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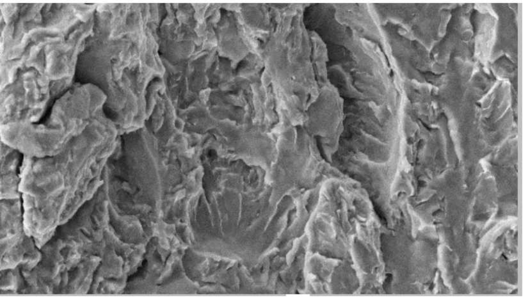

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Oxygen Impurity Effects on Hydrogen Assisted Fatigue and Fracture of X100 Pipeline Steel

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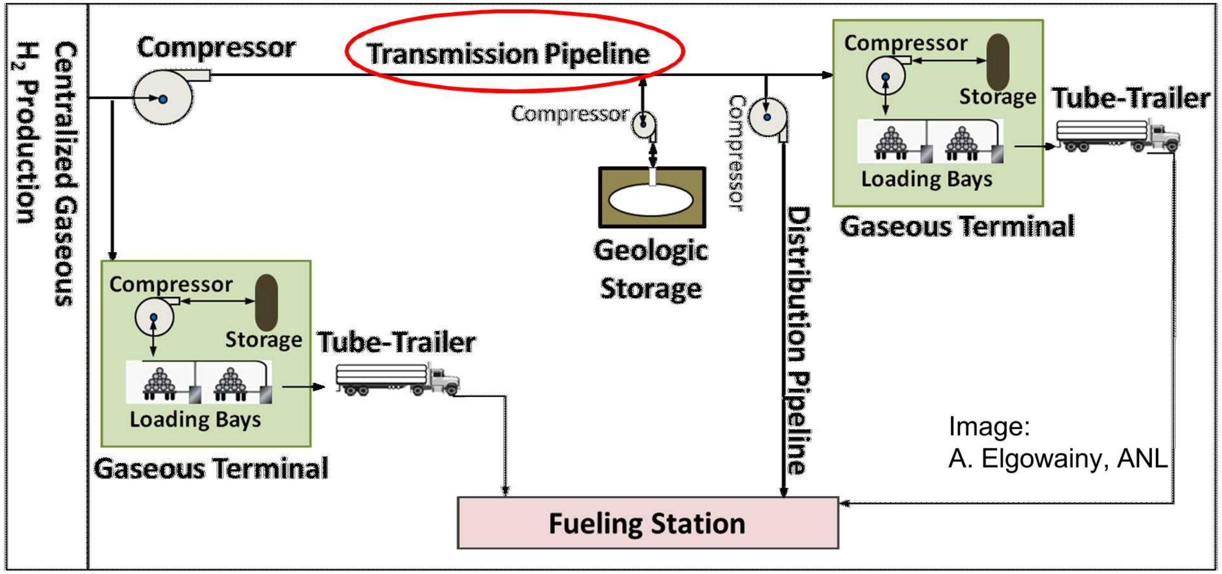
Background: Steel pipelines are used to transport gaseous hydrogen

Gaseous Delivery Pathways



- 1,500 miles of steel hydrogen pipeline in the U.S. today!
- ASME B31.12 Code used for hydrogen pipeline design

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Structural materials are central to the cost and reliability of H_2 infrastructure.

Background: Current H_2 Pipeline Design Codes

- ASME B31.8 ***Natural Gas*** pipeline thickness

$$t = \frac{PD}{2SFET}$$

F= design factor = 0.72 (Class 1)

- ASME B31.12 ***Hydrogen*** pipeline thickness
  - Prescriptive Design Method

$$t = \frac{PD}{2SFETH_F}$$

P = design pressure = 3ksi (21 MPa)
S = specified min yield stress
t = thickness
D = outside diameter = 24 in (610mm)
E = longitudinal joint factor = 1
T = temp derating factor = 1
F= design factor = 0.5 (Class 1)
H_F=Materials Performance Factor

Table IX-5A Carbon Steel Pipeline Materials Performance Factor, H_f

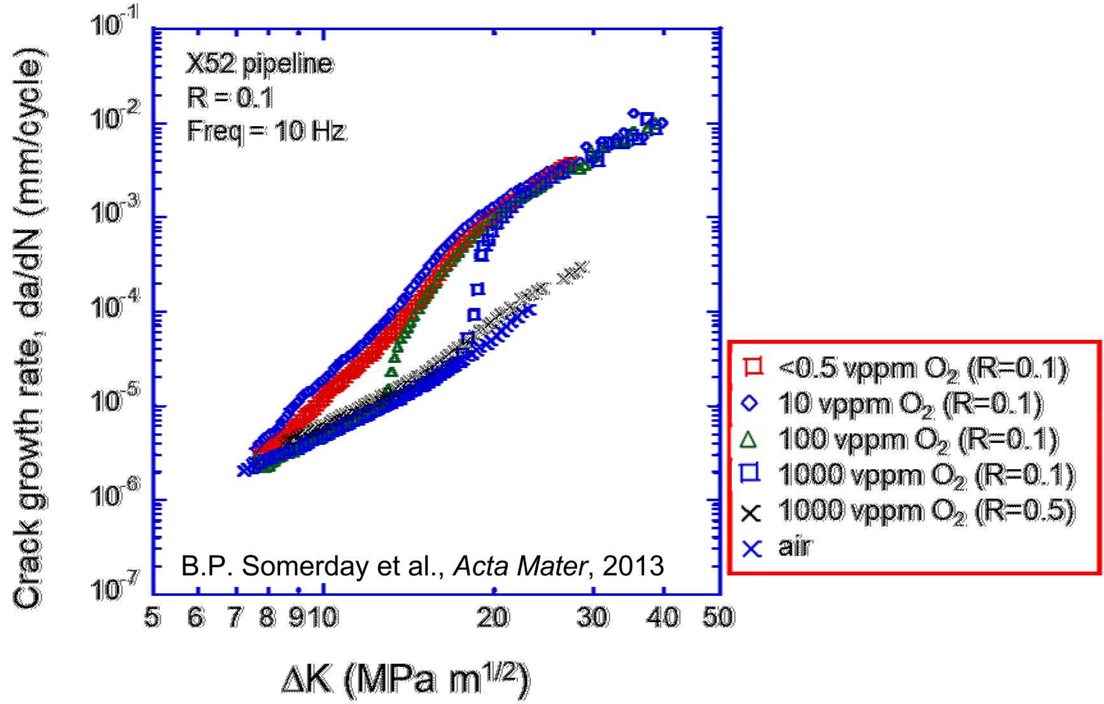
Specified Min. Strength, ksi		System Design Pressure, psig						
Tensile	Yield	≤1,000	2,000	2,200	2,400	2,600	2,800	3,000
66 and under	≤52	1.0	1.0	0.954	0.910	0.880	0.840	0.780
Over 66 through 75	≤60	0.874	0.874	0.834	0.796	0.770	0.734	0.682
Over 75 through 82	≤70	0.776	0.776	0.742	0.706	0.684	0.652	0.606
Over 82 through 90	≤80	0.694	0.694	0.662	0.632	0.610	0.584	0.542

Current Design codes (ASME B31.12) apply thickness premiums to higher strength H_2 pipelines.

Research Question: Are there ways to reduce thickness premiums?

Background: Oxygen impurities can mitigate hydrogen assisted fatigue

- X52 pipeline steel exhibited delayed or in some conditions suppression of hydrogen assisted fatigue (Somerday *et al.* 2013)
- Combination of loading conditions and environment
  - Oxygen partial pressure
  - Frequency, R-ratio, da/dN_{inert}
- Analytical model to describe critical da/dN



$$\left[\frac{da}{dN}f\right]_{crit} = \frac{0.3\chi D p_{tot}(1-\nu^2)}{\pi z_{pass}\theta_O R_g T E \sigma_0}\left(\frac{\Delta K}{\sqrt{a^*}(1-R)}\right)^2$$

da/dN = inert crack growth rate
f = frequency
χ = oxygen concentration
ν = Poisson's ratio = 0.3
T = temperature
z_{pass} = number of oxygen layers required to passivate surface
θ_O = density of oxygen atoms in FeO layer
a* = crack length extending from precrack start notch
E = elastic modulus
s_o = yield strength
R = load ratio
R_g = gas constant

Is this effect applicable to high strength pipeline steel (e.g. X100) where greater cost savings can be achieved?

Purpose: Evaluate oxygen effects on high strength X100 pipeline

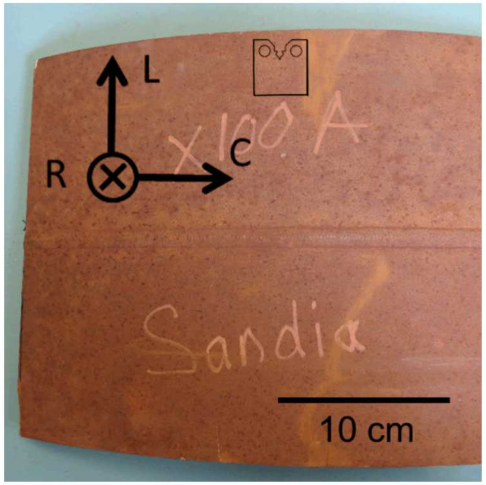
- X100 pipe offers significant cost savings over X52 by use of higher pressures at thinner walls
  - Does X100 have similar behavior to X52 with respect to oxygen?
- Fracture toughness is critical for pipeline design
  - Does oxygen mitigate hydrogen degradation of fracture toughness?
- Oxygen can mitigate hydrogen assisted fatigue
  - What conditions (frequency, R-ratio, test pressure) can oxygen mitigate HA-FCG?

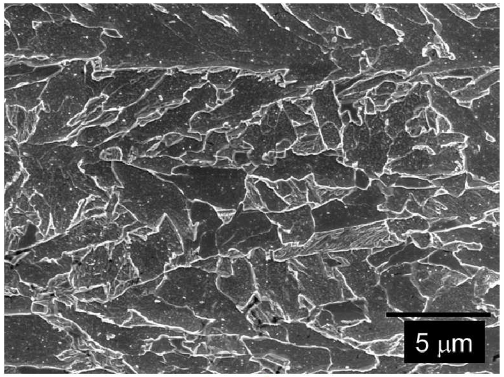
Understanding the loading environments which result in suppression of HA-FCG may lead to reduction of thickness premiums→ Lowering pipeline costs

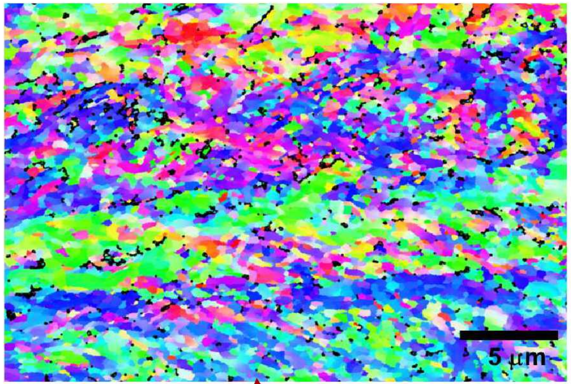
Material: API 5L X100 pipeline steel

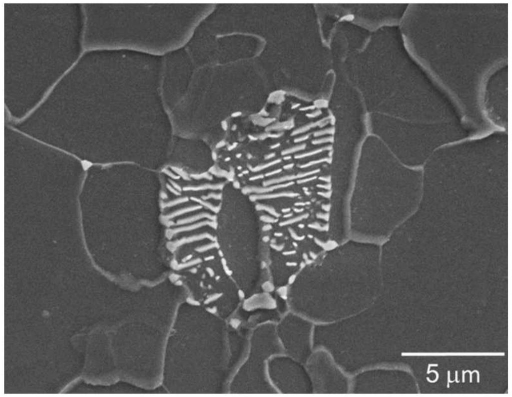
- Compact Tension specimens (12.7 mm thick)
  - YS = 731 MPa, UTS = 868 MPa in longitudinal direction

Fe	C	Mn	P	S	B	Si
Bal	0.085	1.69	0.013	<0.001	0.0015	0.26
Cu	**Ni**	**Cr**	**Mo**	**Nb**	**Ti**	**Al**
0.14	0.24	0.19	0.17	0.047	0.017	0.029









X100

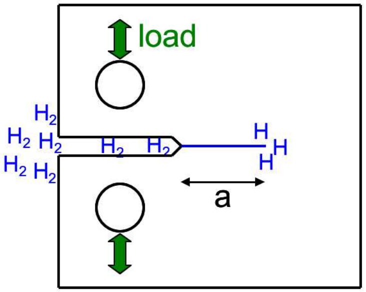
X52 (previous Somerday[1] study)

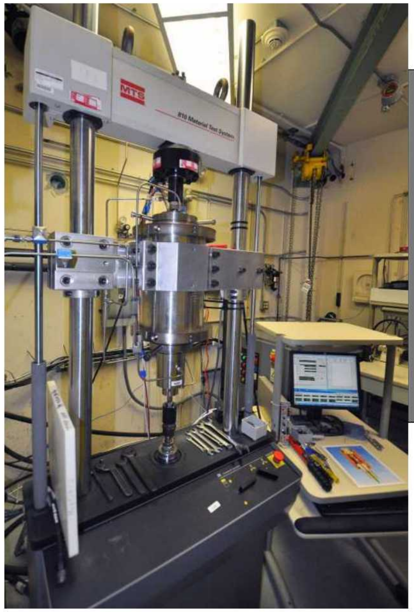
High strength X100 steel consisted of fine ferrite and bainite microstructure compared to coarse ferrite-pearlite in X52

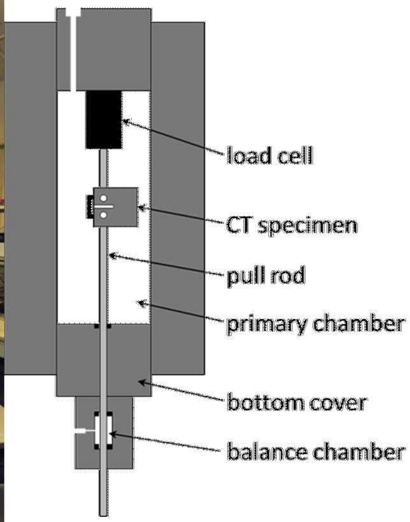
[1]B.P. Somerday et al., *Acta Mater*, 2013

Approach: Fatigue and Fracture measured in service environment, i.e. high-pressure $O_2 + H_2$ gas

Compact Tension
(C(T))



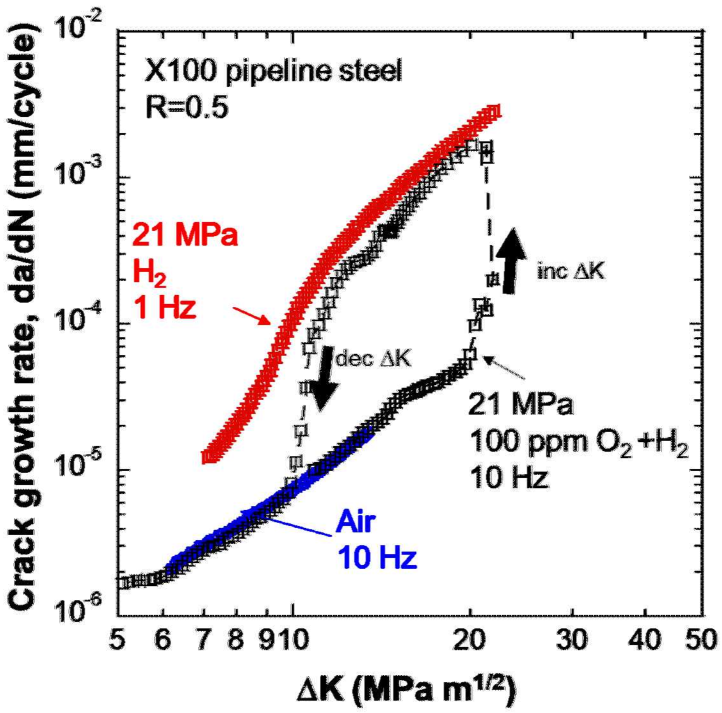




- Instrumentation
  - Internal load cell in feedback loop
  - Crack-opening displacement measured internally using LVDT or clip gauge
  - Crack length calculated from compliance
- Mechanical loading → Fatigue (ASTM E647)
  - K-control or constant load amplitude
  - Frequency: 1 Hz, 10 Hz
  - $R = \frac{P_{min}}{P_{max}} = 0.1, 0.5$
- Mechanical loading → Fracture (ASTM E1820)
  - Rising displacement (0.3 or 2.5 mm/hr)
  - DCPD monitored crack length
- Environment
  - Supply gas
    - Pure H_2 (99.9999%)
    - 100 ppm O_2 + balance H_2
  - Pressures
    - 21 MPa, 2.1 MPa, 1.4 MPa

21 MPa / 100 ppm O_2 / R=0.5 / f=10 Hz

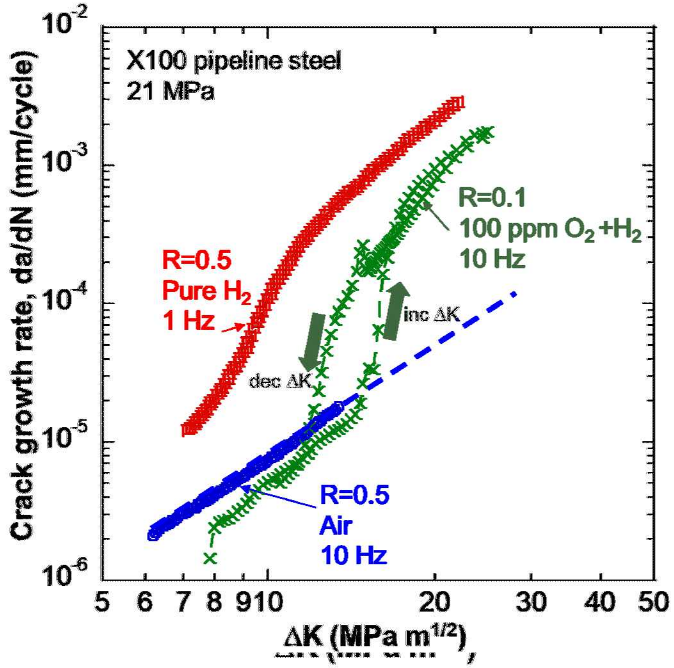
- In pure H_2, accelerated fatigue at onset of test
- In mixed gas (100 ppm O_2) R=0.5, f=10 Hz, onset of HA-FCG delayed to 5.3E-5 mm/cycle during K-increasing test
- Decreasing K used to explore onset of hydrogen-accelerated fatigue crack growth (HA-FCG)
- During K-decreasing, return of FCGR towards inert condition occurs at approximately the same da/dN



Results show dependence of HA-FCG on absolute da/dN

21 MPa / 100 ppm O_2 / R=0.1 / f=10 Hz

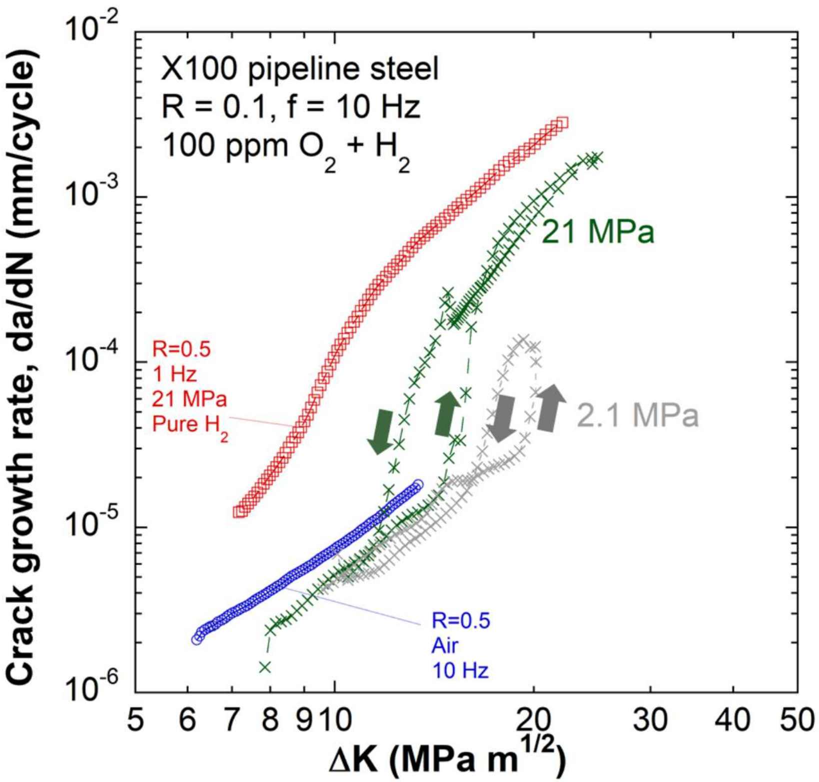
- In mixed gas (100 ppm O_2) R=0.1, f=10 Hz, onset of HA-FCG delayed to 1.6E-5 mm/cycle during K-increasing test
- During K-decreasing, return of FCGR towards inert condition occurs at a *higher* da/dN



Trends are similar to R=0.5 which show onset associated with absolute da/dN

2.1 MPa / 100 ppm O_2 / R=0.1 / f=10 Hz

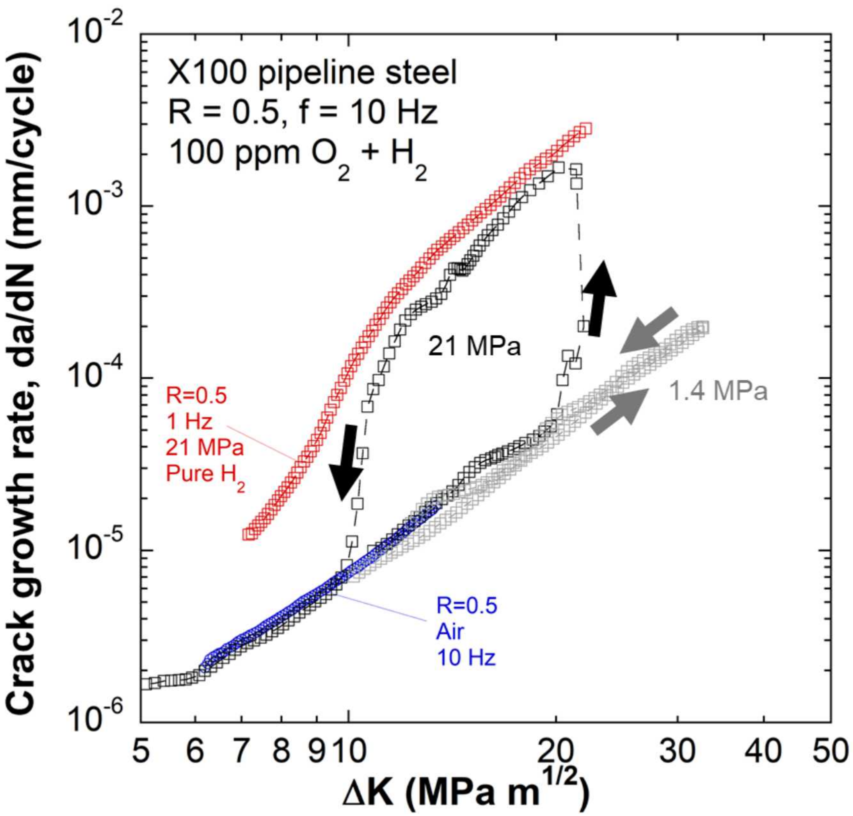
- At lowest pressure (2.1 MPa) mixed gas (100 ppm O_2) R=0.1, f=10 Hz, onset of HA-FCG delayed to 2.9E-5 mm/cycle during K-increasing test.
  - Higher da/dN onset than higher pressure test
- Test was not performed to higher ΔK so absolute da/dN_{H2} was not achieved



At lower pressure, HA-FCG onset trends are similar to R=0.1 but delayed to slightly higher da/dN

1.4 MPa / 100 ppm O_2 / **R=0.5** / f=10 Hz

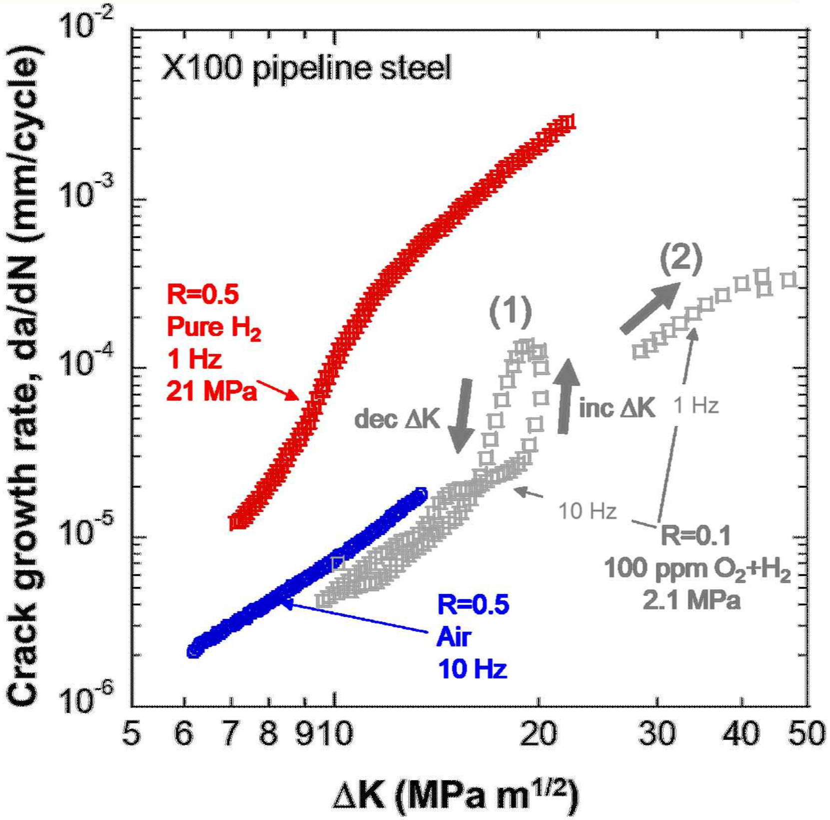
- At lower pressure (1.4 MPa) mixed gas (100 ppm O_2) R=0.5, f=10 Hz, no HA-FCG was observe.
  - K-increasing and K-decreasing overlaid the air curve
- Perhaps HA-FCG would have been observed if higher ΔK would have been imposed



Absence of HA-FCG at 1.4 MPa suggests oxygen may have more dominant effect at lower pressures on mitigating HA-FCG

2.1MPa / 100 ppm O_2 / R=0.1 / f = 10 / 1 Hz

- Frequency effects were investigated at lower pressure 2.1 MPa at R=0.1
- (1) At 10 Hz, HA-FCG was observed on K-inc followed by K-dec
- (2) At 1 Hz, no HA-FCG was observed up to ΔK ~ 47 MPa $m^{1/2}$



Frequency results are consistent with theory that lower frequency allows oxygen to reach greater number of surface sites

Oxygen moderated hydrogen-assisted fracture

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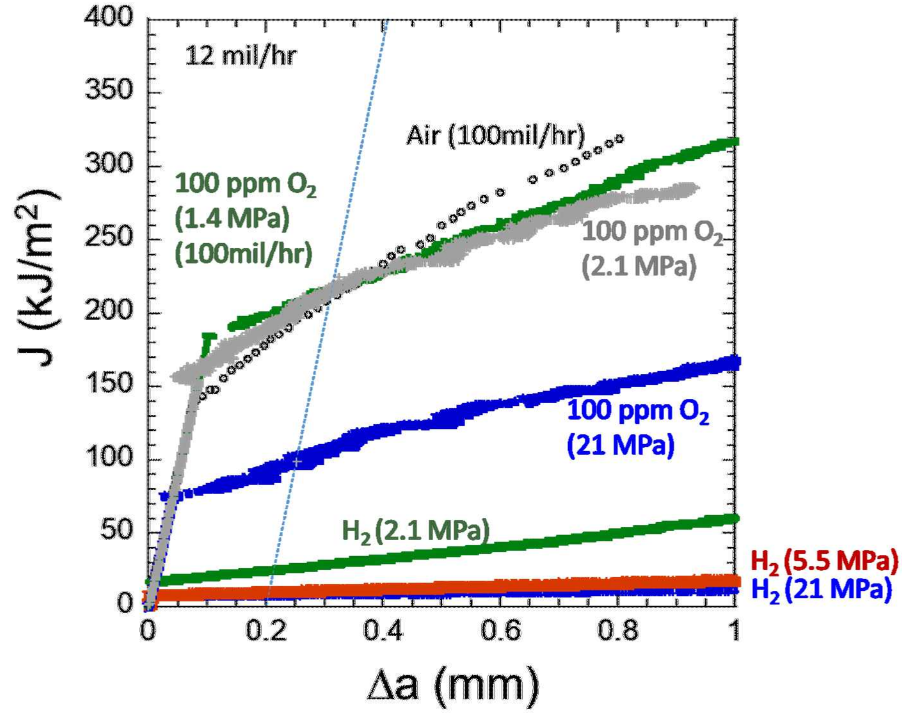
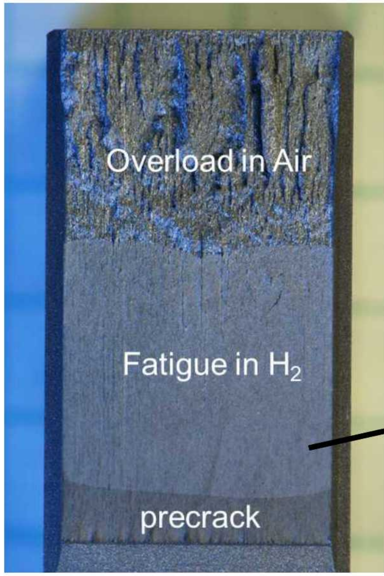
- In 21 MPa pure H_2, fracture toughness K_{JIH} values decreased by 80%.
- In 21 MPa mixed gas, fracture toughness decreased by only 30%.
- At lower pressures (1.4-2.1 MPa) in mixed gas, no effect of hydrogen was measured →(K_{JIH} in air ~ K_{JIH} in mixed gas)
- At lower pressure, test rates of 0.3 and 2.5 mm/hr resulted in similar K_{JIH} ~ air



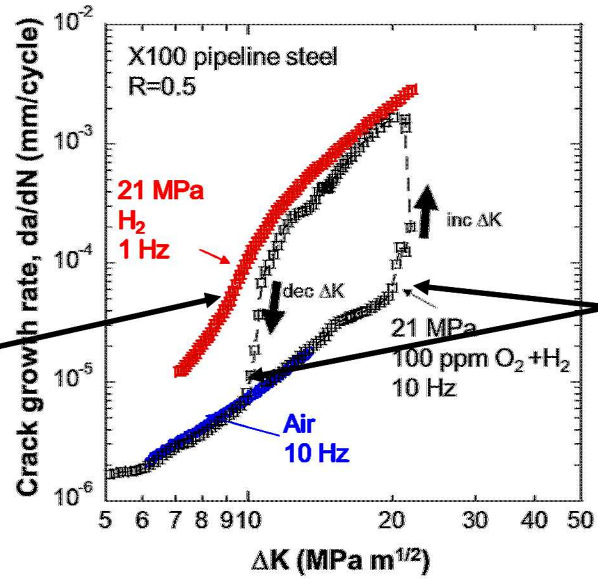
Lower pressure fracture toughness similar to tests in air

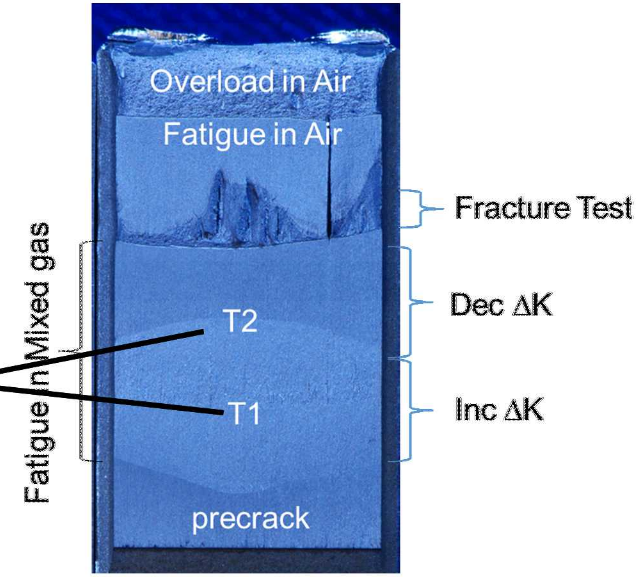
Sample ID	Environment	Test Pressure (MPa)	Actuator rate (mm/hr)	da/dt (mm/s)	K_{JIH} (MPa $m^{1/2}$)
X100-5	H2	21	0.3	8.5E-4	43
X100-6	H2	5.5	0.3	3.6E-4	47
X100-7	H2	2.1	0.3	1.7E-4	75
X100-51	Air	-	2.5	5.0E-4	217
X100-52	Air	-	2.5	1.4E-4	202
X100-53	H2 + 100 ppm O2	21	0.3	1.1E-4	151
X100-55	H2 + 100 ppm O2	2.1	0.3	7.4E-5	222
X100-56	H2 + 100 ppm O2	1.4	2.5	1.0E-4	222

Fracture surfaces reveal effects of O_2 on da/dN



21 MPa H_2
R=0.5, freq=10 Hz

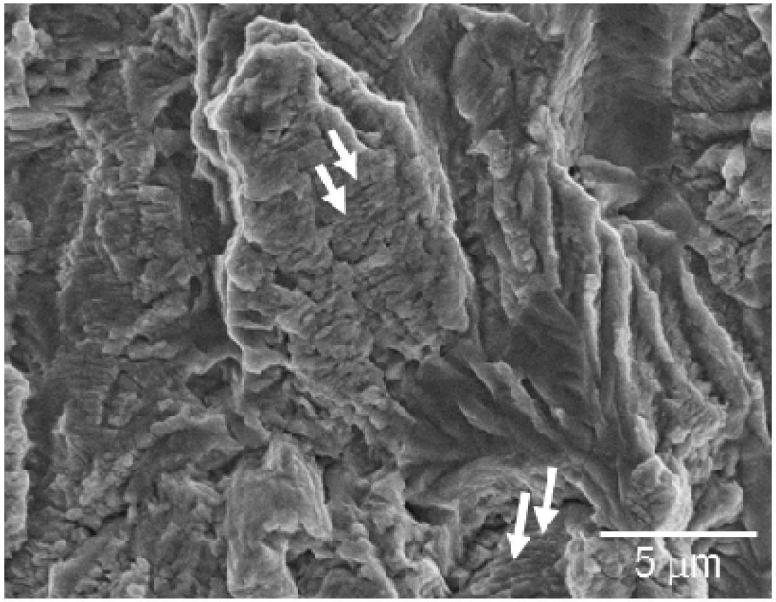




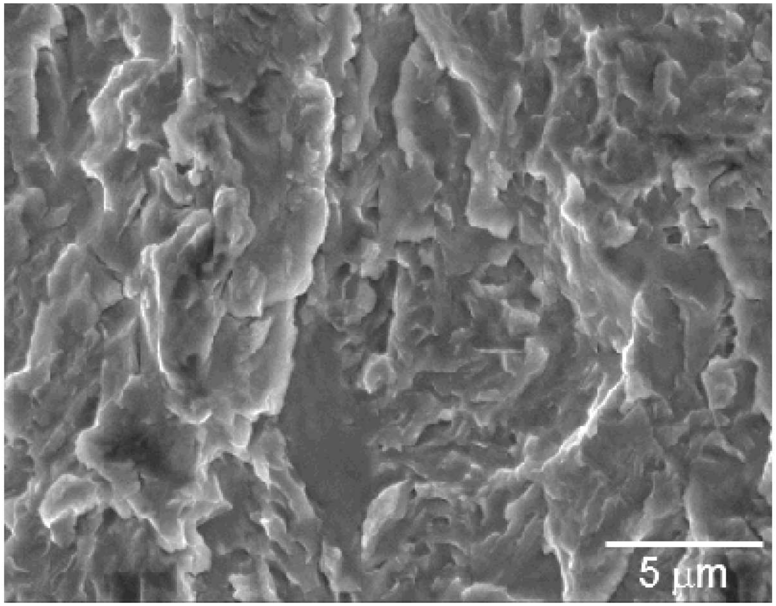
21 MPa O_2 + H_2
R=0.5, freq=10 Hz

- In pure H_2, entire fatigue region appears similar in features
- In mixed gas, two distinct transitions (T1 and T2).
  - T1 – transition from da/dN_{inert} to da/dN_{H2} during K-increasing test
  - T2 – transition from da/dN_{H2} to da/dN_{inert} during K-decreasing test
- Delaminations observed on fracture test

Distinct difference between fatigue in air and H_2



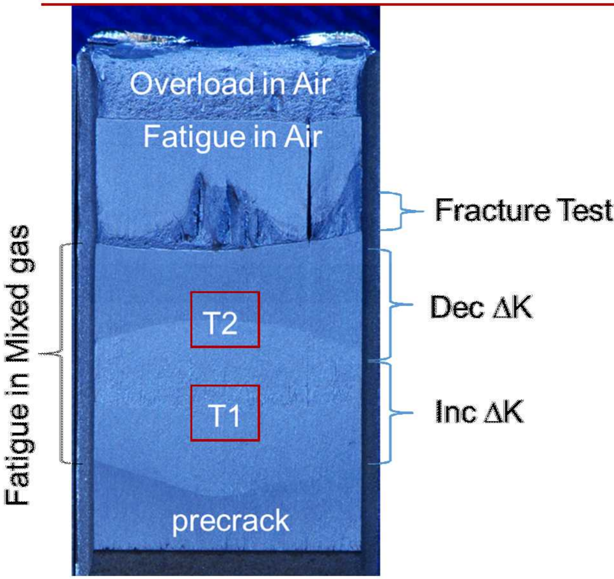
Air
R=0.5, freq=10 Hz

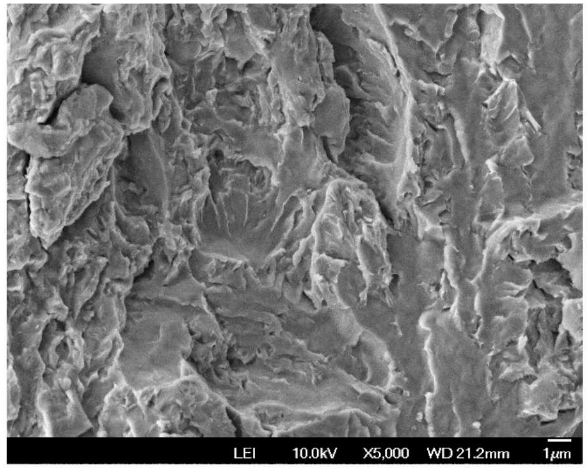


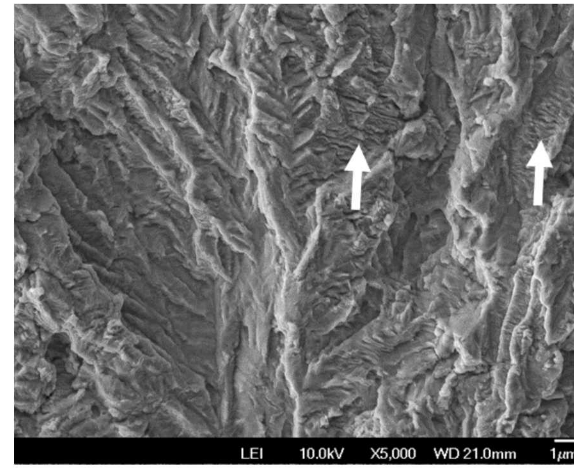
21 MPa H_2
R=0.5, freq=10 Hz

- In air, distinct striations are observed typical of inert fatigue (blunting/sharpening of crack tip)
- In $H_{2,}$ transgranular features absence of fatigue striations

In mixed-gas, oxygen influence is observed on fracture surface



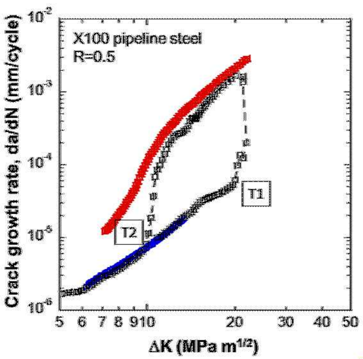


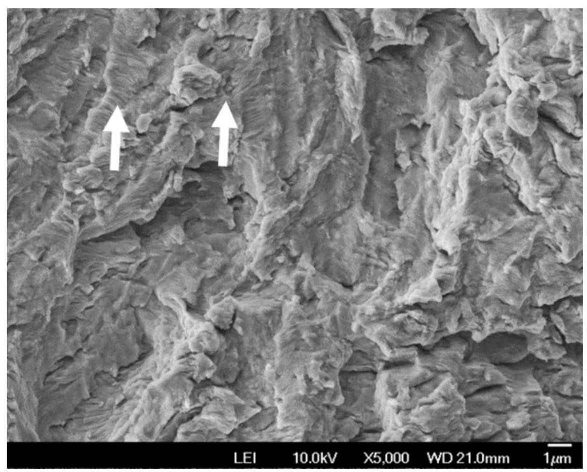


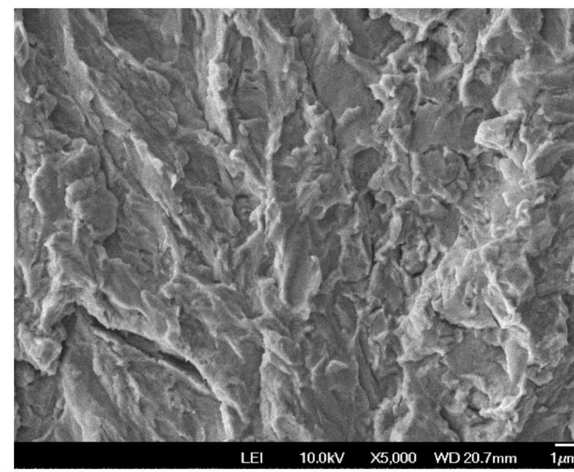
T1

T2

21 MPa O_2 + H_2
R=0.5, freq=10 Hz







The presence or absence of fatigue striations appears to be one of the defining features on fracture surfaces when differentiating the effect of oxygen on FCGR

Summary

- X100 pipeline tested in high pressure mixed gas (100 ppm O_2 + hydrogen) under fatigue and fracture
- Mixed-gas tests exhibited HA-FCG mitigation under specific loading and environmental conditions.
  - Onset of HA-FCG was observed to be dependent on a specific da/dN for given conditions
  - Results in delay onset of HA-FCG: Higher R-ratio, lower frequency, lower pressure
- Fracture tests in mixed gas showed improved toughness relative to pure H_2
  - Lower pressure mixed gas tests showed no signs of hydrogen degradation
- Fracture surfaces show distinct differences when tested in oxygen containing environments when HA-FCG is suppressed.

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Proposed future tests

- 1.4 MPa mixed gas, R=0.5, f=10 Hz ΔK 20→ 50 MPa $m^{1/2}$ → 20
  - Exploring if there is an absolute suppression of HA-FCG at higher ΔK
- 2.1 MPa mixed gas, R=0.1, f=10 Hz, ΔK 12→ 30 MPa $m^{1/2}$ → 12
  - Evaluate the absolute da/dN that we should get at R=0.1 and lower pressure, also serves as duplicate to see what type of repeatability is possible
- 1000 ppm O_2 test 21 MPa, R=0.1, f=10 Hz, ΔK 15→ 50 MPa $m^{1/2}$ → 15
  - This would likely be the earliest onset (if we see one) and could test to high ΔK because Kmax is low due to R=0.1
- Fracture test 21 MPa at faster rate (2.5 mm/hr)
- Fracture test at 100 MPa at 0.3 mm/hr