Proposed test method to establish hydrogen compatibility of materials for fuel cell vehicles

GTR no. 13 Phase 2 IWG
February 5-7, 2018
Torrance, CA

Prepared by: Chris San Marchi, Sandia National Laboratories
In collaboration with SAE Fuel Cell Safety Task Force
Motivation: establish materials compatibility for high-pressure hydrogen service in context of hydrogen fuel cell electric vehicles

Goals of presentation:

• Briefly summarize activity within SAE Fuel Cell Safety Task Force
  • SAE H2 Compatibility Expert Team
  • Collaborative testing and test criteria development
• Present test criteria developed for SAE J2579
  • Brief justification of requirements for materials compatibility
SAE Fuel Cell Safety Task Force

- Meets quarterly with broad representation from automotive OEMs
- Tasked with developing several standards for fuel cell vehicles in context of safety
  - J2579 - *Standard for Fuel Systems in Fuel Cell and Other Hydrogen Vehicles*
    - Includes requirements for materials in contact with high-pressure gaseous hydrogen
  - SAE H2 Compatibility Expert Team
    - Formed to develop requirements for hydrogen compatibility of materials
    - Includes hydrogen compatibility experts identified by Task Force representation
SAE H2 Compatibility Expert Team

• Representation from nationally funded research programs funded to enable deployment of fuel technologies
  - Germany: MPA Stuttgart
  - Japan: Kyushu University and AIST
  - US: Sandia National Laboratories
• Collective learning through so-called “round robin” testing campaign
  - Development of capabilities and examination of procedures to execute fatigue tests in high-pressure hydrogen at low temperature
  - Demonstrate test methodologies at MPA, KU and SNL
Collective learning activity ("round robin")

<table>
<thead>
<tr>
<th>Test</th>
<th>Test conditions</th>
<th>Environment</th>
<th>Number of tests</th>
</tr>
</thead>
<tbody>
<tr>
<td>Slow strain rate tension (SSRT)</td>
<td>( \leq 5 \times 10^{-5} \text{ s}^{-1} )</td>
<td>Control -40°C</td>
<td>3</td>
</tr>
<tr>
<td></td>
<td></td>
<td>90 MPa H2 -40°C</td>
<td>3</td>
</tr>
<tr>
<td>Notched tension-tension fatigue</td>
<td>( S_a = 200 \text{ MPa} ) ( R = 0.1 ) 1 Hz</td>
<td>Control -40°C</td>
<td>3</td>
</tr>
<tr>
<td></td>
<td></td>
<td>90 MPa H2 -40°C</td>
<td>3</td>
</tr>
<tr>
<td>Smooth tension-compression fatigue</td>
<td>( S_a = 320 \text{ MPa} ) ( R = -1 ) 1 Hz</td>
<td>Control -40°C</td>
<td>3</td>
</tr>
<tr>
<td></td>
<td></td>
<td>90 MPa H2 -40°C</td>
<td>3</td>
</tr>
</tbody>
</table>
Test criteria for hydrogen compatibility of materials

SAE J2579, Appendix B.3 is essentially a set of generic test criteria for evaluation of structural metals for service in high-pressure gaseous hydrogen

- Part 1: Definition of materials and environmental test conditions
- Part 2: SSRT
- Part 3: Fatigue life test
- Part 4: Welds

In general, CSA CHMC1 is referenced for the test methods (CHMC1 references ASTM standards)
Part 1: Definition of materials and environmental test conditions

• Material must be defined by and satisfy requirements for
  - Composition
  - Tensile properties: specified minimum Sy, Su, El

• Environmental test conditions
  - Pressure $\geq 1.25$ NWP (nominal working pressure)
  - Test temperature: 228 K (for most materials)
  - Measured gas purity according to CSA CHMC1
    • 2 ppm O$_2$, 10 ppm H$_2$O
**Part 1: Definition of materials and environmental test conditions: test temperature**

Table B.3.1.4 from SAE J2579

<table>
<thead>
<tr>
<th>Alloy type</th>
<th>Test method</th>
<th>Test temperature (K)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Austenitic stainless steel</td>
<td>SSRT</td>
<td>228 ±5</td>
</tr>
<tr>
<td></td>
<td>Fatigue life</td>
<td>228 ±5 and 293 ±5</td>
</tr>
<tr>
<td>Nickel-based alloys</td>
<td>SSRT and Fatigue life</td>
<td>228 ±5</td>
</tr>
<tr>
<td>Aluminum, magnesium and copper alloys</td>
<td>SSRT and Fatigue life</td>
<td>293 ±5</td>
</tr>
<tr>
<td>Other alloys</td>
<td>SSRT and Fatigue life</td>
<td>228 ±5 and 293 ±5</td>
</tr>
</tbody>
</table>
Part 2: Slow strain rate tension test

- Basic tensile test at slow strain rate in the defined hydrogen environment
- Minimum of three (3) tests
- Average property values must be greater than the specified minimum $S_Y$ and $S_u$ values respectively
- Average elongation ($E_l$) > 12%
- Additionally, $S_u/S_Y > 1.07$
Part 3: Fatigue life test

- Force-controlled (axially loaded cylindrical) fatigue test in the defined hydrogen environment
  - Frequency of 1 Hz
  - Maximum stress shall be 1/3 of measured Su (air)
- Minimum of three (3) tests
- Two test configuration options
  - Option 1: smooth test specimen with $R = -1$, or
  - Option 2: notched test specimen with $R = 0.1$
- Cycles to failure $>200,000$ cycles for each test
  - Alternatively, cycles to failure $>100,000$ cycles for each of 5 notched test specimens
Part 3: Fatigue life test: stress cycle

- $S_{\text{max}}$
- Applied stress
- $-S_{\text{max}}$

- $R = -1$ (smooth)
- $R = 0.1$ (notched)

notched tension
smooth tension
compression
Part 4: Welds

• Prepare representative welded structures
• Same testing requirements as for non-welded materials
  - Specified minimum tensile properties must be defined
  - Weld material must satisfy the minimum specified properties
  - Average values from SSRT tests of weld-material must satisfy minimum specified strength properties, $E_I > 12\%$ and $S_u/S_y > 1.07$
  - Fatigue life must be $>200,000$ cycles for each of three (3) smooth or notched fatigue tests; or $>100,000$ cycles for each of (5) notched fatigue tests
### Summary of requirements for compatibility

<table>
<thead>
<tr>
<th>Test configuration</th>
<th>Evaluation parameter</th>
<th>Requirements of tests performed in H2</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Slow strain rate tension tests – SSRT (3 tests)</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Yield strength</td>
<td>Average ≥ Sy</td>
</tr>
<tr>
<td></td>
<td>Tensile strength</td>
<td>Average ≥ Su</td>
</tr>
<tr>
<td></td>
<td>Strain hardening capacity</td>
<td>Average &gt; 1.07</td>
</tr>
<tr>
<td></td>
<td>Elongation</td>
<td>Average ≥ 12%</td>
</tr>
<tr>
<td><strong>Fatigue life tests</strong> (must satisfy 1 of 3 options)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Option 1 (3 tests): Smooth, R = -1</td>
<td>Cycles to failure</td>
<td>Each &gt; 200,000 cycles</td>
</tr>
<tr>
<td>Option 2 (3 tests): Notched, R = 0.1</td>
<td>Cycles to failure</td>
<td>Each &gt; 200,000 cycles</td>
</tr>
<tr>
<td>Option 3 (5 tests): Notched, R = 0.1</td>
<td>Cycles to failure</td>
<td>Each &gt; 100,000 cycles</td>
</tr>
</tbody>
</table>

Note: Sy and Su are specified minimum yield and tensile strength respectively.
Tensile properties are degraded in gaseous hydrogen especially at low temperature.

**Requirement:**
- Minimum specified strength properties are maintained
- Ductility is consistent with pressure applications

**Rationale:**
- Known and ductile tensile response

Tensile strength properties are not degraded in gaseous hydrogen for acceptable materials

• Common stress limitations for fatigue design: minimum of 2/3 $S_y$ and 1/3 $S_u$

• Yield and tensile strengths are typically not affected by hydrogen

• Maximum stress during fatigue testing (J2579) always greater than 1/3 $S_u$
Fatigue life of smooth specimens is typically infinite at stress of $1/3 S_{\text{meas}}$

**Requirement:**
- $N_f > 200,000$ cycles at $S_{\text{max}} = 1/3 S_{\text{meas}}$

**Rationale:**
- Ensure fatigue life at high stress is $>>$ than design life

Data from: M. Nakamura et al., M&M2017 conference, 7-9 October 2017, Hokkaido, Japan
Notched specimens assess sensitivity to stress concentration for typical maximum stress (1/3\(S_u\))

103 MPa H2
Notched specimens
\(R = 0.1\)

Requirement:
• \(N_f > 100,000\) cycles at \(S_{\text{max}} = 1/3\ S_u\)_{\text{meas}}

Rationale:
• Ensure fatigue life at high stress is \(>>\) than design life

Data from: C. San Marchi et al., 43rd MPA Seminar, 11-12 October 2017, Stuttgart, Germany
Diverse range of austenitic stainless steels have been evaluated, including high-strength alloys.

<table>
<thead>
<tr>
<th>material</th>
<th>Yield (MPa)</th>
<th>Tensile (MPa)</th>
<th>Cr</th>
<th>Ni</th>
<th>Mn</th>
<th>N</th>
<th>Typical allowable stress (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>316L</td>
<td>280</td>
<td>562</td>
<td>17.5</td>
<td>12</td>
<td>1.2</td>
<td>0.04</td>
<td>115</td>
</tr>
<tr>
<td>CW 316L</td>
<td>573</td>
<td>731</td>
<td>17.5</td>
<td>12</td>
<td>1.2</td>
<td>0.04</td>
<td>218</td>
</tr>
<tr>
<td>304L</td>
<td>497</td>
<td>721</td>
<td>18.3</td>
<td>8.2</td>
<td>1.8</td>
<td>0.56</td>
<td>195</td>
</tr>
<tr>
<td>XM-11</td>
<td>539</td>
<td>881</td>
<td>20.4</td>
<td>6.2</td>
<td>9.6</td>
<td>0.26</td>
<td>207</td>
</tr>
<tr>
<td>Nitronic 60</td>
<td>880</td>
<td>1018</td>
<td>16.6</td>
<td>8.3</td>
<td>8.0</td>
<td>0.16</td>
<td>218</td>
</tr>
<tr>
<td>SCF-260</td>
<td>1083</td>
<td>1175</td>
<td>19.1</td>
<td>3.3</td>
<td>17.4</td>
<td>0.64</td>
<td>333</td>
</tr>
</tbody>
</table>

Wide range of strength

Wide range of Ni/Mn content
High-strength materials can be evaluated by method and enable higher stress designs

- $1/3 \text{Su}$ of high-strength materials can be more than specified minimum yield strength of annealed material

- Implicitly, increase of design stress enables lower weight and lower cost designs without compromising performance
  - Justified by fatigue performance
Open questions

- **Temperature for fatigue life testing**
  - Most data suggest that austenitic stainless steels show longer fatigue life at low temperature
  - Change temperature of fatigue test to room temperature only?

- **Welding**
  - Additional requirements?

- **Additional testing requirements for aluminum alloys**
  - Stress corrosion cracking (SCC) threshold
  - Test method and evaluation criteria for SCC being formulated by High-Pressure Institute of Japan HPIS E 103:2018
    - Method seems equivalent to ISO 7539-6
    - Criteria should be incorporated in SAE J2579

- **How to incorporate “new” materials into SAE J2579**
  - Replace table B.2 and periodically update with tested materials?
Timeline

- **2018**
  - Present baseline test criteria to GTR IWG

- **2019**
  - Draft of SCC test for aluminum
  - Verification of fatigue test temperature

- **2020**
  - Resolution on Table B.2
  - Publish SAE J2579 revision 4
  - Verification of aluminum testing criteria
Summary

• Materials compatibility test method in SAE J2579 provides performance-based metrics to evaluate materials for hydrogen service
  – J2579 Appendix B.3 requirements for materials do not purport to generate design data
  – Method consists for 4 parts
    • 1: Materials definition
    • 2: Slow strain rate tensile testing (3 tests)
    • 3: Fatigue life testing (3-5 tests)
    • 4: Evaluation of welds (if welded)
  – Tensile testing (SSRT) in H2 demonstrates that materials satisfy the specified minimum properties consistent with pressure application
  – Fatigue life testing in H2 demonstrates that materials have fatigue performance consistent with baseline materials
Fatigue life at low temperature appears to be greater than at room temperature

\[ R = 0.1, f = 1 \text{Hz} \]

- Pressure has modest effect, if any, on fatigue life
- Temperature has either no effect or increases fatigue life
- Nitronic 60 is an exception for both pressure and temperature