

Mechanical Properties of Structural Steels in Hydrogen

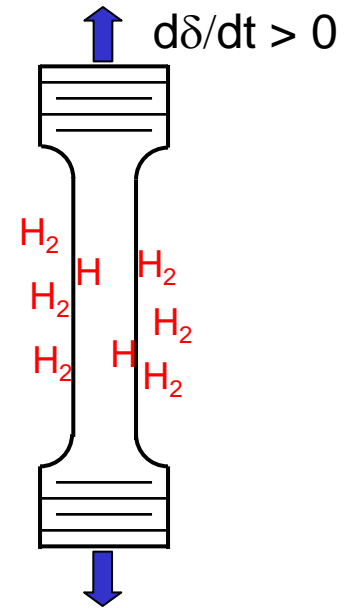
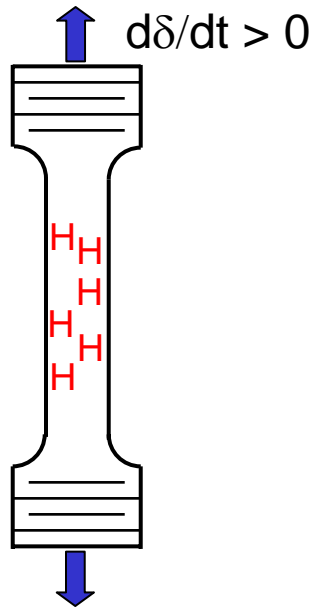
B.P. Somerday, K.A. Nibur, C. San Marchi, and M. Yip
Sandia National Laboratories
Livermore, CA

DOE Hydrogen Pipeline Working Group Meeting
Aiken, SC
September 25-26, 2007

Methods for measuring mechanical properties of structural steels in hydrogen

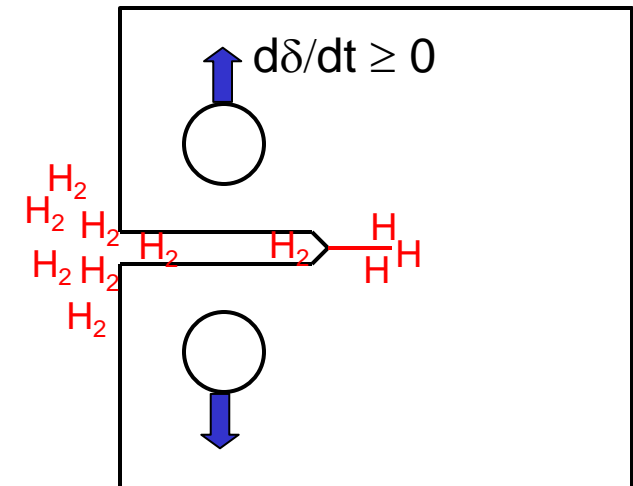
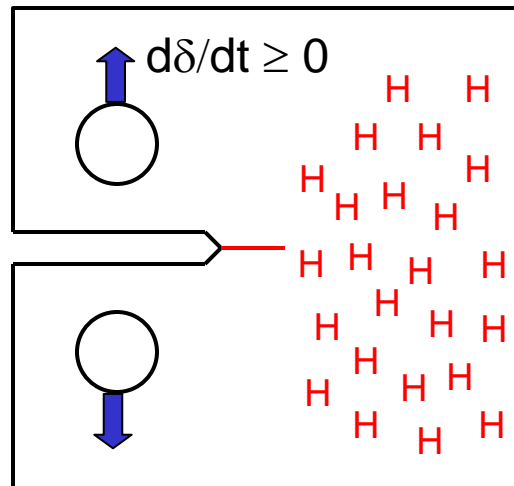
strength of materials:

σ_{UTS} , σ_{YS} , ϵ_f , RA

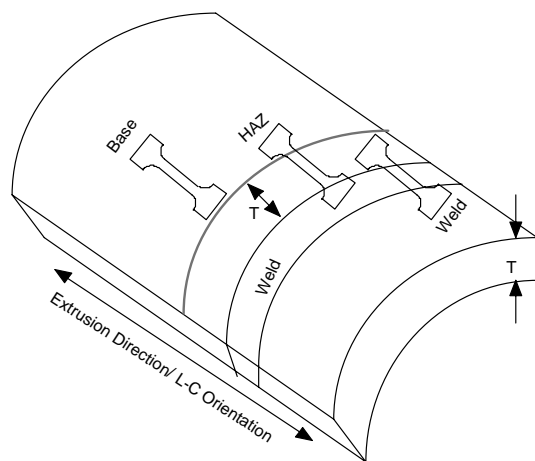


fracture mechanics:

K_{IH} , K_{TH}



Tensile Testing Carbon Steel in H₂



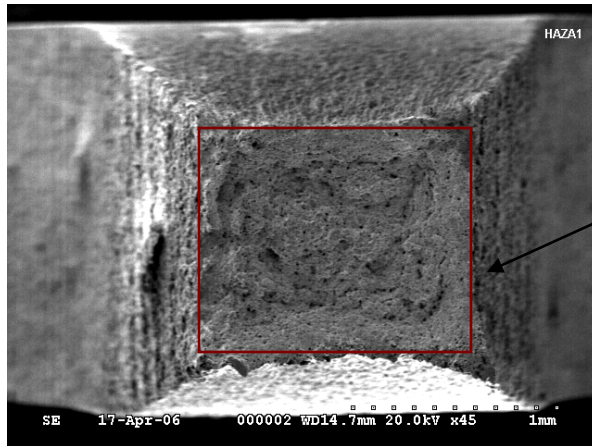
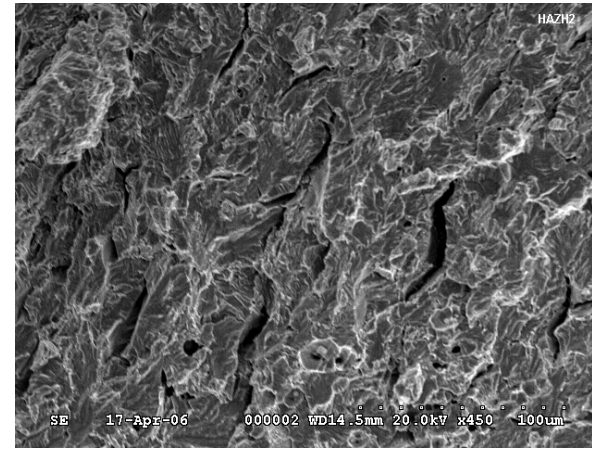
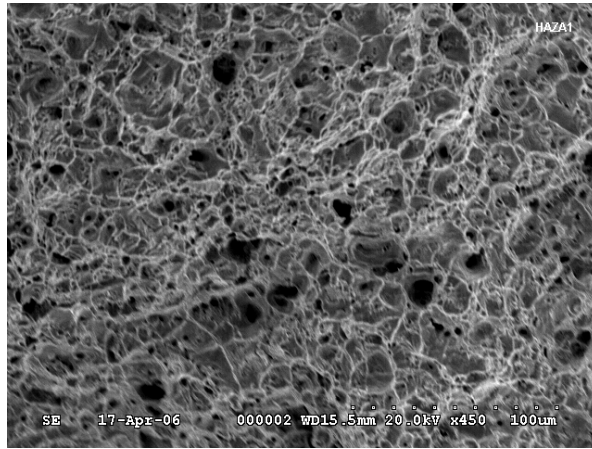
- Alloys: 106 Grade B
- Multi-pass SMAW w/out stress relief
- Specimens machined in 3 conditions: Base metal, Weld and HAZ
- Orientation: L-C
- Atmosphere: 10.3 MPa H₂, ambient pressure Air
- Strain Rate: 10⁻⁴ /sec
- # of samples per matrix point: 6
- Argon purge followed by H₂ pressurization
- Soak (30 min.) at pressure
- Test to failure

Tensile Properties

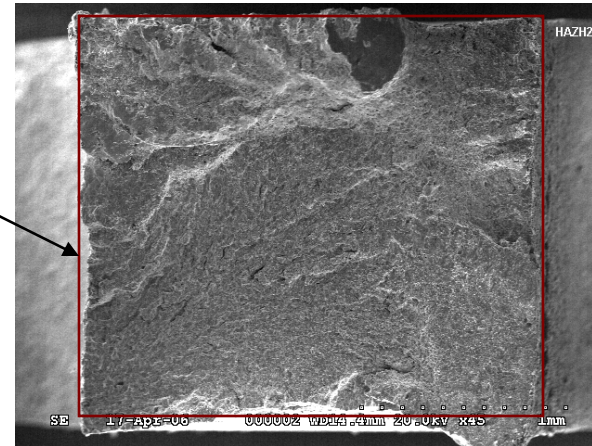
- Mechanical Properties

| | | Average Properties | | | | | | | |
|------|----------------|--------------------|------|--------------|------|----------------------|------|--------------------------|------|
| | | 0.2%Yield (MPa) | Dev. | UTS (MPa) | Dev. | Elong. at failure | Dev. | Reduction of Area (%) | Dev. |
| Base | Air | 355.3 | 15.8 | 484.6 | 8.6 | 0.29 | 0.04 | 68.6 | 1.5 |
| | H ₂ | 357.1 | 32.9 | 486.4 | 17.8 | 0.19 | 0.04 | 30.8 | 5.0 |
| Weld | Air | 343.0 | 20.0 | 490.4 | 9.0 | 0.28 | 0.01 | 74.9 | 2.2 |
| | H ₂ | 350.0 | 16.1 | 480.9 | 12.0 | 0.21 | 0.02 | 30.5 | 6.4 |
| HAZ | Air | 349.3 | 20.8 | 482.3 | 7.6 | 0.27 | 0.04 | 71.0 | 1.7 |
| | H ₂ | 338.2 | 18.5 | 475.5 | 9.6 | 0.19 | 0.04 | 30.4 | 6.8 |

Fractography



Fracture
Surface
Area



Air

Hydrogen

HAZ Samples

Reduction of Area reduced when tested in hydrogen

Crack propagation resistance of X100 steel

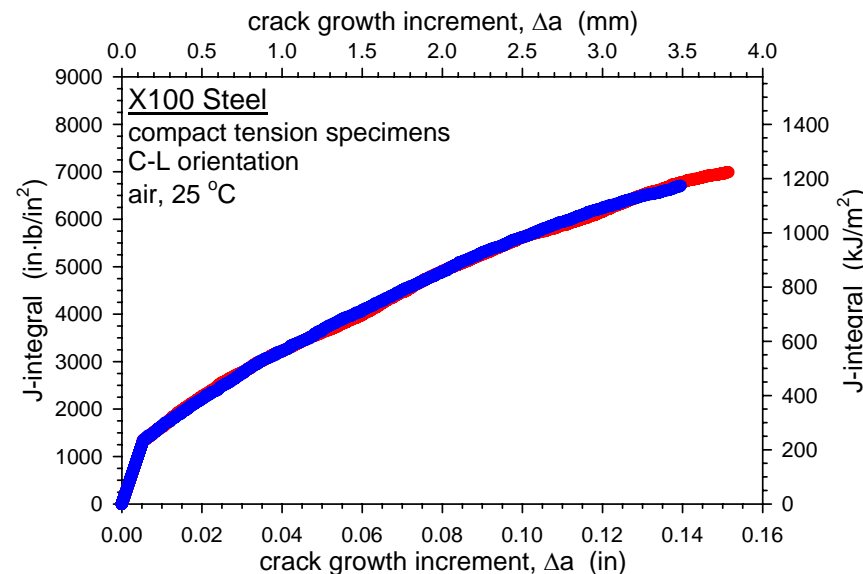
- Alloy composition

| C | Mn | Si | P | S | Ti | V | Ni | Cu | Mo | Cr |
|-------|------|------|-------|--------|------|--------|------|------|------|------|
| 0.073 | 1.86 | 0.11 | 0.009 | <0.002 | 0.01 | <0.005 | 0.48 | 0.27 | 0.17 | 0.02 |

- Yield strength

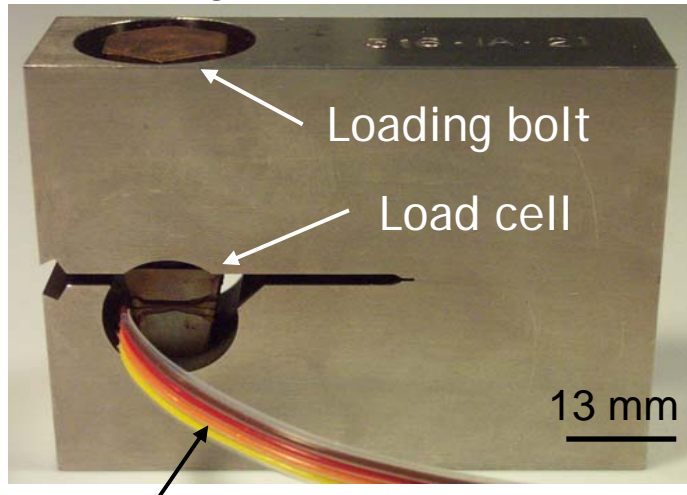
- 96 ksi (662 MPa) in longitudinal orientation
- 114 ksi (787 MPa) in circumferential orientation

- Crack growth resistance curves in air

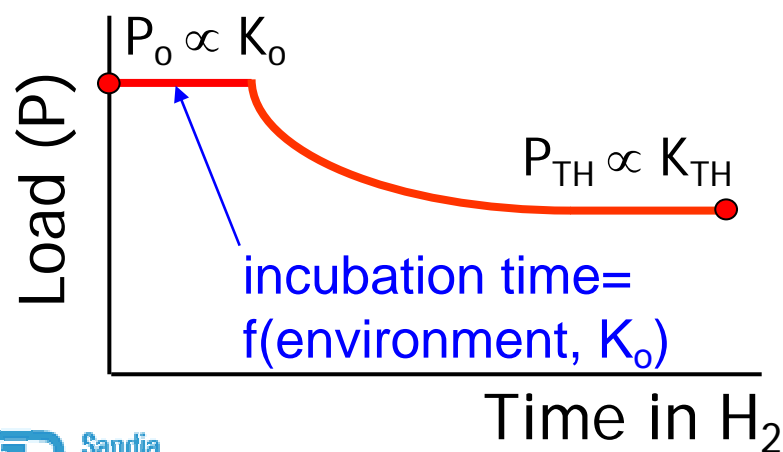


Measurement of sustained-load cracking thresholds

wedge opening load (WOL)
cracking threshold specimen

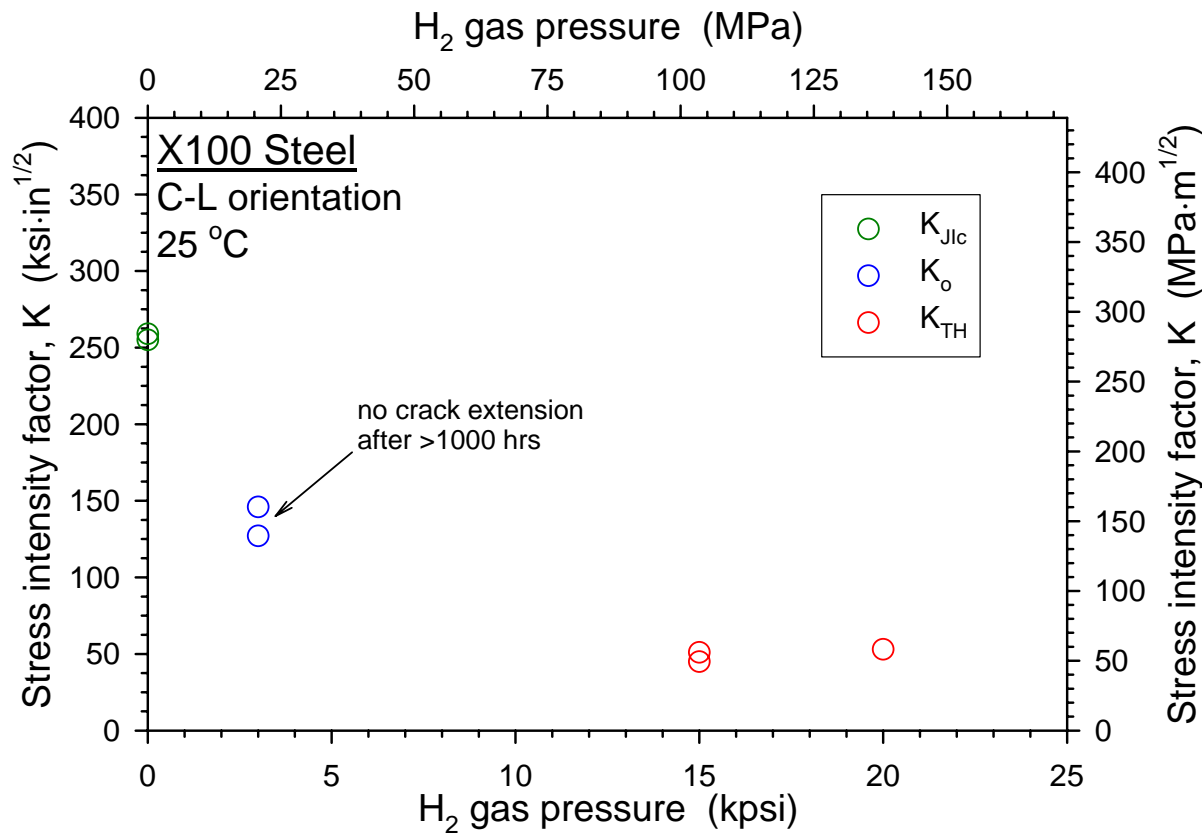


strain gage leads (Excitation and DAQ)

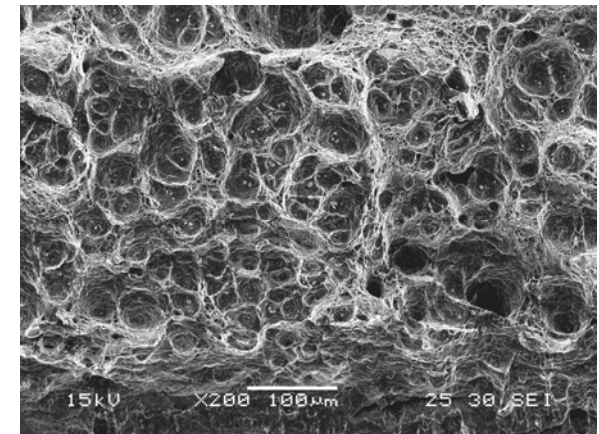


- Specimen loaded to $K_0 > K_{TH}$ using bolt while contained in glove box (Ar with ~1 ppm O₂)
- Loaded specimen exposed to H₂, crack extends after incubation time
- Crack arrests at $K = K_{TH}$

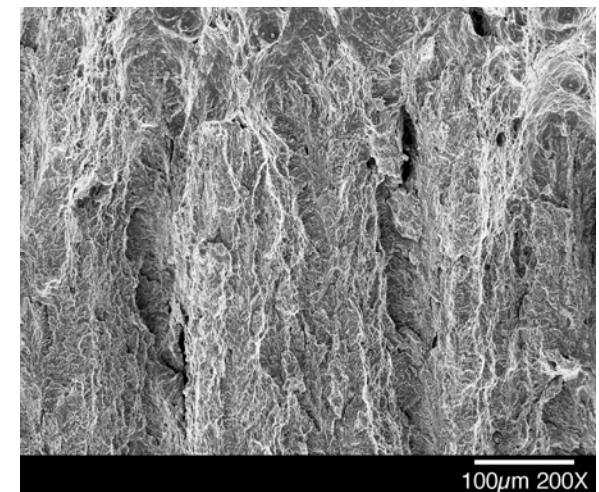
High-pressure H₂ gas severely degrades crack propagation resistance of X100 steel



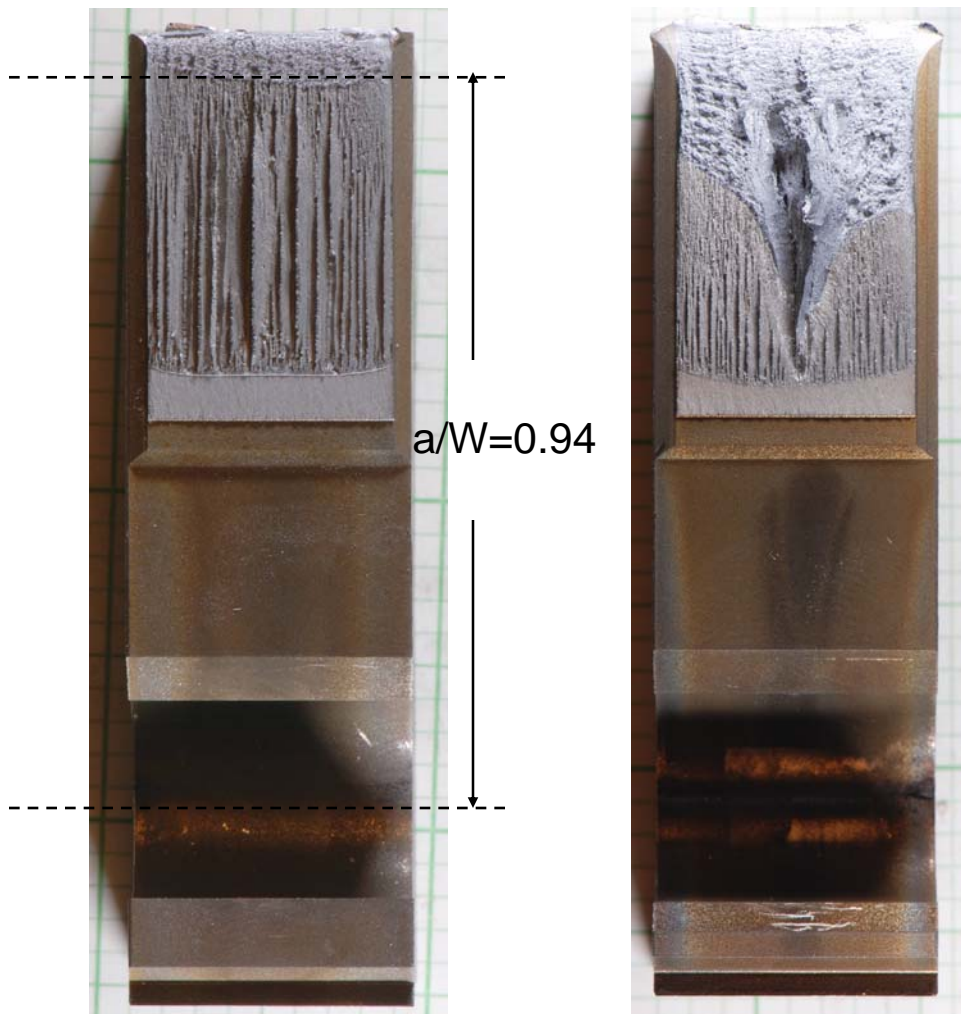
X100 in air



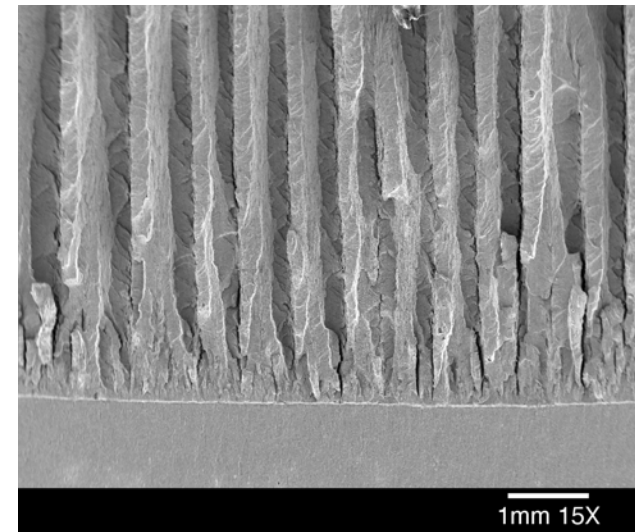
X100 in 15 kpsi H₂ gas



Images of fracture surfaces from X100 tested in H_2 show long crack lengths and delaminations

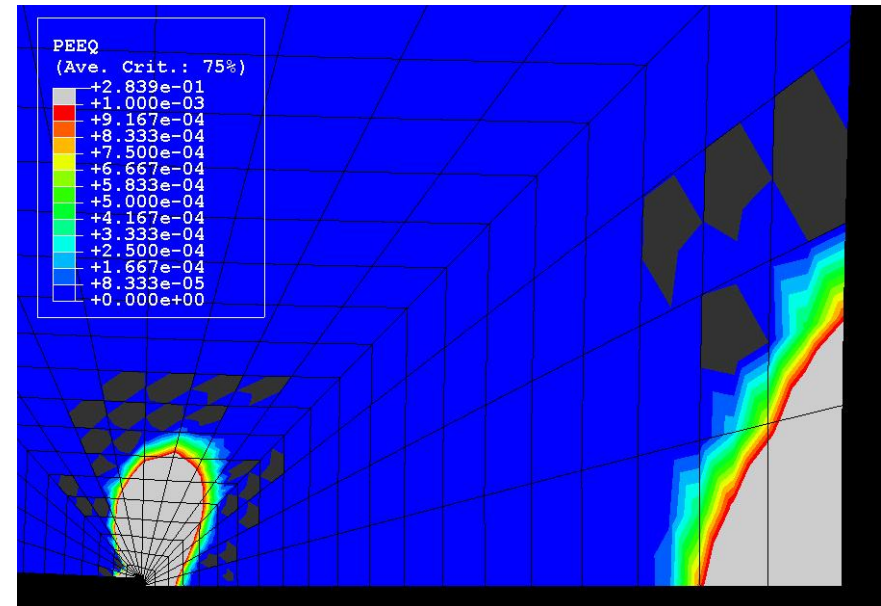
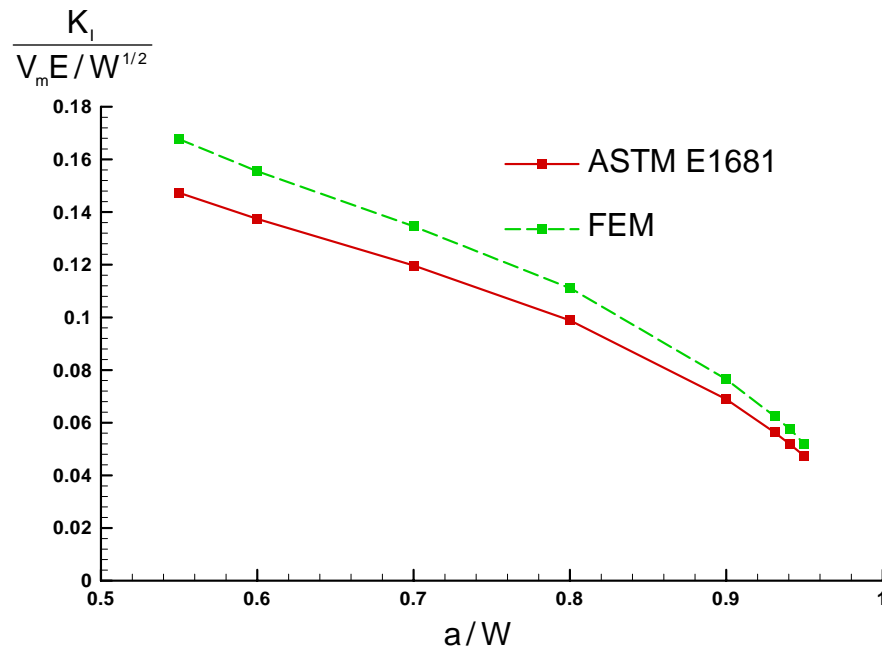


H_2 -assisted crack extension from precrack



FEM calculations verify K solution and demonstrate K dominance for long cracks in WOL specimen

$a/W = 0.941$



ASTM E1681: $K_I = 57.5 \text{ MPa}\sqrt{\text{m}}$

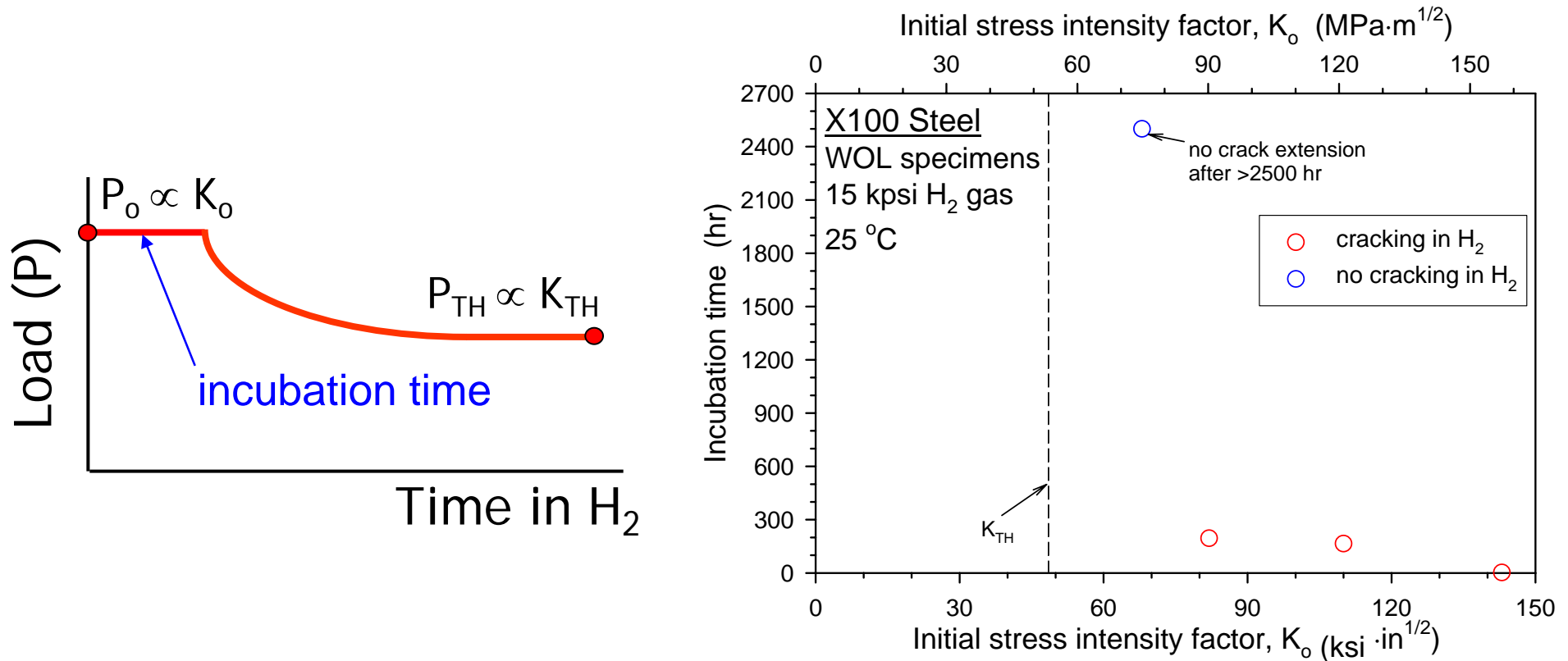
FEM elastic: $K_I = 63.8 \text{ MPa}\sqrt{\text{m}}$

FEM plastic: $J = 16.0 \text{ kJ/m}^2$

$$K_{IJ} = \sqrt{\frac{JE}{1-\nu^2}} \quad K_{IJ} = 62.2 \text{ MPa}\sqrt{\text{m}}$$

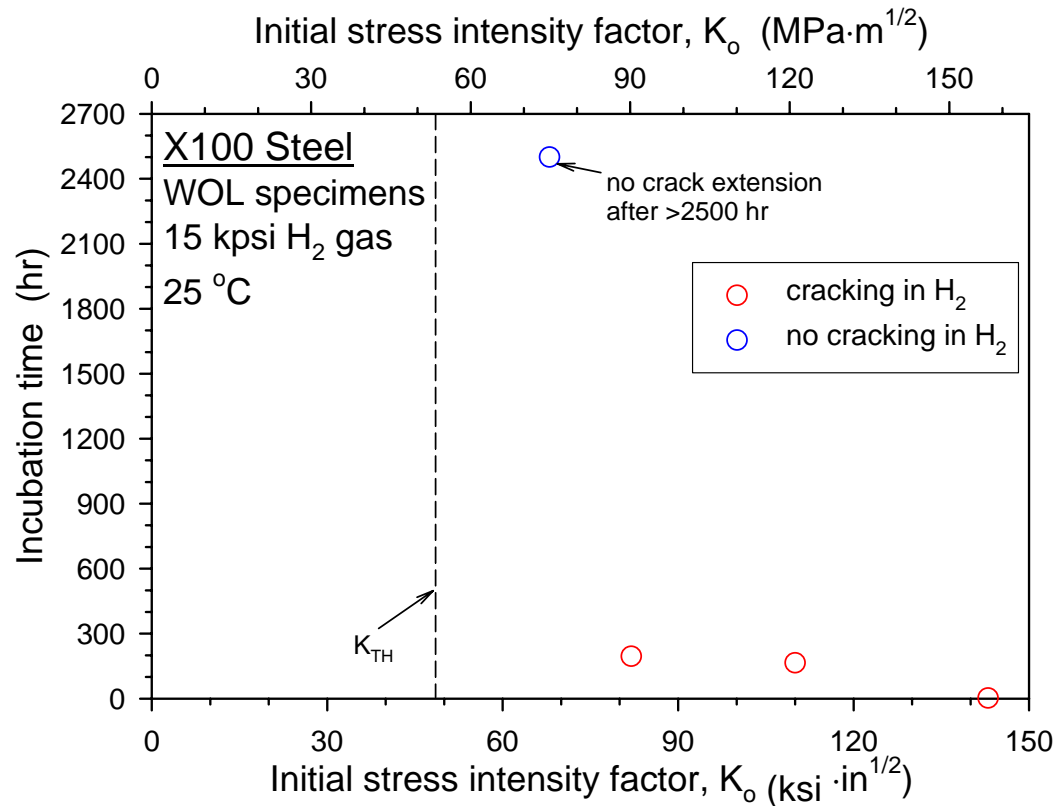
Calculations by M. Dadfarnia and P. Sofronis

Incubation time for crack extension depends on K_0

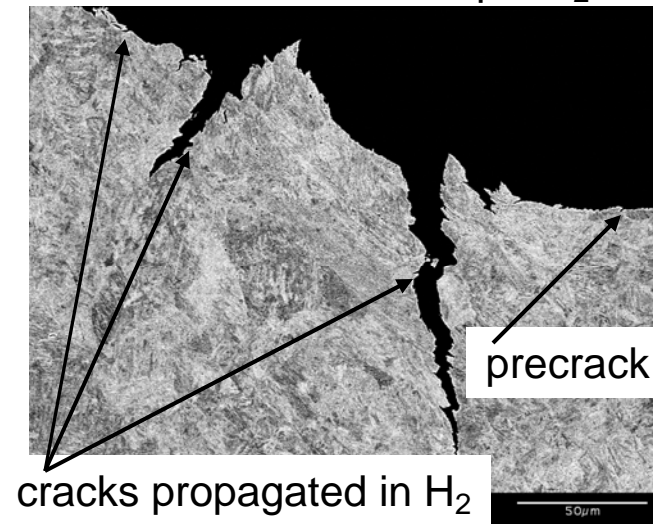


Measurement of sustained-load cracking threshold in H₂ cannot be assured unless crack propagates

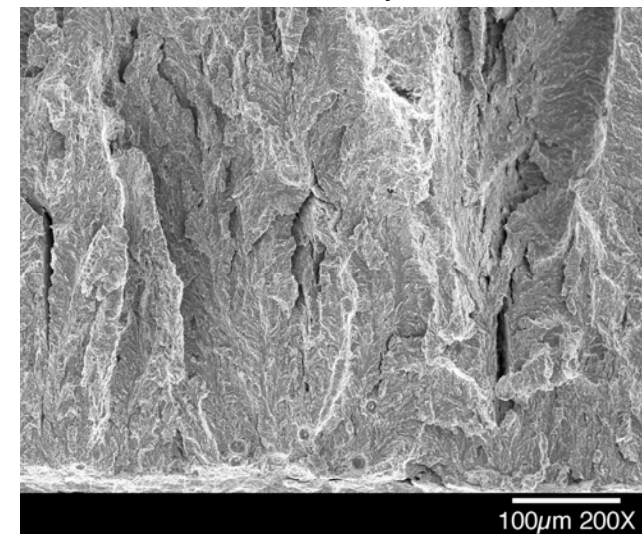
Crack branching may account for no crack extension in specimens with $K_0 > K_{TH}$



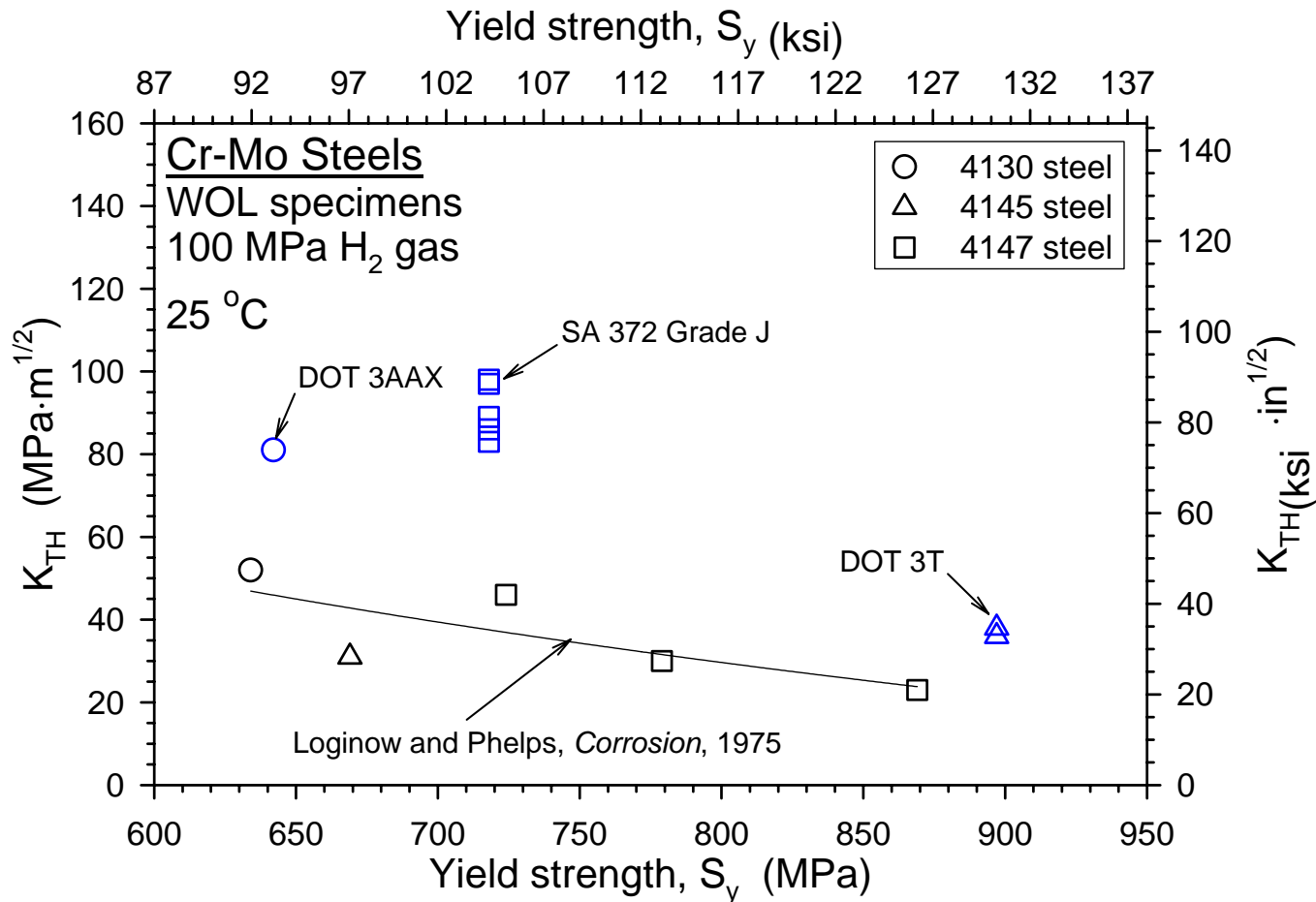
SA 372 Gr. J in 15 kpsi H₂



X100 in 15 kpsi H₂



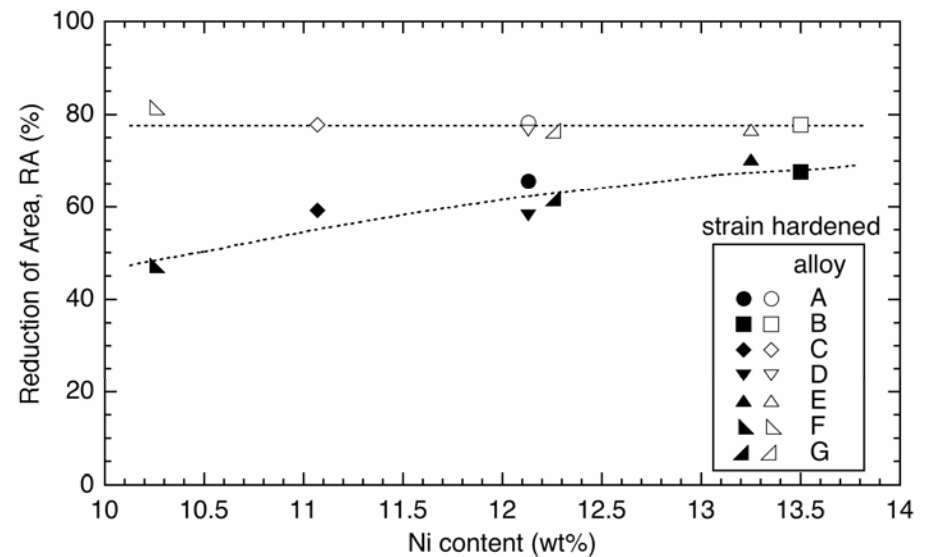
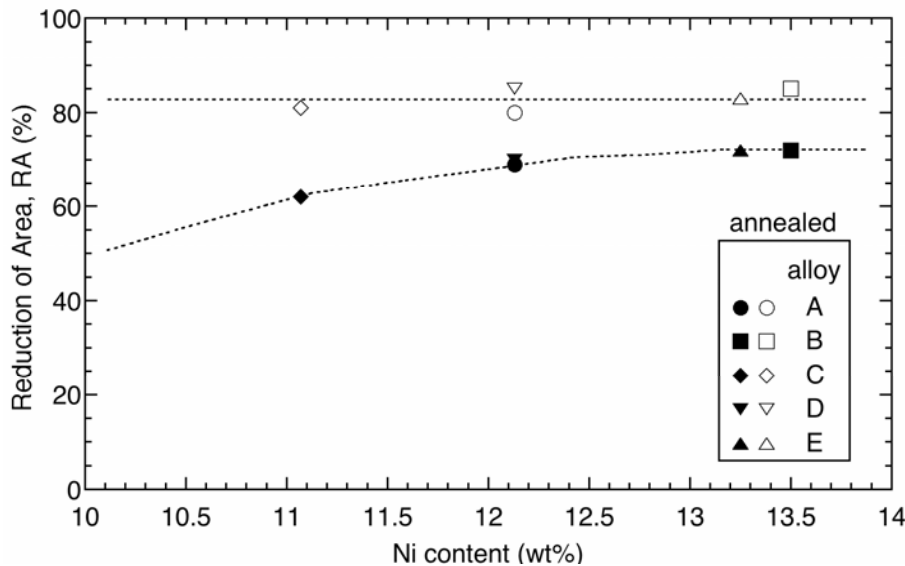
Low-alloy steels for pressure vessels: sustained-load cracking thresholds in H₂ gas



Thresholds in modern Cr-Mo steels are higher than thresholds in older steels

Stainless steels for piping: tensile fracture results for specimens precharged in hydrogen gas

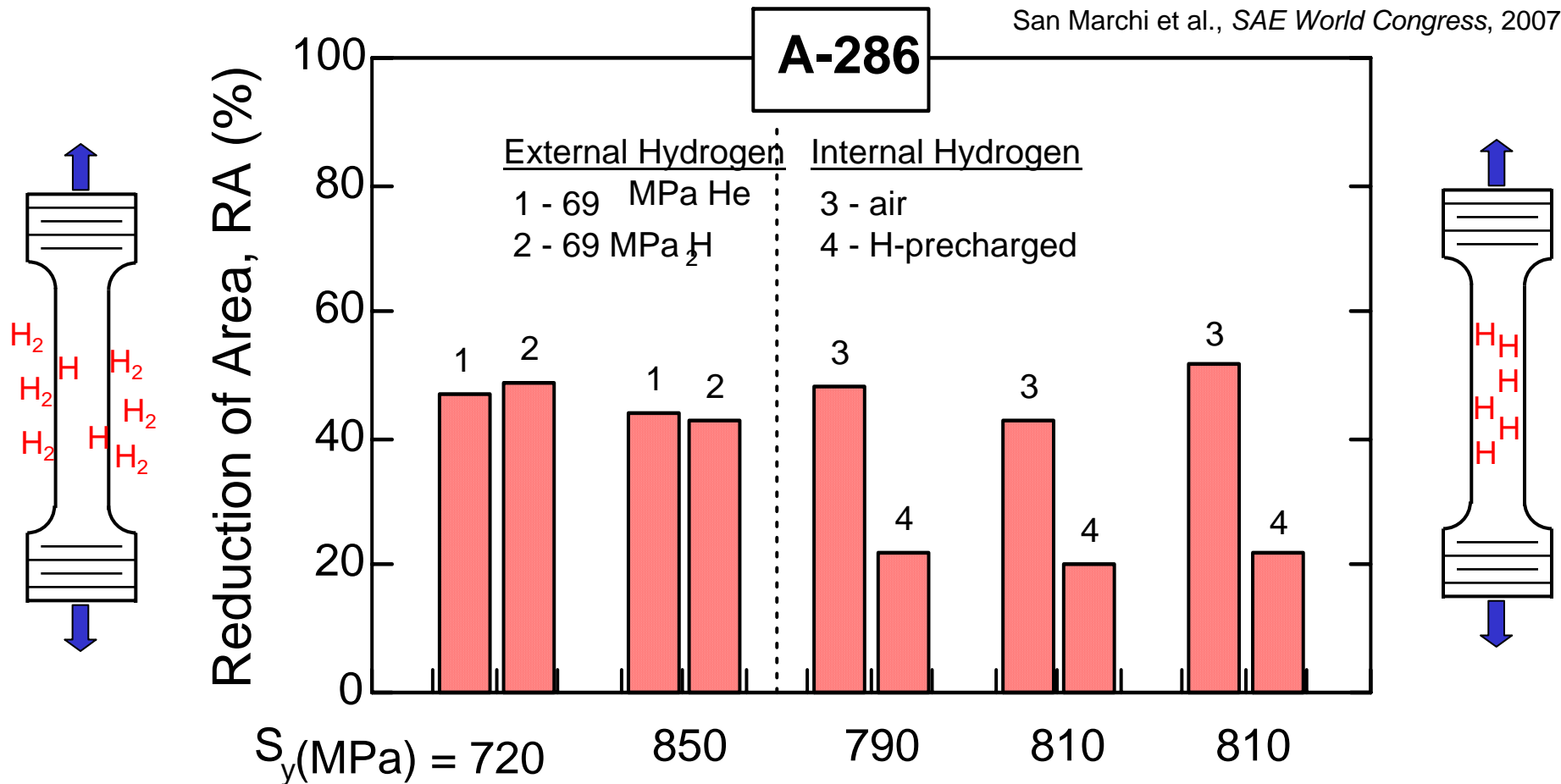
316 stainless steels with ~140 wppm hydrogen



Tensile fracture resistance of H-precharged specimens is sensitive to nickel content but not strain hardening

Stainless steels tested in hydrogen gas vs precharged show different tensile fracture behavior

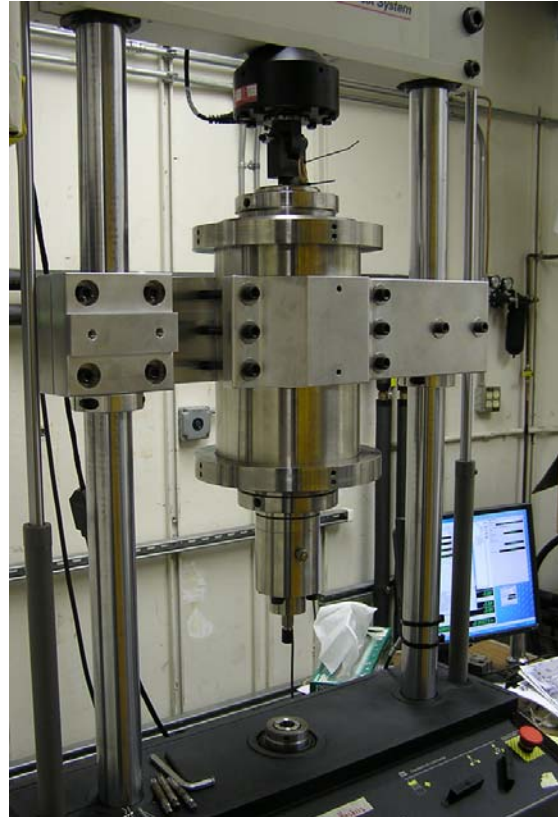
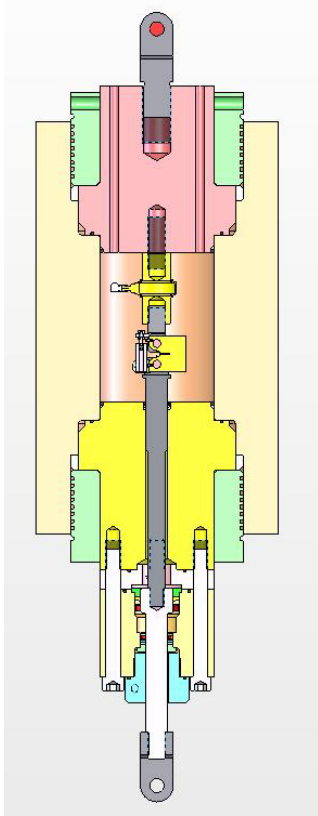
San Marchi et al., SAE World Congress, 2007



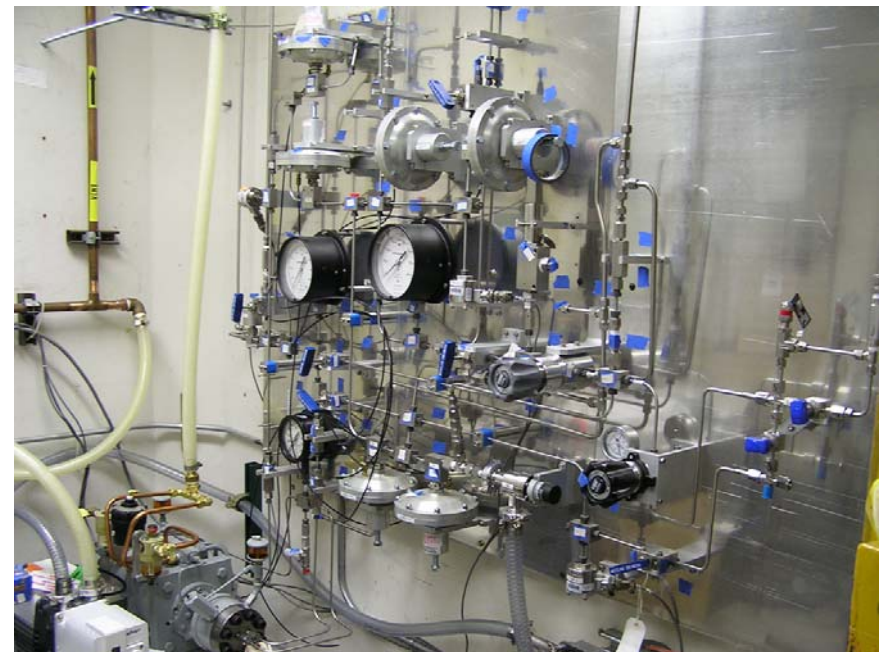
Stable stainless steels may not exhibit loss in RA when strained in H_2 due to limited H uptake

System for measuring fatigue crack growth in high-pressure H₂ nearly complete

vessel on mechanical test frame



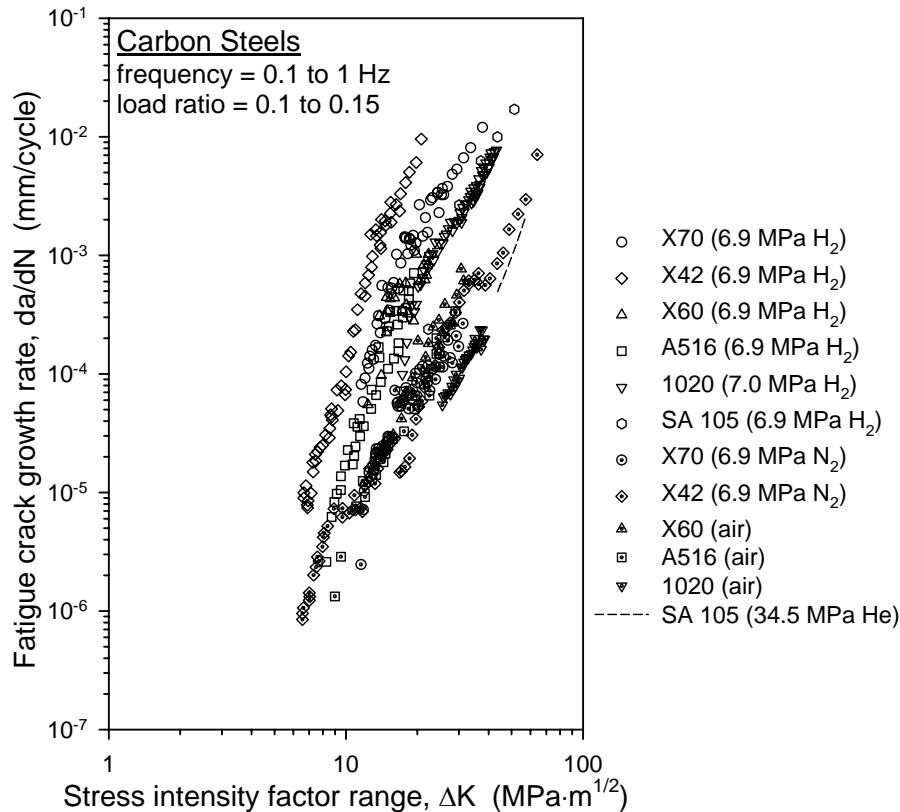
gas manifold



- Pressure vessel can contain H₂ gas up to 20 kpsi (138 MPa)

What is metric for qualifying steels for H₂ pipelines?

Tech. Ref. Hydrogen Compatibility of Materials,
www.ca.sandia.gov/matlsTechRef

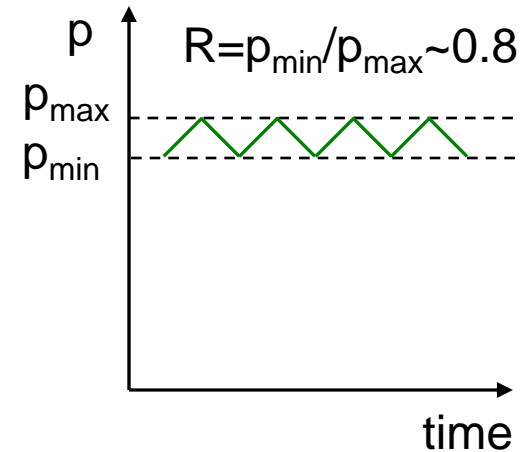
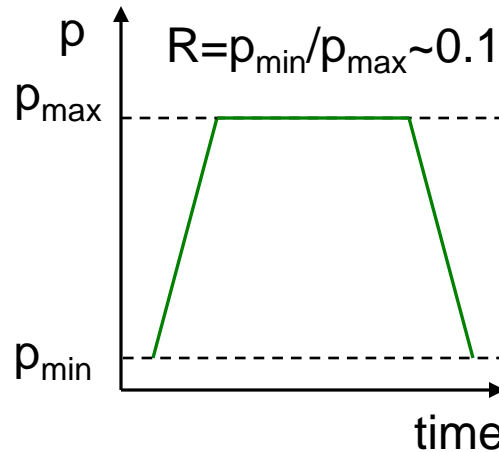
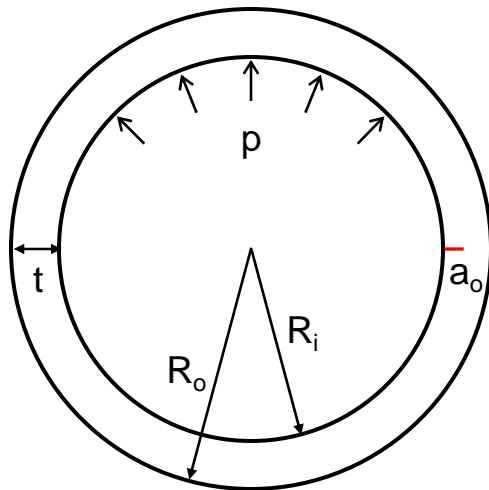


- Fatigue crack growth rates in H₂ greater than growth rates in air or inert gas
- Fatigue crack growth rates in H₂ vary by factor of 10

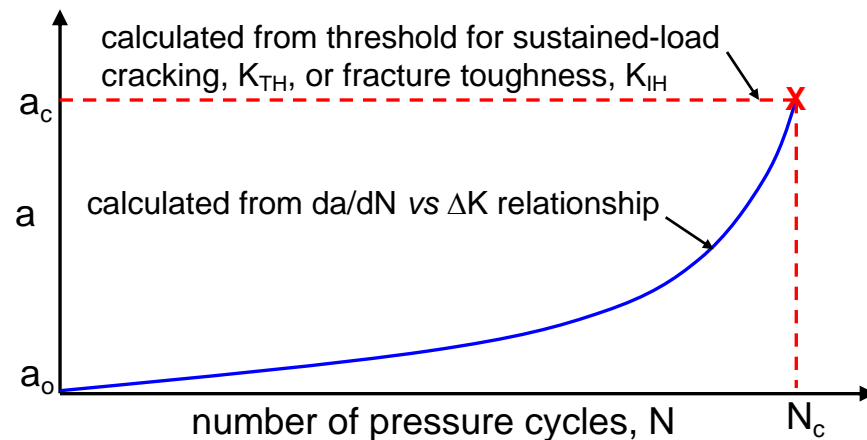
What is acceptable fatigue crack growth rate?

Assess fatigue crack growth data by conducting fracture mechanics analysis of pipeline

- Assume pipeline subjected to pressure cycling



- Calculate number of pressure cycles for crack to reach critical depth

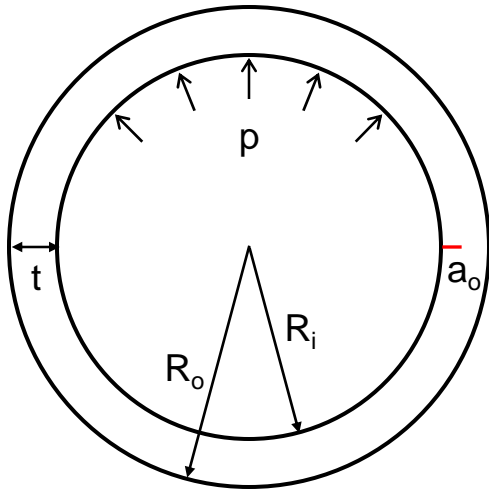


Assume steel, dimensions, and maximum pressure are similar to current H₂ pipelines

- Data for Air Products H₂ pipelines

(ASME, *Hydrogen Standardization Interim Report*, 2005)

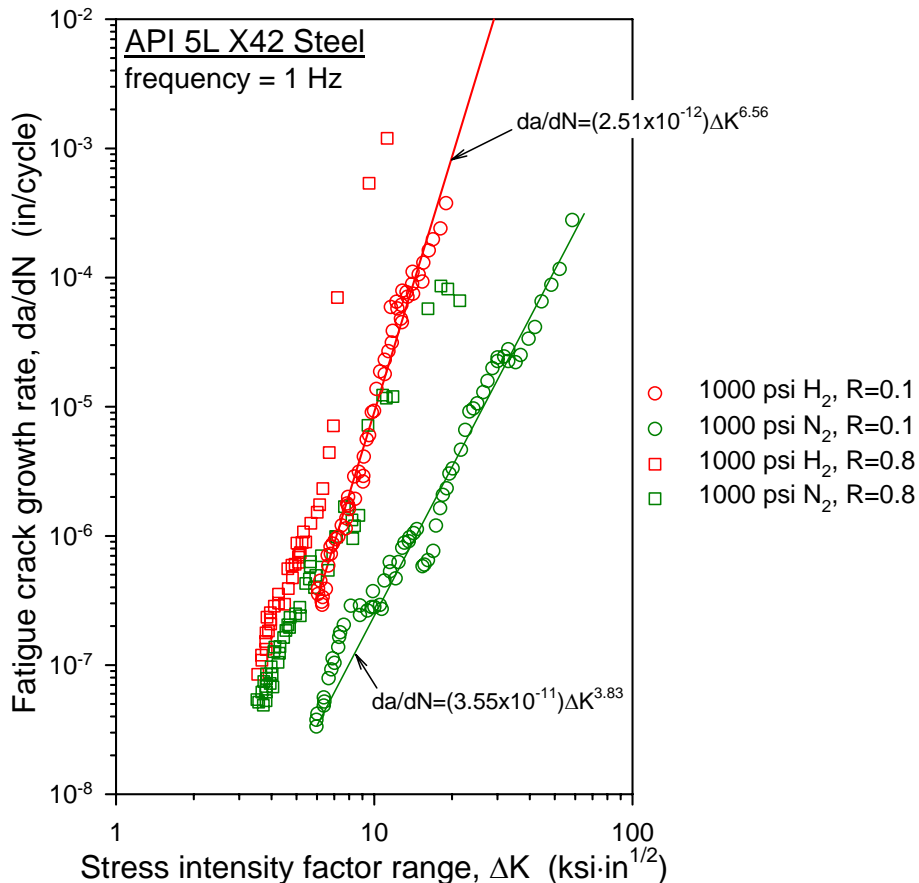
- API 5L X42 steel, manufactured with ERW
- Inner radius, $R_i = 6.0$ in
- Wall thickness, t , from 0.219 to 0.500 in



- Maximum operating pressure, $p = 1500$ psi
- Assume existing defect with depth a_o and length parallel to pipeline axis
 - Simulates defect along manufacturing (seam) weld

Select appropriate material property data

Cialone et al., *Met Trans A*, 1985

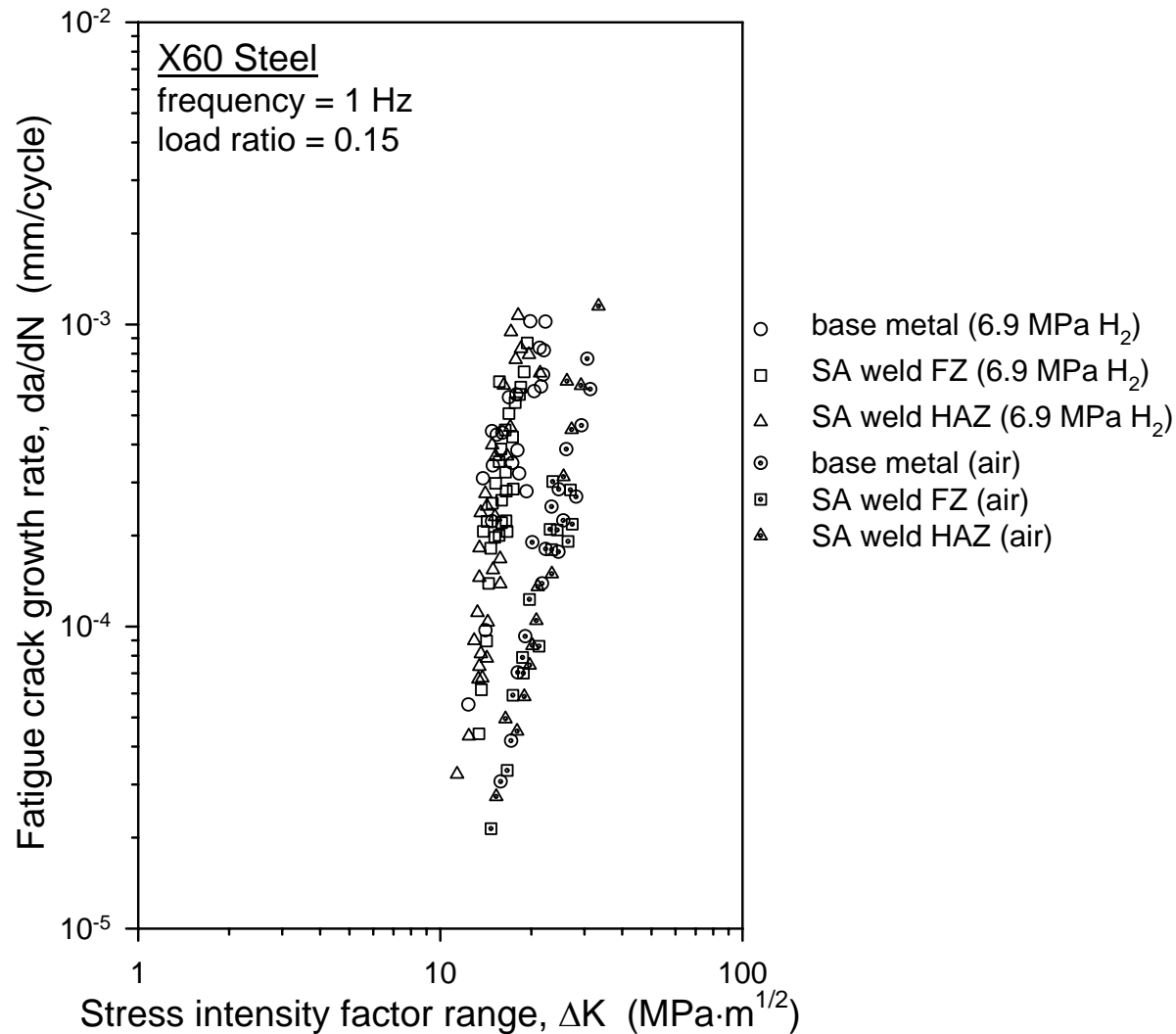


- Need da/dN vs ΔK for X42 ERW in 1500 psi H₂ gas
 - assume data for 1000 psi H₂ gas representative
 - assume data for weld and base metal are similar
- Need da/dN vs ΔK at relevant R ratio and frequency
 - pressure cycling parameters for pipeline unknown
 - use data at R=0.1 and frequency=1 Hz
- Need fracture toughness for X42 ERW in 1500 psi H₂ gas
 - $K_{IH} = 44 \text{ ksi}\cdot\text{in}^{1/2}$ for weld HAZ in 1000 psi H₂

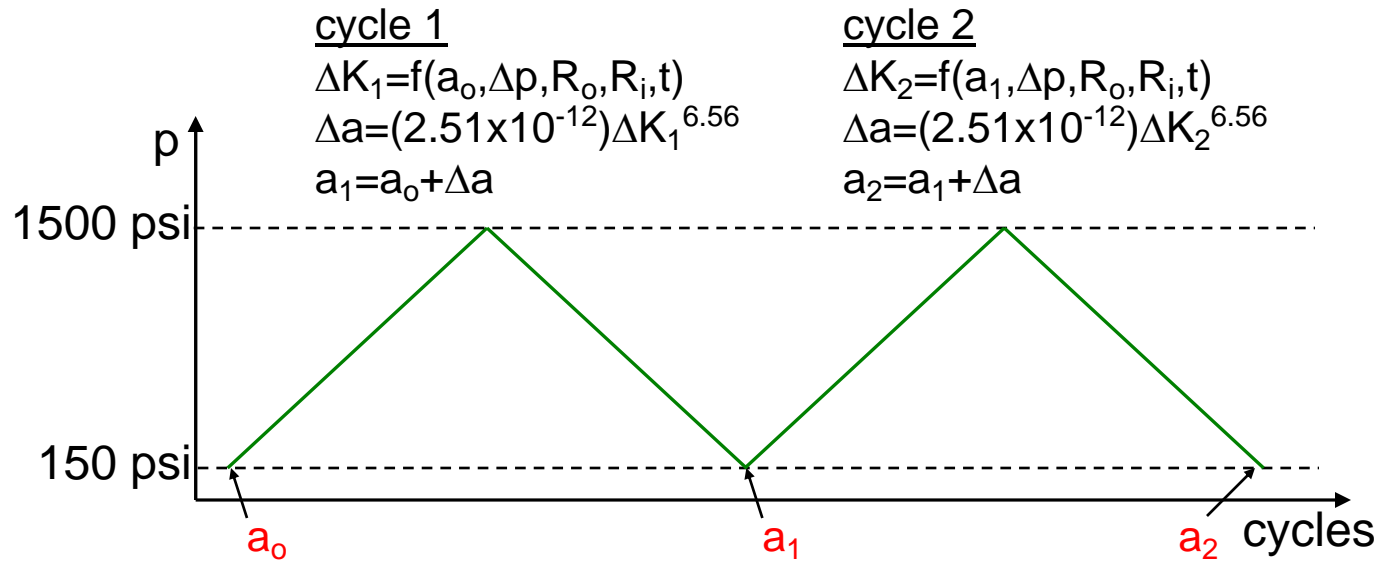
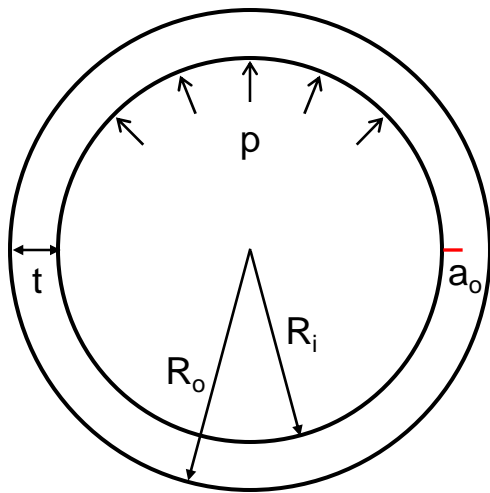
(Cialone et al., *ASTM STP 962*, 1988)

Fatigue crack growth rates similar in base metal, fusion zone, and HAZ for X60 steel

Wachob, NASA-CR-166334, 1981

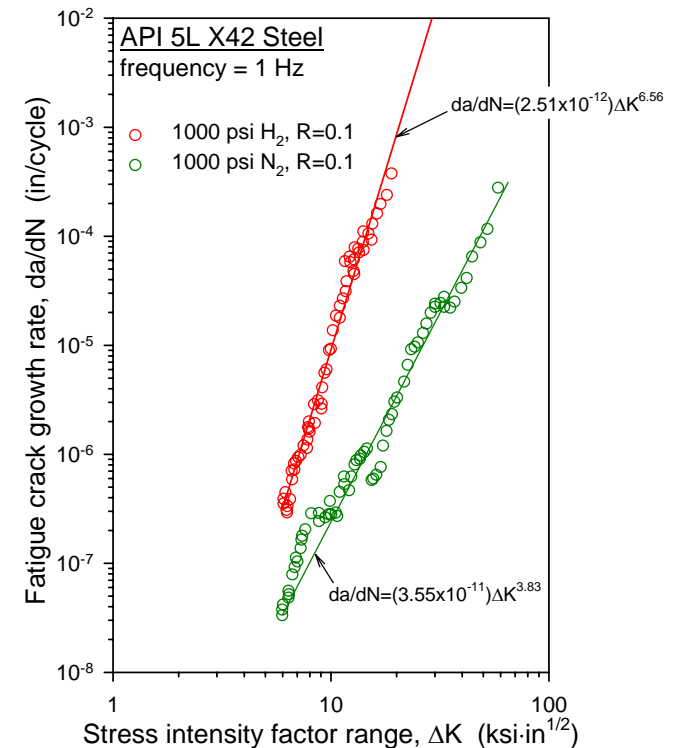
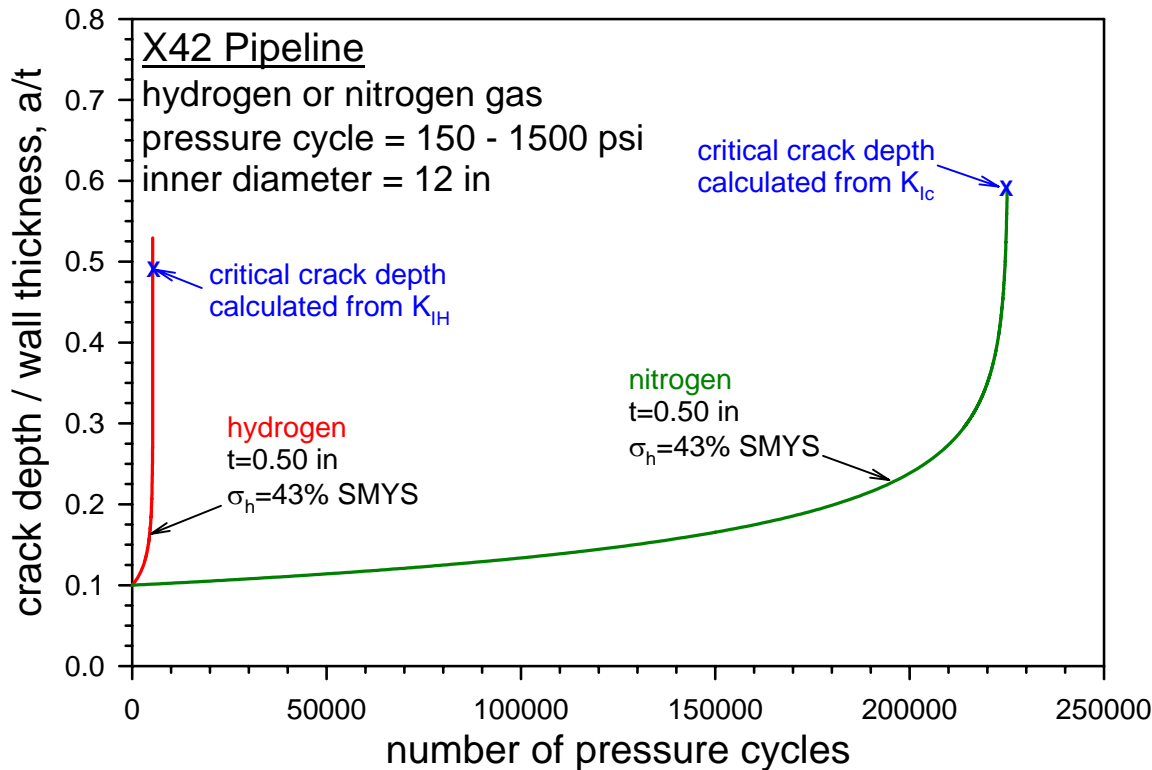


Calculate number of pressure cycles to reach critical crack depth for three cases



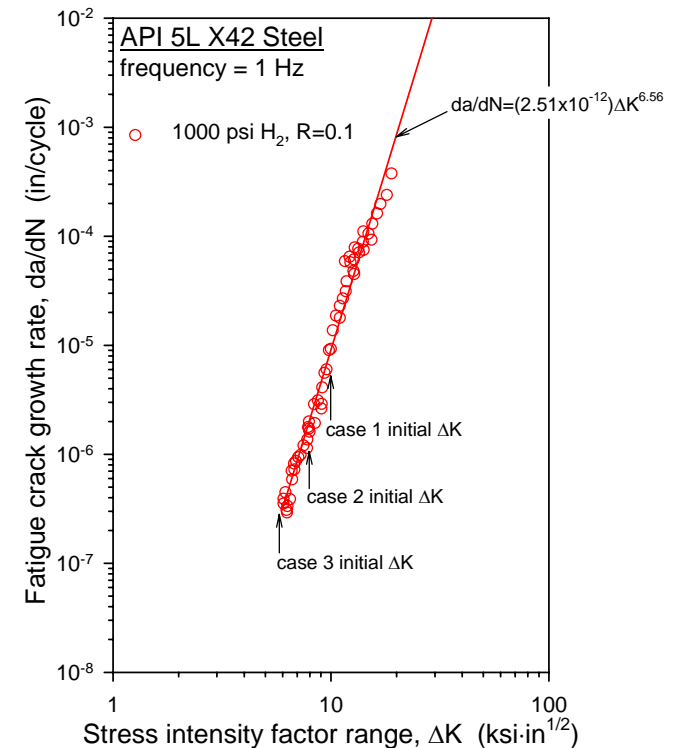
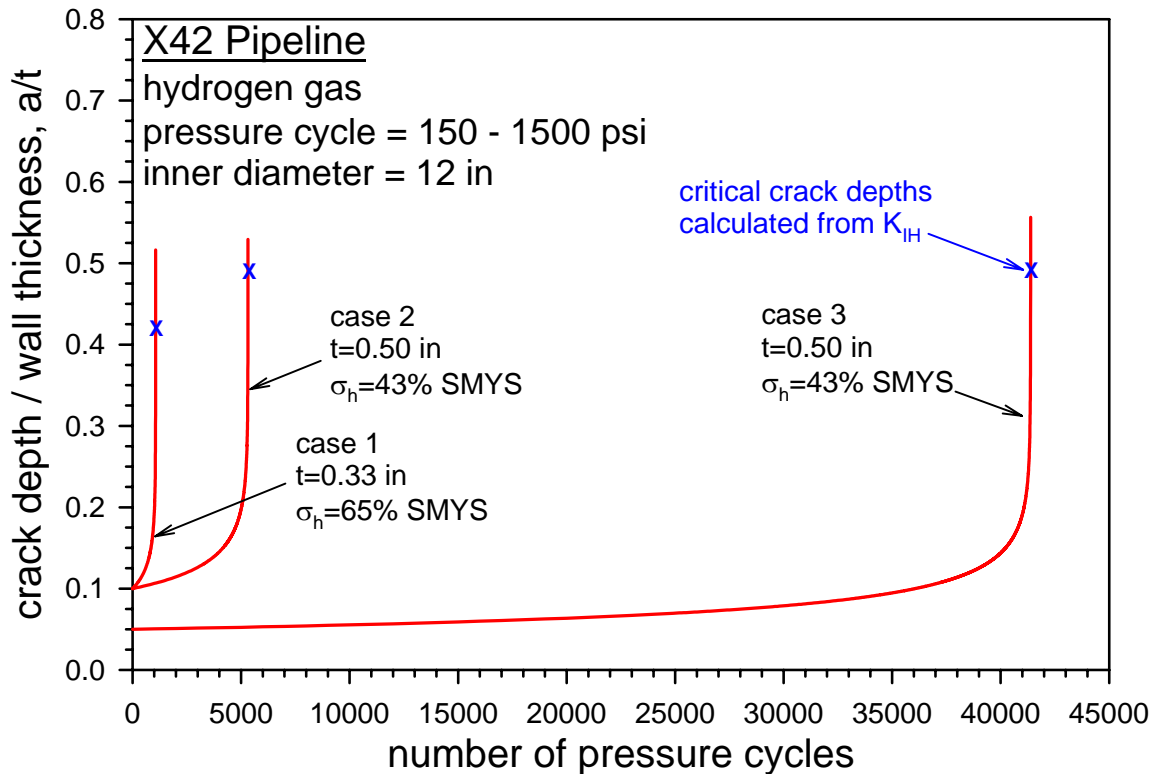
- Case 1: $t=0.330$ in ($\sigma_h=65\%$ SMYS) and $a_o/t=0.10$
- Case 2: $t=0.500$ in ($\sigma_h=43\%$ SMYS) and $a_o/t=0.10$
- Case 3: $t=0.500$ in ($\sigma_h=43\%$ SMYS) and $a_o/t=0.05$

Number of pressure cycles vs crack depth relationships: H₂ compared to inert gas



Number of cycles to critical crack depth reduced by about factor of 40 in H₂ compared to inert gas

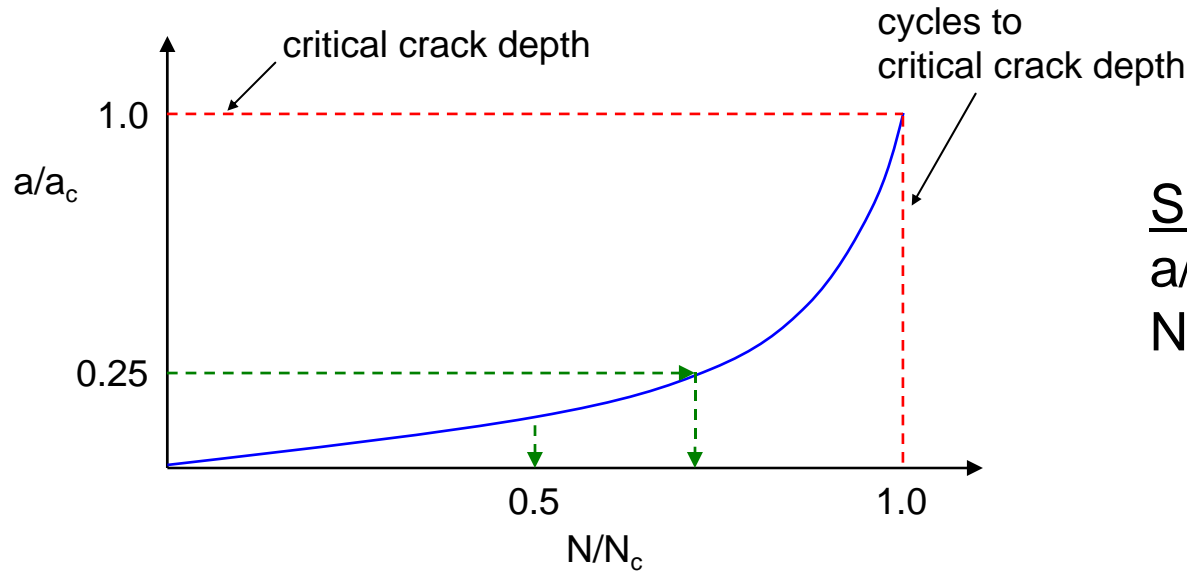
Number of pressure cycles vs crack depth relationships in H₂ for varying dimensions



Number of cycles to critical crack depth is sensitive to applied stress and initial crack depth

ASME applies safety factors on critical crack depth and number of cycles to critical crack depth

M.D. Rana et al., ASME PVP, 2007



Safety factors:

$$a/a_c = 0.25$$

$$N/N_c = 0.5$$

| Environment | t (in) | σ_h | a_0/t | cycles to $0.25 \times a_c$ | $0.5 \times N_c$ |
|----------------|--------|------------|---------|--------------------------------|------------------|
| H ₂ | 0.330 | 65% SMYS | 0.10 | 170 | 535 |
| H ₂ | 0.500 | 43% SMYS | 0.10 | 2,735 | 2,660 |
| H ₂ | 0.500 | 43% SMYS | 0.05 | 38,790 | 20,690 |
| inert | 0.500 | 43% SMYS | 0.10 | 125,260 | 112,545 |

Lifetime of steel hydrogen pipeline depends on both material properties and structural design

- Fatigue crack growth law
 - $da/dN=C\Delta K^n$ in stage II
- Fracture toughness or threshold for sustained-load cracking
 - does not significantly affect cycles to critical crack depth
- Wall thickness
 - affects stress (ΔK)
- Initial crack depth
 - depends on detection limit of NDE
- Operating pressure
 - affects stress (ΔK) and material properties

Fatigue crack growth rates depend on load cycle profile

Walter and Chandler, *Effect of Hydrogen on Behavior of Materials*, 1976

