New and Proposed Changes to ASTM F519 Hydrogen Embrittlement Test Method

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ASTM F07.04 Subcommittee on Hydrogen Embrittlement Testing - ASTM F 519 History

1977 – ASTM F 519 issued (10 pages)
1993 – 1st Revision (7 pages)
1997 – 2nd Revision (12 pages)
2004 – S. Gaydos (Boeing) appointed subcommittee chairman
2005 – 3rd Revision
2006 – 4th Revision
2008 – 5th Revision
2010 – 6th Revision
2012 – 7th Revision
2013 – 8th Revision
2017 – 9th Revision (19 pages)

• This Presentation Will Explain:
  • Significant Changes to ASTM F 519 Since 1997
  • Current Changes Being Discussed for F 519
Introduction


- Issued in 1977
  - Five test specimens available
    - 4 notched specimens: Type 1a (6 inch long bar), 1b, 1c, 1d
      - $K_t = 2.9$ to $3.3$
      - 1 smooth specimen: Type 2a
  - Test specimen material:
    - AISI 4340 steel per MIL-S-5000 heat treated to 260-280 ksi (51-54 HRC)
      - Mag Particle and Nital Etch inspection required on each test specimen
  - Plating test method:
    - 1a, 1b, 1c, 1d: 3 specimens sustained loaded (SL) to 75% NFS for 200 hrs
    - 2a: 3 specimens SL to 92% UTS for 200 hours
  - Maintenance Fluid test method (use Cd plated test specimens):
    - 1a, 1b, 1c, 1d: 3 specimens immersed in test fluid and SL to either 45% NFS (for 1a, 1c) or 55% NFS (for 1b) or 65% NFS (for 1d) for 150 hrs
    - 2a: 3 specimens immersed in test fluid and SL to 80% YS for 150 hrs
    - Test Fluid Volume – Not Specified
  - PASS – FAIL Criteria:
    - PASS = All 3 do not break within the 200 or 150 hour exposure time
    - FAIL = 2 of 3 break during the 150 or 200 hour exposure time
    - RETEST = 1 of 3 fail. Test 3 more. During retest – all 3 need to not break for a PASS
Hydrogen Embrittlement Testing in 1977

1a Sustained Load Test For Plating

1a Sustained Load Test For Maintenance Fluid

1d – SL Plating

1d – SL Maintenance Fluid
1st Revision to ASTM F 519
Issued in 1993 (changes shown in red)

- Five test specimens available
  - 4 notched specimens: Type 1a (6 inch long bar or 3 inch short bar), 1b, 1c, 1d
    - $K_t = 2.9$ to 3.3
    - 1 smooth specimen: Type 2a
- Test specimen material:
  - Same as for 1977 – No Change
- Plating test method:
  - Same as for 1977 – No Change
- Maintenance Fluid test method (use Cd plated test specimens):
  - 1a, 1b, 1c, 1d: 3 specimens immersed in test fluid and SL to either 45% NFS (for 1a, 1c) or 55% NFS (for 1b) or 65% NFS (for 1d) for 150 hrs
  - 2a: 3 specimens immersed in test fluid and SL to 80% YS for 150 hrs
  - Test Fluid Volume – Not Specified for 1a, 1b; 800 ml for 1c, 1d, 2a
- PASS – FAIL Criteria:
  - PASS = All 3 do not break within the 200 or 150 hour exposure time
  - FAIL = 2 of 3 break during the 150 or 200 hour exposure time
  - RETEST = 1 of 3 fail for 1b, 1c, 1d, 2a. Test 3 more specimens. During retest – all 3 need to not break for a PASS
  - RETEST OPTION = 1 of 3 fail for Type 1a. Perform incremental step load (ISL) on other two 1a specimens that did not fail and see if they reach 90% NFS
1) Determine NFS_E8 for Type 1a.1 bare test specimen.
2) Load plated specimen to 75% NFS.
3) Hold at 75% NFS for at least 200 hours.
4) Zn-Ni plating process is non-embrittling if plated specimen does not break before 200 hours.

200 Hour Sustained Load Test

Plating Process is Non-acceptable

Plating Process is Acceptable

ISL Test
5% NFS/Hour
From 75% NFS to 90% NFS

*Plated specimens that pass the ISL Test at 90% NFS are considered to be non-embrittled and equivalent to unplated bare test specimens.
Seven test specimens available
  - 6 notched specimens: Type 1a.2 (6 inch), 1a.1 (2.5 inch), 1b, 1c, 1d, 1e (square bar)
    - $K_t = 2.9$ to 3.3
  - 1 smooth specimen: Type 2a

Test specimen material:
  - Air melted AISI 4340 steel per MIL-S-5000* heat treated to 260-280 ksi (51-53 HRC)
  - Mag Particle and Nital Etch inspection not required on each test specimen

Plating test method (allowed to only plate the notch):
  - 1a.1, 1a.2, 1b, 1c, 1d: 4 specimens sustain loaded (SL) at 75% NFS for 200 hrs
  - 2a: 4 specimens SL to 92% UTS for 200 hours
  - ISL (incremental step load) for Type 1e and all Type 1s added as an alternative to SL if specified by the purchaser

Maintenance Fluid test method (use plated or bare test specimens):
  - 1a.1, 1a.2, 1b, 1c, 1d, 1e: 4 specimens immersed in test fluid and SL to either 45% NFS (for 1a.1, 1a.2, 1b, 1c) or 65% NFS (for 1d) for 150 hrs or do an ISL if specified by the purchaser
  - 2a: 4 specimens immersed in test fluid and SL to 80% YS for 150 hrs
  - Test Fluid Volume – Not Specified

PASS – FAIL Criteria:
  - PASS = All 4 do not break within the 200 or 150 hour exposure time
  - FAIL = 2 of 4 Type 1 or 1 of 4 Type 2a break during the 150 or 200 hour exposure time
  - RETEST = 1 of 4 Type 1 break. Either perform ISL on other 3 and see if they reach 90% NFS or test 4 more

*MIL-S-5000 cancelled in 1995 – use AMS 6415
Incremental Step Load vs. Sustained Load Test for Plating Process Control

1) Determine NFSE8 for Type 1a.1 bare test specimen.
2) Load plated specimen to 75% NFS.
3) Hold at 75% NFS for at least 200 hours.
4) Zn-Ni plating process is non-embrittling if plated specimen does not break before 200 hours.

** ISL = Incremental Step Load

ASTM F 1624-06 Test Method

200 Hr Sustained Load Test

20-30 Hr Incremental Step Load Test
ASTM F 519-17: Changes from 1997 to 2017

– Test Specimens

- Seven test specimens still available for use
  - 6 notched specimens: 1a.1 (2.5 to 3 inch threaded), Type 1a.2 (6 inch button head), 1b, 1c, 1d, 1e (square bar)
  - Drawings were updated and meet ASTM style guide requirements
  - Boeing determined Kt values for all Type 1 notched test specimens using FEA (Finite Element Analysis) software

<table>
<thead>
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<th>Type</th>
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<th>Nominal</th>
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<td>3.31</td>
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<td>3.30</td>
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<td>4.60</td>
</tr>
<tr>
<td>2a</td>
<td>NA</td>
<td>NA</td>
<td>NA</td>
</tr>
</tbody>
</table>

^ Minimum and maximum Kt values calculated from worst case combinations of dimensional tolerances.

- Boeing also determined by FEA that Kt for 60° and 90° notch would be equivalent using the same notch root radius for each Type 1 specimen
  - 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.
- Nital etch and magnetic particle inspection still not required
Comparison of Stresses

Specimen Type 1.a.1

Specimen Type 1.a.2
ASTM F 519-17: Changes from 1997 to 2017

– Test Specimen Material

▪ Grade A and B Steel

▪ Air melted SAE 4340 steel per AMS 6415 (superceded MIL-S-5000 and AMS-S-5000) heat treated to 260-280 ksi (51-53 HRC) still preferred material for hydrogen embrittlement test specimens
  – Designated as Grade A

▪ Vacuum Arc Remelted (VAR) SAE 4340 steel per AMS 6414 heat treated to 260-280 ksi (51-53 HRC) is an acceptable alternative for hydrogen embrittlement test specimens
  – Designated as Grade B

▪ If no Grade is specified by the requestor – then Grade A shall be used

Test conducted by SERDP project (see report WP-2152) showed that VAR 4340 can be used as an alternative to air melted 4340 with no loss in sensitivity
  – Extensive testing has shown that VAR 4340 steel may be used as an alternative to the air melted steel with no loss in sensitivity. *
  – VAR 4340 typically used for aircraft parts because it is a cleaner steel than air melt 4340
  – 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

▪ Other steels, or different hardness ranges, may only be substituted if agreed upon by the cognizant engineering authority.

SERDP Project Results – Steel Alloy Sensitivity for Type 1a.1

Air-melt 4340 - AMS-6415

Aerospace 4340 - AMS-6414
SERDP Project Results – Steel Alloy Sensitivity for Type 1a.2

Air-melt 4340 - AMS-6415

Aerospace 4340 - AMS-6414
ASTM F 519-17: Changes from 1997 to 2017

– Plating Test Method

▪ Guidelines for Plating/Masking Test Specimens:
  ▪ Plating Coverage
    – The entire specimen with the exception of the grip areas shall be covered by plating/coating as determined by a visual inspection.
  ▪ Masking of Grip Areas
    – The grip areas of the specimens may be masked to prevent plating/coating deposit when required to avoid interference with the test fixture.
  ▪ 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.
      – Type 1 Notched Specimens: The entire notch and 0.5 inch on either side of the notch shall be plated/coated.
      – Type 2a Smooth Specimen: Only the edges may be masked. The entire ID and OD shall be plated/coated.
ASTM F 519-17: Changes from 1997 to 2017

– Plating Test Method (Continued)

▪ Plating Test Loading Conditions:

  ▪ Sustained Load (SL) Test Specimens
    – 1a.1, 1a.2, 1b, 1c, 1d, 1e: 4 specimens SL at 75% NFS for 200 hrs
    – 2a: 4 specimens SL to 92% UTS for 200 hours

  ▪ Incremental Step Load (ISL) Test Specimens
    – ISL is an alternative to SL if approved by the cognizant engineering authority
    – 1a.1, 1a.2, 1b, 1c, 1d, 1e: 4 specimens ISL tested at 15/5/1* + 5/5/2** step protocol (21 hour test to reach 90% NFS)
    – Test specimens that meet or exceed 90% NFS threshold are considered to be non-embrittled

▪ Series (Gang) Loading – Samples, that have the same certified NFS, may be loaded on a test frame either individually or by multiples in series
  – 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.
    – ETUL (Extended Time Under Load) - If the total time-off-load (wall clock) exceeds 10 % of the total test duration (for example, 20 hours for a 200 hour 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 hours).

*15/5/1 = 15 steps at 5% NFS per step held for 1 hour per step
**5/5/2 = 5 steps at 5% NFS per step held for 2 hour per step
Series or Gang Loading of Type 1a.1 Test Specimens
ASTM F 519-17: Changes from 1997 to 2017

– Maintenance Fluid Test Method

▪ Maintenance Fluid Test Loading Conditions

▪ Passive Chemicals (deicers, cleaners and neutral paint strippers)
  – 1a.1, 1a.2, 1b, 1c, 1d, 1e: 4 specimens immersed in test fluid and SL to either 45% NFS (for 1a.1, 1a.2, 1b, 1c) or 65% NFS (for 1d) for 150 hrs
  – 2a: 4 specimens immersed in test fluid and SL to 80% YS for 150 hrs

▪ Aggressive Chemicals (temper etch solution and acid paint strippers)
  – Apply pre-load 45% NFS, 65% NFS or 80% YS (depending on specimen type) and hold for 1.5 times the recommended fluid exposure time for this chemical
  – Remove specimen from test fluid, rinse and dry and then load to 75% NFS or 92% UTS (depending on specimen type) and hold for 200 hours

▪ If approved by the cognizant engineering authority, ISL may be used as an alternative on Type 1 notched test specimens for maintenance fluid testing
ASTM F 519-17: Changes from 1997 to 2017

– Maintenance Fluid Test Method (Continued)

▪ Test Specimen Type
  ▪ If approved by the cognizant engineering authority, Type 1d test specimens loaded to 45% NFS may be used as an alternative to Type 1a.1, 1a.2, 1b, 1c, and 1e test specimens loaded to 45% NFS for passive chemical testing, or loaded to 45% NFS during the pre-load phase, for aggressive chemical testing.
    – Extensive testing has shown that Type 1d test specimens can be used as an alternative to the other type test specimens with no loss in sensitivity.*

▪ Test specimens shall be plated or bare
  ▪ If surface finish is not specified by the requestor – then specimens shall be LHE (Low Hydrogen Embrittlement) cadmium plated and conversion coated

▪ Test Fluid Volume – 25 ml (minimum) per cm\(^2\) of exposed surface

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SERDP Project Results – Test Specimen Sensitivity

Predicted Median Lifetime
Strength=T5 (280 KSI)

1d

Predicted Median Lifetime
Strength=T5 (280 KSI)

1a2

Predicted Median Lifetime
Strength=T5

1a1

Predicted Median Lifetime
Strength=T5 (280 KSI)

1e

Predicted Median Lifetime
Strength=T5 (280 KSI)

1c
ASTM F 519-17: Changes from 1997 to 2017

– PASS / FAIL Criteria

▪ PASS/FAIL Criteria for Plating and Maintenance Fluid Test Methods
  ▪ PASS = All 4 do not break within the 200 or 150 hour exposure time
  ▪ FAIL = 2 of 4 break during the 200 or 150 hour exposure time

▪ RESTEST OPTIONS
  – RETEST OPTION 1 = 1 of 4 Type 1 test specimens break. Perform ISL on other 3 and see if they reach 90% NFS
    – The remaining three specimens shall be step loaded every 2 hours in 5 % NFS increments to 90 % of the NFS after completion of the 200 hour sustained load.
    – If the three remaining specimens all sustain 90 % NFS for 2 hours, the plating process shall be considered non-embrittling.
  – RETEST OPTION 2 = 1 of 4 break. Test 4 more.
Proposed Changes to ASTM F 519 for 2018

SLF (Self Loading Frame)

- F07.04 Subcommittee ballot submitted to add the ability to test the Type 1a.1 specimen in a self-loading fixture (SLF) such as is used with the Type 1c specimen.
  - Negatives received and need to be resolved
- Ballot will be revised and resubmitted before November 2018 ASTM F07.04 subcommittee meeting

![SLF with a 1a.1 Test Specimen Installed](image)
Proposed Changes to ASTM F 519 for 2018 (Continued)

ISL (Incremental Step Load)
- F07.04 ISL Task Group Formed to Make ISL an Acceptable Alternative for SL (Sustained Load) Testing
- Proposed change to be balloted before November 2018 ASTM F07.04 subcommittee meeting

Was:
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.

To:
1.5.2 An accelerated incremental step load (ISL) test as defined in Annex A3 may be used as an alternative to SLT unless specifically disallowed by a cognizant engineering authority. (i.e. the authority must specifically state that ISL is not allowed as a substitute, or that the 200 hour SLT test is required).
Acknowledgements:
ASTM F07.04 Hydrogen Embrittlement Subcommittee Members and Subcommittee Secretary
Ed Babcock – Boeing-Mesa

Next ASTM F07.04 Subcommittee Meetings:
November 7 and 8, 2018 – Washington, D.C.
Washington Hilton
November 7 – F07.04 Subcommittee Meeting
November 8 – 14th Annual Hydrogen Embrittlement Workshop
For more information – go to:
www.astm.org