



Standard Test Method for Mechanical Hydrogen Embrittlement Evaluation of Plating/Coating Processes and Service Environments¹

This standard is issued under the fixed designation F519; 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.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope

1.1 This test method describes mechanical test methods and defines acceptance criteria for coating and plating processes that can cause hydrogen embrittlement in steels. Subsequent exposure to chemicals encountered in service environments, such as fluids, cleaning treatments or maintenance chemicals that come in contact with the plated/coated or bare surface of the steel, can also be evaluated.

1.2 This test method is not intended to measure the relative susceptibility of different steels. The relative susceptibility of different materials to hydrogen embrittlement may be determined in accordance with Test Method [F1459](#) and Test Method [F1624](#).

1.3 This test method specifies the use of air melted AISI E4340 steel per SAE AMS-S-5000 (formerly MIL-S-5000) heat treated to 260 to 280 ksi (pounds per square inch $\times 1000$) as the baseline. This combination of alloy and heat treat level has been used for many years and a large database has been accumulated in the aerospace industry on its specific response to exposure to a wide variety of maintenance chemicals, or electroplated coatings, or both. Components with ultimate strengths higher than 260 to 280 ksi may not be represented by the baseline. In such cases, the cognizant engineering authority shall determine the need for manufacturing specimens from the specific material and heat treat condition of the component. Deviations from the baseline shall be reported as required by [12.1.2](#). The sensitivity to hydrogen embrittlement shall be demonstrated for each lot of specimens as specified in [9.5](#).

1.4 Test procedures and acceptance requirements are specified for seven specimens of different sizes, geometries, and loading configurations.

1.5 *Pass/Fail Requirements*—For plating/coating processes, specimens must meet or exceed 200 h using a sustained load test (SLT) at the levels shown in [Table 3](#).

¹ This test method is under the jurisdiction of ASTM Committee [F07](#) on Aerospace and Aircraft and is the direct responsibility of Subcommittee [F07.04](#) on Hydrogen Embrittlement.

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1.5.1 The loading conditions and pass/fail requirements for service environments are specified in [Annex A5](#).

1.5.2 If approved by the cognizant engineering authority, a quantitative, accelerated (≤ 24 h) incremental step-load (ISL) test as defined in [Annex A3](#) may be used as an alternative to SLT.

1.6 This test method is divided into two parts. The first part gives general information concerning requirements for hydrogen embrittlement testing. The second is composed of annexes that give specific requirements for the various loading and specimen configurations covered by this test method (see section [9.1](#) for a list of types) and the details for testing service environments.

1.7 The values stated in the foot-pound-second (fps) system 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.

1.8 *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:²

- [B374 Terminology Relating to Electroplating](#)
- [B851 Specification for Automated Controlled Shot Peening of Metallic Articles Prior to Nickel, Autocatalytic Nickel, or Chromium Plating, or as Final Finish](#)
- [D1193 Specification for Reagent Water](#)
- [E4 Practices for Force Verification of Testing Machines](#)
- [E8/E8M Test Methods for Tension Testing of Metallic Materials](#)
- [E18 Test Methods for Rockwell Hardness of Metallic Materials](#)

² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

TABLE 1 Lot Acceptance Criteria for Notched Specimens

Type	Item	Sampling of Each Lot	Requirement/Method
1	Tensile Strength	4 each	Test Method E8. Tensile strength of each specimen must be within 10 ksi of the average.
1	Hardness ^A	5 %	51 to 53 HRC per Test Methods E18. Round the average of three readings per specimen per Practice E29.
1	Dimensions	100 %	Meet tolerances of corresponding drawings. Notch dimension verified with shadow graphic projection at 50 to 100× or equivalent.
1	Notched Fracture Strength (NFS)	10 ea	Test Methods E8/E8M. NFS of each specimen must be within 10 ksi of the average.
1c	Self-loading notched round specimen bend fixture, Fig. A2.7	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. A2.8	10 ea	Alternate: The change in diameter at fracture load for each specimen must be within 0.008 inches 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.

- [E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications](#)
- [E292 Test Methods for Conducting Time-for-Rupture Notch Tension Tests of Materials](#)
- [E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method](#)
- [E709 Guide for Magnetic Particle Testing](#)
- [E1417 Practice for Liquid Penetrant Testing](#)
- [E1444 Practice for Magnetic Particle Testing](#)
- [E1823 Terminology Relating to Fatigue and Fracture Testing](#)
- [F1459 Test Method for Determination of the Susceptibility of Metallic Materials to Hydrogen Gas Embrittlement \(HGE\)](#)
- [F1624 Test Method for Measurement of Hydrogen Embrittlement Threshold in Steel by the Incremental Step Loading Technique](#)
- [F2078 Terminology Relating to Hydrogen Embrittlement Testing](#)
- [G5 Reference Test Method for Making Potentiodynamic Anodic Polarization Measurements](#)
- [G38 Practice for Making and Using C-Ring Stress-Corrosion Test Specimens](#)

2.2 *SAE AMS Standard*:³

[AMS 2430 \(R\) Shot Peening, Automatic](#)

[AMS 2759/2 Heat Treatment of Low-Alloy Steel Parts Minimum Tensile Strength 220 ksi \(1517 MPa\) and Higher](#)

[AMS 2759/11 Stress Relief of Steel Parts](#)

[AMS 6360 Steel Tubing, Seamless 0.95Cr – 0.20Mo \(0.28 – 0.33C\) \(SAE 4130\) Normalized or Stress Relieved](#)

[AMS-QQ-P-416 Plating, Cadmium \(Electrodeposited\)](#)

[AMS-S-5000 Steel, Chrome-Nickel-Molybdenum \(E4340\) Bars and Reforging Stock](#)

2.3 *Military and Federal Standards and Commercial Item Descriptions*:⁴

[MIL-PRF-16173 Corrosion Preventive Compound, Solvent Cutback, Cold-Application](#)

[Commercial Item Description \(CID\) A-A-55827 Chromium Trioxide, Technical](#)

3. Terminology

3.1 *Definitions*—Definitions of terms used in this test method may be found in Terminology B374, Terminology E1823 and Terminology F2078.

3.2 *Acronyms and Abbreviations*:

AIISI = American Iron and Steel Institute

a/W = notch depth-to-width ratio

d/D = ratio of minor to major diameters at the notch

ESH = Environmental, Safety and Health

fps = foot pound second

HRC = Rockwell Hardness Scale C

ID = inside diameter

ISL = incremental step load

kip = pounds load multiplied by 1000

ksi = pounds-force per square inch multiplied by 1000

K_t = stress concentration factor

LS = longitudinal short transverse

NFS = notched fracture strength

OD = outside diameter

psi = pounds-force per square inch

RMS = root mean square

SAE AMS = Society of Automotive Engineers Aerospace Material Specification

SCE = saturated calomel electrode

SLT = sustained load test

T.I.R. = total indicated runout

4. Summary of Test Methods

4.1 *Plating/coating Processes*—Unstressed test specimens are cleaned, plated/coated, and baked (if applicable) in accordance with the specification to which the process is to be

³ Available from Society of Automotive Engineers (SAE), 400 Commonwealth Dr., Warrendale, PA 15096-0001, <http://www.sae.org>.

⁴ Copies of these documents are available at <http://assist.daps.dla.mil/> quicksearch/ or <http://assist.daps.dla.mil/> or from the Standardization Document Order Desk, 700 Robins Ave., Building 4D, Philadelphia, PA 19111-5094.

TABLE 2 Electroplating Bath Compositions and Operating Conditions for Sensitivity Test

Item	Treatment A		Treatment B
Bath composition:	Range		Optimum
Cadmium (as CdO)	2.9 to 5.5 oz/gal (22 to 41 g/L)	4.5 oz/gal (33.7 g/L)	same as Treatment A
Total Sodium cyanide (NaCN)	12.0 to 16.0 oz/gal (89.9 to 120 g/L)	14 oz/gal (104 g/L)	same as Treatment A
Ratio NaCN to CdO	2.8/1 to 6.0/1	3.0/1	same as Treatment A
pH	12.0 or greater	12.0	same as Treatment A
Temperature	70–90°F (21–32°C)	75°F (24°C)	same as Treatment A
Sodium hydroxide (NaOH) ^A	1.0 to 3.2 oz/gal (7.5 to 24.0 g/L)	2.5 oz/gal (18.7 g/L)	same as Treatment A
Brightener such as Colcad 100 ^B or equivalent	Manufacturer's suggested range		None
Electroplating current	10 A/ft ² (108 A/m ²)		60 A/ft ² (645 A/m ²)
Electroplating time	30 minutes		6 minutes
Baking			
Baking temperature	375 ± 25°F (190 ± 14°C)		same as Treatment A
Baking time: Type 1 Specimen	Do Not Bake		23 h
Baking time: Type 2a Specimen	8 h		23 h
Chromate Treatment ^C	Yes		same as Treatment A

^A Addition of sodium hydroxide may not be required on solution makeup, since the addition of 1 oz/gal of cadmium oxide is equivalent to 0.6 oz/gal of free hydroxide.

^B The sole source of manufacture of Colcad 100 known to the committee at this time is Columbia Chemical in Brunswick, Ohio, www.columbiachemical.com. If you are aware of alternative manufacturers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend.

^C After baking, the specimens shall be dipped into any appropriate chromate conversion coating solution for minimum time that will produce an adherent and continuous coating as described in AMS-QQ-P-416 Type II.

TABLE 3 Pass/Fail Loading Requirements of Test Specimens

Type 1a, 1b, 1c, 1d, 1e	75 % of the tensile or bend NFS (Table 1).
Type 2a	92 % of the Test Methods E8/E8M, E4 ultimate strength, obtained by deflecting a 2.300-inch diameter O-Ring specimen with a 2.525-inch stressing bar.

qualified. Specimens are then maintained under a sustained load in air to measure the time to rupture/completion of the test period.

4.2 Service Environments—Specimens are tested in the service environment. The sequence of exposure to the environment and loading shall be as defined in Annex A5. If plated/coated test specimens are to be utilized for evaluating a service environment, then the plating/coating process must first be determined to be acceptable in accordance with 4.1.

5. Significance and Use

5.1 Plating/coating Processes—This test method provides a means by which to detect possible hydrogen embrittlement of steel parts during manufacture by verifying strict controls during production operations such as surface preparation, pretreatments, and plating/coating. It is also intended to be used as a qualification test for new plating/coating processes and as a periodic inspection audit for the control of a plating/coating process.

5.2 Service Environment—This test method provides a means by which to detect possible hydrogen embrittlement of steel parts (plated/coated or bare) due to contact with chemicals during manufacturing, overhaul and service life. The details of testing in a service environment are found in Annex A5.

6. Apparatus

6.1 Testing Machine—Testing machines shall be within the guidelines of calibration, force range, resolution, bending strain, and verification of Practices E4 and Test Methods E292 as applicable.

6.1.1 The force measurement on test machines used for notched round tension specimens, shall be via analytical calculations such as for dead weight or lever arm type hanging weight machines, or by routine empirical measurement (electronic output) such as for strain gauge load cells. Estimating test loads that are derived from load ring deflections only is not allowed, unless the effects of specimen grip joint compliance is known, is accounted for, and within Practice E4 and Test Method E8/E8M tolerances. Strain gauge load cell measurements/results can be used in conjunction with load ring deflections.

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

6.3 Series Loading—Samples may be loaded on a test frame either individually or by multiples in series. All specimens loaded onto an individual test frame shall have the same certified NFS ± 1 %. In the event that one or more of the specimens fracture during the designated test period, the fractured specimen(s) shall be removed, replaced with a dummy specimen and the load chain placed back under load. The total time under load (that is, test duration) for the remaining specimens shall be extended by any time-off-load as detailed in 6.3.1. The procuring specification or cognizant engineering authority may elect to restrict loading to one specimen per test frame.

6.3.1 *Extended Time Under Load (ETUL)*—If the total time-off-load (wall clock) exceeds 10 % of the total test duration (for example, 20-h for a 200-h test); then the time under load shall be extended by the amount of time-off-load in excess of 10 % (that is, in excess of 20 h). The procuring specification or cognizant engineering authority may elect to waive this extended time under load (ETUL) protocol.

EXAMPLE 1 for SLT

Four cadmium-plated specimens representing a single plating bath are loaded on an individual SLT test frame as part of a monthly plating bath verification. One of the four specimens fractures after 180 h (initial time under load = 180 h). It takes 8 h to discover the fractured specimen, replace it with a dummy link, and put the remaining three specimens back under load (time off-load = 8 h). No extension of time under load is required since the time off-load (8 h) was less than 10 % of the total test duration (10 % of 200 h = 20 h). Total time under load will therefore be 200 h for the remaining specimens.

EXAMPLE 2 for SLT

Twenty cadmium-plated specimens representing five distinct plating baths (A, B, C, D, and E) are loaded on an individual SLT test frame as part of a monthly plating bath verification. One specimen from bath A fractures after 100 h; one specimen from bath B fractures after 120 h; and one specimen from bath C fractures after 140 h. Each time a specimen fractures, it takes 8 h to discover the fractured specimen, replace it with a dummy link, and put the remaining specimens back under load (total time off-load = 8+8+8 h = 24 h). The total time off-load (24 h) exceeds 10 % of the total test duration (10 % of 200 = 20 h) by 4 h. Therefore, the time under load must be extended by 4 h. Total time under load will therefore be 204 h for the remaining specimens.

6.4 *Service Environment 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 used for testing in aqueous environments. The corrosion potential of the specimen may be controlled with a reference Saturated Calomel Electrode (SCE) or equivalent reference electrode such as with Ag/AgCl in accordance with Reference Test Method G5.

7. Materials and Reagents

7.1 *Materials:*

7.1.1 Air melted AISI E4340 per AMS-S-5000 (formerly MIL-S-5000).

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

7.1.3 Conditioned shot, in accordance with AMS 2430.

7.2 *Reagents:*

7.2.1 Corrosion preventive compound, meeting the requirements of MIL-PRF-16173, Grade 2.

7.2.2 Cadmium cyanide electroplating bath (SAE AMS-QQ-P-416, formerly Federal Specification QQ-P-416. (Table 2).

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

7.2.4 Chromic acid (CID A-A-55827, formerly Federal Specification O-C-303D).

7.2.5 Water (Specification D1193 Type IV).

8. Hazards

8.1 *Environmental, Safety and Health (ESH):*

8.1.1 Equipment, materials, solutions, and emissions (if applicable) shall be controlled, handled, used, and disposed in accordance with the applicable governing ESH regulations.

9. Test Specimens

9.1 *Configuration:*

9.1.1 Dimensional drawings with tolerances are given for the two types of specimens in the following annexes:

9.1.1.1 Type 1—Notched Specimens

Type 1a: Notched, Round, Tension

Type 1a.1—Standard Size—per Fig. A1.1

Annex A1

Type 1a.2—Oversized—per Fig. A1.2

Annex A1

Type 1b: Notched, Round, Tension Self Loading Fixture—per

Annex A2

Figs. A2.1-A2.5

Type 1c: Notched, Round, Bend Self Loading Fixture—per

Annex A2

Fig. A2.6, Fig. A2.7

Type 1d: Notched, C-Ring, Bend Self Loading Fixture—per

Annex A2

Fig. A2.8, Fig. A2.9

Type 1e: Notched, Square, Bend—per Fig. A3.1

Annex A3

9.1.1.2 Type 2—Smooth Specimens

Type 2a: O-Ring, Bend Self Loading Fixture—per Fig. A4.1, Fig. A4.2

Annex A4

9.1.2 The notched round tension, round bend and square bend specimens 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.

9.2 *Manufacture:*

9.2.1 Stress concentration factors (K_t) for each of the specimen types were calculated using Finite Element Analysis (FEA) and the dimensions in the drawings.

NOTE 1—An ASTM Research Report documenting the analysis is in process and a reference number will be included in a future revision.

Type	K_t		
	Minimum ^A	Nominal	Maximum ^A
1a.1	3.16	3.30	3.48
1a.2	3.31	3.59	3.76
1b	2.86	3.07	3.33
1c	3.30	3.45	3.62
1d	3.73	4.03	4.42
1e	3.95	4.27	4.60
2a	NA	NA	NA

^A Minimum and maximum K_t s calculated from worst case combinations of dimensional tolerances.

9.2.2 If the 60° notch angle does not permit plating/coating to the root of the notch, then an angle of 90° ± 1° shall be used only with prior approval of the cognizant engineering authority.

NOTE 2—The FEA determined that changing the notch angle from 60° to 90° had a negligible affect on the K_t . Therefore, no dimensional changes are required to compensate for this deviation. However, such deviation shall be reported as required in 12.1.2.

9.2.3 The baseline material/condition for test specimens is normalized and tempered, hot or cold drawn bar stock, air melted AISI E4340 steel per AMS-S-5000 (formerly MIL-S-5000) heat treated per AMS 2759/2 Class A to a tensile strength between 260 and 280 ksi (51 to 53 Rockwell C Hardness Scale (HRC)) as determined according to Test Methods E18. Rounding per Practice E29 permits an absolute hardness range of 50.6 to 53.4 HRC of the average of three measurements.

9.2.3.1 Other steels or final hardnesses may only be substituted if agreed upon by the cognizant engineering authority. Deviations from the baseline shall be reported as required in 12.1.2.

9.2.4 Rough machine blanks as required.

9.2.5 Heat treat as a finished part to a strength level of 260 to 280 ksi per AMS 2759/2 Class A or B as follows:

9.2.5.1 Austenitize at 1500 – 1550°F in a protective atmosphere and then quench in oil.

9.2.5.2 Double temper, 2 h each, at 425 – 490°F.

9.2.6 Verify heat treat response by pulling 4 specimens in accordance with Test Method E8/E8M and Table 1.

9.2.7 Finish machine the reduced section and notch configuration.

9.2.7.1 The entire notch shall be ground to size.

9.2.7.2 Single-point machining of the notch is not permitted.

9.2.7.3 Burnishing of the notch is not permitted.

9.2.7.4 Shot peening/blasting/mechanical cleaning of the notch during specimen manufacturing is not permitted.

9.2.8 After grinding, all specimens shall receive a stress relief bake at 375 ± 25°F (190 ± 14°C) per AMS 2759/11 for 4–5 h. A suitable protection from discoloration due to surface oxidation shall be used during the stress relief bake.

9.2.9 Acid or cathodic electrolytic cleaning is prohibited.

9.2.10 Straightening after final heat treatment is prohibited.

9.3 Storage:

9.3.1 After stress relief baking, 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-PRF-16173, Grade 2.

9.4 Inspection:

9.4.1 A lot shall consist of those specimens cut from the same heat of steel in the same orientation, heat treated together in the same furnace load, quenched and tempered together and subjected to the same manufacturing processes together.

9.4.2 All notched specimens in the lot shall be suitable for test purposes if the inspection results on samples from that lot conform to the requirements of Table 1 and 9.5.

9.5 Sensitivity Test:

9.5.1 The sensitivity to hydrogen embrittlement shall be demonstrated for each lot of specimens by exposing six trial specimens to two different embrittling environments after manufacture and inspection in accordance with Section 9.

9.5.1.1 Three specimens shall be electroplated under the highly embrittling conditions produced in a bright cadmium cyanide bath by Treatment A (Table 2) and the remaining three specimens shall be electroplated under the less embrittling conditions of Treatment B (Table 2). An equivalent plating/coating or imposed potential may be used only with the approval of the cognizant engineering authority if its sensitivity is demonstrated to be equivalent to that found in Table 2.

9.5.2 Using the SLT method, each lot of specimen 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.

9.5.3 Using the ISL test method, each lot of specimens shall be of suitable sensitivity only if all three specimens plated by Treatment A initiate a crack at less than 50 % NFS and all of the three specimens plated by Treatment B fracture above 90% NFS identified in Table 3. The loading profile to be used with this standard is fifteen load increments of 5 % NFS sustained

for one hour plus five increments of 5 % NFS for 2 h each or (15/5/1) + (5/5/2), which totals to a maximum of 24 hour loading profile for a maximum anticipated load of 100 % NFS.

9.6 Certification:

9.6.1 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:

9.6.1.1 Manufacturer of specimen lot.

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

9.6.1.3 Heat treat process certification for specimen lot.

9.6.1.4 Test results for requirements in Table 1 and 9.6, including the corresponding average rupture load in units of X.XX kips (1 kip = 1000 pounds load).

10. Procedure

10.1 Plating/coating Processes:

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

10.1.2 *Re-Use of Specimens*—Test specimens produced and tested per this test method shall only be used once. Stripping and reuse of specimens are prohibited.

10.1.3 *Plating/Coating Coverage*—The entire specimen with the exception of the grip areas shall be covered by plating/coating as determined by a visual inspection.

10.1.4 *Masking of Grip Areas of Specimens*—The grip areas of the specimens may be masked to prevent plating/coating deposit when required to avoid interference with the test fixture. If the grip area is masked, this fact shall be reported.

10.1.5 *Masking To Provide Hydrogen Escape Path*—Non-porous plating/coatings such as nickel or bright cadmium can inhibit the outward diffusion of hydrogen. Therefore, when plating specimens with these types of plating/coatings it is necessary to leave a portion of the specimen unplated/coated so that the hydrogen can be baked out. The following guidelines are established for masking specimens to be plated/coated with hard, non-porous coatings:

10.1.5.1 *All notched Types (1a.1, 1a.2, 1b, 1c, 1d, and 1e)*—The entire notch and 0.5 inch on either side of the notch shall be plated/coated.

10.1.5.2 *Type 2a Smooth Ring*—Only the edges may be masked. The entire ID and OD shall be plated/coated.

10.1.6 *Dimensions for Stress Calculations*—The dimensions of the bare metal specimen shall be used in stress calculations.

10.1.7 SLT Method:

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

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

10.1.8 The alternate, ISL accelerated test (≤24 h) as described in Annex A3 of this document and as further defined in Test Method F1624 may be used only if specified by the cognizant engineering authority.

10.2 Service Environments:

10.2.1 The testing protocol (number, load, and time) shall be as specified in Annex A5.

11. Interpretation of Results

11.1 *No Fracture*—For the SLT method, a plating/coating process shall be considered non-embrittling if none of the plated/coated specimens fracture within 200 h after loading as specified in [Table 3](#). Self-loading specimen Types (1b, 1c, 1d and 2a) may need to be examined using magnetic particle per Practice [E1444](#) or liquid penetrant per Practice [E1417](#) to determine if a crack initiated but did not propagate due to load relaxation.

11.2 *One of Four Fracture*—If only one of a minimum of four specimens fractures within the 200-h sustained load exposure time, the remaining three specimens may be step loaded every 2 h in 5 % increments to 90 % of the NFS after completion of the 200-h sustained load. If the three remaining specimens all sustain 90 % NFS for 2 h, the plating/coating process shall be considered non-embrittling.

11.3 *Two or More Fracture*—If two or more specimens fracture within the sustained load exposure time, the plating/coating process shall be considered embrittling.

11.4 *Replacement Testing*—Replacement testing shall only be conducted after the root cause of any failure has been determined to be anomalous.

11.4.1 *Root Cause Determination*—A failure may be ruled anomalous if it is determined that an error was made in the plating/coating process of the specimens or if a metallurgical examination of the fractured specimen indicates some defect in the manufacture of the specimen.

11.4.1.1 Pre-existing specimen defects such as cracks, grinding burns, or nonmetallic inclusions can be considered a basis for invalidation of test results and replacement testing.

11.4.1.2 Fracture at any location other than the notch invalidates the test and is a basis for replacement testing for all notched specimens (that is, all types except 2a).

11.4.2 *Number of Specimens*—A replacement test shall use four specimens. If any specimen fractures during the replacement test, the plating/coating process shall be considered embrittling. If the failure is determined to be anomalous, retesting per [11.4](#) may be repeated.

12. Report

12.1 A test record or report shall be produced upon completion of testing that bears the minimum information specified in [12.1.1](#) through [12.1.6](#).

12.1.1 A lot acceptance and sensitivity certification record or report in accordance with the requirements of [Table 1](#) and [9.5](#) that clearly identifies the notched fracture strength (NFS) for the specimen lot(s) being tested.

NOTE 3—The lot acceptance and sensitivity record or report may be provided by the specimen manufacturer.

12.1.2 The type and number of specimens tested including deviations from baseline geometry, material, temper, K_t , or masking practices.

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

12.1.4 The sustained or threshold load applied, or percent of NFS or notch bend strength of unplated specimens, or displacement as appropriate for the type of specimen tested.

12.1.5 The number of specimens on the test frame together; their certified NFSs; details of any specimen fractures along with associated time-off-load; and any Extended Time Under Load (ETUL).

12.1.6 The time under load in the test environments.

12.1.7 If fractures were observed, they shall be reported. If samples conform to requirements, the report shall so state. If step-loading protocol of [11.2](#) is invoked, it shall state why with reference to fractures, and it shall be clearly noted on the face of the certification.

13. Precision and Bias

13.1 *Precision*—The sensitivity test requirement in [9.5](#) and the acceptance and rejection criteria in Section [11](#) are the judgments for precision, and may vary with the precision on applied stress and test specimen geometry.

13.2 *Bias*—Random lot-to-lot bias in the properties of specimens related to raw material or specimen manufacture may exist.

14. Keywords

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

ANNEXES

(Mandatory Information)

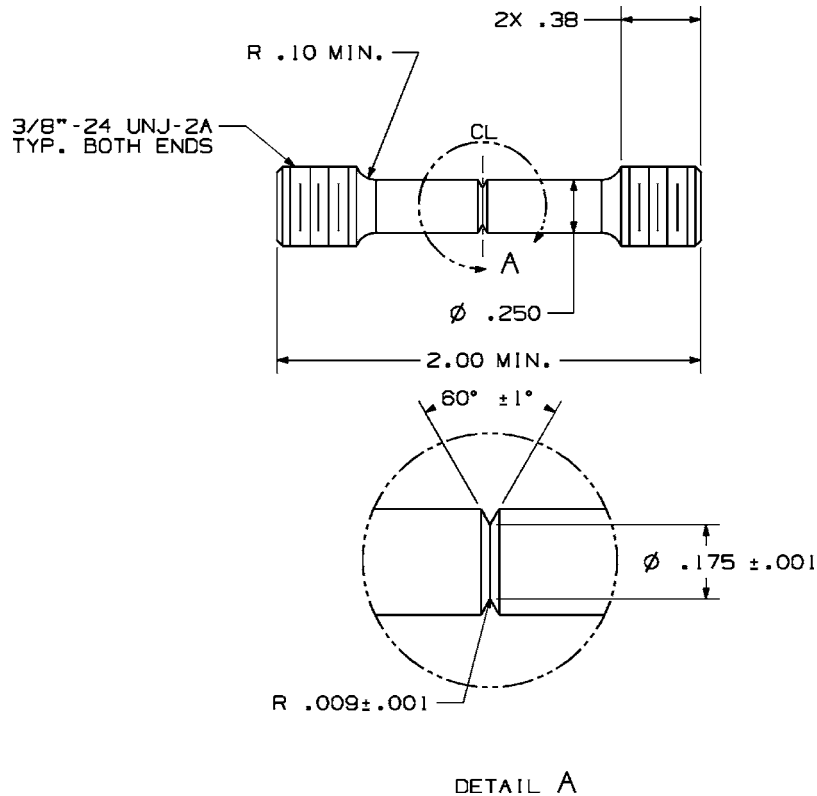
A1. SPECIAL REQUIREMENTS FOR THE TESTING OF NOTCHED ROUND TENSION SPECIMENS

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

A1.1.1 *Type 1a.1 Standard*—The dimensions for the standard sized specimen for the sustained load test (SLT) are shown in Fig. A1.1. The test machine shall have a minimum load capacity of 10 kips to measure the NFS in accordance with Table 1. Correspondingly, a test machine with a minimum load

capacity of 7.5 kips is required to maintain a sustained load of 75 % of the NFS in accordance with Table 3.

A1.1.2 *Type 1a.2*—The dimensions for the oversized specimen for SLT are shown in Fig. A1.2. The test machine must have a minimum capacity of 16 kips to measure the NFS in accordance with Table 1. Correspondingly, a minimum 12-kip test machine is required to maintain a sustained load of 75 % of the NFS in accordance with Table 3.



Tolerances unless otherwise specified:

- X.X ± 0.1
- X.XX ± 0.03
- X.XXX ± 0.005

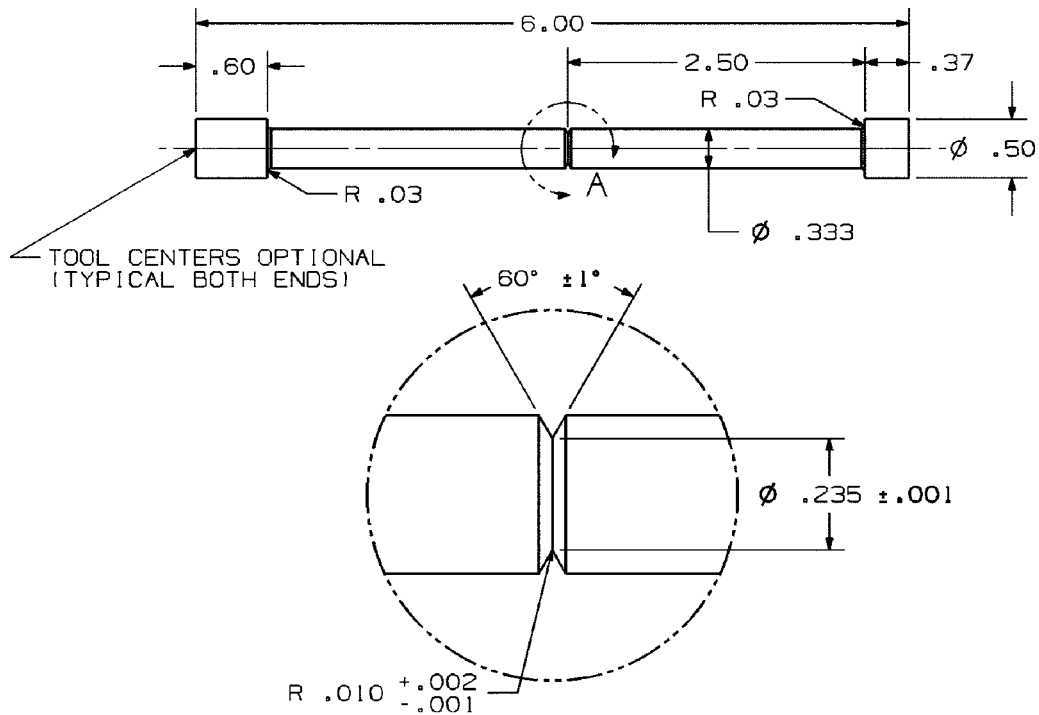
NOTE 1—Specimen preparation must comply with 9.2.

NOTE 2—Surface finish of notch shall be 32 RMS or better, all other surfaces shall be 63 RMS or better.

NOTE 3—Configuration of the ends of the specimen is optional so long as any external threaded ends have the minor diameter greater than 0.25 inch.

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

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



DETAIL A

Tolerances unless otherwise specified:

- X.X ± 0.1
- X.XX ± 0.05
- X.XXX ± 0.007

NOTE 1—Specimen preparation must comply with 9.2.

NOTE 2—Surface finish of notch shall be 32 RMS or better, all other surfaces shall be 63 RMS or better.

NOTE 3—Configuration of the ends of the specimen is optional so long as any external threaded ends have the minor diameter greater than 0.35 inch.

NOTE 4—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

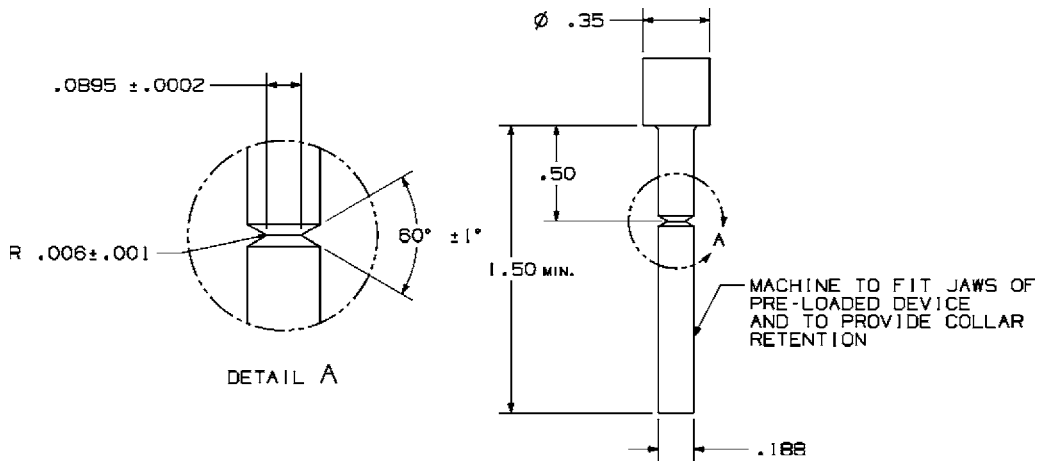
A2. SPECIAL REQUIREMENTS FOR THE TESTING OF NOTCHED SPECIMENS IN SELF-LOADING FIXTURE

A2.1 *Type 1b—Notched Tension Specimen*—Type 1b is a sub-sized specimen where the ratio of minor to major diameters at the notch ($d/D = 0.0895/0.188$). The specimen is loaded under a constant displacement and requires a special sustained loading device for loading to 75 % of the NFS. The capacity of the testing machine for determination of the NFS in accordance with Table 1 must exceed 2.5 kips. Consideration should also be given to Test Methods E8/E8M – 04, Note 5, Figure 8 concerning the use of subsize specimens.

A2.1.1 The method of spring-loading notched round specimens in tension uses a pre-calibrated ring as detailed in Figs. A2.1-A2.5. 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 % NFS.

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 levels as high as 75 % NFS 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



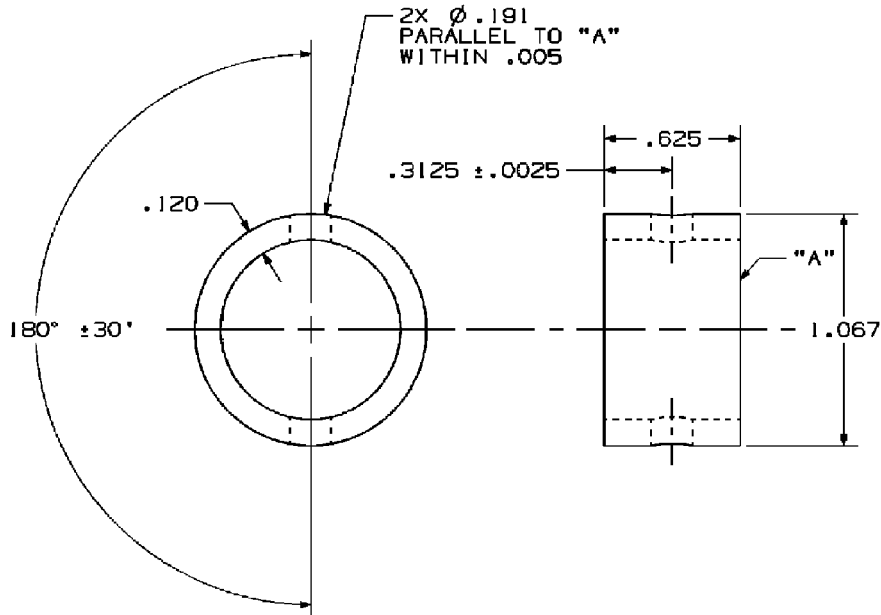
Tolerances unless otherwise specified:
 X.X \pm 0.1
 X.XX \pm 0.03
 X.XXX \pm 0.001

NOTE 1—Specimen preparation must comply with 9.2.

NOTE 2—Surface finish of notch shall be 32 RMS or better, all other surfaces shall be 63 RMS or better.

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

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



Tolerances unless otherwise specified:
 X.X \pm 0.1
 X.XX \pm 0.03
 X.XXX \pm 0.005

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 per AMS 6360 or equivalent, heat treated in accordance with AMS 2759/2 Class A to a tensile strength between 200 and 220 ksi (1380 and 1520 MPa).

NOTE 3—See Annex A5 for galvanic isolation requirements when used for fluid testing.

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

measured ring diameter must be within 0.001 in. of that measured after initiating the test.

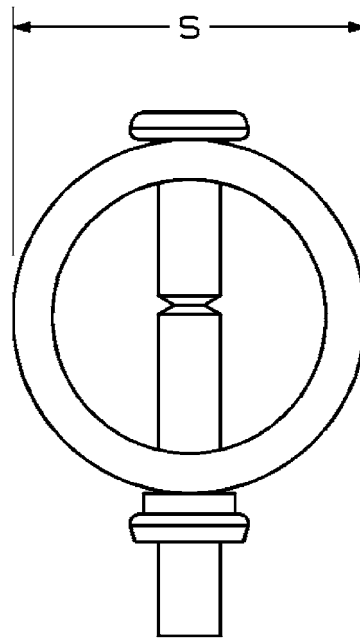
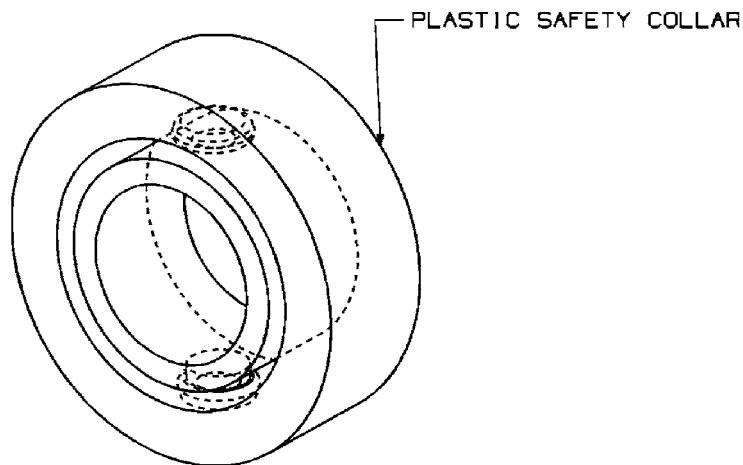


FIG. A2.3 Assembly of Type 1b Tensile Specimen with Type 1b Stress Ring and Swaged Retaining Collar



Tolerances unless otherwise specified:
 X.X ± 0.1
 X.XX ± 0.03
 X.XXX ± 0.010

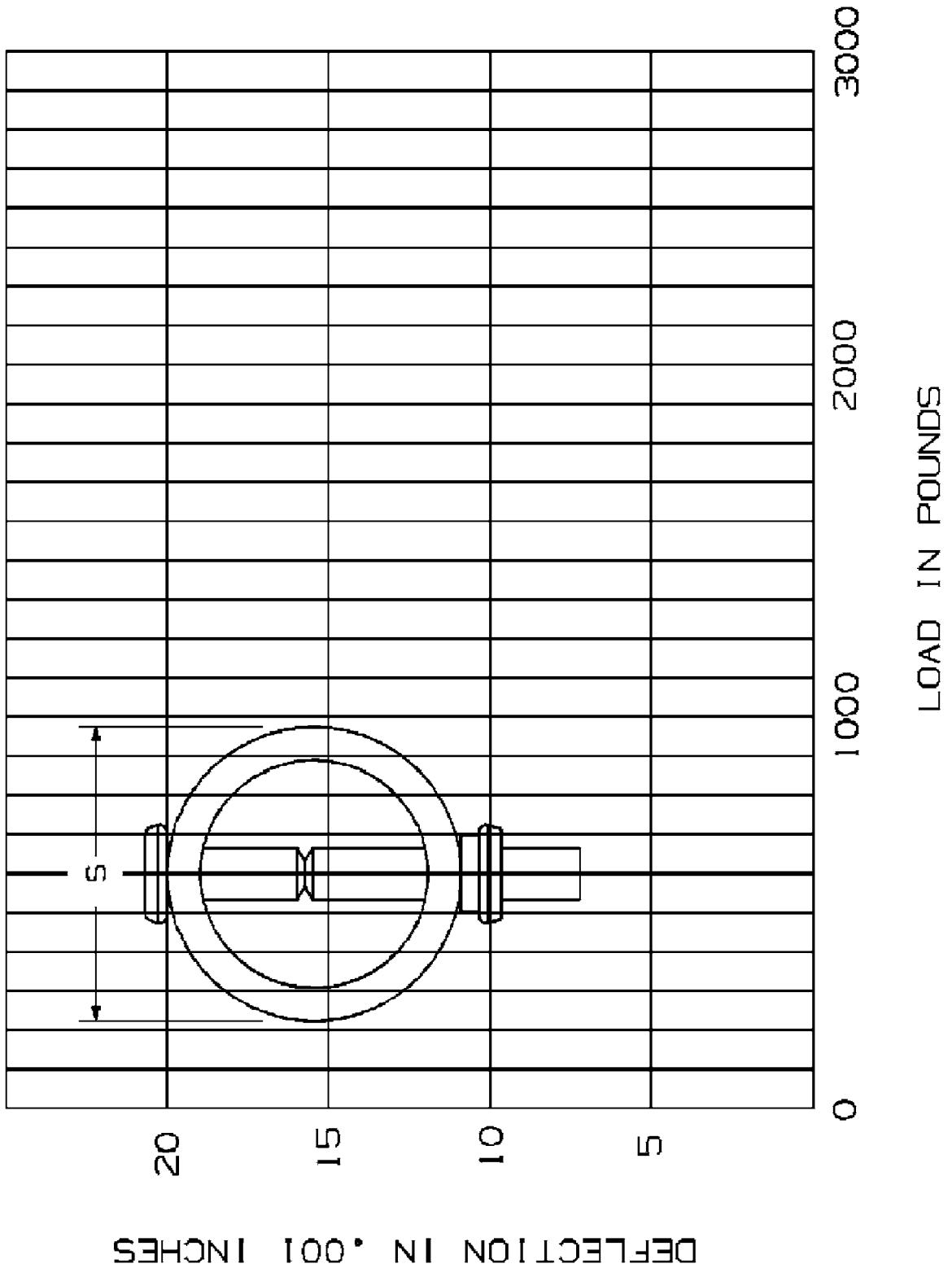
NOTE 1—A flexible collar (1¾-in. outside diameter with ¼-in. wall) is inserted over the specimen and stressing ring after loading to the required stress level.

FIG. A2.4 Protective Collar Installation on Type 1b Specimens and Stressing Ring

A2.1.4 **Warning**—To prevent possible injury from a failure of a loaded specimen, mount a protective collar over the specimen and ring immediately after loading as shown in Fig. A2.4 and Fig. A2.5.

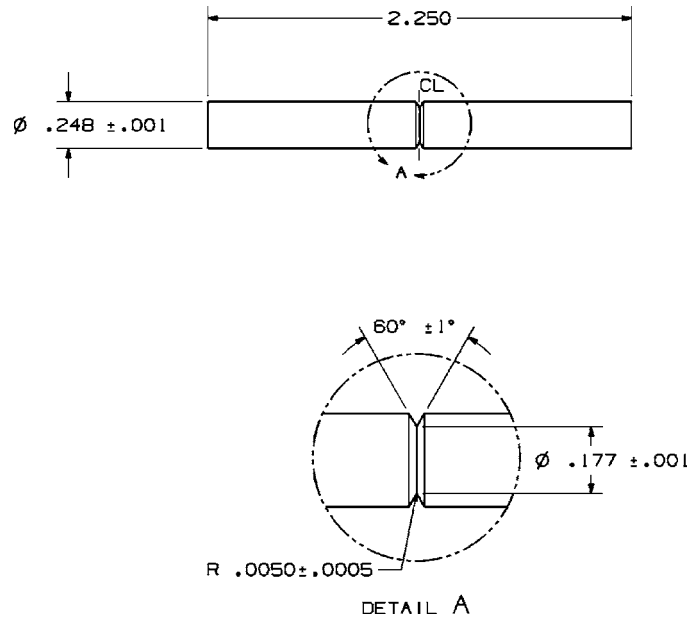
A2.2 *Type 1c—Notched Round Bend Specimen*—The specimen dimensions are given in Fig. A2.6. A self-loading device that can be used to subject notched round specimens to simulated pure bending loads is shown in Fig. A2.7. The device

RING CALIBRATION



Dimension 'S' During Swage Procedure ___ Inches
 Final 'S' Dimension After Swage Procedure ___ Inches

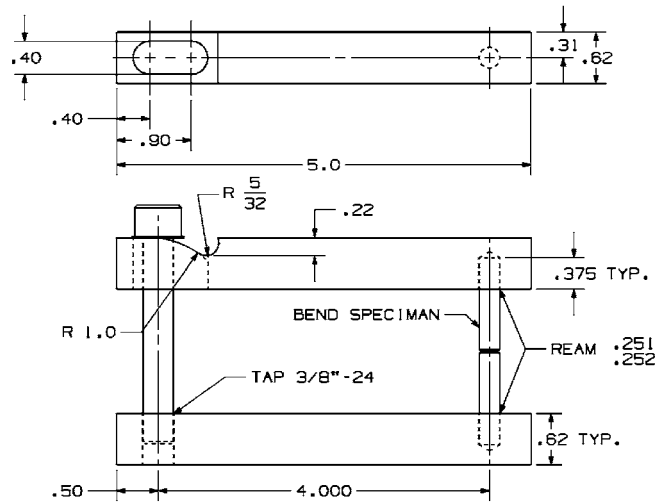
FIG. A2.5 Type 1b Information Card Showing Deflection of Stress Ring Necessary to Achieve Percent of Ultimate Desired



Tolerances unless otherwise specified:
 X.X ± 0.1
 X.XX ± 0.03
 X.XXX ± 0.005

- NOTE 1—Specimen preparation must comply with 9.2.
- NOTE 2—Surface finish of notch shall be 32 RMS or better, all other surfaces shall be 63 RMS or better.
- NOTE 3—Root radii, reduced section, and notch root radius must be concentric with centerline of specimen within 0.002 T.I.R.

FIG. A2.6 Dimensional Requirements for the Type 1c Specimen



Tolerances unless otherwise specified:
 X.X ± 0.1
 X.XX ± 0.01
 X.XXX ± 0.002

- NOTE 1—Loading bars shall be made from stainless steel (>35 HRC) 5/8-inch square bar.
- NOTE 2—See Annex A5 for galvanic isolation requirements when used for fluid testing.

FIG. A2.7 Self-Loading Bend Fixture for Type 1c

shown in Fig. A2.7 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. The distance

between the loading bars shall be measured at each end in addition to the number of turns of the loading bolt, to ensure proper seating of the specimen and loading bolt.

NOTE A2.1—Users are cautioned that debris may accumulate in the

bottom of the loading bolt recess for accepting the specimen. This can potentially alter the stress values if not removed. Users are further cautioned that specimens may move out of position as load is applied and measures shall be taken to preclude this movement.

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 the notched bend C-Ring specimen are given in Fig. A2.8. The notched bend C-Ring specimen consists of a 1/4-inch long section machined from 2-inch diameter or larger bar stock of air-melted AISI E4340 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. A2.8. Determine the average diameter of the notched C-Ring at fracture with unexposed specimens (see Table 1) with the loading device in Fig. A2.9. Stress levels can be related to the percentage change in diameter required to fracture the specimens.

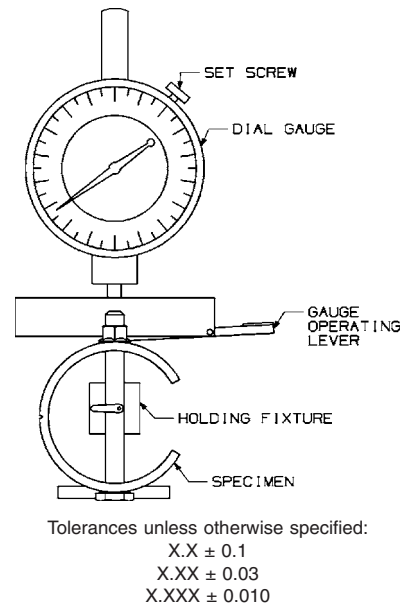
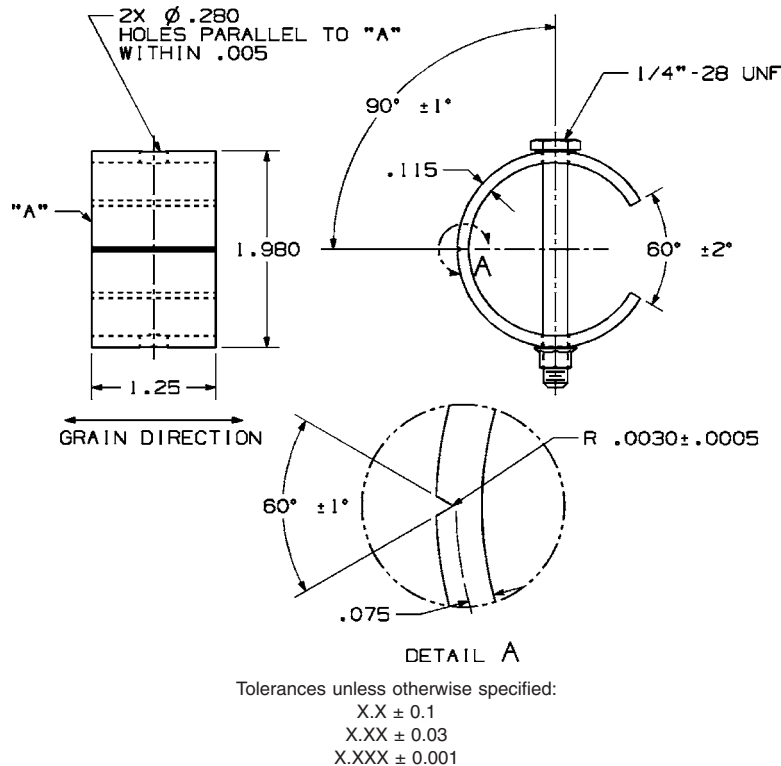


FIG. A2.9 Device for Measuring Deflection During Stressing of Type 1d Specimen



NOTE 1—Specimen preparation must comply with 9.2.

NOTE 2—Surface finish of notch shall be 32 RMS or better, all other surfaces shall be 63 RMS or better.

NOTE 3—See Annex A5 for galvanic isolation requirements when used for fluid testing.

FIG. A2.8 Dimensional Requirements for Type 1d Specimens with Loading Bolt

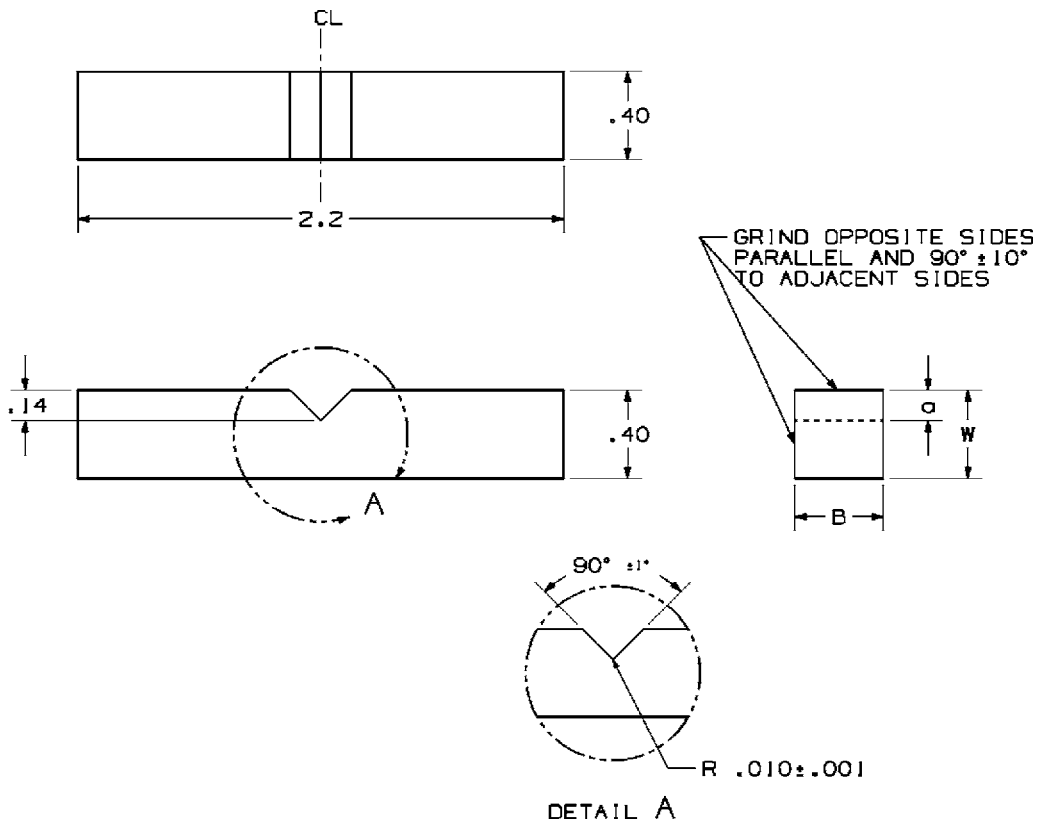
A3. SPECIAL REQUIREMENTS FOR THE TESTING OF SPECIMENS WITH THE ISL TEST METHOD

A3.1 *Specimens*—All Test Method F519 specimen types may be tested in accordance with the accelerated ISL testing protocol of Test Method F1624 in either tension or in bending. However, a specific ISL and holding time protocol in accordance with Test Method F1624 is prescribed only for the Type 1e, four-point bend test specimens manufactured from AISI 4340 steel heat treated to 52–54 HRC. Specific ISL and holding time protocols for the other specimen types, materials and heat treat conditions would have to be developed by following the methodology contained in Test Method F1624. The specimen Type 1e is scaled to match the same a/W (notched depth-to-width ratio) 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 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 square bar, which is rough machined slightly oversize to allow any decarburization from the heat treatment to be removed. One transverse section shall be microstructurally examined to insure that if any orientation effects exist, the



Tolerances unless otherwise specified:
 X.X ± 0.1
 X.XX ± 0.01
 X.XXX ± 0.001

- NOTE 1—Specimen preparation must comply with 9.2.
- NOTE 2—Notch must be in the LS orientation per Terminology E1823.
- NOTE 3—Surface finish of notch shall be 32 RMS or better, all other surfaces shall be 63 RMS or better.
- NOTE 4—Within the tolerances of the specimen, the thickness of the net section $W - a = 0.26$ shall be maintained.

FIG. A3.1 Dimensional Requirements for Type 1e Specimen

notch will be in the LS orientation per Terminology **E1823**. The specimen shall then be ground to final dimensions to eliminate decarburization of the sides.

A3.3 Loading Protocol:

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

A3.3.2 The recommended loading protocol for this test method consists of 15 steps of 5 % NFS per hour or (15/5/1) up to 75 % NFS and then steps at 5 % NFS for 2 h until rupture.

A3.3.3 For serial-loading operations using the ISL protocol, refer to Test Method **F1624**.

A3.4 Threshold:

A3.4.1 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 NFS for that specimen configuration and not as pass/fail as with the sustained load test, time-to-fracture criterion.

A3.5 Equivalence Between SLT and ISL:

A3.5.1 The equivalence between the SLT and the ISL test methods has not been established in accordance with Practice **E691**. However, it has been demonstrated that unplated specimens tested in air using the ISL method will fracture at 90 % NFS. In so far as this represents a nominally hydrogen free state, it is reasonably assumed that plated/coated specimens that sustain 90 % NFS for 2 h can be considered to be non-embrittled.

A3.6 Interpretation of Results:

A3.6.1 For test specimens that meet or exceed 90 % of their ISL NFS for 2 h or more using the (15/5/1 + 5/5/2) step protocol, the plating/coating process is considered to be non-embrittling.

A3.7 Tolerance of Parts:

A3.7.1 Since hydrogen tolerance varies with hardness, actual parts made of low-strength steel might have more tolerance for residual hydrogen because of plating/coating and might not need the same threshold as high-hardness steels. Therefore, adjustments in threshold requirements may be made.

A3.7.2 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 **F1624**.

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 non-marring material such as aluminum or rubber having a 60 Shore A Durometer hardness.

A4.2 Manufacture:

A4.2.1 The Type 2a specimens are machined to size from normalized and stress relieved tubing or bar stock.

A4.2.1.1 Specimens shall be deburred by hand, using silicon-carbide paper, before heat treatment.

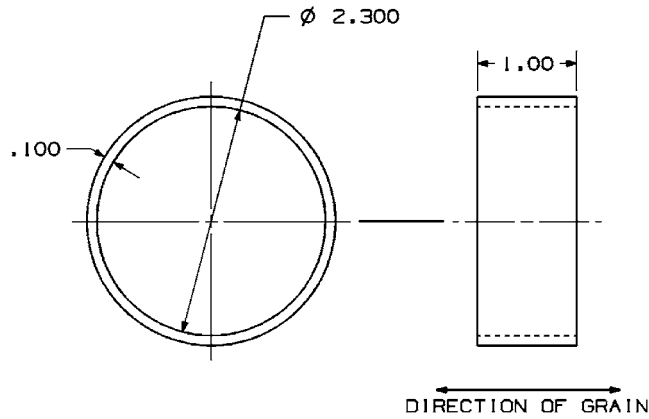
A4.2.1.2 Specimens shall be heat treated from 51 to 53 HRC. One specimen per lot shall be microstructurally examined to determine that no evidence of decarburization is present in accordance with AMS 2759/2 Class A.

A4.2.1.3 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-inch nozzle, 30 to 50-psi pressure, approximately 10 inches from part surface and moved continuously.

A4.2.1.4 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 inches in accordance with Specification **B851**.

A4.2.2 Grinding after final heat treatment is prohibited for Type 2a specimens.

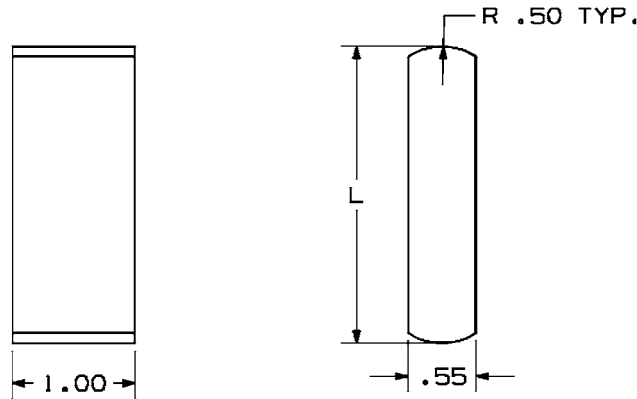
A4.3 Type 2a specimens shall be suitable for test purposes if each specimen is inspected and found to be in conformance with the requirements of **Table A4.1**.



Tolerances unless otherwise specified:
 X.X ± 0.1
 X.XX ± 0.03
 X.XXX ± 0.002

- NOTE 1—Specimen preparation must comply with 9.2.
- NOTE 2—Surface finish of 63RMS or better to be held before shot peening.
- NOTE 3—Ovality of ring must be within tolerance before and after heat treatment and shot peening.
- NOTE 4—Ovality of ring is determined by measuring the diameter of the ring at three locations oriented 60° apart.

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



Tolerances unless otherwise specified:
 X.X ± 0.1
 X.XX ± 0.03
 X.XXX ± 0.010

- 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/coating being evaluated for hydrogen embrittlement; but for sensitivity testing per Table 1, bars must be cadmium-electroplated with bright cadmium per Treatment A in accordance with AMS-QQ-P-416 (formerly QQ-P-416) before use.
- NOTE 2—Length “L” to be 2.415 ± 0.003 inches to load Type 2a specimens to 80 % of the yield strength.
- NOTE 3—Length “L” to be 2.525 ± 0.002 inches to load Type 2a specimens to 92 % of the ultimate strength.
- NOTE 4—See Annex A5 for galvanic isolation requirements when used for fluid testing.

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 (Test Methods E18)
Dimensions ^A	Conformance to Fig. A4.1
Surface quality ^A	No magnetic indications greater than 0.031 inches per Guide E709
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*—Use of this section pertains to both application-specific processes and evaluation of commercial-off-the-shelf (COTS) products. Suggested guidance is provided in this annex for evaluating specific applications already in use as well as COTS. However, thorough evaluation of specific applications typically requires additional testing utilizing relevant exposure parameters (pre-loading exposure, concentration, time, temperature, etc). These parameters vary with application and must be approved by the Cognizant Engineering Authority (CEA). Such testing shall be performed in accordance with any applicable testing specification. Any deviation from the parameters or test conditions herein shall be reported per 12.1.2 through 12.1.6.

A5.1.1 *Surface Finish*—Specimens shall be plated/coated or bare as specified by the procuring agency. Unless otherwise specified, the specimens shall be cadmium-plated and conversion-coated per Treatment B of this specification (or equivalent). The plating/coating process shall be determined to be non-embrittling per Section 11. Bare specimens shall also meet the surface finish requirements in 9.1.1 of this standard. The finish of the specimens shall be documented.

NOTE A5.1—Service environments found to be non-embrittling to plated/coated specimens may be highly embrittling to bare specimens and vice versa. Therefore, it is highly recommended that the service environment be tested for both surface finishes.

A5.1.2 *Number*—A minimum of four specimens shall be used per test.

A5.1.3 *Re-Use of Specimens*—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 either passive or aggressive.

A5.2.1.1 Passive chemicals typically do not result in any measurable corrosive attack on plated/coated or bare air melted AISI E4340 steel. Most cleaners and paint strippers are examples of passive chemicals.

A5.2.1.2 Aggressive chemicals have a measurable corrosive reaction with plated/coated or bare air melted AISI E4340 steel. Contact time of aggressive chemicals with plated/coated or bare AISI E4340 steel parts or coupons is intentionally restricted to limit detrimental reactions. Nitric acid used in temper etching is an example of an aggressive chemical.

A5.3 *Service Chemical Testing Procedure*:

A5.3.1 *Passive Chemicals*—Test specimens shall be immersed in passive chemicals for the full duration of the test (150 h) while stressed as specified in Table A5.1. After the test is completed, the test specimen and fixtures shall be rinsed and dried with compressed air.

A5.3.2 *Aggressive Chemicals*—The test specimen shall be immersed under a preload stress as specified in Table A5.1 and

TABLE A5.1 Load Requirements for Evaluating Passive Service Environments

Type 1a, 1b, 1c & 1e	45 % NFS.
Type 1d	65 % NFS.
Type 2a	80 % of 0.2 % yield strength per Test Methods E8/E8M, obtained by deflecting a 2.300-in. diameter O-Ring specimen with a 2.415-in. stressing bar.

shall be limited to 1.5 times the manufacturer’s maximum recommended exposure time for the application at the maximum recommended temperature for the application. (Exposure time may be a fraction of a minute). Time of exposure shall be recorded and reported. After the specified exposure under a preload, the test specimens and fixture shall be neutralized (per the manufacturer’s recommendations if no governing specification applies), rinsed and dried with compressed air. The load of the test specimen shall then be increased to the levels specified in Table 3 for a cumulative total of 200 h (including the preload exposure time).

A5.4 *Test Conditions*:

A5.4.1 *Environment/Containment*—The test shall be conducted in air or any other controlled environment using an appropriate container that is inert to the service environment.

A5.4.2 *Galvanic Isolation of Test Specimen*—Precaution shall be taken to ensure that the test specimen is suitably galvanically isolated from both the container and the test fixture or compensated to prevent galvanic coupling interactions.

NOTE A5.2—This is of particular concern when using self-loading type specimens (Types 1b, 1c, 1d and 2a). One example of acceptable practices for isolating these types of specimens from their self-loading frames can be found in Practice G38, paragraph 12.2, Fig. 5 and Notes 5 and 6. Another suitable practice is the use of an “isolation-cup” that restricts the test fluid from contacting any surface other than the specimen.

A5.4.3 *Concentration of Materials Supplied as Liquids*—Test materials supplied as liquids in the concentrated (as-received) condition and at the manufacturer’s minimum recommended concentration to determine the full range of embrittling effects of exposure. Dilute with Specification D1193 Type IV water unless otherwise specified.

A5.4.4 *Concentration of Materials Supplied as Solids*—Test materials supplied as solid in the saturated condition and at the manufacturer’s minimum recommended concentration to determine the full range of embrittling effects of exposure. Dissolve materials in Specification D1193 Type IV water unless otherwise specified.

A5.4.5 *Volume*—The exposure volume shall be 25 mL of solution per cm² of exposed surface. The containment system shall be covered to reduce evaporative losses. Fluid level shall be checked at least once per work day. Fluid level shall be

maintained using the diluent rather than the maintenance chemical unless otherwise specified (in the case of maintenance chemicals diluted with water the diluent shall be Specification **D1193** Type IV water.)

A5.4.6 Temperature—Conduct all tests at 68 to 86°F (20 to 30°C) or the operating service temperature of the maintenance chemicals. The temperature used shall be recorded.

NOTE A5.3—When elevated temperature testing is required for a specific application, the temperature shall be at least 50°F (27.8°C) below the lower of:

- Tempering temperature of the specimen material.
- Melting/sublimation point of any coating applied.

A5.5 Loading Protocol—Install the test specimens into the loading frame. Apply the loads specified in **Table A5.1**. Any containment chamber shall be isolated around the notched section of the test specimen. If the entire loading frame is immersed into the chemical, 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—See Section **11** in addition to the following:

A5.6.1 With no Fractures:

A5.6.1.1 Passive Chemicals—A passive chemical shall be considered non-embrittling if none of the specimens immersed

and tested per **A5.3.1** fracture within 150 h at the loads specified in **Table A5.1**. Record the time to failure if less than 150 h.

A5.6.1.2 Aggressive Chemicals—An aggressive chemical shall be considered non-embrittling if none of the specimens immersed and tested per **A5.3.2** fracture within 200 h after being stressed to 75 % NFS for Type 1 specimens, or 92 % of ultimate strength for Type 2a specimens (see **Table 3**). Record the time to failure if less than 200 h.

A5.6.2 With One or More Fractures:

A5.6.2.1 One of Four Fracture—If only one of a minimum of four specimens fractures within the exposure time (150 h for passive or 200 h for aggressive chemicals), the remaining three specimens may be step loaded, every 2 h in 5 % increments to 90 % NFS. If the three remaining specimens all sustain 90 % NFS for 2 h, the service environment shall be considered non-embrittling.

NOTE A5.4—Passive chemicals do not have to be in contact with the specimens during step loading.

A5.6.2.2 Two or More Fracture—If two or more specimens fracture within the exposure time, the service environment shall be considered embrittling.

A5.7 Replacement Testing—A5.7 Refer to Section **11**.

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