 1—The notched round bar tension, bend, and square bar bend shall be loaded in the longitudinal grain direction, but the C-ring and O-ring can only be loaded normal to the longitudinal grain direction.

7.2 *Manufacture*:

7.2.1 For all notched round specimens, a stress concentration factor (K_t) of 3.1 ± 0.2 shall be maintained.

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

7.2.2 If the 60° notch angle does not permit plating to the root of the notch, then an angle of 90° ± 1° shall be used. For the Type 1.1 specimen, the K_t of 3.1 ± 0.2 shall be maintained by reducing the root radius of the notch to 0.008 ± 0.001 in. for the 90° notch.

7.2.3 The test specimens shall be produced from normalized and tempered, hot or cold drawn bar stock, AISI 4340 steel per MIL-S-5000 and heat treated per MIL-H-6875 so as to meet the hardness requirement of 51 to 53 HRC per Test Method E 18. Rounding per Practice E 29 permits an absolute hardness range of 50.6 to 53.4 HRC of the average of three measurements.

7.2.4 Tolerances unless otherwise specified are:

X.X	±0.1 in.
X.XX	±0.01 in.
X.XXX	±0.001 in.

7.2.5 The surface finish of all notches shall be 32 RMS or better. The other surfaces shall have a finish of 63 RMS or better.

7.2.6 The entire notch shall be ground to size after quenching and tempering to final hardness before plating. Single-point turning of the notch configuration is not permitted. Burnishing of the notch is not permitted. The notch shall not be shot peened or receive any blasting/mechanical cleaning operation after the notch is ground to size.

7.2.7 After grinding, all specimens shall receive a stress relief bake at 375 ± 25°F (190 ± 14°C) for 4 h. A suitable protection from heat treating discoloration should be used.

7.2.8 Acid or cathodic electrolytic cleaning is prohibited.

7.2.9 Straightening after final heat treatment is prohibited.

7.3 *Storage*:

7.3.1 Before plating, 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 MIL-C-16173, Grade 2.

7.4 *Inspection*:

7.4.1 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.

7.4.2 All notched specimens shall be suitable for test purposes if the sampling and inspection results conform to the requirements of Table 2.

7.5 *Sensitivity Test*:

7.5.1 The sensitivity to hydrogen embrittlement must be demonstrated for each heat of AISI 4340 steel by exposing six trial specimens to two different embrittling environments after manufacture and inspection in accordance with Section 7.

7.5.2 Three specimens shall be electroplated under the highly embrittling conditions produced in a cadmium cyanide bath by Treatment A (Table 1) and the remaining three specimens shall be electroplated under the less embrittling conditions of Treatment B (Table 1). An equivalent plating or imposed potential may be used if its sensitivity is demonstrated to be equivalent to that found in Table 1.

7.5.3 Each heat of steel shall be of suitable sensitivity only if all three specimens plated by Treatment A fracture within 24 h and none of the three specimens plated by Treatment B fracture within 200 h after applying the sustained loads specified in Table 3.

7.5.4 To verify further the quality of the manufactured lot of specimens, a minimum of five specimens plated per Treatment

TABLE 2 Lot Acceptance Criteria for Type 1 Notched Specimens

Type	Item	Sampling of Each Lot	Requirement/Method
1	Hardness[A]	5 %	51 to 53 HRC per ASTM Test Method E 18. Round the average of three readings per specimen per Practice E 29.
1	Dimensions	100 %	Meet tolerances of corresponding drawings. Notch dimension verified with shadow graphic projection at 50 to 100×.
1	Notched Fracture Strength (NFS)	10 ea	NFS of each specimen must be within 10 ksi of the average.
1c	Self-loading Notched round bar bend fixture, Fig. A2.5	10 ea	Alternate: The number of turns of the loading bolt, which is required to produce fracture in each specimen, must be within 5 % of the average.
1d	Self-loading Notched C-Ring bend fixture, Fig. A2.6	10 ea	Alternate: The change in diameter at fracture load specimens for each specimen must be within 0.005 in. 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 3 Pass/Fail Loading Requirements of Test Specimens

Type 1a, 1b, 1c, 1d	75 % of the tensile or bend notched fracture strength (Table 2).
Type 2a	92 % of Test Method E 8, obtained by deflecting a 2.300-in. diameter O-ring specimen with a 2.525-in. stressing bar.

B shall be tensile tested per Test Methods E 8 as in Table 2 and all of the tensile test results shall be within ±10 ksi of the mean of the ten unplated specimens. The diameter or dimensions of the bare metal specimen shall be used in the stress calculations.

7.6 *Certification*:

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

7.6.1.1 Manufacturer of specimen lot.

7.6.1.2 Steel supplier, heat number, and certificate for chemical composition and heat treatment response.

7.6.1.3 Test results for requirements in Table 2, 7.5.3, and 7.5.4, including the corresponding average rupture load in units of X.XX kips.

8. Testing Protocol

8.1 *Plating Processes*:

8.1.1 *Number*—A minimum of four specimens shall be used per test.

8.1.1.1 Test specimens produced and tested per this Test Method shall only be used once. Stripping and reuse of specimens is prohibited.

8.1.1.2 Sometimes it is necessary to plate only the notched area of the specimen to provide an escape path for hydrogen during bake.

8.1.1.3 The diameter or dimensions of the bare metal specimen shall be used in stress calculations.

8.1.2 *Load*—Sustained load specimens shall be loaded in accordance with Table 3.

8.1.3 *Time*—Sustained loads shall be maintained for a minimum of 200 h. Actual fracture times can be electronically monitored with a relay switch system.

8.1.4 An alternate rising step load (RSL) accelerated test (≤24 h) as described in Annex A4 of this document and as further defined in Test Method F 1624 can be used if specified by the purchaser.

8.2 *Service Environments*:

8.2.1 The testing protocol (number, load, and time) are specified in Annex A5.

9. Interpretation

9.1 A plating process shall be considered acceptable quality if none of the plated specimens fracture within 200 h after loading as specified in Table 3.

9.2 If only one of a minimum of four specimens fractures within the sustained load exposure time, continue to step load the remaining three specimens every hour in 5 % increments to 90 % of the fracture tensile/bend load after completion of a minimum 200-h sustained load. After 1 h at 90 %, the process shall be considered nonembrittling if no fracture occurs in the three remaining specimens.

9.3 If two or more specimens fracture within the sustained load exposure time, the plating process shall be considered as having excessive embrittling characteristics.

9.4 The cause of any premature failure shall be determined and corrected before retest.

9.5 Retest the plating process with four unused specimens. If no fracture occurs within the sustained load exposure time, the material or process shall be considered acceptable quality. If any specimen fractures during retest, the plating process shall be considered embrittling.

9.6 Another retest can only be considered after a metallurgical examination of the fractured specimens indicates some defect in the processing of the specimens.

9.7 Preexisting specimen defects such as cracks, grinding burns, or nonmetallic inclusions can be considered as a basis for invalidation of rejectable test results and retest.

10. Report

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

10.1.1 A lot acceptance and sensitivity certification report.

10.1.2 The type and number of specimens tested.

10.1.3 A description of the plating process and test environment (concentration, temperature, and so forth) if other than ambient air.

10.1.4 The sustained or threshold load, or percent of notched fracture strength or notch bend strength of unplated specimens, or displacement as appropriate for the type of specimen tested.

10.1.5 The time under load in the test environments.

11. Precision and Bias

11.1 No information is presented about either the precision or bias of Test Method F 519 for measuring the time to failure, since the test results are nonquantitative pass/fail measurements.

11.2 The precision and bias of this test method for measuring the threshold are essentially as specified in Test Method F 1624.

12. Keywords

12.1 cleaner; coating; delayed failure; fluids; hydrogen embrittlement; maintenance chemicals; plating; steel; stress cracking; threshold

ANNEXES

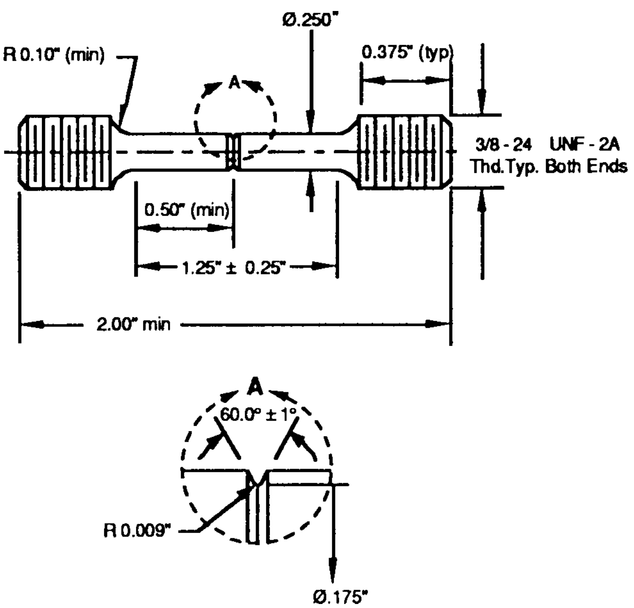
(Mandatory Information)

A1. SPECIAL REQUIREMENTS FOR THE TESTING OF NOTCHED ROUND BAR TENSION SPECIMENS UNDER CONSTANT LOAD

A1.1 *Type 1a*—The notched round bar tension specimen is given in two sizes. If the specimen geometry is not called out, the Type 1a.1, standard notched round bar specimen under load control shall be used.

A1.2 *Type 1a.1 Standard*—The dimensions for the standard sized specimen for the sustained load test (SLT) is shown in Fig. A1.1. The test machine shall have a minimum load capacity of 10 kips to measure the notched fracture strength in accordance with Table 2. Correspondingly, a test machine with a minimum load capacity of 7 kips is required to maintain a sustained load of 75 % of the notched fracture strength in accordance with Table 3.

A1.3 *Type 1a.2*—The dimensions for the oversized specimen for the sustained load test (SLT) is shown in Fig. A1.2. The test machine must have a minimum capacity of 16 kips to measure the notched fracture strength in accordance with Table 2. Correspondingly, a minimum 12-kip test machine is required to maintain a sustained load of 75 % of the notched fracture strength in accordance with Table 3.

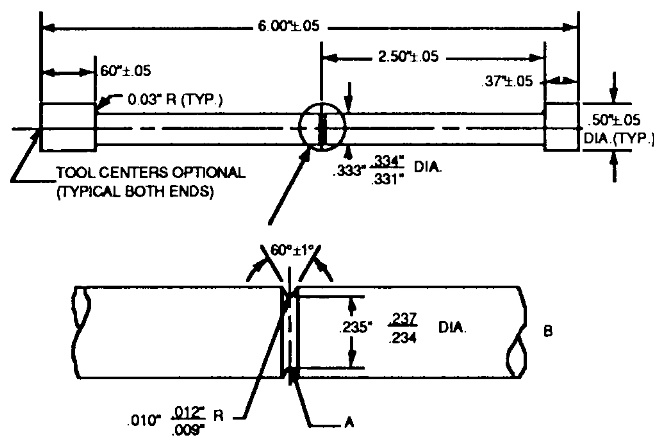


NOTE 1—Specimen preparation must comply with Section 7.2.

NOTE 2—End configuration is optional, except that external threaded ends must have the minor diameter greater than 0.25 in.

NOTE 3—Root radii, reduced section, and notch root radius must be concentric with centerline of specimen within 0.002 T.I.R.

FIG. A1.1 Dimensional Requirements for the Type 1a.1 Specimen



NOTE 1—Specimen preparation must comply with Section 7.2.

NOTE 2—End configuration is optional, such as the use of external threaded ends, where the minor diameter must be greater than 0.35 in.

NOTE 3—Root radii, reduced section, and notch root radius must be concentric with centerline of specimen within 0.002 T.I.R.

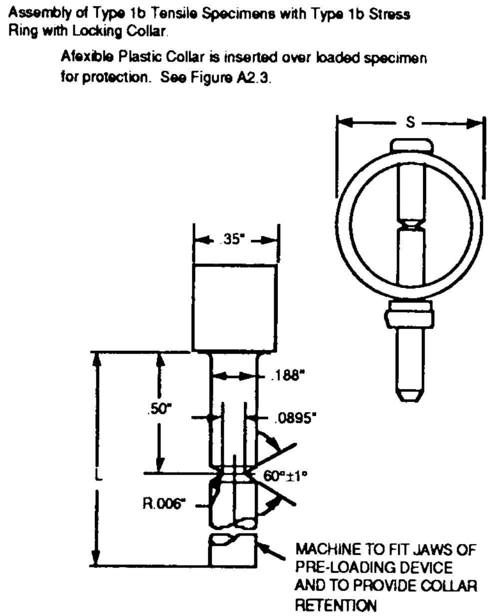
FIG. A1.2 Dimensional Requirements for the Type 1a.2 Specimen

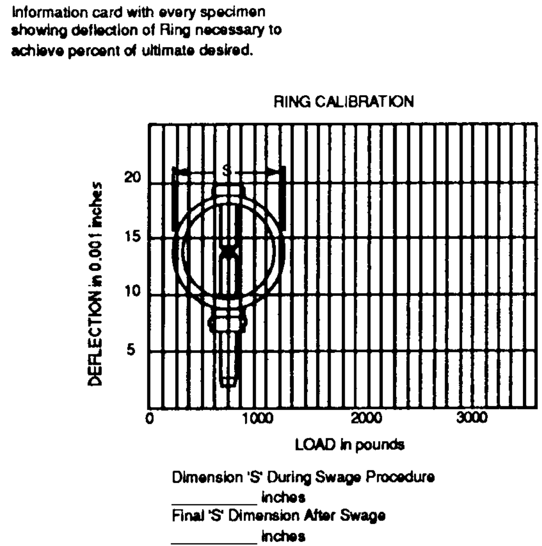
A2. SPECIAL REQUIREMENTS FOR THE TESTING OF NOTCHED SPECIMENS IN SELF-LOADING FIXTURE

A2.1 *Type 1b—Notched Bar Tension*—Type 1b is a sub-sized specimen ($d/D = 0.0895/0.188$) under a constant displacement requiring a special sustained loading device for loading to 75 % of the notched ultimate tensile strength. The capacity of the testing machine for determination of the notched ultimate tensile strength in accordance with Table 2 must exceed 2.5 kips (11 kN). Consideration should also be given to Test Methods E 8 Note 5. "The use of specimens smaller than 0.250-in. diameter shall be restricted to cases where the material to be tested is of insufficient size to obtain larger specimens or when all parties agree to their use for acceptance testing. Smaller specimens require suitable equipment and greater skill in both machining and testing."

A2.1.1 The method of spring-loading notched round bars in tension uses a precalibrated ring as detailed in Fig. A2.1. The ring manufacturer calibrates each ring by determining the change in diameter of the ring when subjected to a load equivalent to that required to stress a test specimen to 75 % of its notched ultimate strength.

A2.1.2 Mechanisms used to load a test specimen in a stressed ring must be able to hold the test specimen at a stress level of 90 % of the maximum load applied to the specimen. A method that allows loading and locking test specimens at stress





NOTE 1—"L" to be 1.5 in. (38.1 mm) or greater.
NOTE 2—Tolerance (unless otherwise specified):
X.XX ± 0.05 in. (127 mm)
X.XXX ± 0.001 in. (0.025 mm)
X.XXXX ± 0.0002 in. (0.005 mm)

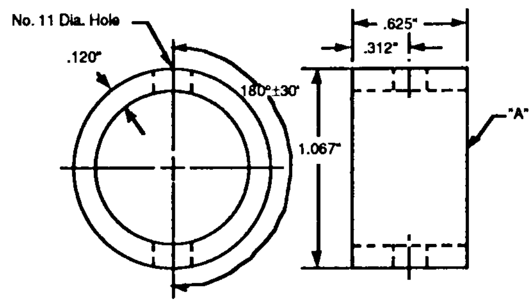
FIG. A2.1 Dimensional Requirements for Type 1b Specimens

levels as high as 75 % of their notched tensile strength is described as follows.

A2.1.3 Insert the test specimen in a calibrated ring. Fit the end of the specimen extending through the ring with a stainless steel retaining collar and insert it into the jaws of a hydraulic loading device. As the load is applied, the collar is swaged to the shaft of the specimen. No torsional loads shall be introduced during loading. The change in ring diameter after removal from the hydraulic loading device indicates the actual stress level obtained (test load), and this must be at least 90 % of the change in ring diameter produced during initial loading. If the specimen has not failed by the end of the test period, the measured ring diameter must be within 0.001 in. (0.025 mm) of that measured after initiating the test. (**Warning**—To prevent possible injury from a premature failure of a loaded specimen, mount a protective collar over the specimen and ring immediately after loading as shown in Fig. A2.2 and Fig. A2.3.)

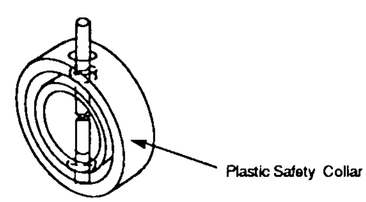
A2.2 *Type 1c—Notched Round Bar Bend*—The specimen dimensions are given in Fig. A2.4. A self-loading device can be used to subject notched round bars to four-point bending loads is shown in Fig. A2.5. The device shown in Fig. A2.5 is calibrated by counting the number of turns of the loading bolt required to fracture the specimen. Start the counting at the point where the tightening of the loading bolt first eliminates all slack in the mechanism. This position of the loading bolt measures the distance between the loading bars at each end to ensure that the same starting point is used for each loading. Stress levels are then indicated as a percentage of the average number of turns required to cause fracture in the calibration process.

A2.3 *Type 1d—Notched C-Ring Bend*—The dimensions for



NOTE 1—No. 11 diameter holes located a true position within 0.005-in. (0.127-mm) diameter and drilled parallel to face "A" within 0.003 in. (0.076 mm).
NOTE 2—Material to be AISI 4130 steel heat treated in accordance with MIL-H-6875 to a tensile strength between 200 and 220 ksi (1380 and 1520 MPa).
NOTE 3—Tolerances (unless otherwise specified):
X.XXX ± 0.005 in. (0.125 mm)
X.XXXX ± 0.0025 in. (0.065 mm)

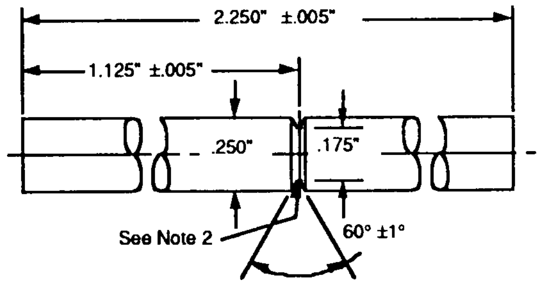
FIG. A2.2 Dimensional Requirements for Type 1b Stressing Ring



NOTE 1—A flexible collar (1¾-in. outside diameter with ¼-in. wall) is inserted over the specimen and stressing ring after loading to the required stress level.

FIG. A2.3 Protective Collar Installation on Type 1b Test Specimens and Stressing Ring

the notched bend C-ring specimen are given in Fig. A2.6. The notched bend C-ring specimen consists of a 1¼-in. long section cut from 2-in. outside diameter, ⅛-in. wall thickness, machined from 2-in. diameter bar stock of AISI 4340 steel. For a self-loading device, use a loading bolt to place the specimen under load. The bolt shall be oriented across the diameter of the specimen opposite the notch as shown in Fig. A2.6. Determine the average diameter of the notched C-ring at fracture with



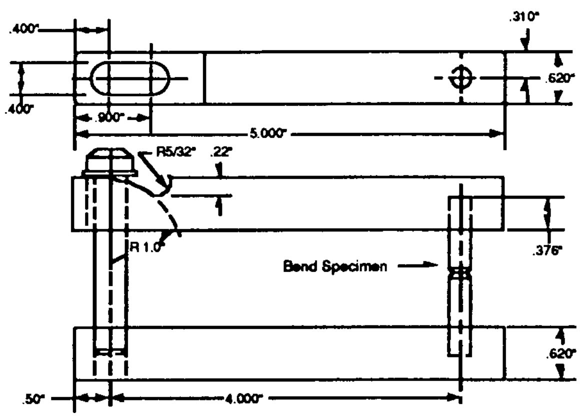
NOTE 1—Specimen preparation must comply with Section 7.2.

NOTE 2—Notch root radius to be 0.0050 ± 0.0005 in.

NOTE 3—Notch angle is also optional per Section 7.2.2.

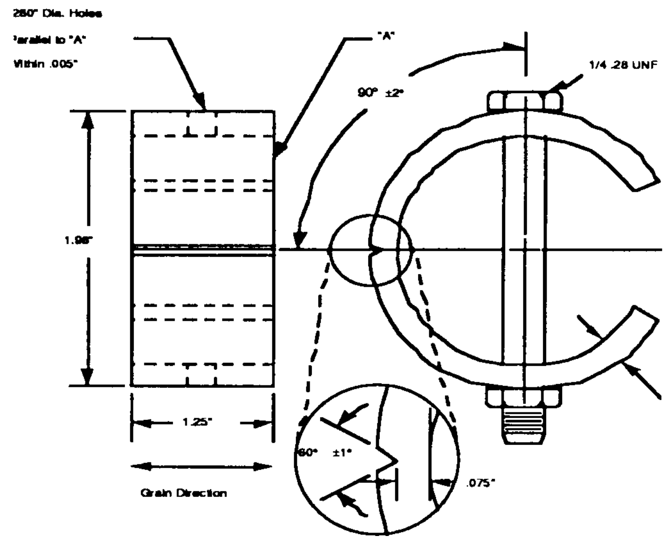
NOTE 4—Root radii, reduced section, and notch root radius must be concentric with centerline of specimen within 0.002 T.I.R.

FIG. A2.4 Dimensional Requirements for the Type 1c Notched Round Bar Bend Specimen



NOTE 1—Loading bars shall be made from stainless steel (>35 HRC) ⅝-in. square bar.

FIG. A2.5 Notched Round Bar Bend Self-Loading Fixture



NOTE 1—Specimen preparation must comply with Section 7.2.

NOTE 2—Notch root radius to be 0.0030 ± 0.0005 in.

NOTE 3—Notch angle is also optional per Section 7.2.2.

FIG. A2.6 Dimensional Requirements for Type 1d Notched C-Ring Bend Specimens with Loading Bolt

unexposed specimens (see Table 2) with the loading device in Fig. A2.7. Stress levels can be related to the percentage change in diameter required to fracture the specimens.

A3. A QUANTITATIVE, ACCELERATED (≤24 h) TEST METHOD THAT MEASURES THE THRESHOLD LOAD FOR THE ONSET OF HYDROGEN ASSISTED STRESS CRACKING

A3.1 Specimens—All Test Method F 519 specimens can be tested in accordance with the accelerated incrementally increasing or rising step-loading testing protocol of Test Method F 1624 in either tension or in bending. The specimen Type 1e is scaled to match the same a/W and K_t values as the Type 1d, notched C-ring bend specimen, although Type 1e is oriented with the crack plane transverse, like the Type 1a, 1b, and 1c specimens.

A3.1.1 *Type 1e*—Notched Square Bar Bend Specimen: The specimen dimensions are shown in Fig. A3.1. This specimen shall be tested with appropriate four-point bend adapters under displacement control.

A3.2 *Manufacture*—The specimens shall be manufactured from hot-drawn square bar, which is rough machined to allow for any anticipated decarburization. The specimens shall be

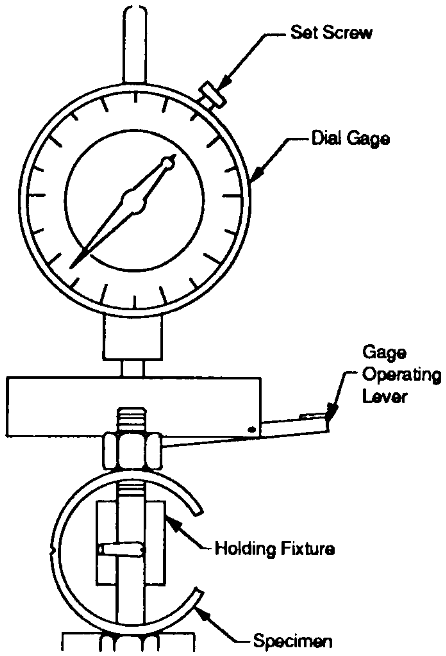
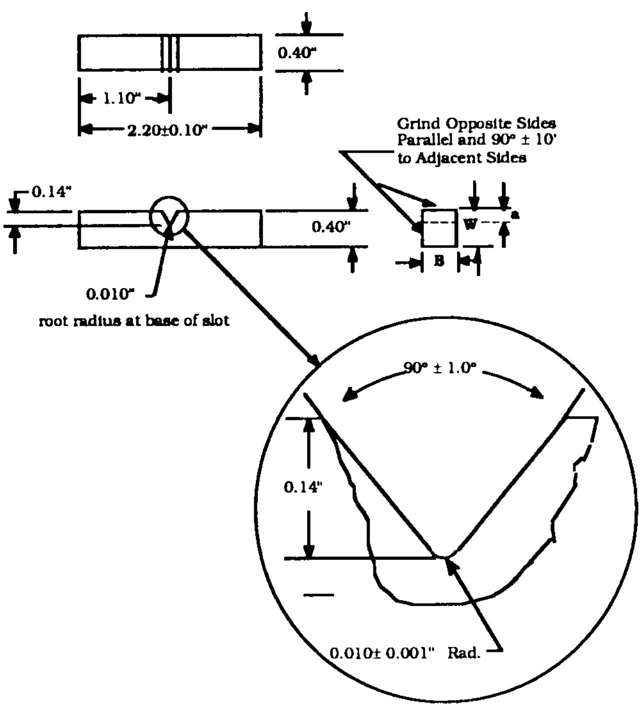


FIG. A2.7 Device for Measuring Deflection During Stressing of Type 1d Notched C-Ring Bend Specimen



NOTE 1—Specimen preparation must comply with Section 7.2.

NOTE 2—Notch must be in the LS orientation per ASTM E 616.

FIG. A3.1 Dimensional Requirements for a Type 1e, Notched Square Bar Bend Specimen

heat treated to 51 to 53 HRC. One transverse section shall be microstructurally examined to insure that if any orientation effects exist, the notch will be in the LS orientation per Terminology E 616. The specimen shall then be ground to final dimensions to eliminate decarburization of the sides. The entire notch shall be ground to size after quenching and tempering to final hardness before plating. Single-point turning of the notch configuration is not permitted. Burnishing of the notch is not permitted. Within the tolerances of the specimen, a notched depth-to-width ratio of $a/W = 0.35$ shall be maintained. A 4-h stress relief at 375 ± 25°F shall be applied. Suitable protection to prevent discoloration as a result of a surface oxide may be used.

A3.3 *Loading Protocol*:

A3.3.1 The RSL test method is a combined sustained load and slow strain rate test for hydrogen embrittlement by using incremental loads and hold times under displacement control to detect the onset of subcritical crack growth to measure a threshold stress that is used to quantify the amount of residual hydrogen in a specimen.

A3.3.2 A specific rising step load and holding time protocol in accordance with Test Method F 1624 is prescribed only for the Test Method F 519, AISI Type 4340, Type 1e, four-point bend test specimens. Instrumented testing equipment with adjustable constant displacement loading is required as described in Test Method F 1624.

NOTE A3.1—A loading device that has been found to meet the displacement control step load test requirements of Test Method F 1624 is available from Fracture Diagnostics, Inc., P.O. Box 6401, Denver, CO, 80206.[11]

A3.3.3 The loading protocol for this standard, consists of steps of no less than 10 % of the fracture strength (10 % FS) per hour up to 50 % FS and then steps no greater than 5 % FS/h until rupture. Longer holding times or smaller % FS may be used. The minimum requirements for a step load profile are listed in Table A3.1.

A3.4 *Threshold*—When a specimen is 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 will be 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 will be recorded. The test results will be recorded as a threshold, which is a percentage of the fracture stress for that specimen configuration and not as pass/fail as with the sustained load test, time-to-fracture criterion.

[11] The loading device is covered by a patent. Interested parties are invited to submit information regarding the identification of an alternative(s) to this patented item to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend.

TABLE A3.1 Minimum Requirements for a Step Loading Profile for Accelerated (≤24 h) Rising Step Load Threshold Determination

%FS	#h	Σh	%FS	#h	Σh	%FS	#h	Σh	%FS	#h	Σh
10	1	1	50	1	5	70	1	9	90	1	13
20	1	2	55	1	6	75	1	10	95	1	14
30	1	3	60	1	7	80	1	11	100	1	15
40	1	4	65	1	8	85	1	12	105	1	16

A3.5 *Sensitivity Testing*—Based on the loading profile schedule in Fig. A3.1, the requirements for sensitivity (Section 7.5) of the heat-treated lot of specimens shall be demonstrated if the Treatment A plated specimens fracture in less than 5 h (<50 % FS) and no fracture of Treatment B plated specimens occurs in less than 13 h (>90 % FS).

A3.6 As an alternative to using a plated specimen, based on the loading profile schedule in Table A3.1, the requirements for sensitivity (Section 7.5) of the heat-treated lot of specimens shall be demonstrated if unplated (bare) specimens fracture in less than 5 h at an imposed potential of −1.2 V versus SCE and no delayed fracture occurs in less than 13 h (>90 % FS) on unplated (bare) specimen tested in air.

A3.7 *Equivalence Between SLT and RSL*:

A3.7.1 Specimen testing of the plating process and sensitivity testing consists of the same loading profile as in Table A3.1. Since the threshold is measured with the RSL test method, equivalent loads are related to the percentage of the fracture strength; therefore, to meet the requirements of the SLT, only the specified percentage of the fracture strength needs to be attained with the RSL test. As an example, a threshold of 80 % of the notched fracture strength is equivalent to the 200-h SLT plating requirement of 75 %.

A3.7.2 Based on the loading profile schedule in Fig. A3.1, the 75 % load tests can be conducted in 10 h.

A3.7.3 For thresholds in service environments that are less than 75 %, a loading rate of 5 % of the fracture strength per hour shall be used. Then a threshold of 70 % of the notched fracture strength is equivalent to the 200-h SLT service environment requirement of 65 %, and so forth. When the threshold exceeds the sustained load test requirement, they are considered to be equivalent.

A3.7.4 Alternately, time can be used as a criterion for achieving a threshold. As an example, specimens achieve a 75 % threshold per Table A3.1 when the test time of 10 h is attained or exceeded. For thresholds less than 75 %: such as 65 % with the modified 5 %/h protocol (13 h); or for 45 % (9 h).

A3.8 *Interpretation of Results*:

A3.8.1 For Test Method F 519 test specimens that exceed 90 % of their RSL fracture strength or (≥13 h), the plating bath is considered to be nonembrittling.

A3.8.2 For Test Method F 519 test specimens that exceed 75 % of their RSL fracture strength or (≥13 h), the plating bath is considered to be of acceptable quality.

A3.9 *Tolerance of Parts*:

A3.9.1 Since hydrogen tolerance varies with hardness, actual parts made of low-strength steel might have more tolerance for residual hydrogen because of plating and might not need the same threshold as high-hardness steels. Therefore, adjustments in threshold requirements can be made. As an example, the conventional pass/fail 200-h acceptance level of 75 % of the fracture strength is not necessarily hydrogen free but can be considered adequate for many applications of high-strength steels that have low applied service stresses and no residual stress as a result of processing.

A3.9.2 To obtain a correlation between actual production hardware and threshold levels in this standard, the threshold level or hydrogen tolerance level for the production hardware can be measured using Test Method F 1624.

A4. SPECIAL REQUIREMENTS FOR THE TESTING OF SMOOTH O-RING SPECIMENS WITH CONSTANT DISPLACEMENT STRESS BARS

A4.1 *Specimen*:

A4.1.1 *Type 2a*—The dimensions of the specimen are shown in Fig. A4.1. The specimen loading configuration involves the insertion of an oversized stressing bar into an O-ring, while the O-ring is compressed in a vise. The stressing bars are made according to Fig. A4.2. The vise jaws should be faced with nonmarring material such as aluminum or rubber having a 60 Shore A Durometer hardness.

A4.2 *Manufacture*:

A4.2.1 The smooth O-ring specimen loaded is processed as follows:

A4.2.2 The Type 2a specimens are machined to size from normalized and stress relieved tubing or bar stock.

A4.2.2.1 Specimens shall be deburred by hand, using silicon-carbide paper, before heat treatment.

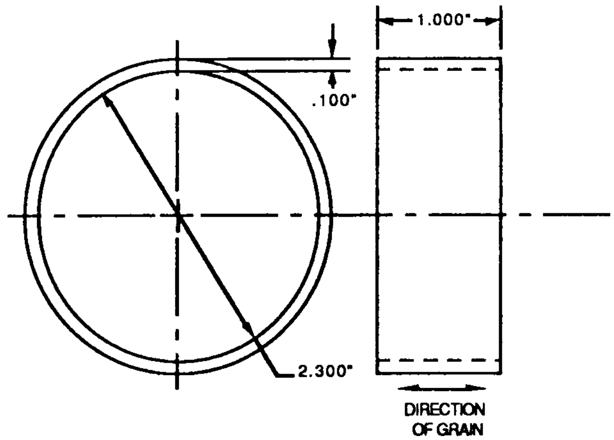
A4.2.2.2 Specimens shall be heat treated from 51 to 53 HRC. One specimen per lot shall be microstructurally examined to determine evidence of decarburization.

A4.2.2.3 Clean all surfaces by blasting lightly with 150 grit or finer aluminum oxide after final heat treatment and again after shot peening. Use a 0.375- to 0.5-in. nozzle, 30- to 50-psi pressure, approximately 10 in. from part surface and moved continuously.

A4.2.2.4 All surfaces of specimens shall be shot peened using conditioned shot in accordance with AMS 2430. The shot intensity shall be Almen “A” 0.006 to 0.010 in. in accordance with Specification B 851.

A4.2.3 Grinding after final heat treatment is prohibited for Type 2a specimens.

A4.3 Type 2a specimens shall be suitable for test purposes if each specimen is inspected and found to be in conformance with the requirements of Table A4.1.



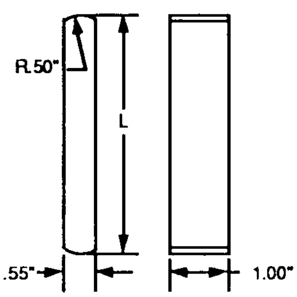
NOTE 1—Surface finish of 63RMS or better to be held before shot peening.

NOTE 2—Ovality of ring must be within tolerance before and after heat treatment and shot peening.

NOTE 3—Ovality of ring is determined by measuring the diameter of the ring at three locations oriented 60° apart.

NOTE 4—Tolerance: X.XXX ± 0.002 in.

FIG. A4.1 Dimensional Requirements for Type 2a Specimens



NOTE 1—Bars should be made from plain carbon or low alloy steel. Both the stressing bars and the O-ring specimens are to be coated with the plating being evaluated for hydrogen embrittlement; but for sensitivity testing per Table 2, bars must be cadmium-electroplated with bright cadmium in accordance with QQ-P-416 before use.

NOTE 2—Length "L" to be 2.415 ± 0.003 in. to load Type 3.1 specimens to 80 % of the yield strength.

NOTE 3—Length "L" to be 2.525 ± 0.002 in. to load Type 3.1 specimens to 92 % of the ultimate strength.

FIG. A4.2 Dimensional Requirements for Type 2a Stressing Bars

TABLE A4.1 Acceptance Criteria for Type 2a Specimen

Property	Requirement/Method
Hardness[A]	51 to 53 HRC (ASTM Test Method E 18)
Dimensions[A]	Conformance to Fig. A4.1
Surface quality[A]	No magnetic indications greater than 0.031 in. Guide ASTM E
Scratches[A]	Not allowed

[A] Only specimens meeting the listed criteria shall be acceptable for testing.

A5. DETAILS OF TESTING IN SERVICE ENVIRONMENTS

A5.1 *Service Environments*—Notice: Because of the extensive variety of newly developed cleaners and maintenance chemicals, loading sequences and times of exposure, the requirements are often user specific. As a result, the testing protocol described in this section should only be used as a guideline. A more extensive protocol and details for use in specific applications is currently in process.

A5.1.1 *Surface Finish*—Specimens shall be coated or plated as specified by the procuring agency. Unplated specimens can be specified by the procuring agency for specific service environment testing. The unplated specimens shall also meet the surface finish requirements of this standard.

A5.1.2 *Number*—A minimum of four specimens shall be used per test. If plated specimens are required, they shall meet the testing requirements in Part 1 of this test method before being subjected to evaluation in a service environment. Test specimens produced and tested per this specification shall only be used once. Stripping and reuse of specimens is prohibited.

A5.2 *Classification of Chemicals—Definitions:*

A5.2.1 Process or service chemicals shall be classified as aggressive or passive.

A5.2.1.1 With aggressive chemicals (such as temper etching with nitric acid), the time of exposure is intentionally limited because of their aggressive corrosive reaction with AISI E4340 steel.

A5.2.1.2 Passive chemicals include cleaners and paint strippers, which typically do not result in any measurable corrosive attack of AISI E4340 steel.

A5.3 *Service Chemical Testing Procedure*:

A5.3.1 *Aggressive Chemicals*—The test specimen shall be immersed under a stress of 45 % of the notched fracture strength and shall be limited to the exposure time of the relevant service specification (sometimes to a fraction of a minute). After the specified exposure under a preload, the test specimens and fixture are neutralized (per the relevant service specification), rinsed and dried with compressed air. The load of the test specimen is then increased to 75 % of net fracture strength for 200 h for Type 1 specimens. The Type 2a specimen is loaded to 92 %.

A5.3.2 *Passive Chemicals*—Test specimens under the specified load shall be coated or immersed in passive chemicals for the duration of the test. After the test is completed, the test specimen and fixtures are rinsed and dried with compressed air.

A5.4 *Test Conditions*—The test shall be conducted in air or any other controlled environment using an appropriate inert container and fixture that is suitably electrically isolated from the specimen or compensated to prevent a galvanic coupling.

A5.4.1 Test liquids in the concentrated condition and at the maximum specified dilution to determine the full embrittling effect of exposure in service.

A5.4.2 Test solids in the saturated condition and at the minimum specified concentration to determine the full embrittling effect of exposure in service.

A5.4.3 Conduct all tests at 68 to 86°F (20 to 30°C) or the operating service temperature of the maintenance chemicals. Do not exceed 150°F (66°C).

A5.5 *Loading Protocol*—Install the test specimens into the loading frame. Apply the loads specified in Table 3. For testing maintenance chemicals, any containment chamber shall be isolated around the notched section of the test specimen. If the entire loading frame is immersed into the chemicals, the loading frames shall be designed to avoid any galvanic reaction with the test specimen. The sequence of exposure to the environment and load application shall be documented.

A5.6 *Interpretation of Results*:

A5.6.1 An aggressive chemical shall be considered nonembrittling if none of the exposed specimens fracture within 200 h after being stressed to 75 % of their fracture strength for Type 1 specimens, or 92 % for Type 2a specimens (see Table 3).

A5.6.2 A passive chemical shall be considered nonembrittling if none of the immersed specimens fail within 150 h after immersion into the chemical at the loads specified in Table A5.1. Record the time to failure if less than 150 h. The test may be discontinued after 150 h.

A5.6.3 If only one of a minimum of four specimens fractures within the exposure time, continue to step load the remaining three specimens, every hour in 5 % increments to 90 % of the fracture tensile/bend load after completion of a minimum 200- or 150-h sustained load. After 1 h at 90 %, the process shall be considered nonembrittling if no fracture occurs.

A5.6.4 If two or more specimens fracture within the exposure time, the service environment shall be considered as having excessive embrittling characteristics.

A5.6.5 The root cause and corrective action must be determined before any retest.

A5.6.6 Retest the service environment with four unused specimens. If no fracture occurs within the exposure time, the material or process shall be considered nonembrittling. If any specimen fractures during retest, the service environment shall be considered embrittling.

A5.6.7 Another retest can only be reconsidered after a metallurgical examination of the failed specimens indicates some defect in the processing of the specimens.

A5.6.8 A retest can only be considered after a metallographic examination of the fractured specimens indicates some defect in the specimens.

A5.6.9 Preexisting specimen defects such as cracks, grinding burns, or nonmetallic inclusions can be considered as an invalidation of fracture.

TABLE A5.1 Load Requirements for Evaluating Passive Service Environments

Type 1a, 1b	45 % notched tensile fracture load.
Type 1c	45 % notched bend fracture load.
Type 1d	65 % notched bend fracture load.
Type 2a	80 % of 0.2 % YS per ASTM E 8, obtained by deflecting a 2.300-in. diameter O-ring specimen with a 2.415-in. stressing bar.