

R&D for Safety, Codes and Standards: Materials Compatibility

Chris San Marchi
Sandia National Laboratories

Codes and Standards Tech Team
March 28, 2018

Outline

- **Materials Qualification for FCEVs**
 - **SAE H2 Compatibility Expert Team**
 - **SAE J2579 Appendix B**
 - **Summary**
 - **Justification**
- **Fatigue crack growth rate (FCGR) laws for high-pressure storage tanks**
 - **ASME BPVC VIII.3 KD-10**
 - **Extrapolation to other ferritic steels**
 - **Effects of pressure**

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: test temperature

Table B.3.1.4 from SAE J2579

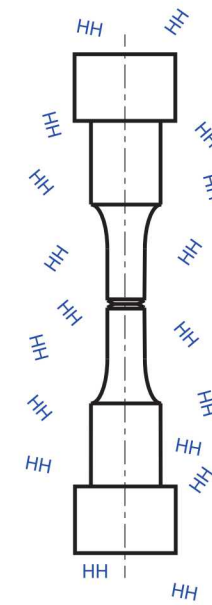
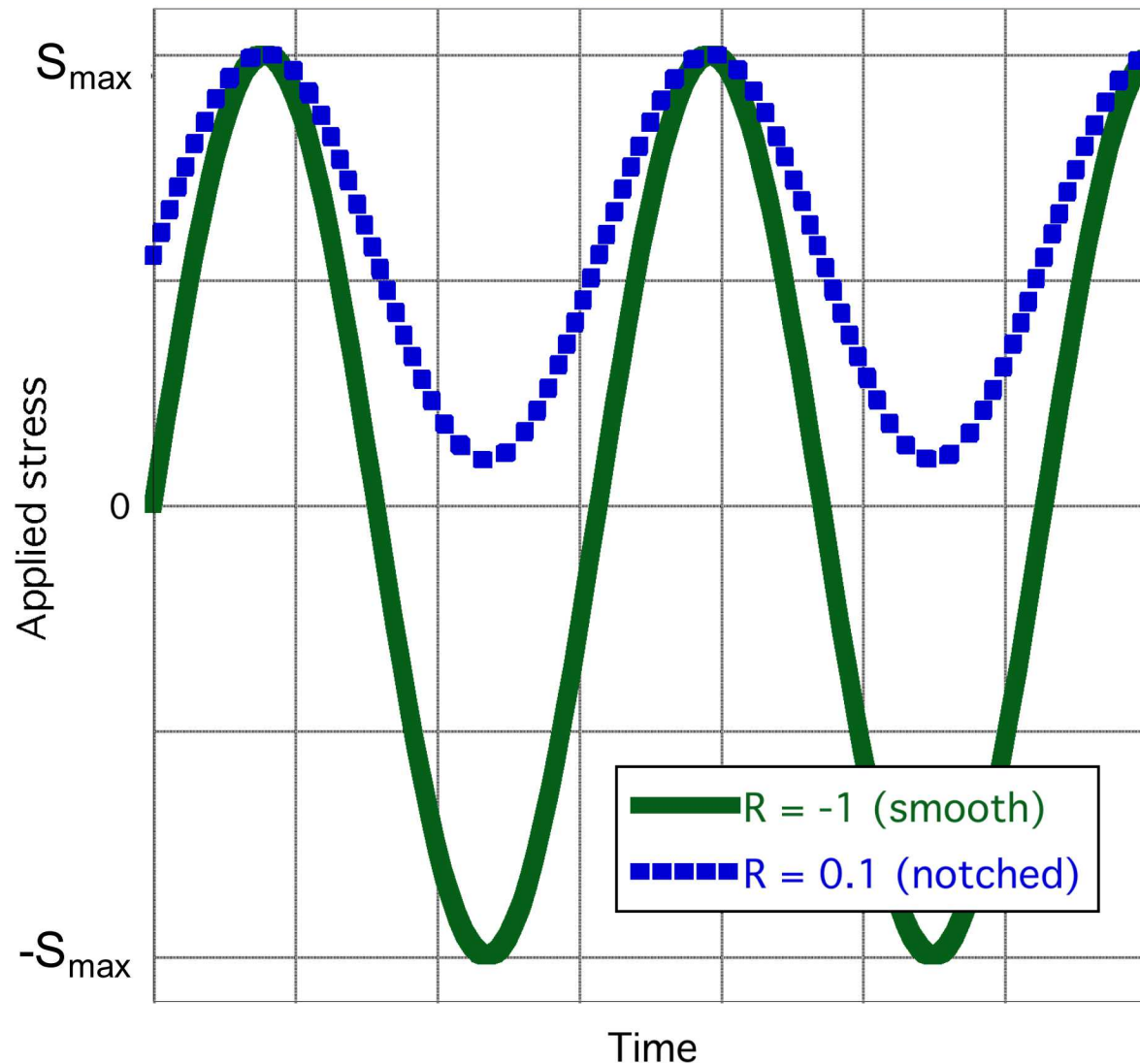
Alloy type	Test method	Test temperature (K)
Austenitic stainless steel	SSRT	228 ± 5
	Fatigue life	228 ± 5 and 293 ± 5
Nickel-based alloys	SSRT and Fatigue life	228 ± 5
Aluminum, magnesium and copper alloys	SSRT and Fatigue life	293 ± 5
Other alloys	SSRT and Fatigue life	228 ± 5 and 293 ± 5

Summary of requirements for compatibility

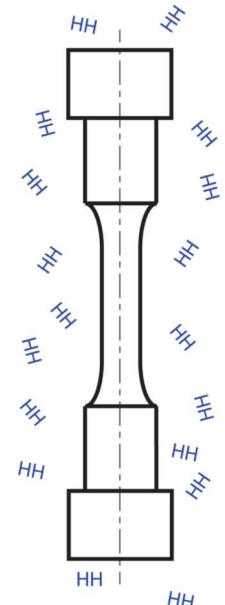
Test configuration		Evaluation parameter	Requirements of tests performed in H2
Slow strain rate tension tests – SSRT (3 tests)		Yield strength	Average $\geq S_y$
		Tensile strength	Average $\geq S_u$
		Strain hardening capacity	Average > 1.07
		Elongation	Average $\geq 12\%$
Fatigue life tests (must 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

Part 3: Fatigue life test: stress cycle

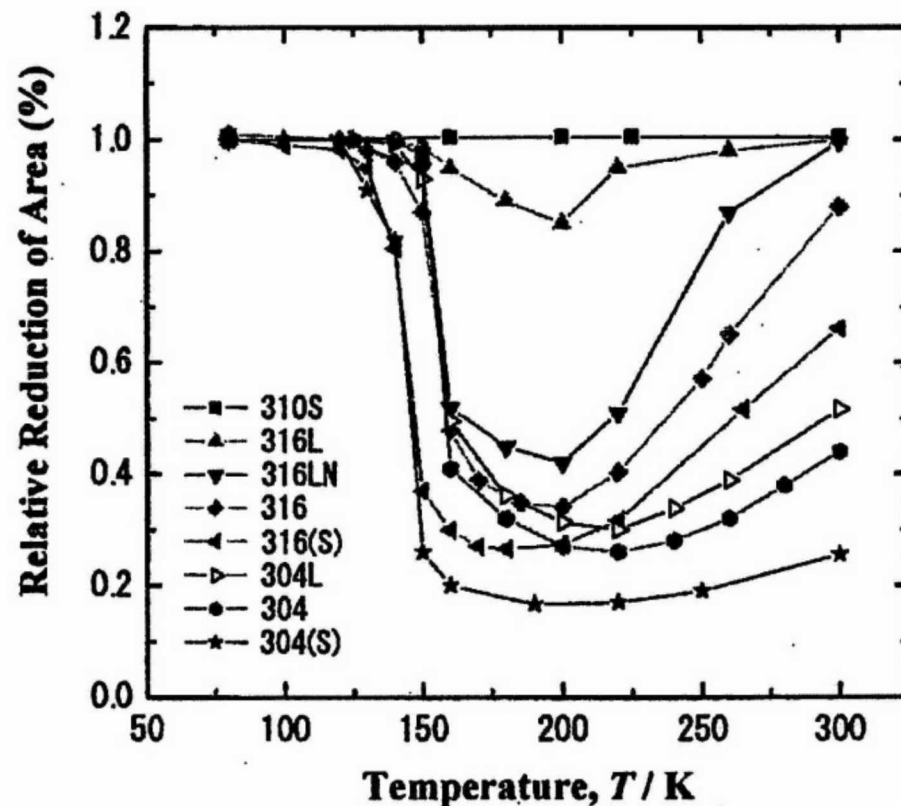


**notched
tension
tension**



**smooth
tension
compression**

Tensile properties are degraded in gaseous hydrogen especially at low temperature



Requirement:

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

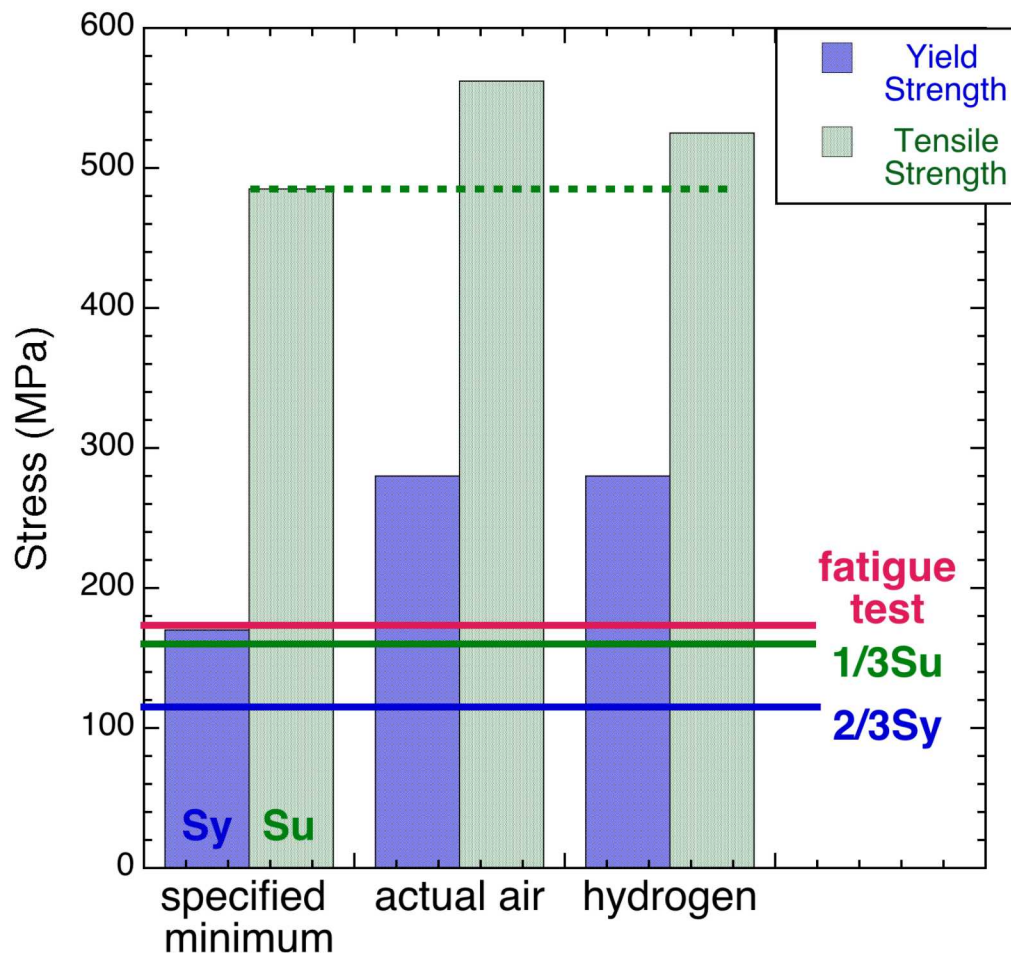
Rationale:

- Known and ductile tensile response

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

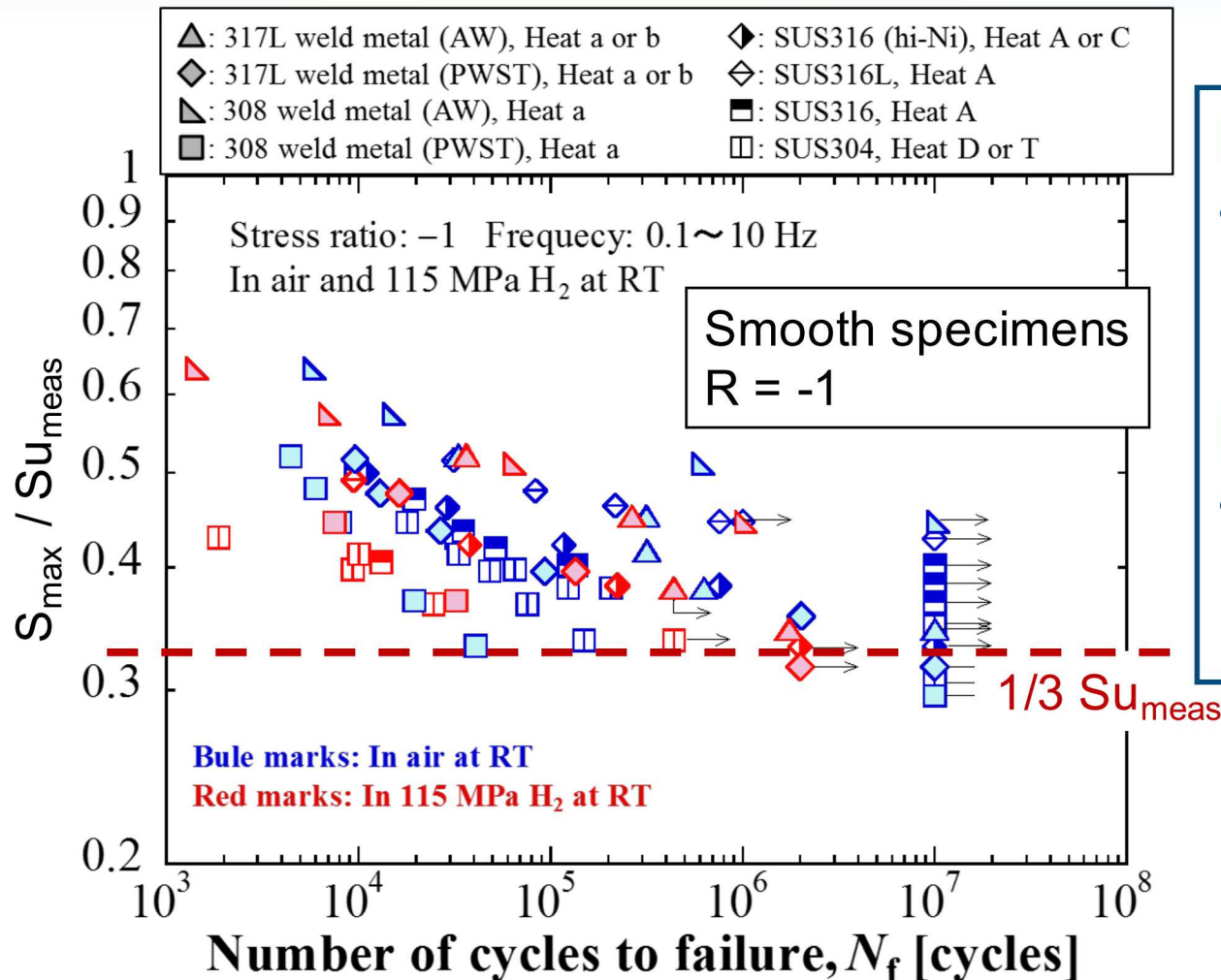
Tensile strength properties are not degraded in gaseous hydrogen for acceptable materials

Annealed austenitic stainless steel



- Common stress limitations for fatigue design: minimum of $\frac{2}{3} S_y$ and $\frac{1}{3} S_u$
- Yield and tensile strengths are typically not affected by hydrogen
- Maximum stress during fatigue testing (J2579) always greater than $\frac{1}{3} S_u$

Fatigue life of smooth specimens is typically infinite at stress of $1/3 S_{u_{meas}}$



Requirement:

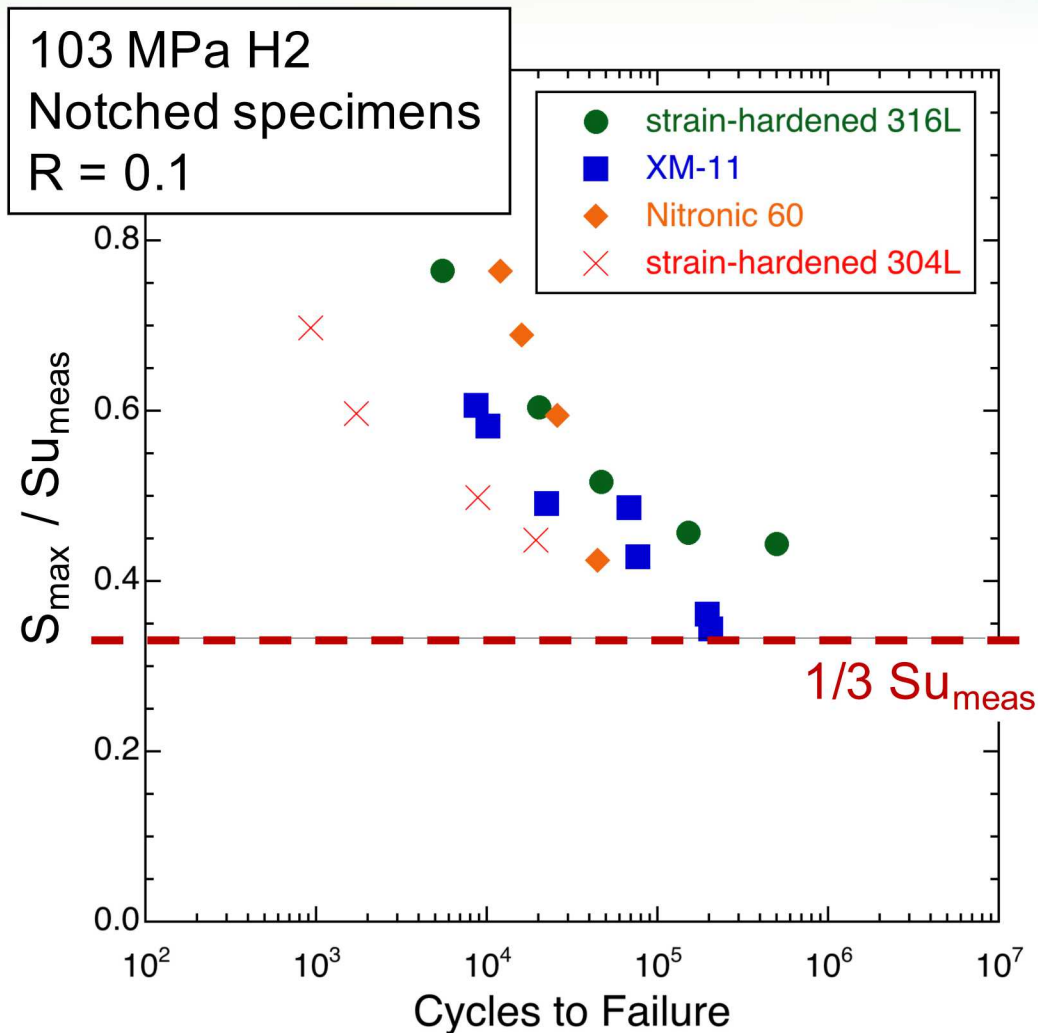
- $N_f > 200,000$ cycles
at $S_{max} = 1/3 S_{u_{meas}}$

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 ($1/3 S_u$)



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

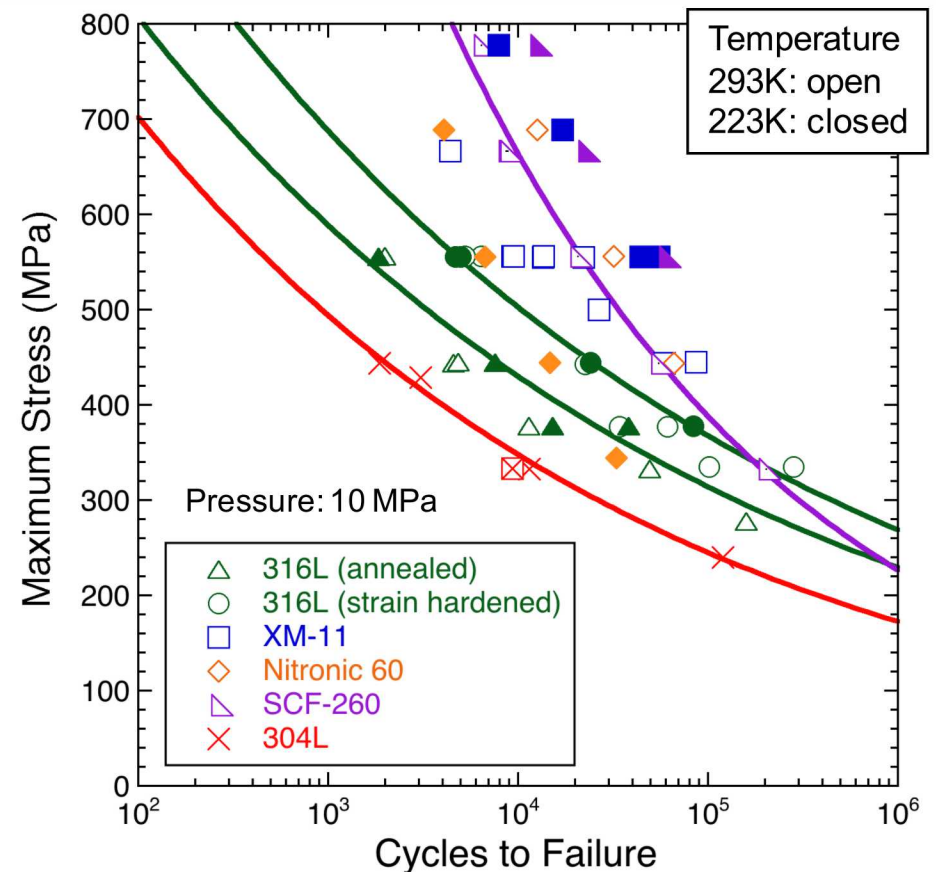
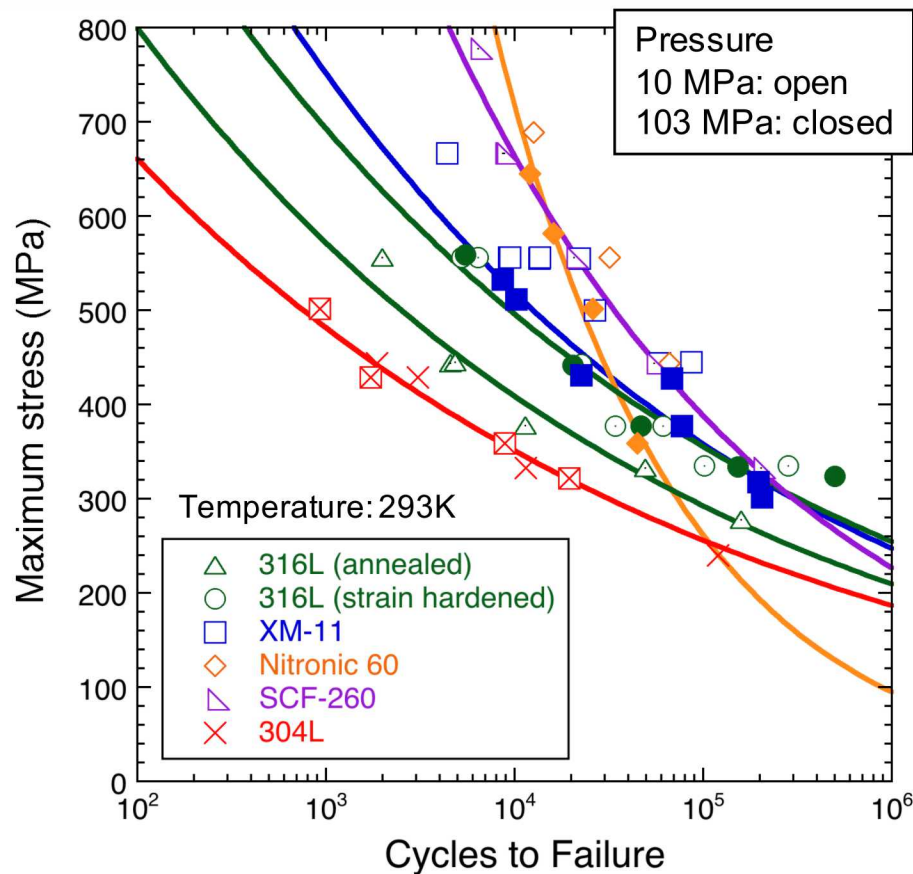
material	Yield (MPa)	Tensile (MPa)	Cr	Ni	Mn	N	Typical allowable stress (MPa)
316L	280	562	17.5	12	1.2	0.04	115
CW 316L	573	731	17.5	12	1.2	0.04	218
304L	497	721	18.3	8.2	1.8	0.56	195
XM-11	539	881	20.4	6.2	9.6	0.26	207
Nitronic 60	880	1018	16.6	8.3	8.0	0.16	218
SCF-260	1083	1175	19.1	3.3	17.4	0.64	333

Wide range of strength

Wide range of Ni/Mn content

Fatigue life at low temperature appears to be greater than at room temperature

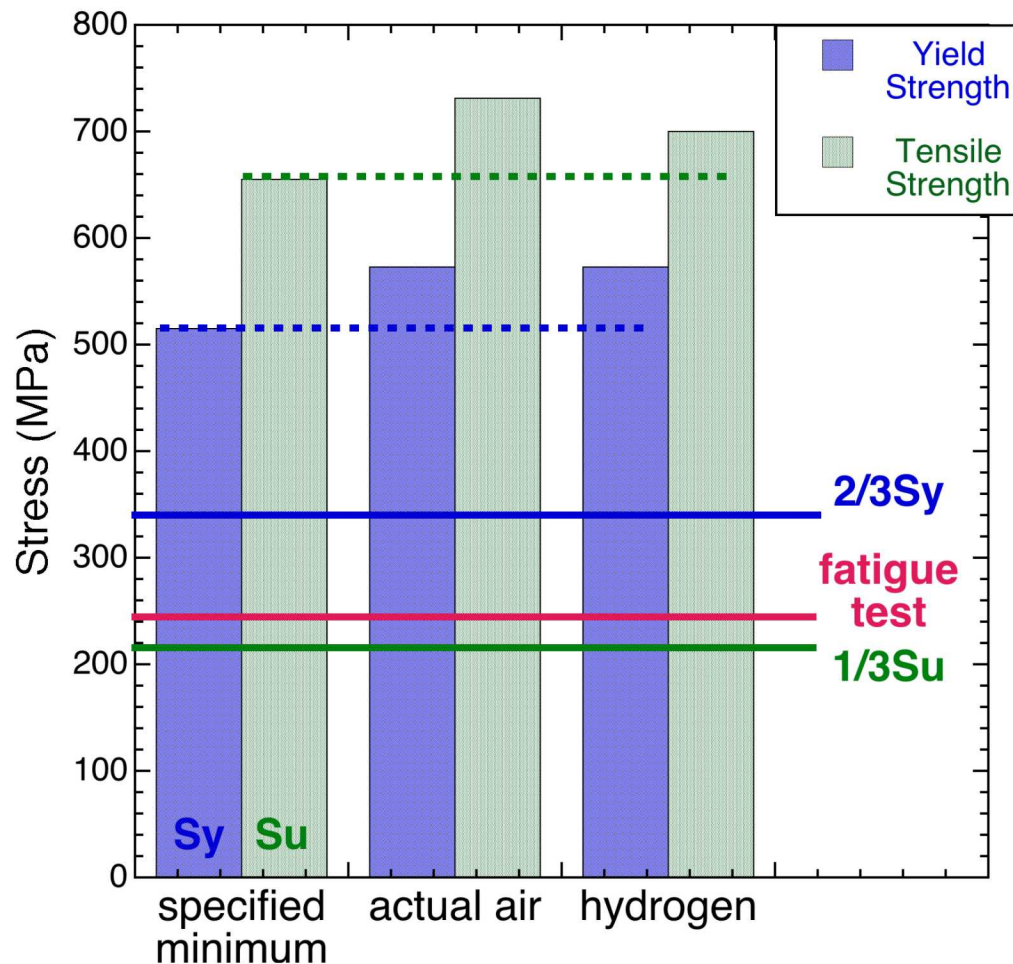
$R = 0.1, f = 1\text{ Hz}$



- 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

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

Strain-hardened austenitic stainless steel

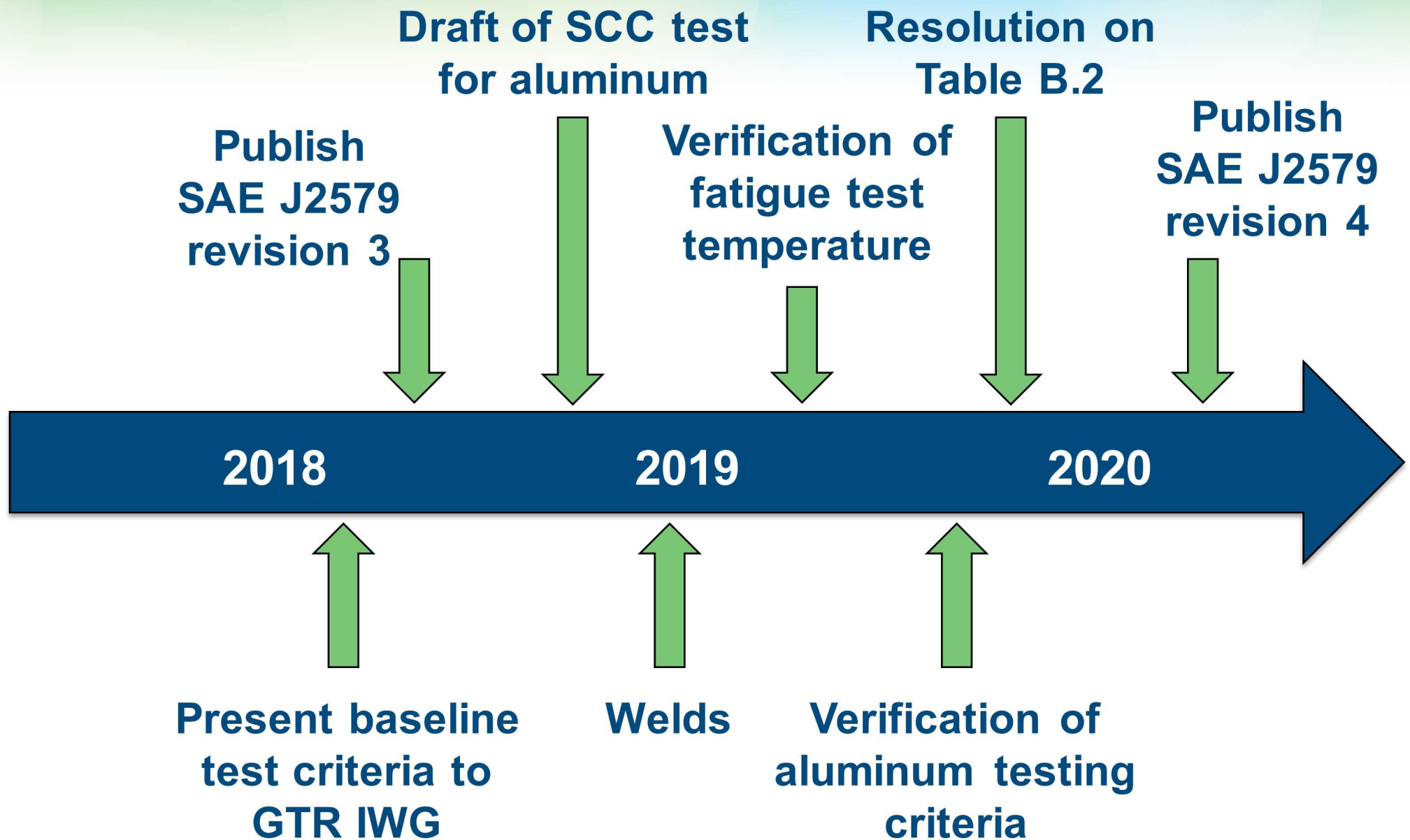


- 1/3 Su of high-strength materials can be more than specified minimum yield strength of annealed material
- Implicitly, increase of design stress enables lower weight and lower cost designs without compromising performance
 - Justified by fatigue performance

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 of SAE activities related to materials



Summary of test criteria in SAE J2579

- **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

Outline

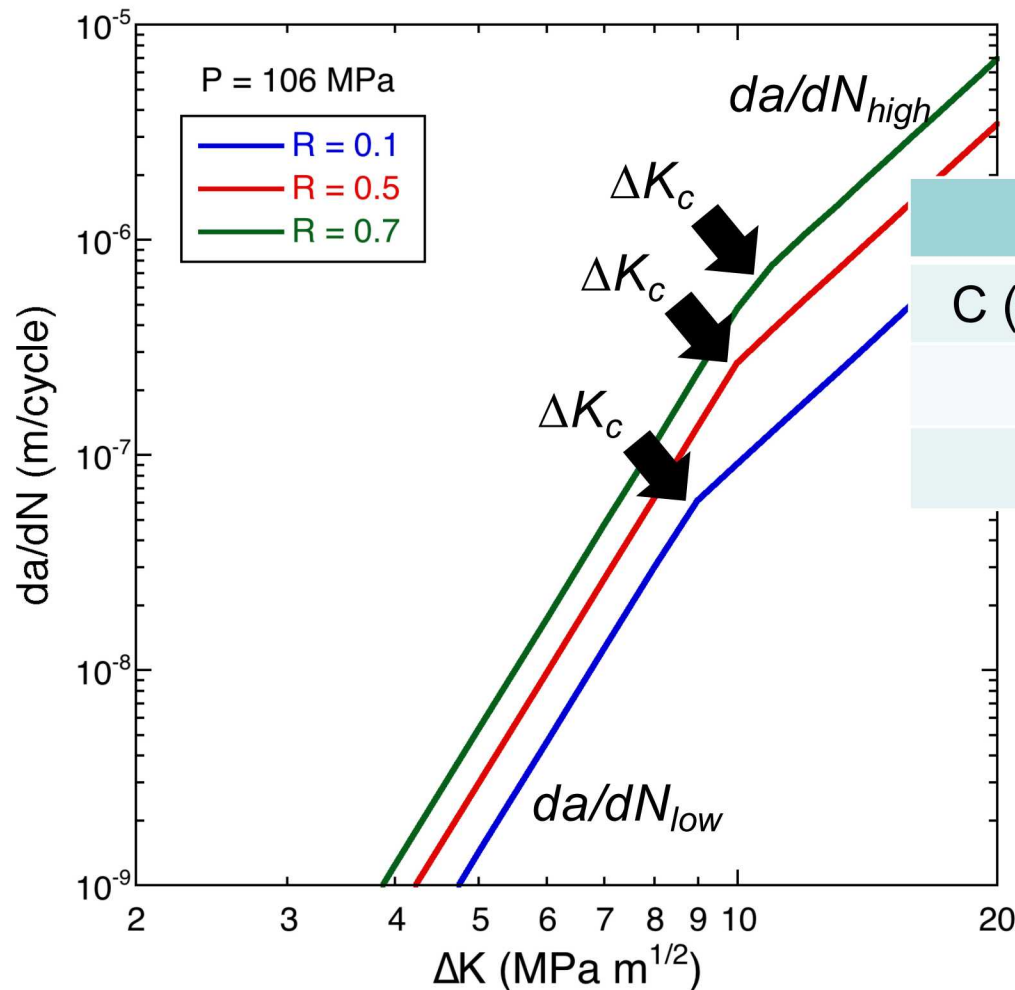
- **Materials Qualification for FCEVs**
 - **SAE H2 Compatibility Expert Team**
 - **SAE J2579 Appendix B**
 - **Summary**
 - **Justification**
- **Fatigue crack growth rate (FCGR) laws for high-pressure storage tanks**
 - **ASME BPVC VIII.3 KD-10**
 - **Extrapolation to other ferritic steels**
 - **Effects of pressure**

Pressure vessel steels fatigue tested at Sandia in gaseous H₂ at pressure of ≥ 103 MPa (15 ksi)

Designation	Tensile strength (MPa)	Yield Strength (MPa)
Cr-Mo steels		
SA-372 Grade J (A71)	839	642
SA-372 Grade J (B50)	871	731
SA-372 Grade J (A72)	908	784
SA-372 Grade J (AV60Z)	890	760
34CrMo4	1045	850
Ni-Cr-Mo steels		
SA-372 Grade L	1149	1053
SA-372 Grade L-LS [†]	873 [†]	731 [†]
SA-723 Grade 1 – Class 1	860	715
SA-723 Grade 3 – Class 2	978	888

[†] Does not meet SA-372 (low strength)

Formulation of power law relationship for fatigue crack growth



$$\frac{da}{dN} = C \left[\frac{1 + C_H R}{1 - R} \right] \Delta K^m$$

	da/dN_{low}	da/dN_{high}
C (m/cycle)	3.5×10^{-14}	1.5×10^{-11}
m	6.5	3.66
C_H	0.4286	2.00

$$\Delta K < \Delta K_c: \quad da/dN = da/dN_{low}$$

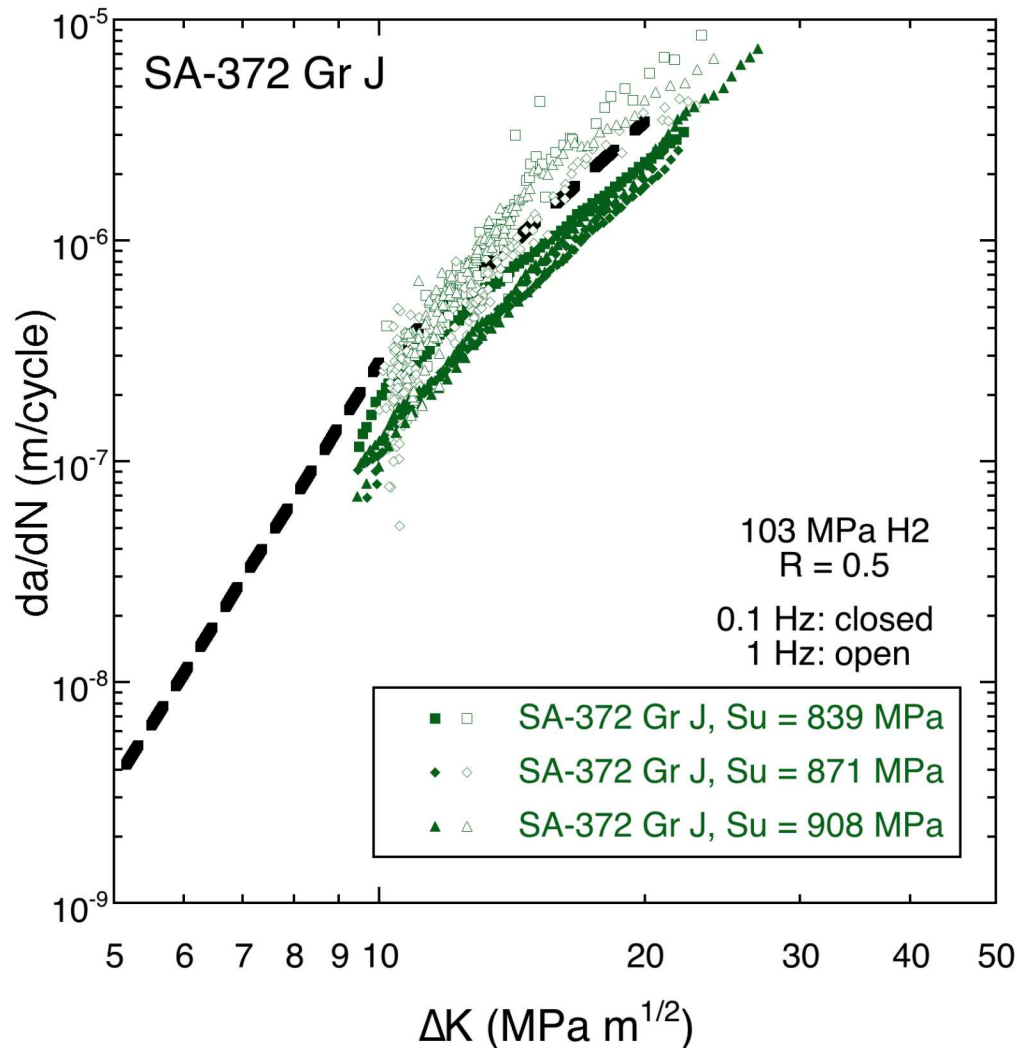
$$\Delta K \geq \Delta K_c: \quad da/dN = da/dN_{high}$$

$$\Delta K_c = 8.475 + 4.062R - 1.696R^2$$

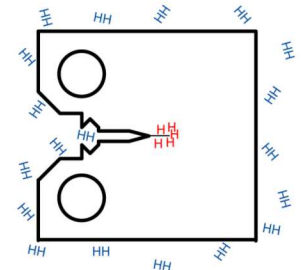
$\Delta K, \Delta K_c$ units: MPa $m^{1/2}$

Fatigue crack growth rates of SA-372 Grade J

R = 0.5

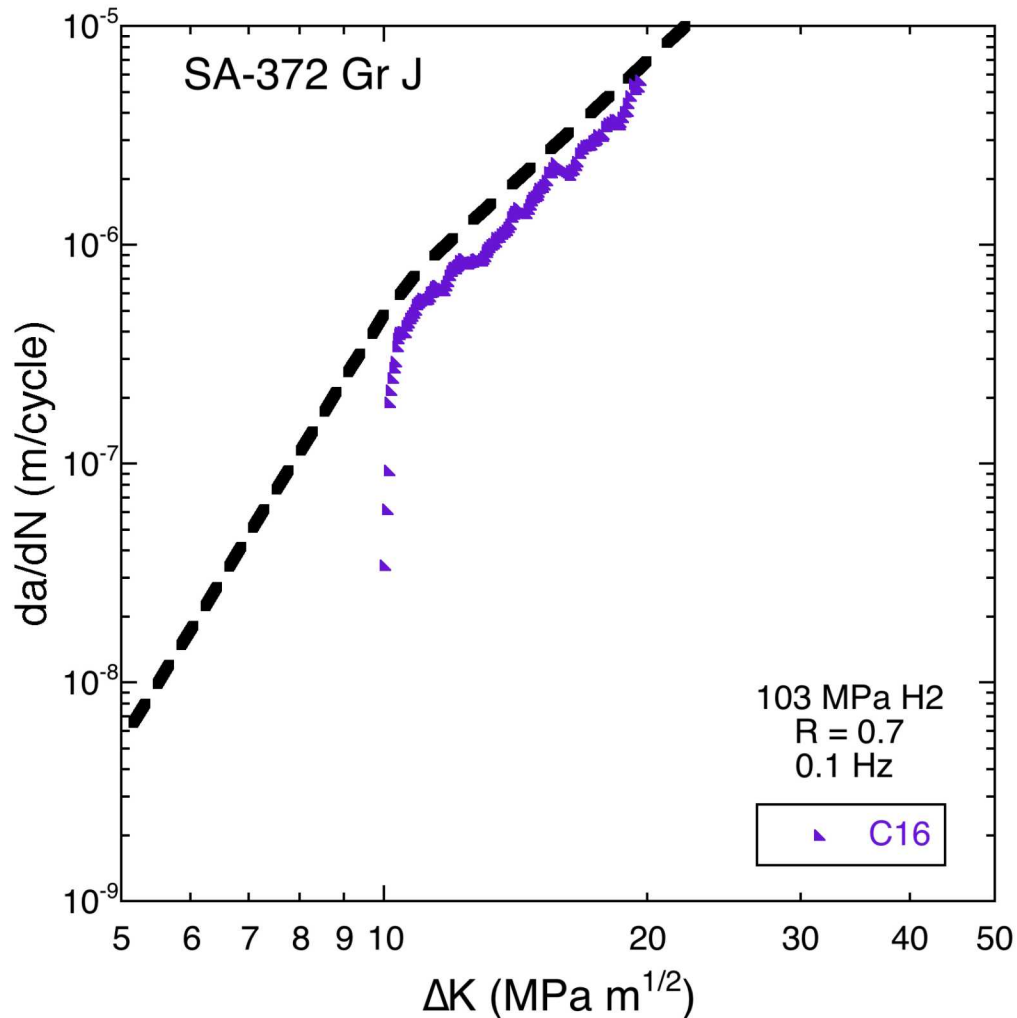


- Gaseous hydrogen at pressure of 103 MPa
- R = 0.5
- 0.1 Hz data from PVP2013-97455
- 1 Hz data from ICHS 2009



Fatigue crack growth rates of SA-372 Grade J

R = 0.7

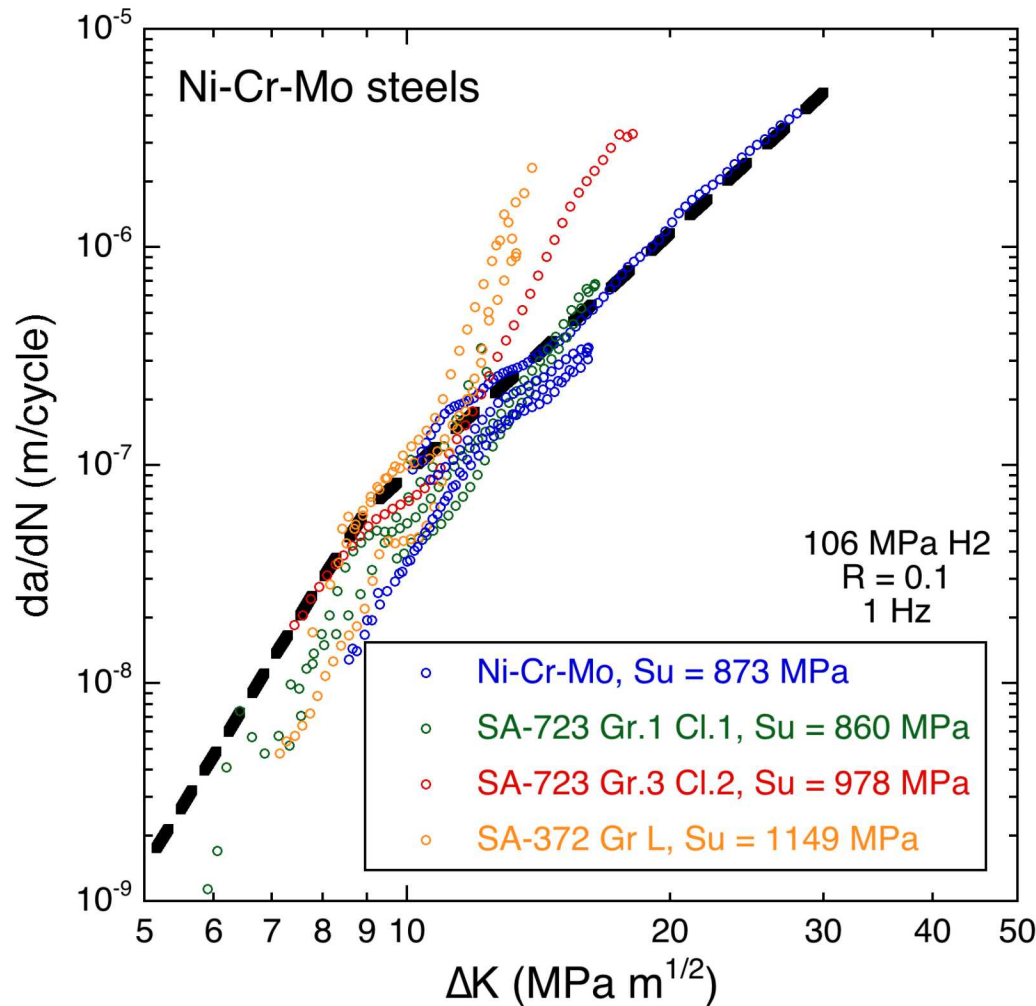


- Gaseous hydrogen at pressure of 103 MPa
- R = 0.7
- 0.1 Hz
- Data not published?
 - Not from SNL

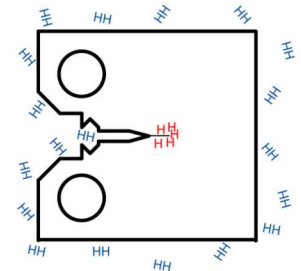
Designation	Tensile strength (MPa)	Yield Strength (MPa)
SA-372 Gr J (C16)	884	711
SA-372 Gr J (C57)	904	787
SA-372 Gr J (C58)	913	764

Fatigue crack growth rates of Ni-Cr-Mo steels

$R = 0.1$

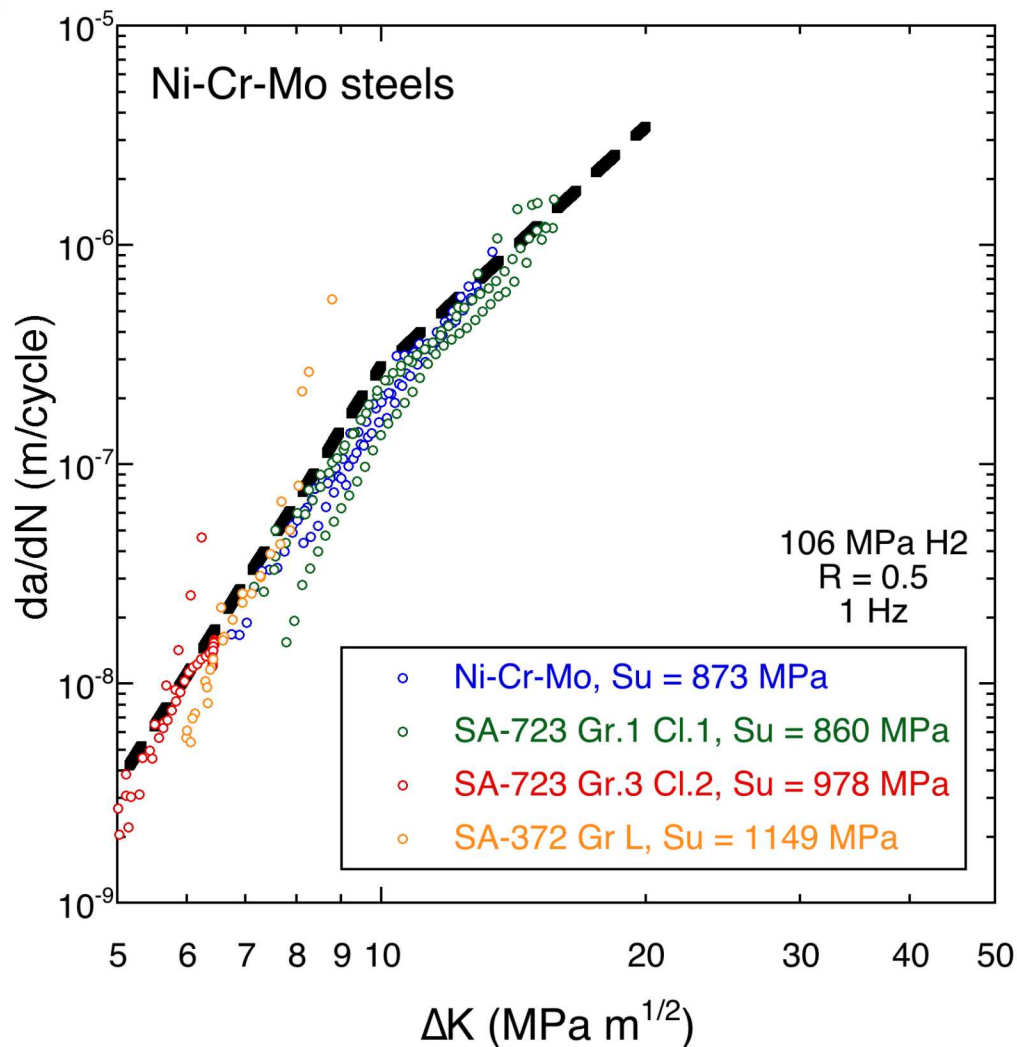


- Gaseous hydrogen at pressure of 106 MPa
- $R = 0.1$
- Frequency of 1 Hz
- Data from ICHS 2017

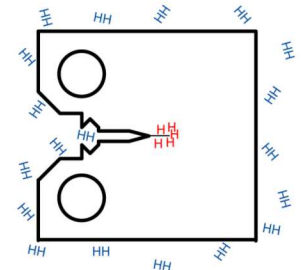


Fatigue crack growth rates of Ni-Cr-Mo steels

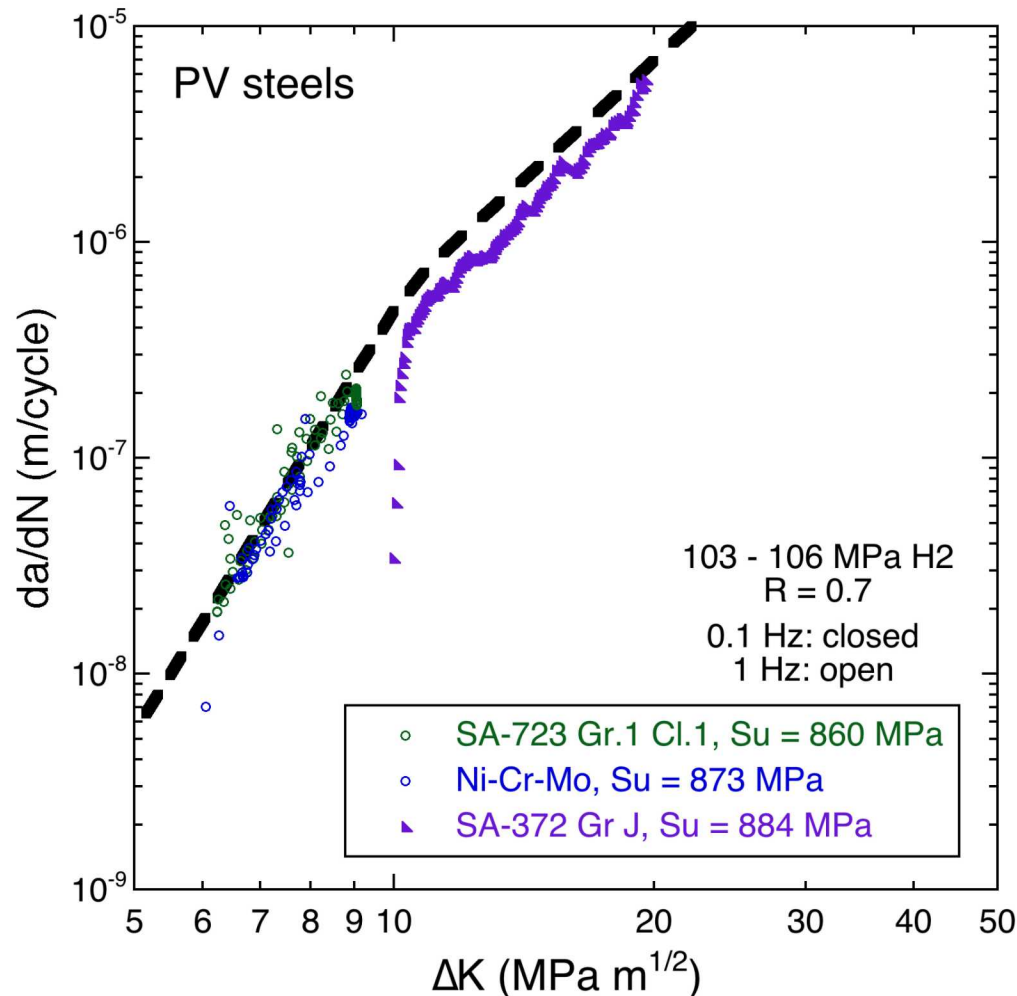
$R = 0.5$



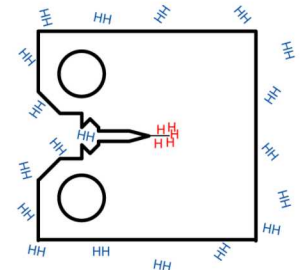
- Gaseous hydrogen at pressure of 106 MPa
- $R = 0.5$
- Frequency of 1 Hz
- Data from ICHS 2017



Fatigue crack growth rates at $R = 0.7$ both Cr-Mo and Ni-Cr-Mo PV steels



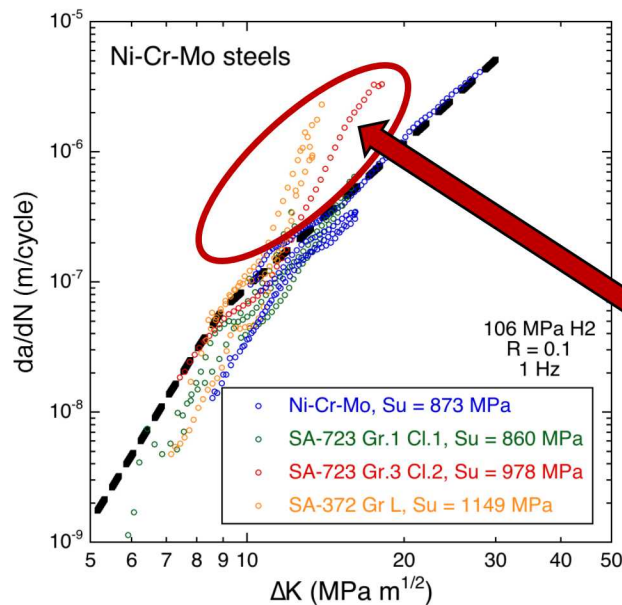
- Gaseous hydrogen
- $R = 0.7$
- Cr-Mo steel data (0.1 Hz)
from NIST (unpublished?)
– P = 103 MPa
- Ni-Cr-Mo steel data (1 Hz)
from ICHS 2017
– P = 106 MPa



Basis for limiting strength

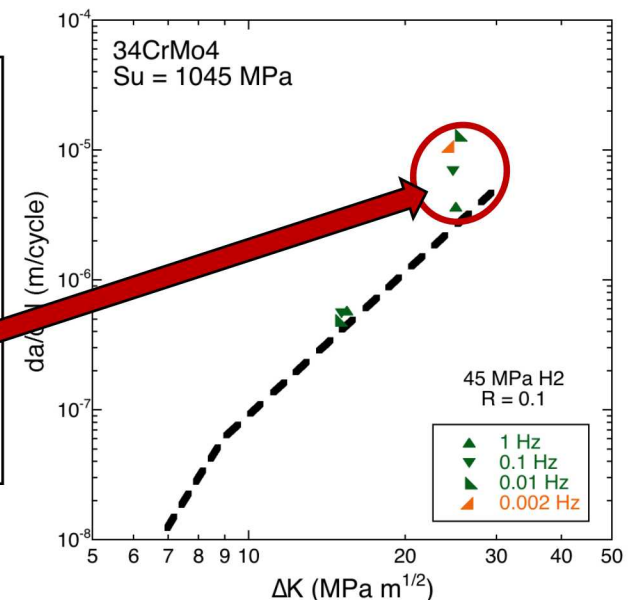
Hydrogen-assisted fatigue crack growth

- High-strength steels show transition to accelerated crack growth related to baseline behavior (eg, stage III)
 - only observed in tests of high-strength steels: tensile strength > 950 MPa
 - Related to fracture resistance: as $K_{\max}$ approaches K_{JH} (where K_{JH} is measured as J_{IC} from ASTM E1820 in gaseous hydrogen)



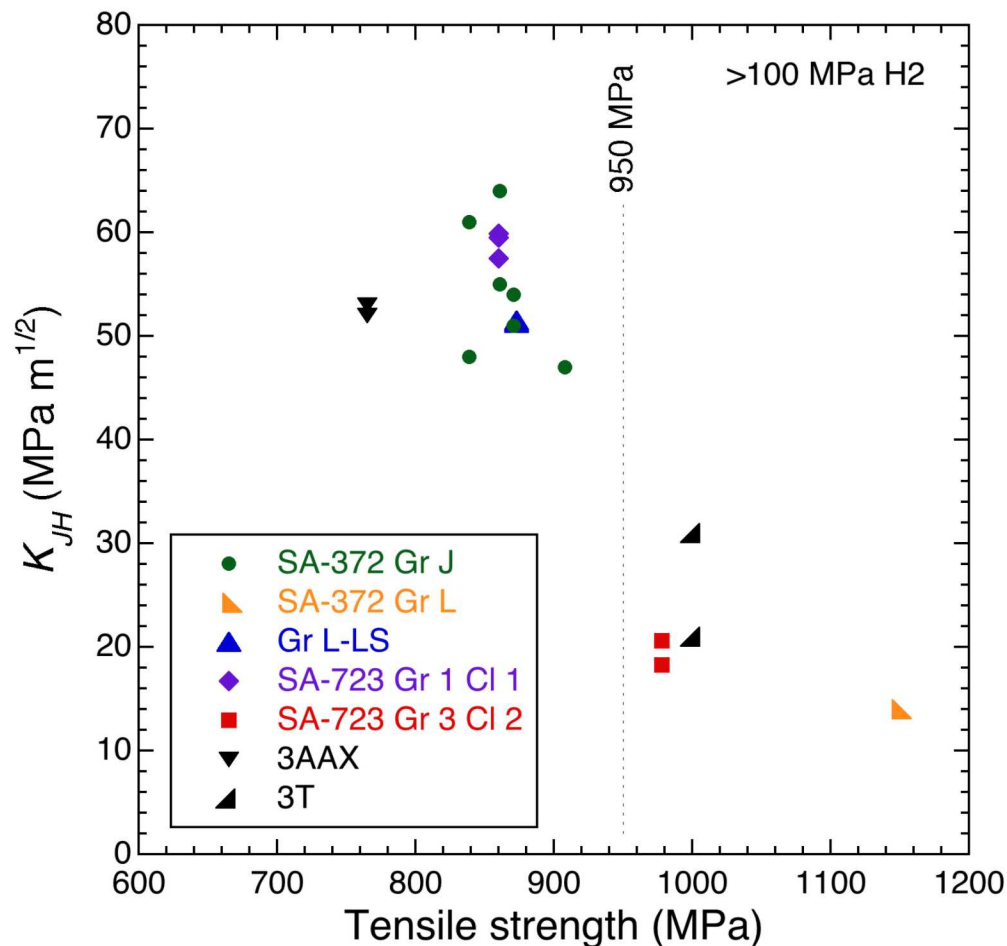
For PV steels with
Su > 950 MPa

- Accelerated fatigue crack growth rate is observed
- $K_{JH} < 30$ MPa m^{1/2}



Basis for limiting strength

Fracture resistance – rising load (K_{JH})

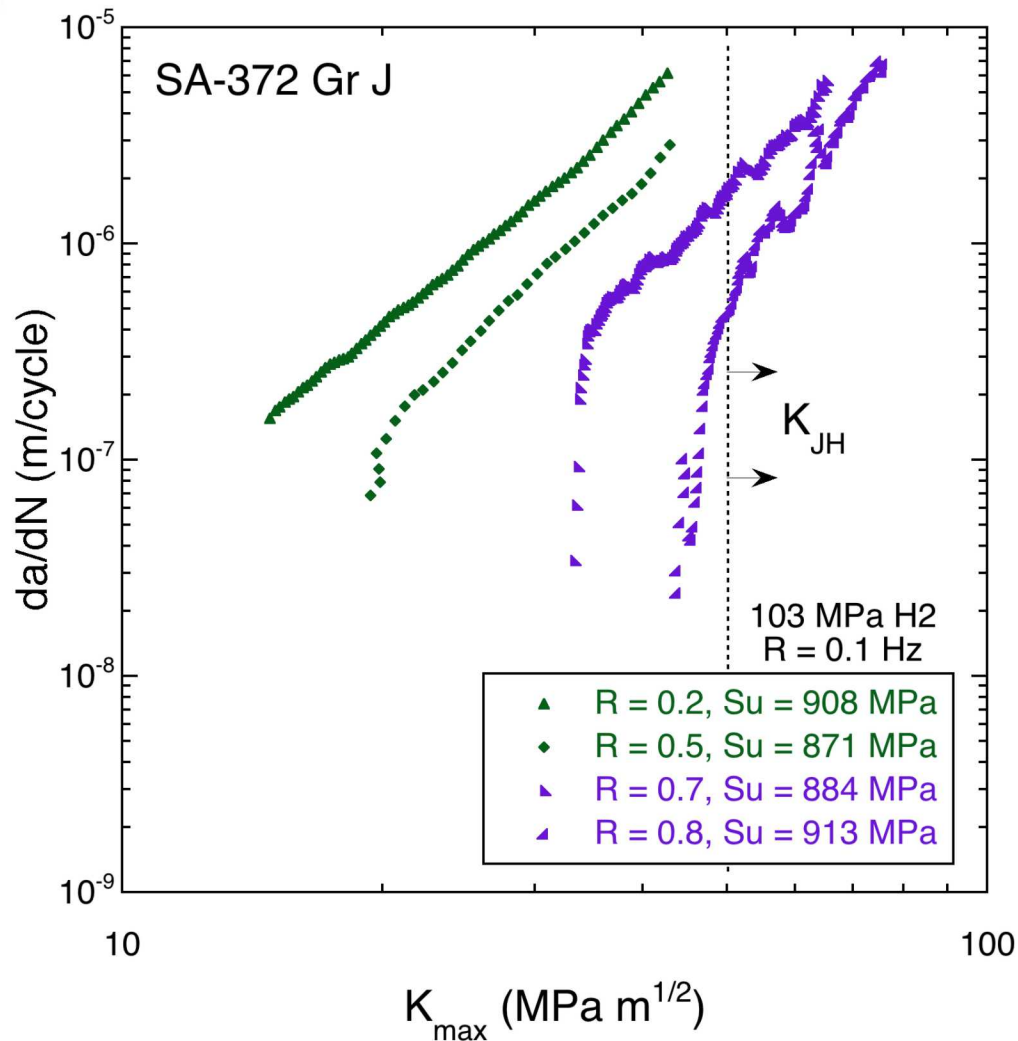


PV steels display low fracture resistance in high strength condition

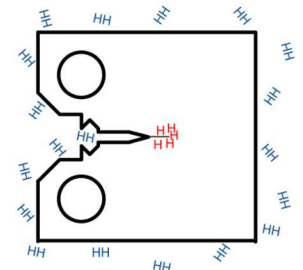
- For tensile strength < 950 MPa
– $K_{JH} > 45 \text{ MPa m}^{1/2}$
- For tensile strength > 950 MPa
– $K_{JH} < 30 \text{ MPa m}^{1/2}$

K_{JH} = elastic-plastic plane-strain fracture toughness in gaseous hydrogen (ASTM E1820)

Basis for limiting K_{max}

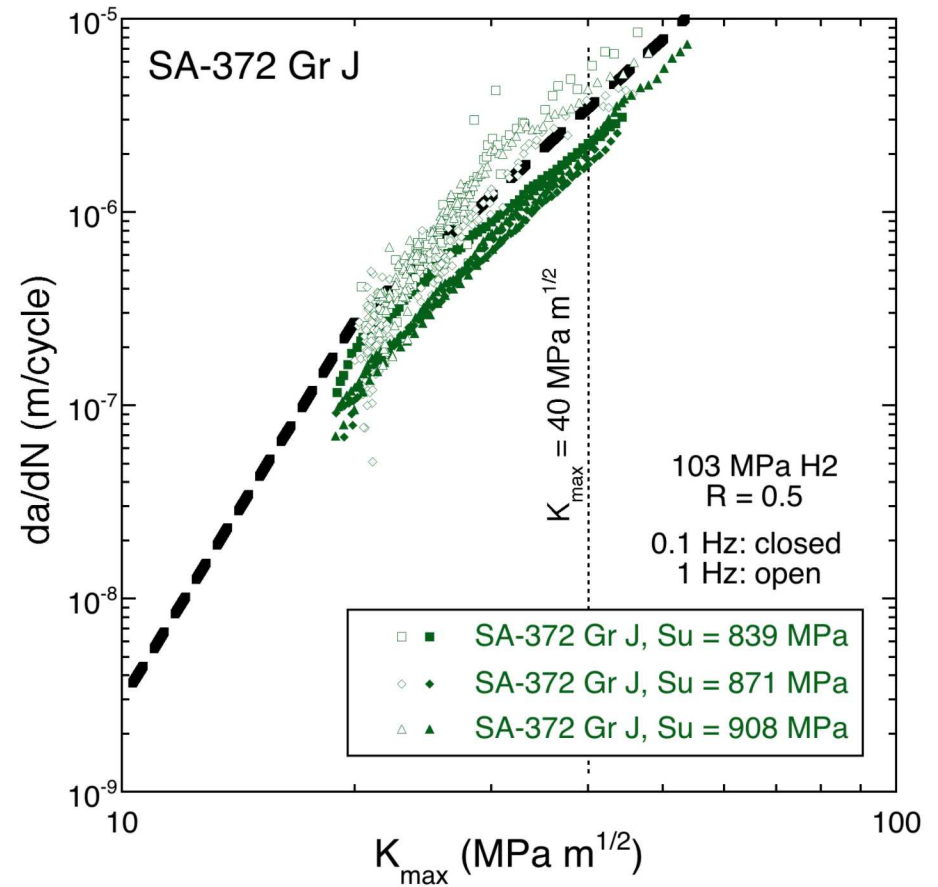
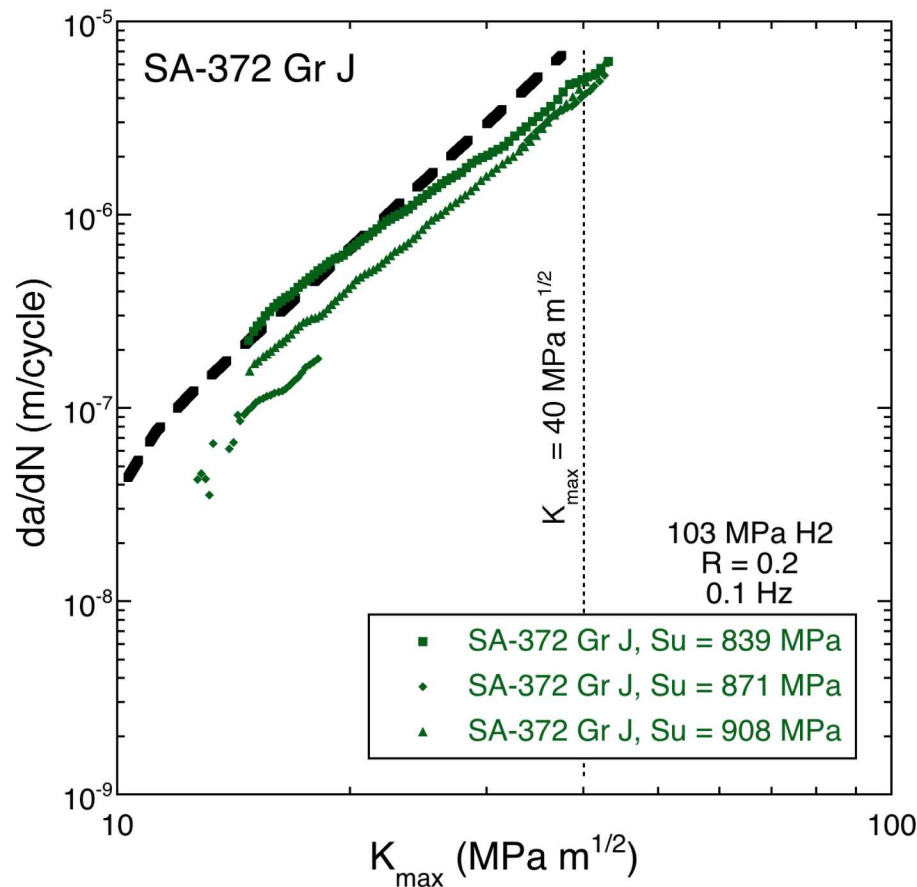


- "Stage III" fatigue crack growth begins at $K_{max} < K_{JH}$
 - The proposed relationships do not capture stage III
- $K_{JH} \geq 45\text{-}50 \text{ MPa } m^{1/2}$ for tensile strength < 950 MPa
- $K_{max} \leq 40 \text{ MPa } m^{1/2}$ provides a bound on the proposed relationships to ensure $K_{max} \leq K_{JH}$ for steels with tensile strength < 950 MPa



Basis for limiting K_{max}

- No evidence of transition to stage III for K_{max} up to $40 \text{ MPa m}^{1/2}$ in SA-372 Gr J steels
- For steels shown below:
 - Measured K_{JH} values in 103 MPa H2 are within the range of $47\text{-}61 \text{ MPa m}^{1/2}$ (5 measurements)



Summary

- Review of fatigue crack growth rate for PV steels generally shows consistency of fatigue response independent of alloy and strength

- *Exception:* tensile strength > 950 MPa shows transition to stage III crack growth at low $K_{\max}$

- Two-part power law was established to bound FCGR behavior as a function of load ratio, R

$$\frac{da}{dN} = C \left[\frac{1 + C_H R}{1 - R} \right] \Delta K^m$$

- Transition between “two parts” also quantitatively established

$$\Delta K_c = 8.475 + 4.062R - 1.696R^2$$

- Proposed constraints for use of established relationships

- Tensile strength < 950 MPa

- $K_{\max} \leq 40 \text{ MPa m}^{1/2}$

Outlook: Pressure term can be added to extend applicability to lower pressure and other steels

$$\frac{da}{dN} = C \left[\frac{1 + C_H R}{1 - R} \right] \Delta K^m \left(\frac{f}{f_{ref}} \right)^{1/2}$$

- f is fugacity (related to pressure) and f_{ref} is the reference fugacity for which other terms were developed

