

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
Slow strain rate tension (SSRT)	$\leq 5 \times 10^{-5} \text{ s}^{-1}$	Control -40°C	3
		90 MPa H2 -40°C	3
Notched tension-tension fatigue	Sa = 200 MPa R = 0.1 1 Hz	Control -40°C	3
		90 MPa H2 -40°C	3
Smooth tension-compression fatigue	Sa = 320 MPa R = -1 1 Hz	Control -40°C	3
		90 MPa H2 -40°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 , EI**
- **Environmental test conditions**
 - **Pressure ≥ 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)
Austenitic stainless steel	SSRT	228 ± 5
	Fatigue life	228 ± 5 and 293 ± 5
Nickel-based alloys	SSRT and Fatigue life	228 ± 5
Aluminum, magnesium and copper alloys	SSRT and Fatigue life	293 ± 5
Other alloys	SSRT and Fatigue life	228 ± 5 and 293 ± 5

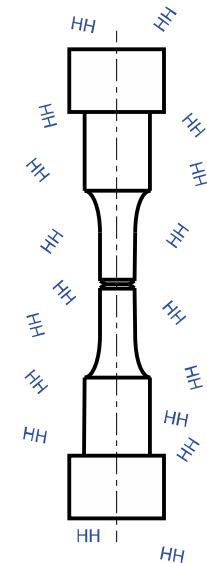
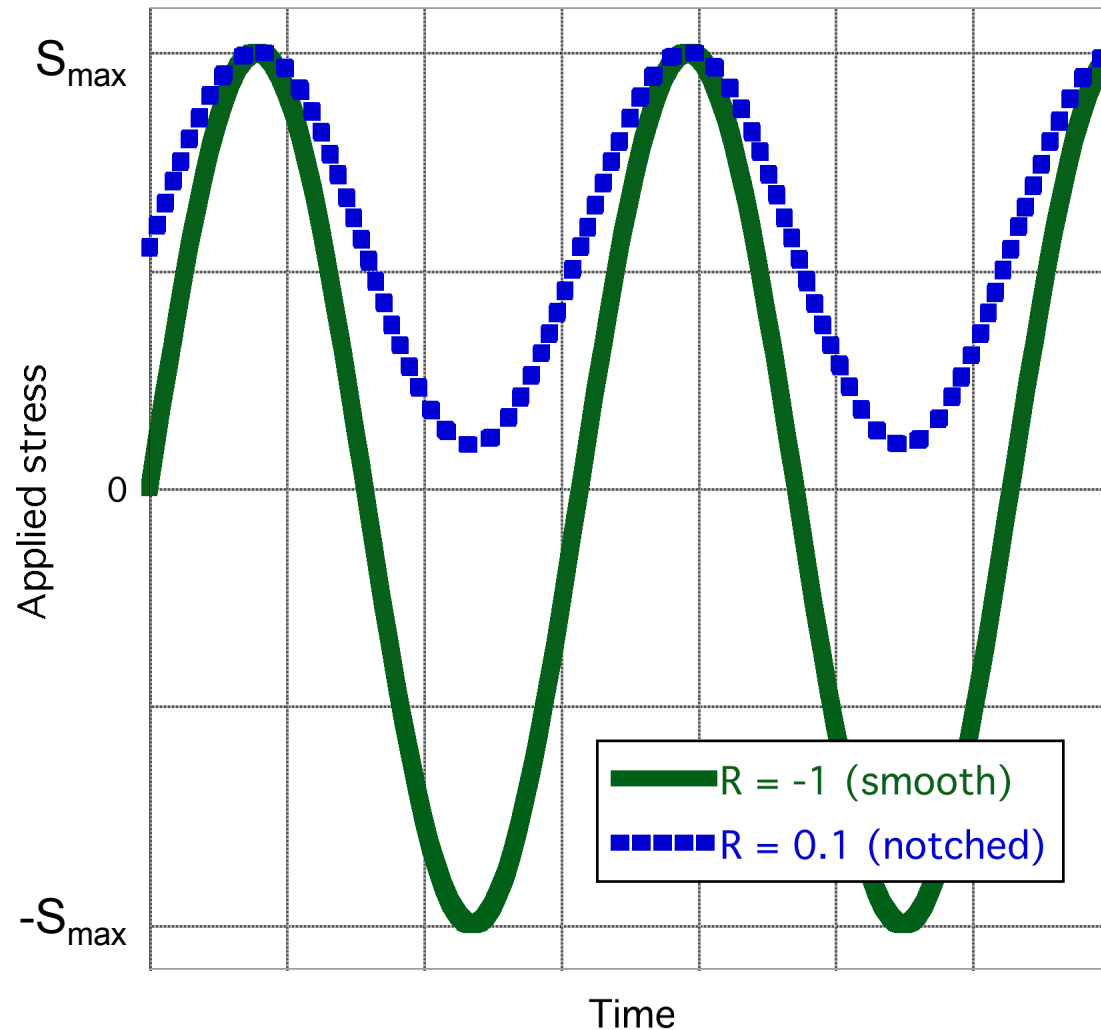
Part 2: Slow strain rate tension test

- **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, $S_u/S_y > 1.07$**

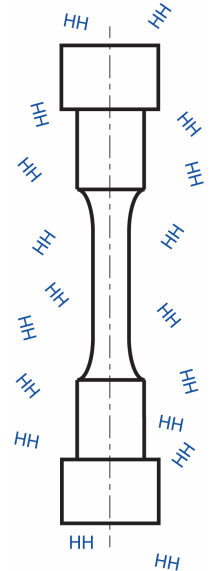
Part 3: Fatigue life test

- **Force-controlled (axially loaded cylindrical) fatigue test in the defined hydrogen environment**
 - **Frequency of 1 Hz**
 - **Maximum stress shall be 1/3 of measured 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



**notched
tension
tension**



**smooth
tension
compression**

Part 4: Welds

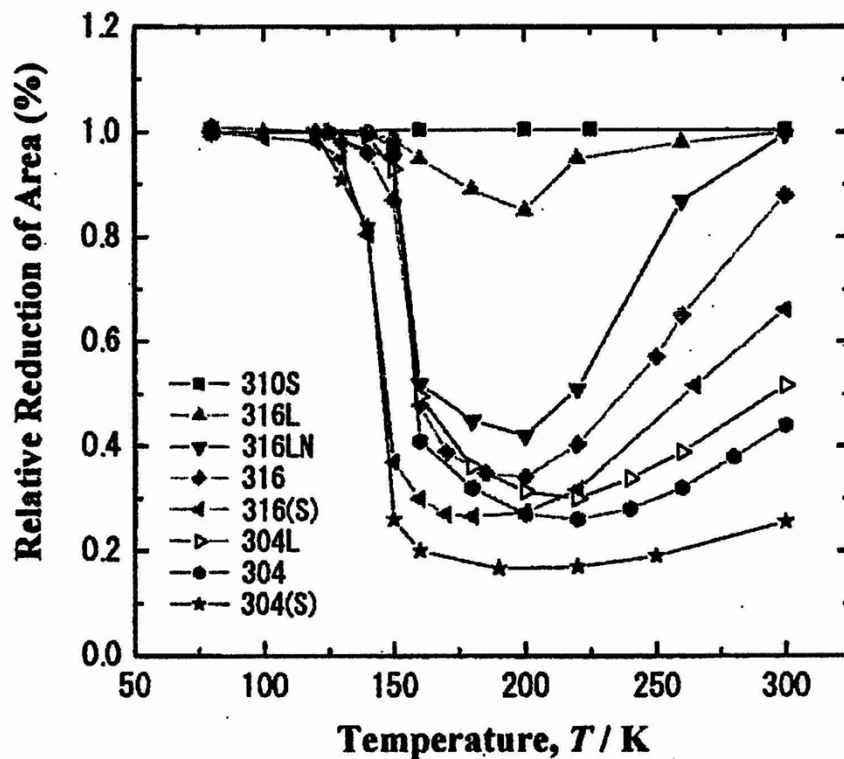
- **Prepare representative welded structures**
- **Same testing requirements as for non-welded materials**
 - **Specified minimum tensile properties must be defined**
 - **Weld material must satisfy the minimum specified properties**
 - **Average values from SSRT tests of weld-material must satisfy minimum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

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

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

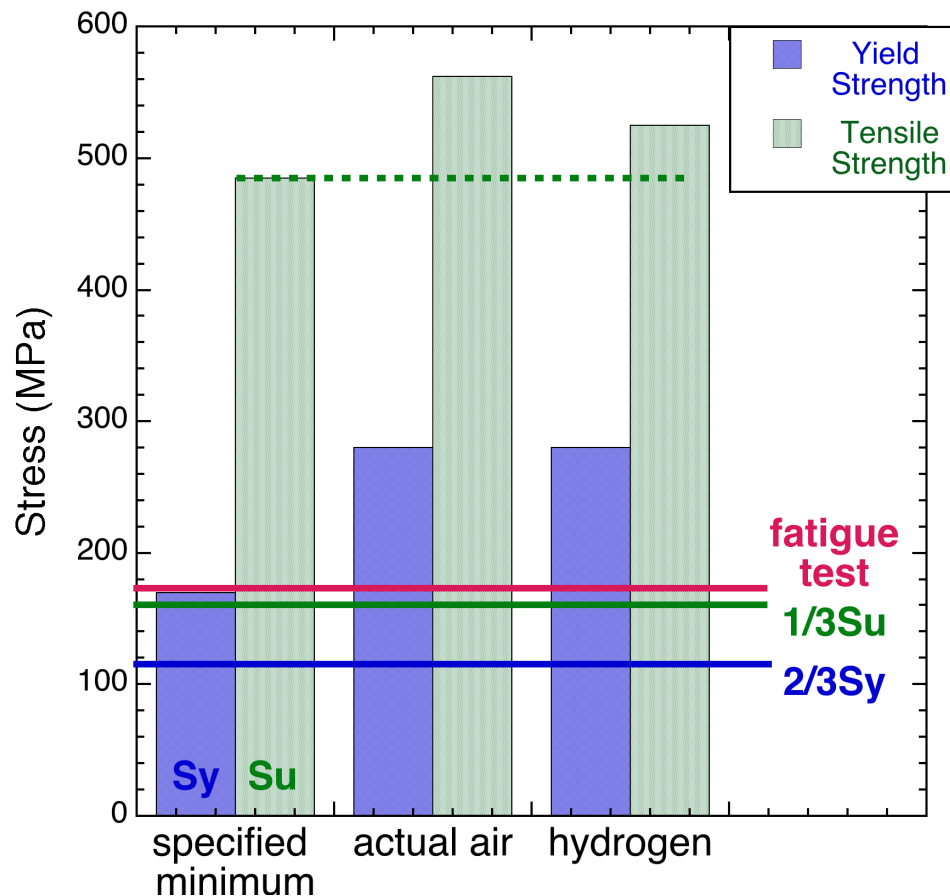
Rationale:

- Known and ductile tensile response

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

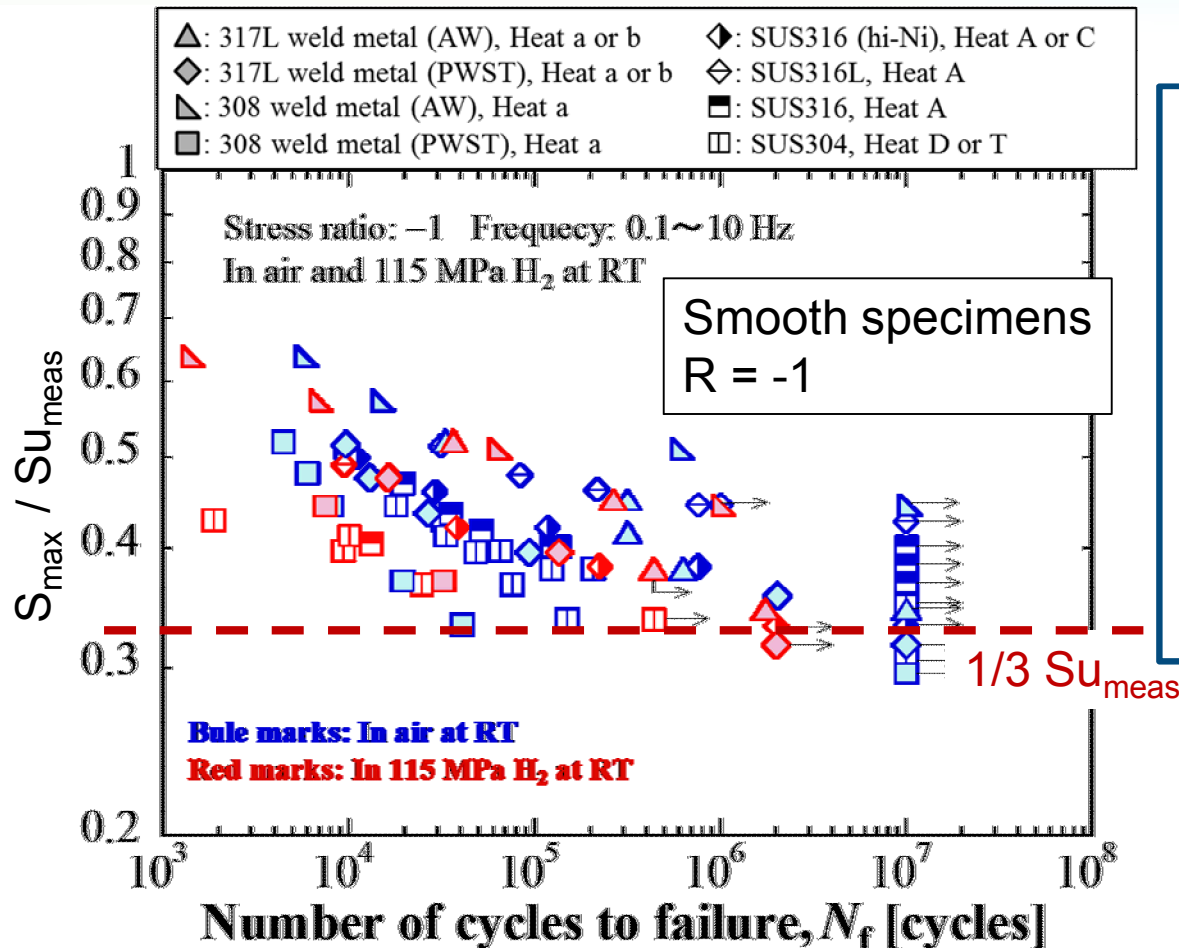
Tensile strength properties are not degraded in gaseous hydrogen for acceptable materials

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 $1/3 S_{u_{meas}}$



Requirement:

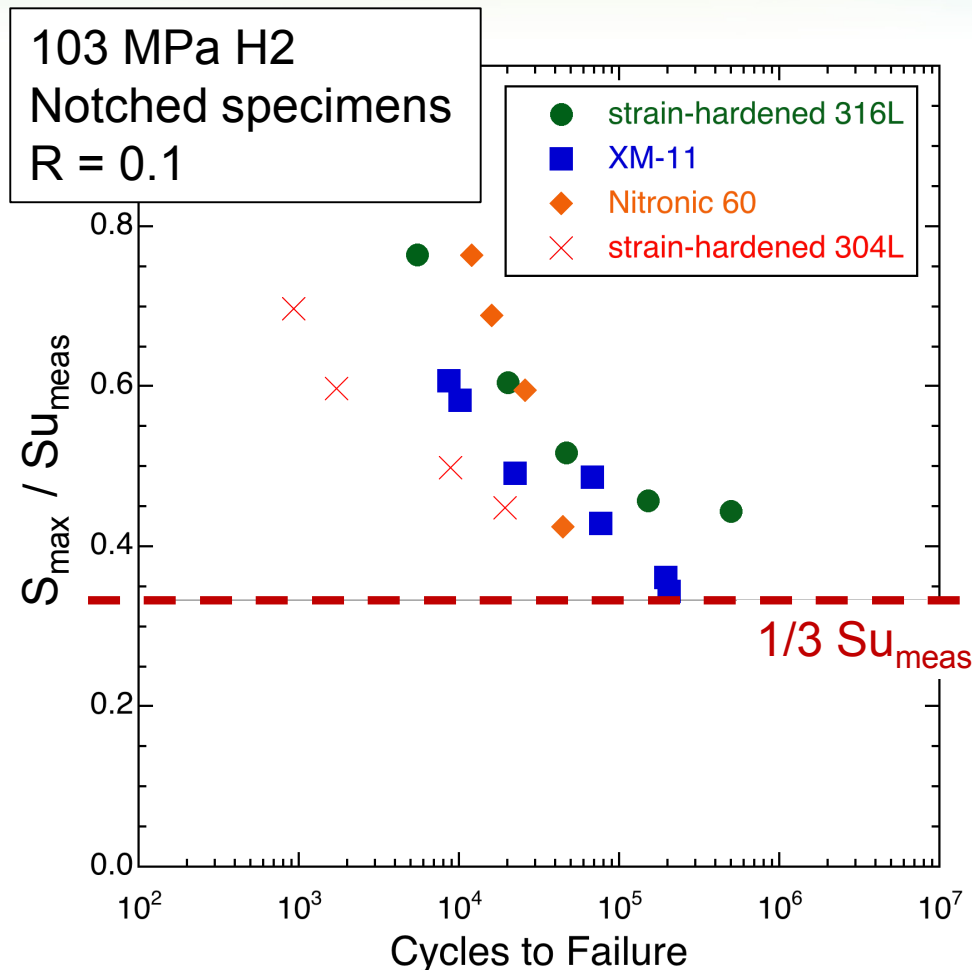
- $N_f > 200,000$ cycles
at $S_{max} = 1/3 S_{u_{meas}}$

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 ($1/3S_u$)



Requirement:

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

Rationale:

- Ensure fatigue life at high stress is \gg 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

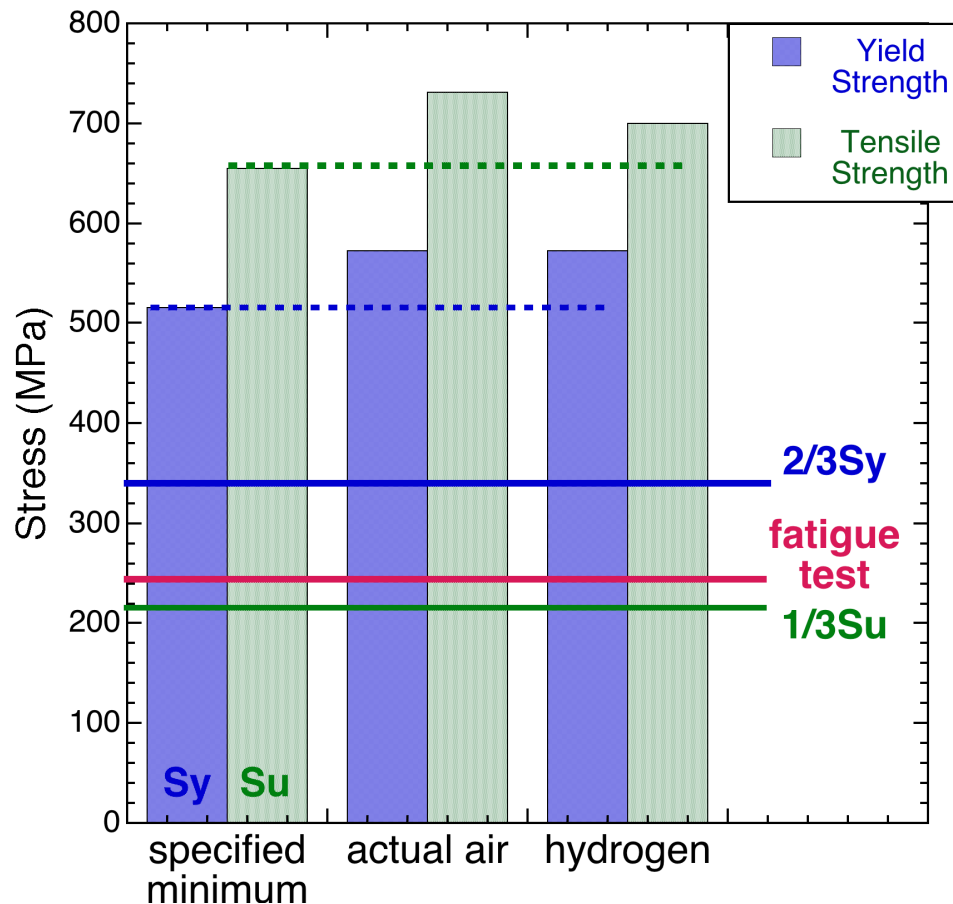
material	Yield (MPa)	Tensile (MPa)	Cr	Ni	Mn	N	Typical allowable stress (MPa)
316L	280	562	17.5	12	1.2	0.04	115
CW 316L	573	731	17.5	12	1.2	0.04	218
304L	497	721	18.3	8.2	1.8	0.56	195
XM-11	539	881	20.4	6.2	9.6	0.26	207
Nitronic 60	880	1018	16.6	8.3	8.0	0.16	218
SCF-260	1083	1175	19.1	3.3	17.4	0.64	333

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

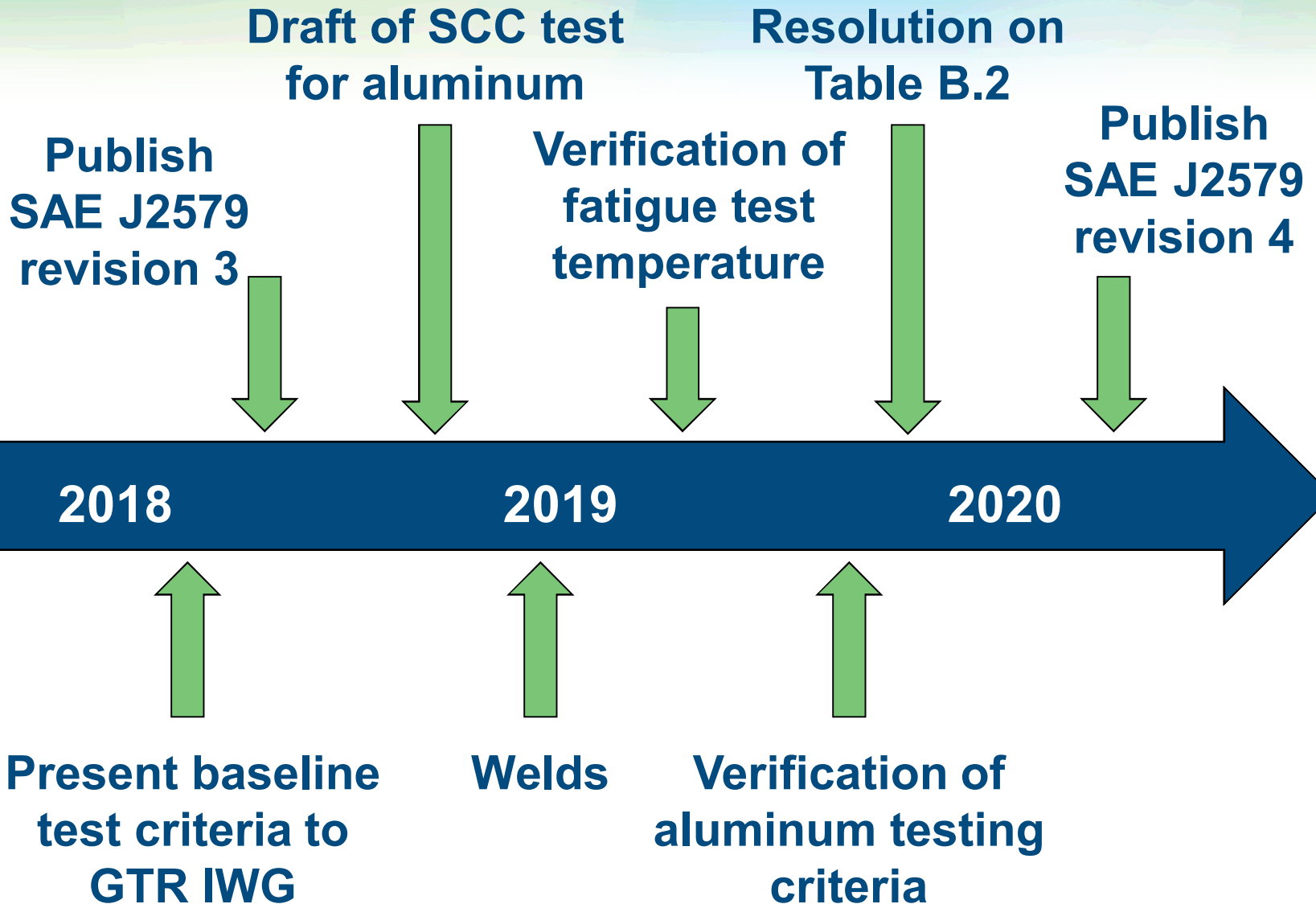


- $1/3 S_u$ of high-strength materials can be more than specified minimum yield strength of annealed material
- Implicitly, increase of design stress enables lower weight and lower cost designs without compromising performance
 - Justified by fatigue performance

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



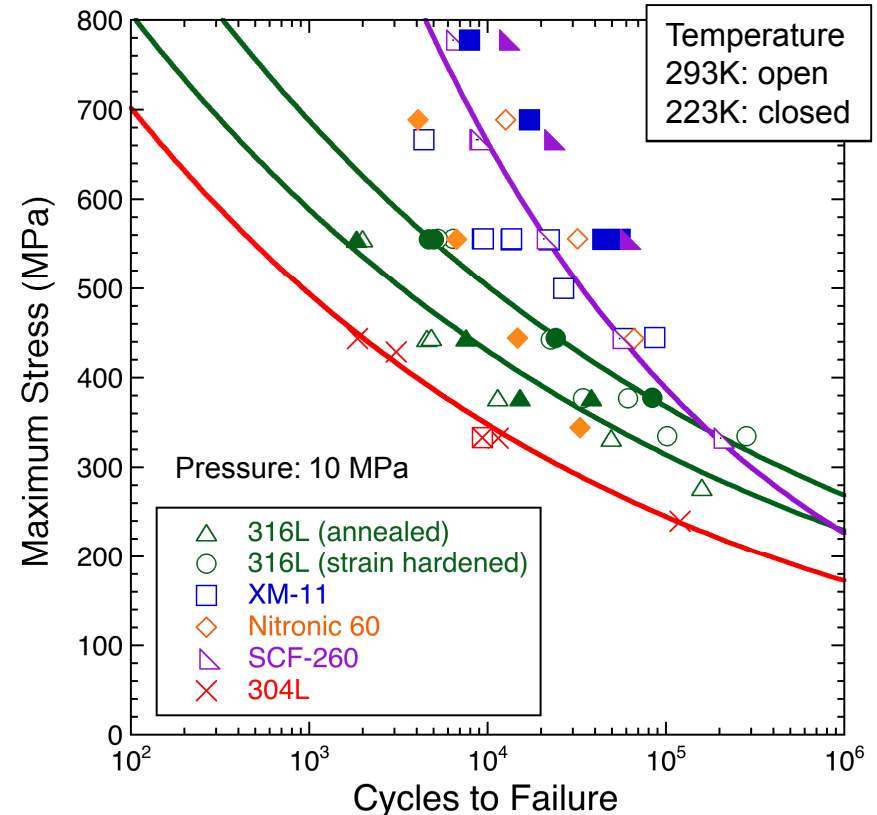
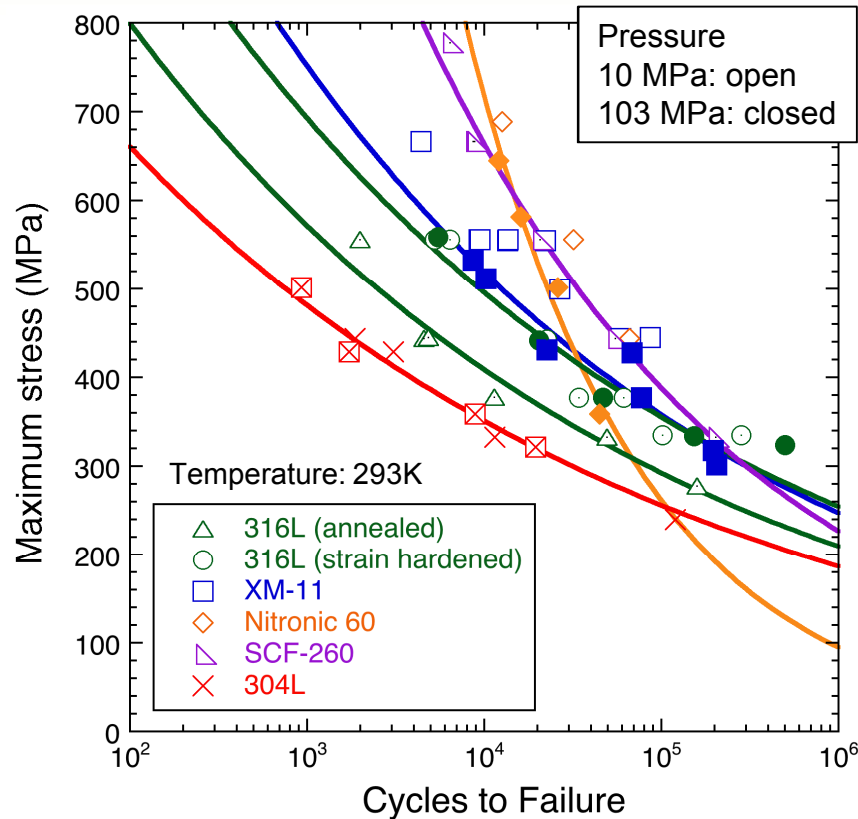
Summary

- **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 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**

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