

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

**GTR no. 13 Phase 2 IWG**

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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")

Test	Test conditions	Environment	Number of tests
<b>Slow strain rate tension (SSRT)</b>	$\leq 5 \times 10^{-5} \text{ s}^{-1}$	<b>Control</b> $-40^\circ\text{C}$	3
		<b>90 MPa H2</b> $-40^\circ\text{C}$	3
<b>Notched tension-tension fatigue</b>	$\text{Sa} = 200 \text{ MPa}$ $R = 0.1$ 1 Hz	<b>Control</b> $-40^\circ\text{C}$	3
		<b>90 MPa H2</b> $-40^\circ\text{C}$	3
<b>Smooth tension-compression fatigue</b>	$\text{Sa} = 320 \text{ MPa}$ $R = -1$ 1 Hz	<b>Control</b> $-40^\circ\text{C}$	3
		<b>90 MPa H2</b> $-40^\circ\text{C}$	3

# 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  $S_y$ ,  $S_u$ ,  $E_l$**
- **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  $H_2O$**

# Part 1: Definition of materials and environmental test conditions: test temperature

Table B.3.1.4 from SAE J2579

Alloy type	Test method	Test temperature (K)
<b>Austenitic stainless steel</b>	SSRT	<b>228 ±5</b>
	Fatigue life	<b>228 ±5 and 293 ±5</b>
<b>Nickel-based alloys</b>	<b>SSRT and Fatigue life</b>	<b>228 ±5</b>
<b>Aluminum, magnesium and copper alloys</b>	<b>SSRT and Fatigue life</b>	<b>293 ±5</b>
<b>Other alloys</b>	<b>SSRT and Fatigue life</b>	<b>228 ±5 and 293 ±5</b>

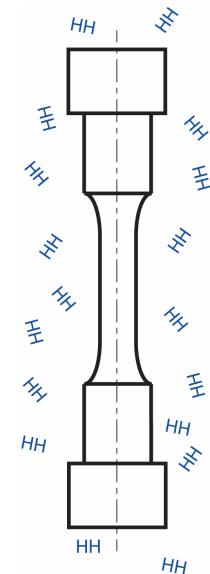
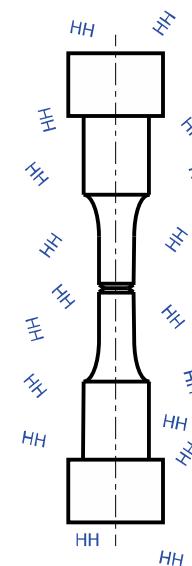
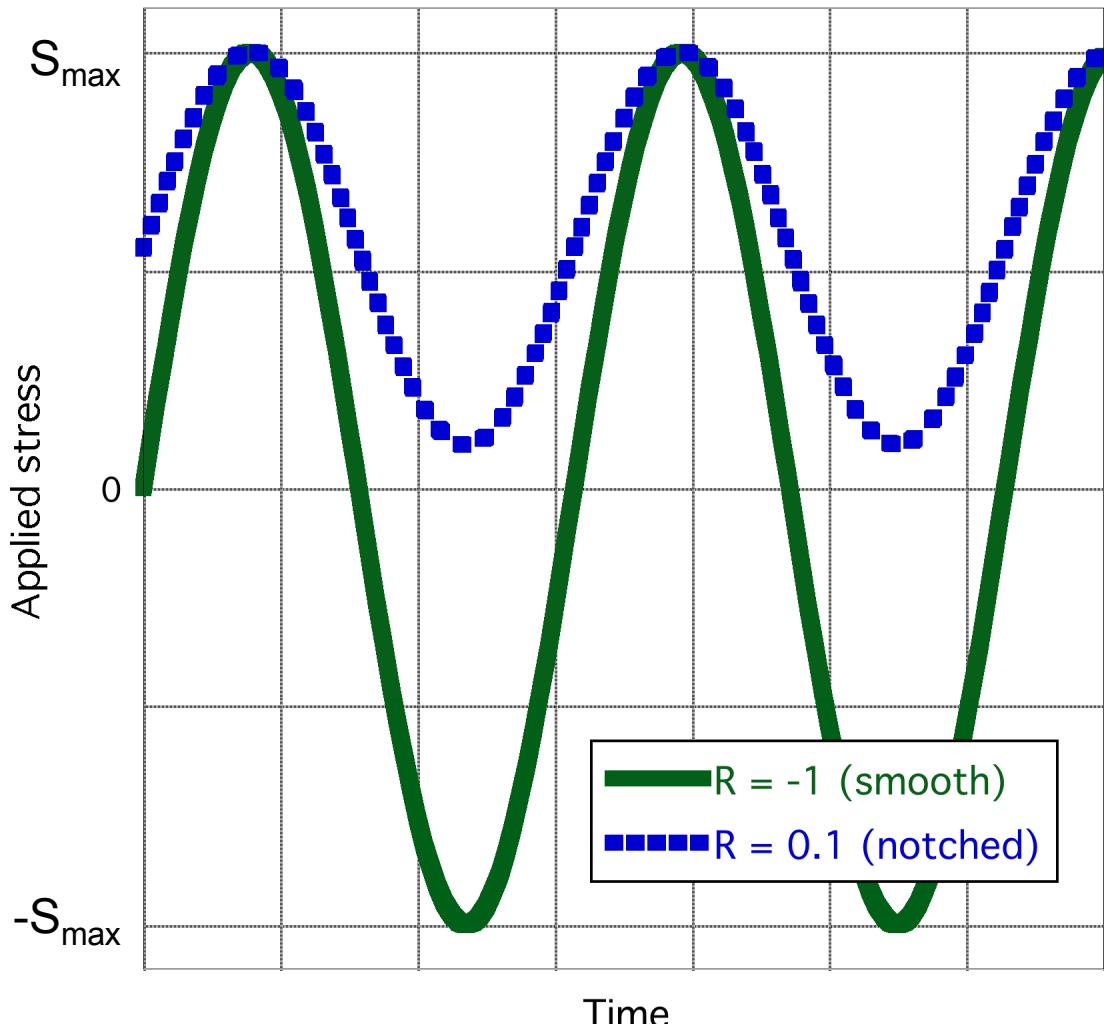
## Part 2: Slow strain rate tension test

- **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 (EI)  $> 12\%$**
- **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  $S_u$  (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



**notched  
tension  
tension**

**smooth  
tension  
compression**

## Part 4: Welds

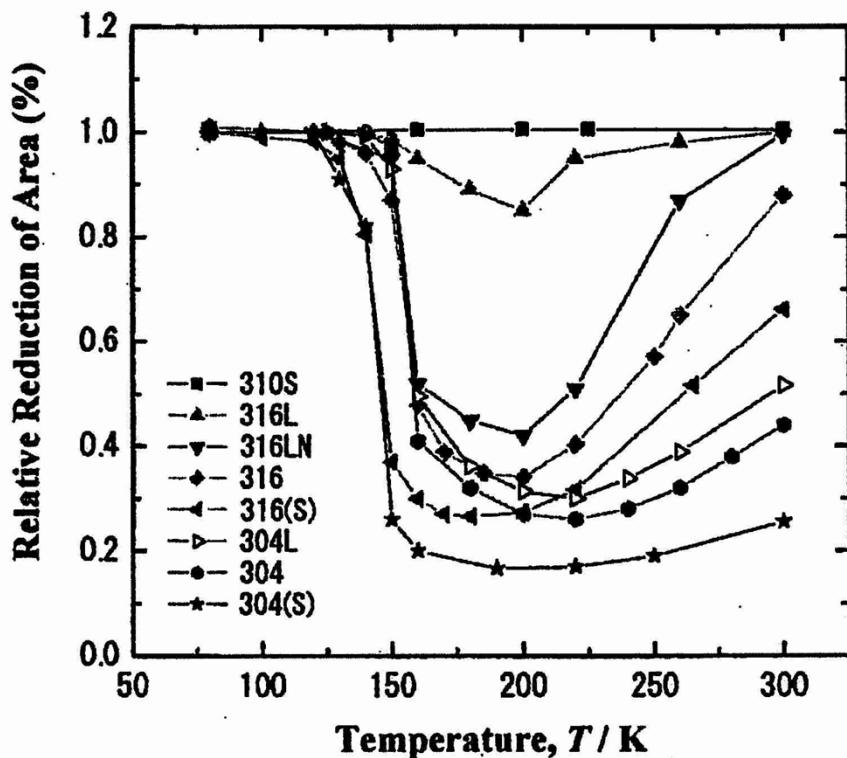
- Prepare representative welded structures
- Same testing requirements as for non-welded materials
  - Specified minimum tensile properties must be defined
  - Weld material must satisfy the minimum specified properties
  - Average values from SSRT tests of weld-material must satisfy minimum specified strength properties,  $EI > 12\%$  and  $Su/Sy > 1.07$
  - 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

Test configuration	Evaluation parameter	Requirements of tests performed in H2	
<b>Slow strain rate tension tests – SSRT (3 tests)</b>	<b>Yield strength</b>	<b>Average <math>\geq</math> <math>S_y</math></b>	
	<b>Tensile strength</b>	<b>Average <math>\geq</math> <math>S_u</math></b>	
	<b>Strain hardening capacity</b>	<b>Average <math>&gt; 1.07</math></b>	
	<b>Elongation</b>	<b>Average <math>\geq 12\%</math></b>	
<b>Fatigue life tests</b> (must satisfy 1 of 3 options)	Option 1 (3 tests): <b>Smooth, <math>R = -1</math></b>	<b>Cycles to failure</b>	<b>Each <math>&gt; 200,000</math> cycles</b>
	Option 2 (3 tests): <b>Notched, <math>R = 0.1</math></b>	<b>Cycles to failure</b>	<b>Each <math>&gt; 200,000</math> cycles</b>
	Option 3 (5 tests): <b>Notched, <math>R = 0.1</math></b>	<b>Cycles to failure</b>	<b>Each <math>&gt; 100,000</math> cycles</b>

Note:  $S_y$  and  $S_u$  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

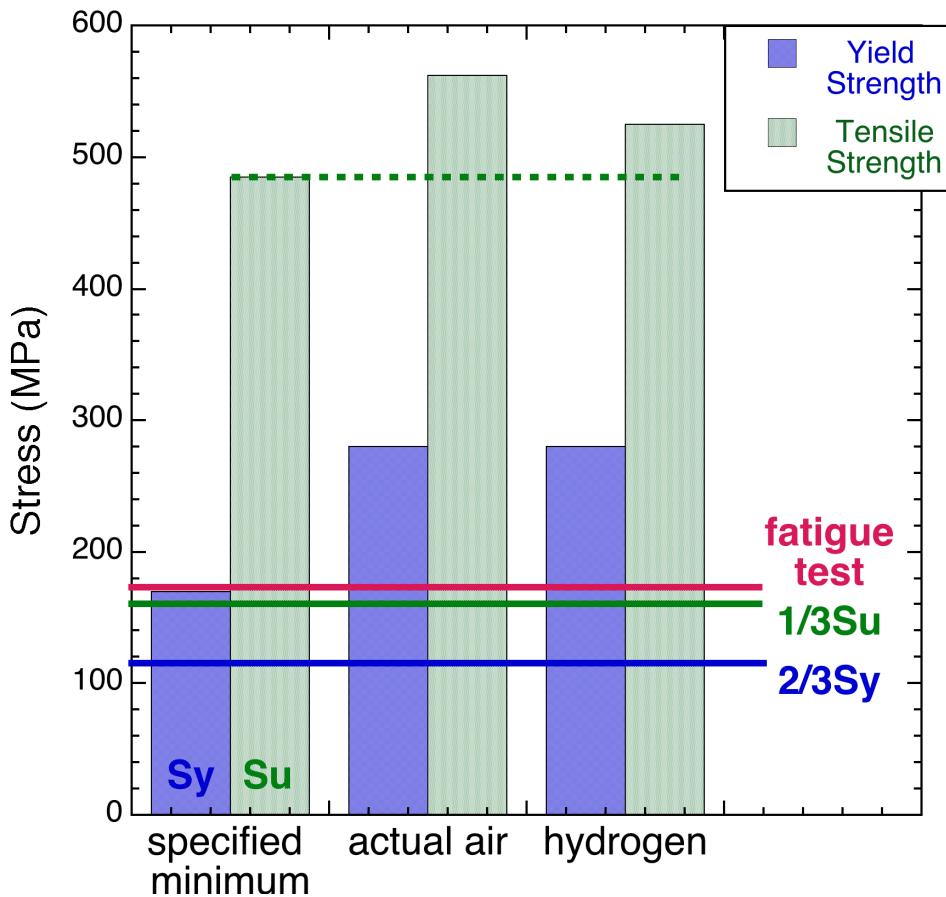
## Rationale:

- Known and ductile tensile response

Data from: Fukuyama et al., *J Japan Inst Metals* 67 (2003) 456-459.

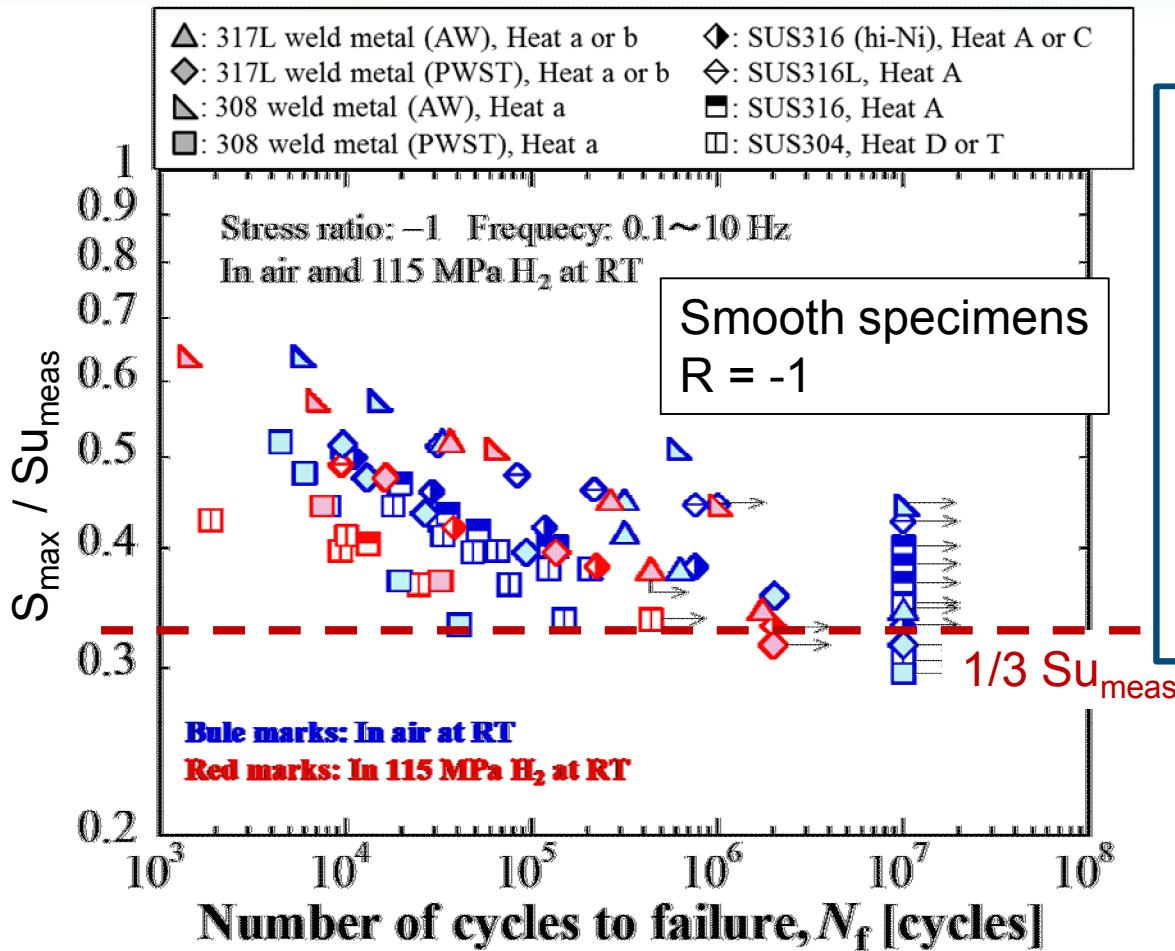
# Tensile strength properties are not degraded in gaseous hydrogen for acceptable materials

## Annealed austenitic stainless steel



- 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 $S_{u\text{meas}}$



## Requirement:

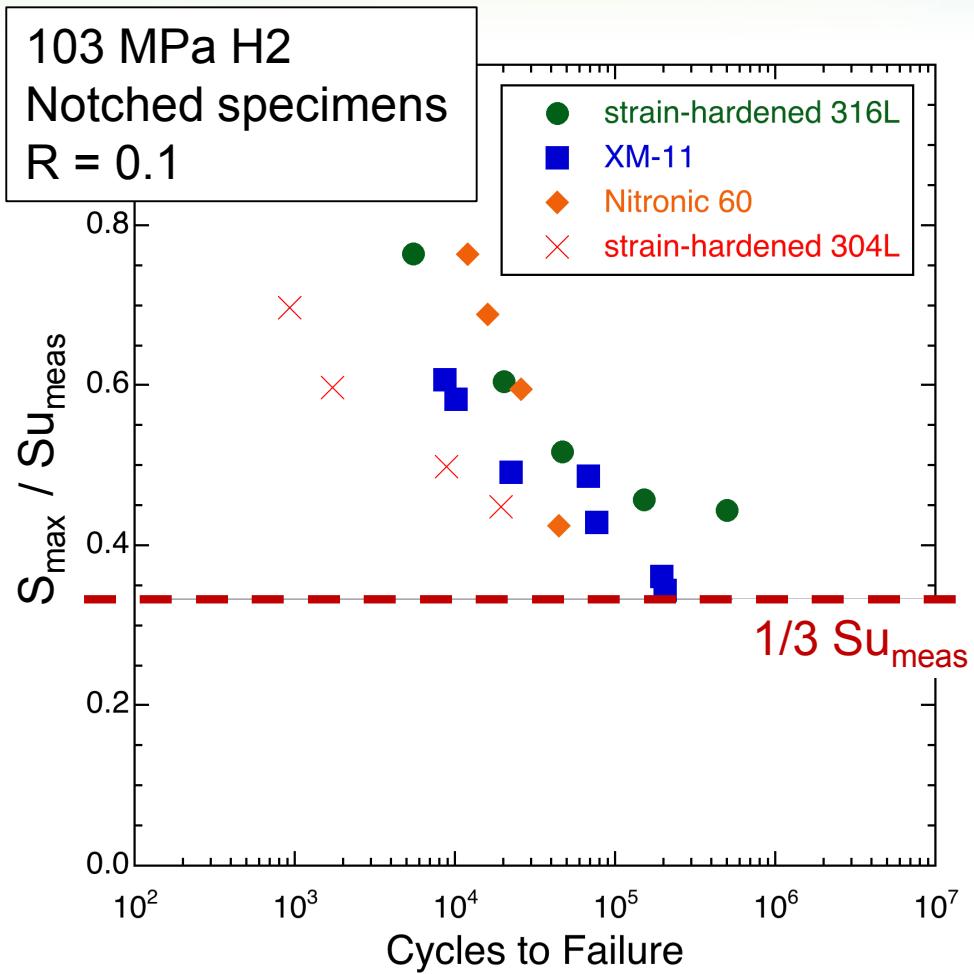
- $N_f > 200,000$  cycles at  $S_{\max} = 1/3 S_{u\text{meas}}$

## Rationale:

- Ensure fatigue life at high stress is  $\gg$  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 $S_u$ )



## Requirement:

- $N_f > 100,000$  cycles  
at  $S_{\max} = 1/3 S_{u\text{meas}}$

## Rationale:

- Ensure fatigue life at high stress is  $\gg$  than design life

Data from: C. San Marchi et al.,  
43<sup>rd</sup> MPA Seminar, 11-12 October  
2017, Stuttgart, Germany

# Diverse range of austenitic stainless steels have been evaluated, including high-strength alloys

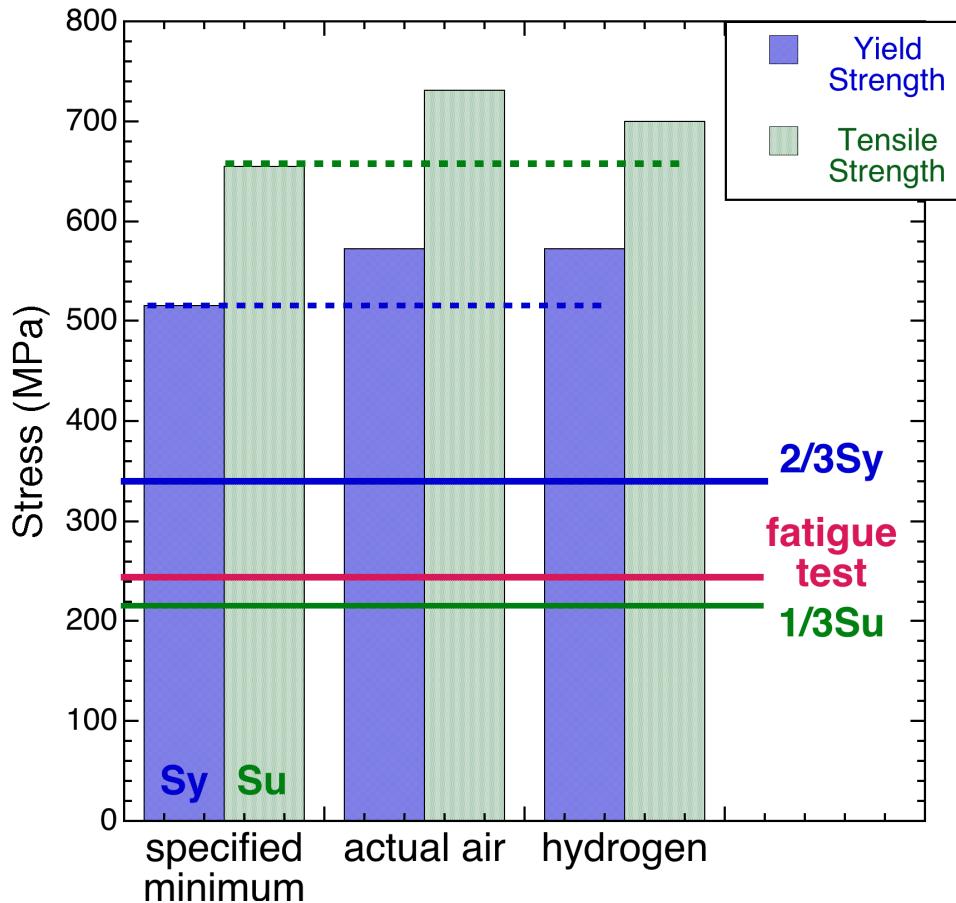
material	Yield (MPa)	Tensile (MPa)	Cr	Ni	Mn	N	Typical allowable stress (MPa)
316L	280	562	17.5	12	1.2	0.04	115
CW 316L	573	731	17.5	12	1.2	0.04	218
304L	497	721	18.3	8.2	1.8	0.56	195
XM-11	539	881	20.4	6.2	9.6	0.26	207
Nitronic 60	880	1018	16.6	8.3	8.0	0.16	218
SCF-260	1083	1175	19.1	3.3	17.4	0.64	333

*Wide range of strength*

*Wide range of Ni/Mn content*

# High-strength materials can be evaluated by method and enable higher stress designs

## Strain-hardened austenitic stainless steel

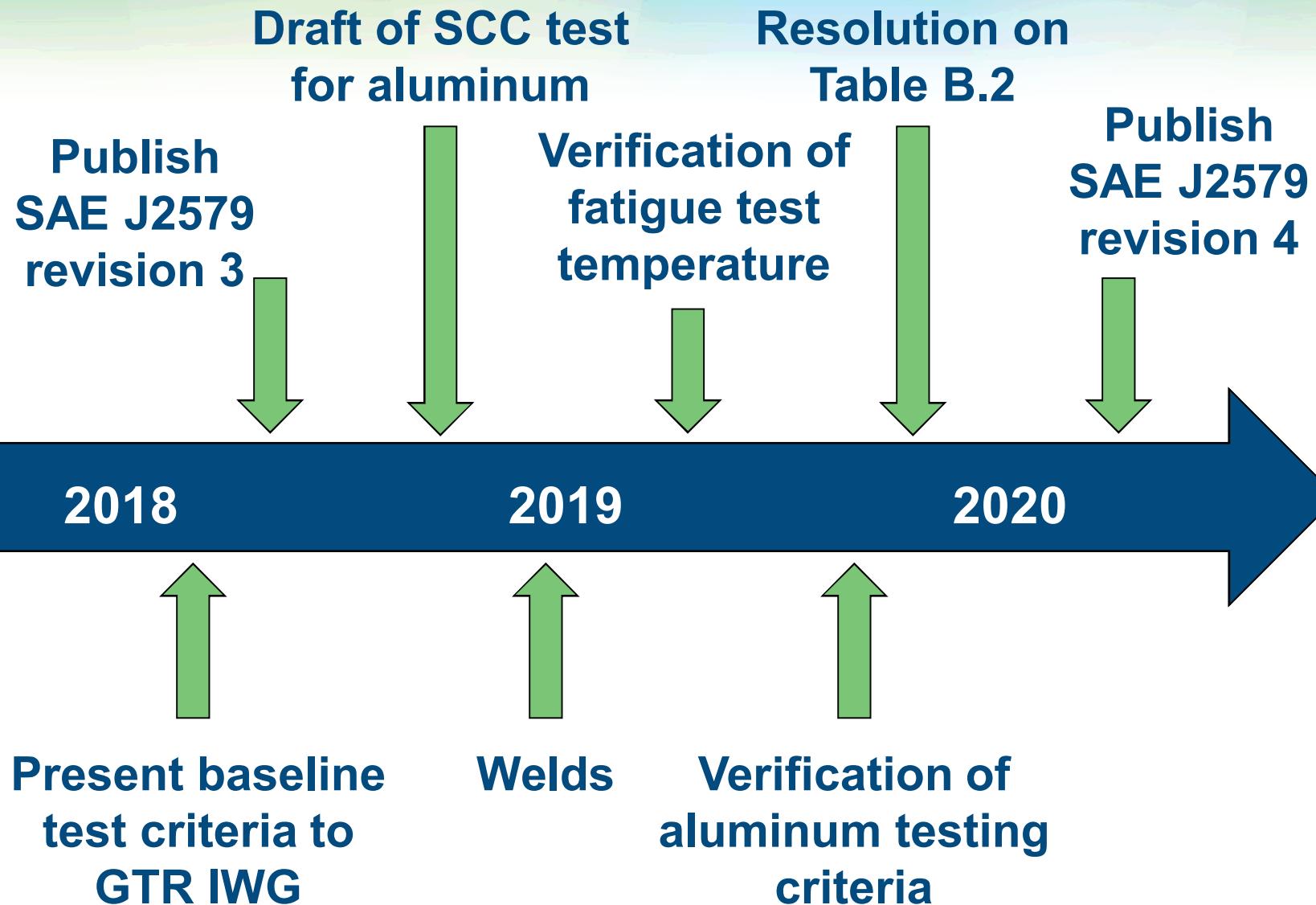


- **1/3  $S_u$  of high-strength materials can be more than specified minimum yield strength of annealed material**
- **Implicitly, increase of design stress enables lower weight and lower cost designs without compromising performance**
  - Justified by fatigue performance

# 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



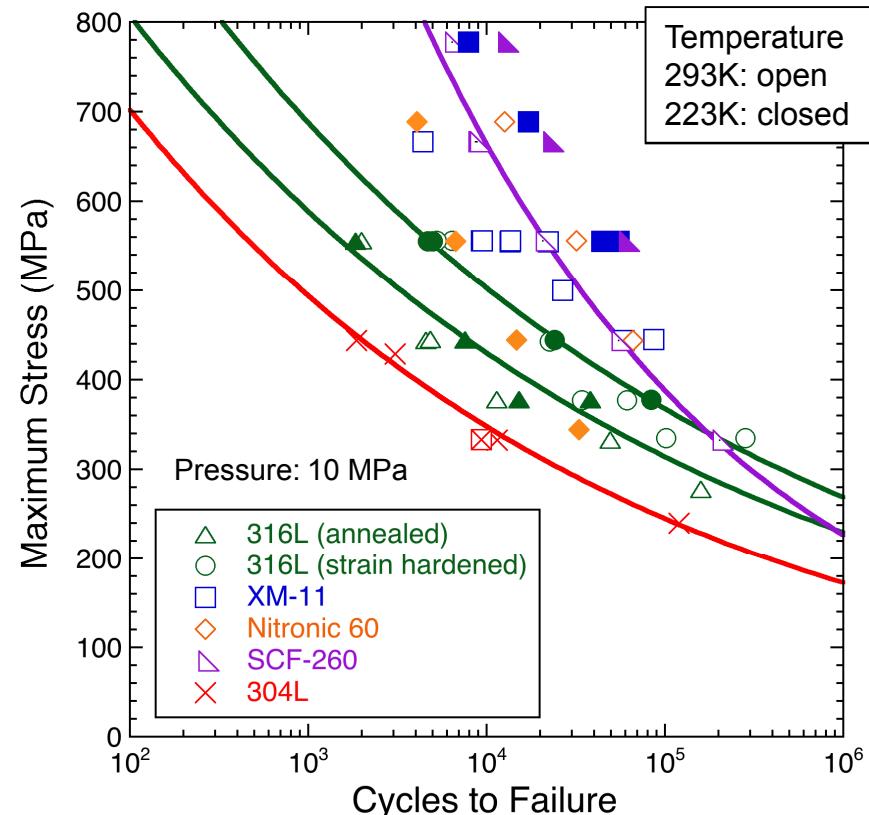
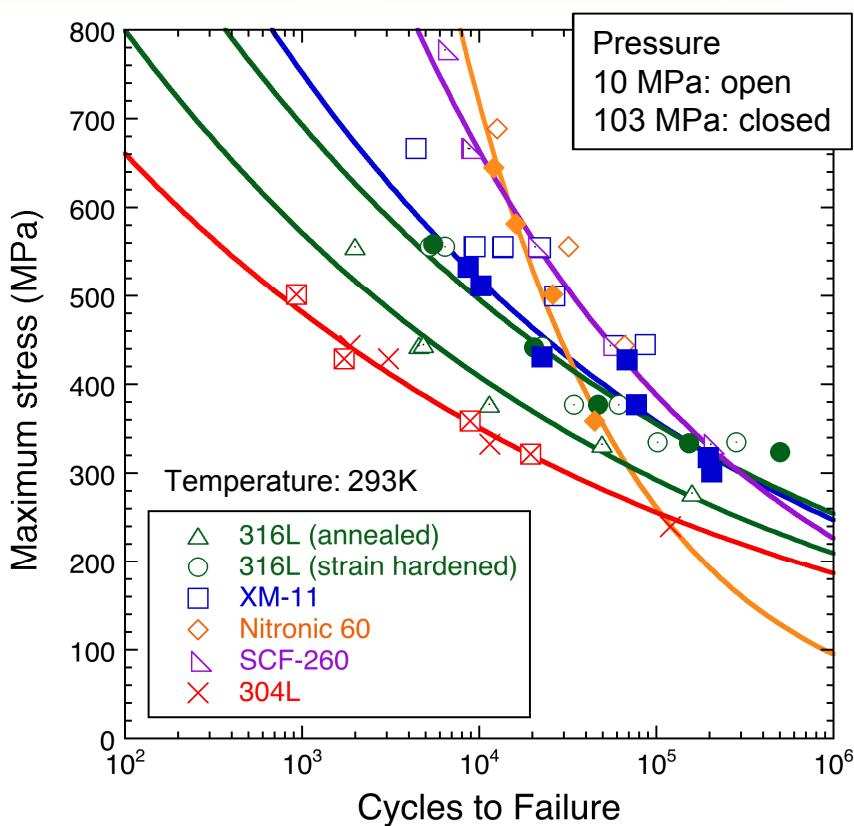
# Summary

- **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 H<sub>2</sub> demonstrates that materials satisfy the specified minimum properties consistent with pressure application
  - Fatigue life testing in H<sub>2</sub> demonstrates that materials have fatigue performance consistent with baseline materials

# Backup slides

# 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