

Designation: F519 – 18

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 SAE 4340 steel (Grade A, see 7.1.1) per SAE AMS 6415 (formerly SAE AMS-S-5000 and formerly MIL-S-5000) or an alternative VAR (Vacuum Arc Remelt) SAE 4340 steel (Grade B, see 7.1.1) per SAE AMS 6414, and both are heat treated to 260 to 280 ksi (pounds per square inch ×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.

NOTE 1-Extensive testing has shown that VAR 4340 steel may be used

as an alternative to the air melted steel with no loss in sensitivity.²

Note 2—VAR 4340 also meets the requirements in AMS 6415 and could be used as an alternative to air melt steel by the steel suppliers because AMS 6415 does not specify a melting practice.

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.

1.5.1 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 (\leq 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.

¹This 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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² "Final Report - Design of Experiment Approach to Hydrogen Re-Embrittlement Evaluation WP-2152"; S.M Grendahl, H. Nguyen, F. Kellogg, S. Zhu, S. Jones; Strategic Environmental Research and Development Program (SERDP); Project WP-2152; July 2015; https://www.serdp-estcp.org/Program-Areas/Weapons-Systems-and-Platforms/Surface-Engineering-and-StructuralMaterials/WP-2152.

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TABLE 1 Lot Acceptance Criteria for Notched Specimens

Туре	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 ⁴	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.

1.9 This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

2. Referenced Documents

- 2.1 ASTM Standards:³
- **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
- 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:⁴
- 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 6414 Steel, Bars, Forgings, and Tubing (SAE 4340) Vacuum Consumable Electrode Remelted
- AMS 6415 Steel, Bars, Forgings, and Tubing (SAE 4340) AMS-QQ-P-416 Plating, Cadmium (Electrodeposited)

2.3 Military and Federal Standards and Commercial Item Descriptions:⁵

- 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:
- AISI = 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

³ 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.

⁴ Available from Society of Automotive Engineers (SAE), 400 Commonwealth Dr., Warrendale, PA 15096-0001, http://www.sae.org.

⁵ 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 ^{<i>B</i>} 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	23 h	
Baking time: Type 2a Specimen	8	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. ^BThe 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.

- 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 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.