SAND2024-09901C

Sandia National Laboratories

Exceptional service in the national interest

Effect of Oxygen Impurities on Subcritical Crack Growth in High-Pressure Hydrogen Environments

Robert W. Wheeler, Chris San Marchi, and Joseph Ronevich

Hydrogen and Materials Science Department, Sandia National Laboratories Livermore, CA, USA

ASME Pressure Vessels and Piping Conference July 2024

Sandia National Laboratories is a multimission laboratory managed and operated by National Technology and Engineering Solutions of Sandia LLC, a wholly owned subsidiary of Honeywell International Inc. for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-NA0003525.

U.S. DEPARTMENT OF ENERGY

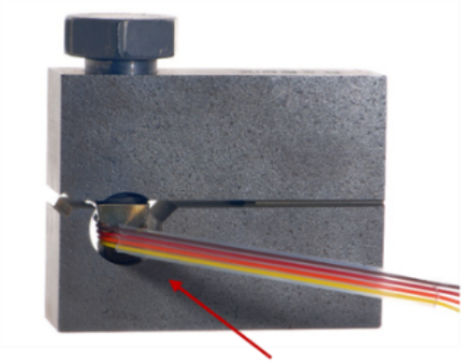
SAND2024-09901C

NNSA

Overview

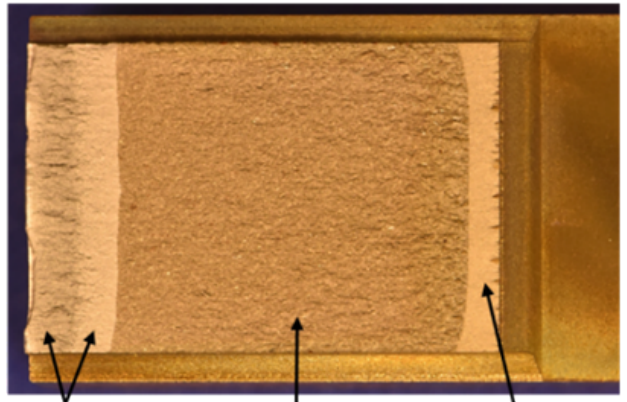
- Background and motivation
  - Mitigating effects of oxygen impurities on hydrogen embrittlement in laboratory testing
- Experimental methods
  - Long-term, constant displacement fracture tests in high pressure gaseous hydrogen environments
  - Commercial pressure vessel and pipeline steels
- Experimental results
  - Comparison of subcritical crack growth in high-pressure hydrogen and hydrogen with varying degrees of oxygen impurities
- Conclusions and future work

Wedge-opened Loaded (WOL) Sample



Reaction pin (load tup) with externally monitored strain gauge

Grade L: WOL Fracture Surface

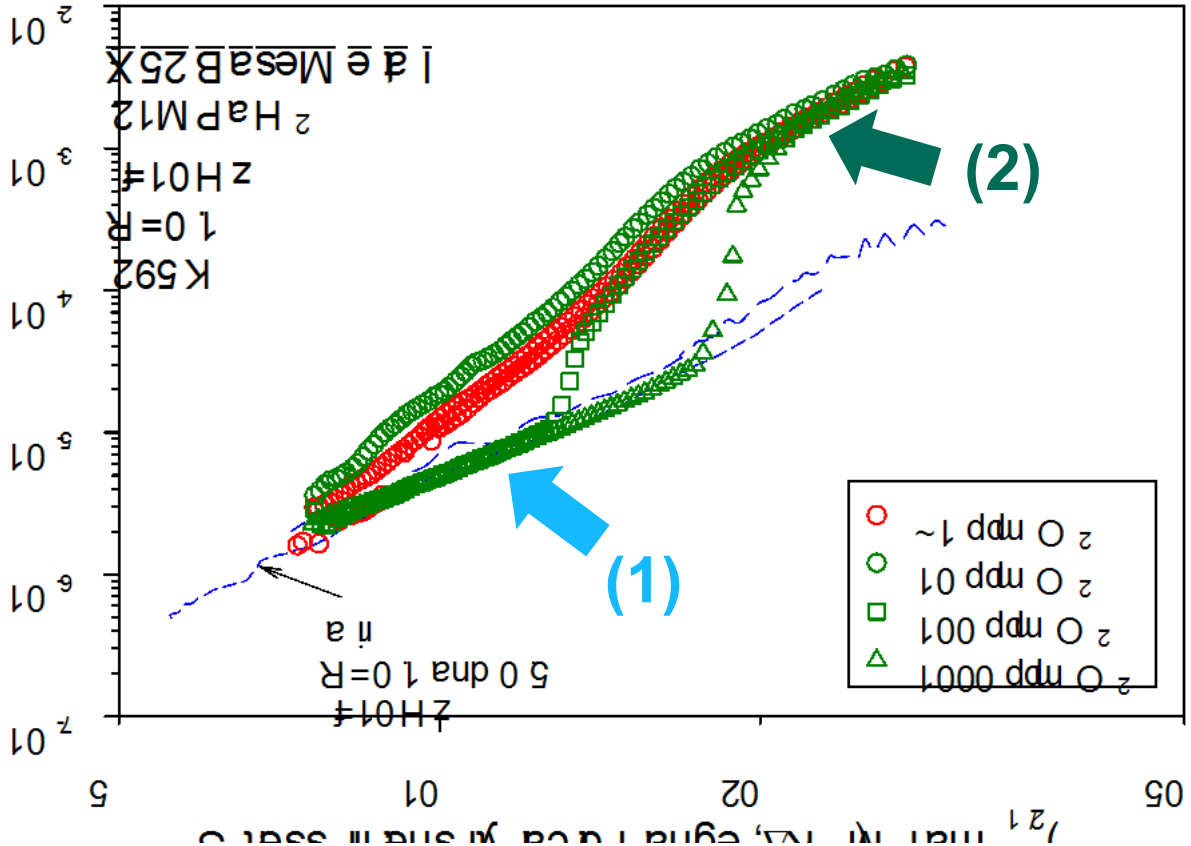


Post-test fatigue and LN_2 fracture

Fatigue precrack (in air)

Crack growth in H_2 + 100PPM O_2

Oxygen is known to affect measurements of fatigue

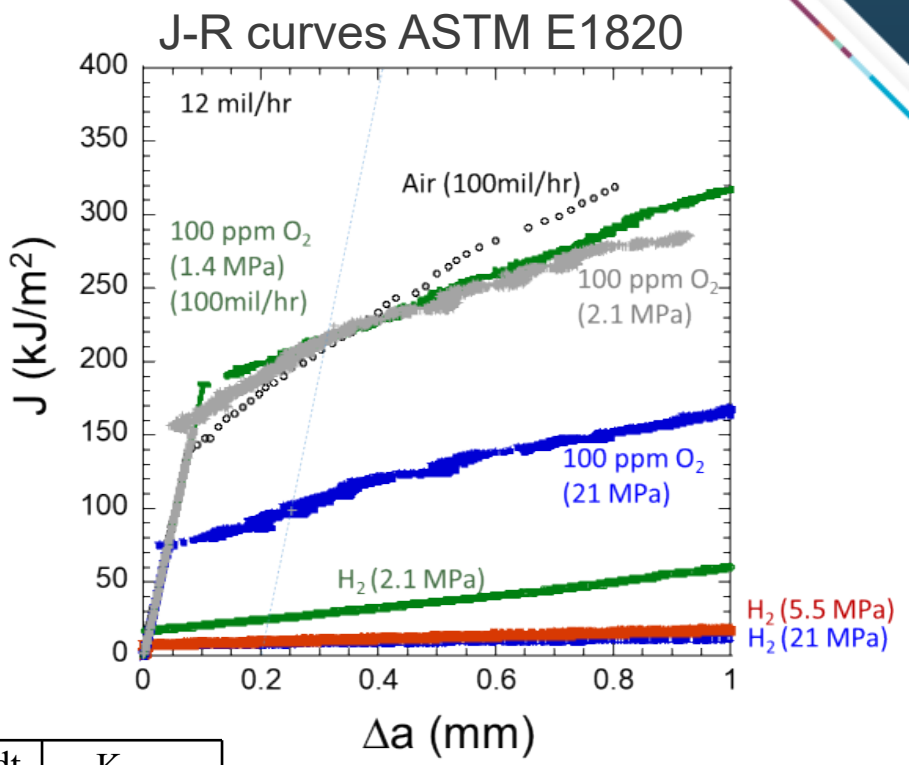


- Numerous examples of trace gases mitigating fatigue crack growth rate (FCGR) in laboratory conditions
- Example:
  - (1) Oxygen reduces FCGR comparable to air
  - (2) Oxygen has no effect on FCGR in H_2

Refs.: Somerday et al, *Acta Mater* 61 (2013) 6153. Nibur et al, SAND2010-4633 (2010).

Oxygen moderated hydrogen-assisted fracture

- Fracture toughness K_{JQH} values decreased by over 60% in pure H_2 at 2.1 MPa
- In 21 MPa mixed gas (100 ppm O_2), fracture toughness decreased by only 30% relative to air
  - In pure H_2 at 21 MPa, relative decrease was 80%
- At lower pressures (1.4-2.1 MPa) in mixed gas, no effect of hydrogen was measured



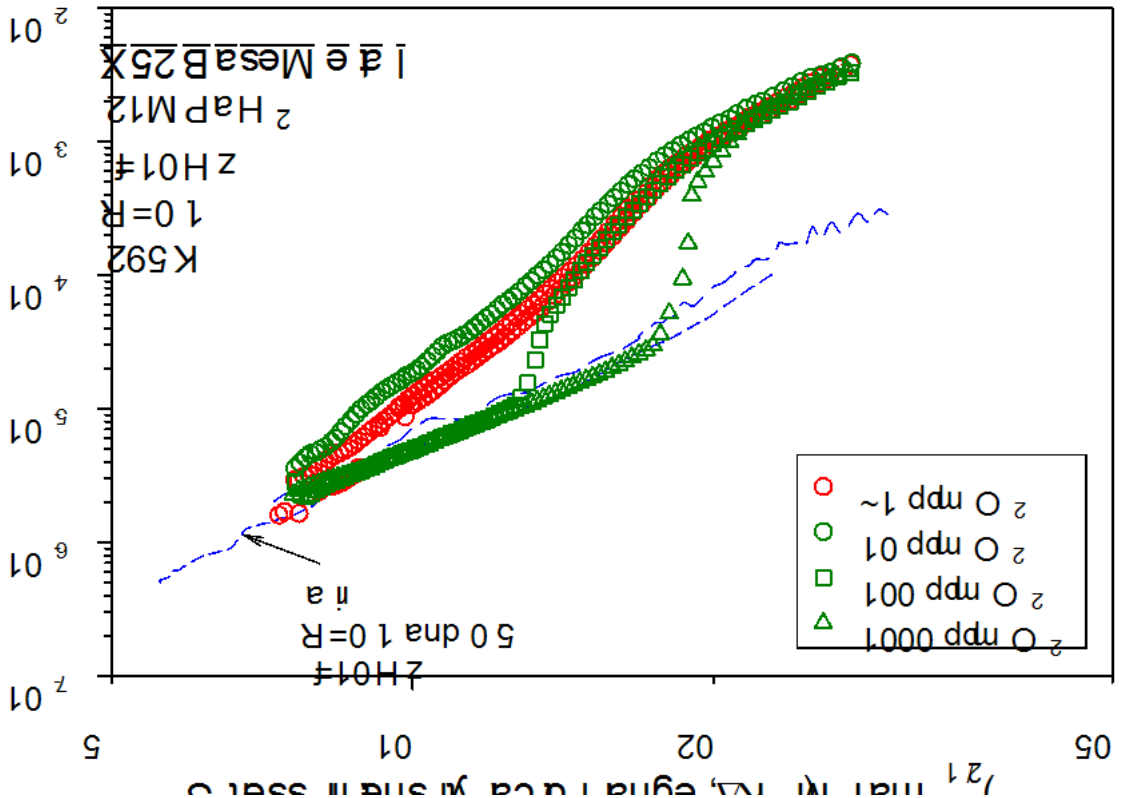
Sample ID	Environment	Test Pressure (MPa)	Actuator rate (mm/hr)	da/dt (m/s)	K_{JQH} (MPa $m^{1/2}$)
X100-51	Air	-	2.5	5.0E-7	217
X100-52	Air	-	2.5	1.4E-7	202
X100-5	H2	21	0.3	8.5E-7	43
X100-6	H2	5.5	0.3	3.6E-7	47
X100-7	H2	2.1	0.3	1.7E-7	75
X100-53	H2 + 100 ppm O2	21	0.3	1.1E-7	151
X100-55	H2 + 100 ppm O2	2.1	0.3	7.4E-8	222
X100-56	H2 + 100 ppm O2	1.4	2.5	1.0E-7	222

60 - 80% reduction

30% reduction

Ref: Ronevich et al, PVP2018-84163

Oxygen has been shown to mitigate hydrogen embrittlement in laboratory tests



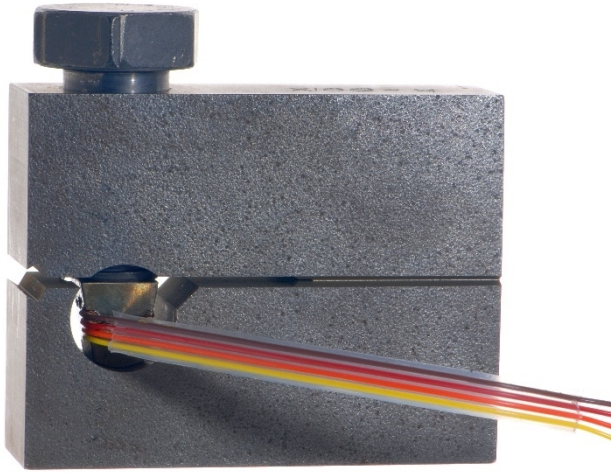
- Fatigue and fracture measurements can be significantly impacted by oxygen impurities
- Fatigue crack growth tests are typically performed at 1 Hz (±decade)
  - da/dN = 10^{-5} mm/cycle
  - Time for Δa = 1mm: ~1 day
  - 1 day = 0.02% of 10 year life
- Are the time scales of a typical laboratory fatigue test sufficient to demonstrate kinetics over decades?
  - More accurately simulate the mechanical/environmental conditions that components see when in use
  - Does trace oxygen have long term mitigation effects on hydrogen embrittlement?

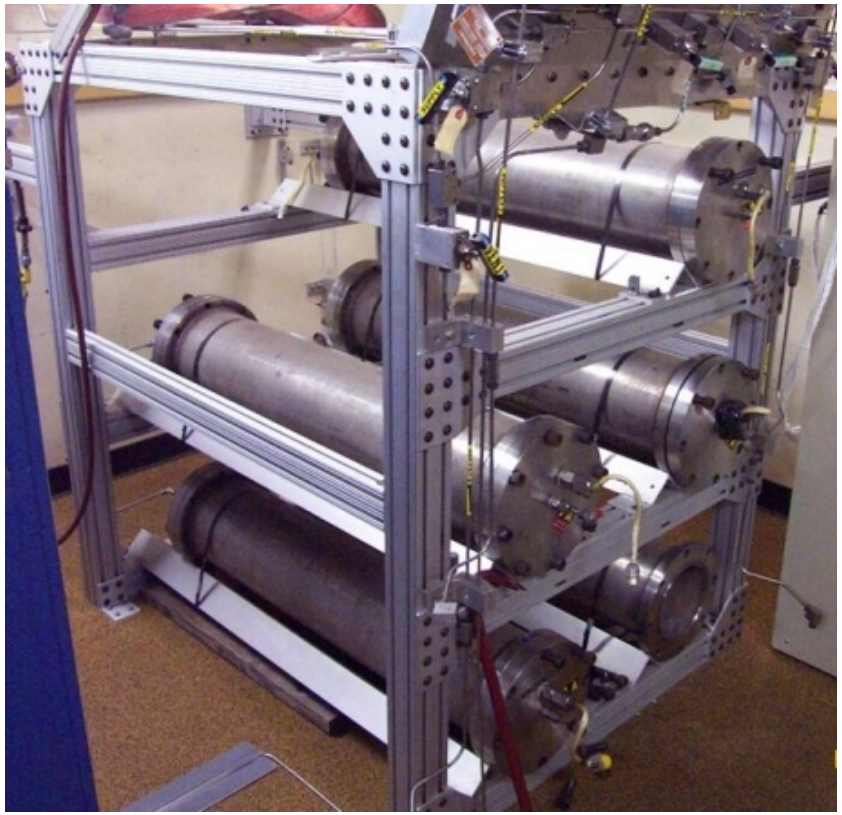
Ref.: Somerday et al, *Acta Mater* 61 (2013) 6153.
Ref.: Nibur et al, SAND2010-4633 (2010).

Sustained load testing can be executed over periods of days to weeks to months to years

- Fixed displacement tests
- Placed in pressure vessels & pressurized up to 140 MPa gaseous environment
  - Experiments in this study were performed at 103MPa
- Instrumented reaction pins allows us to determine incubation time
- Directly compare subcritical crack growth in hydrogen and mixed gas environments

Wedge-opened loaded (WOL)

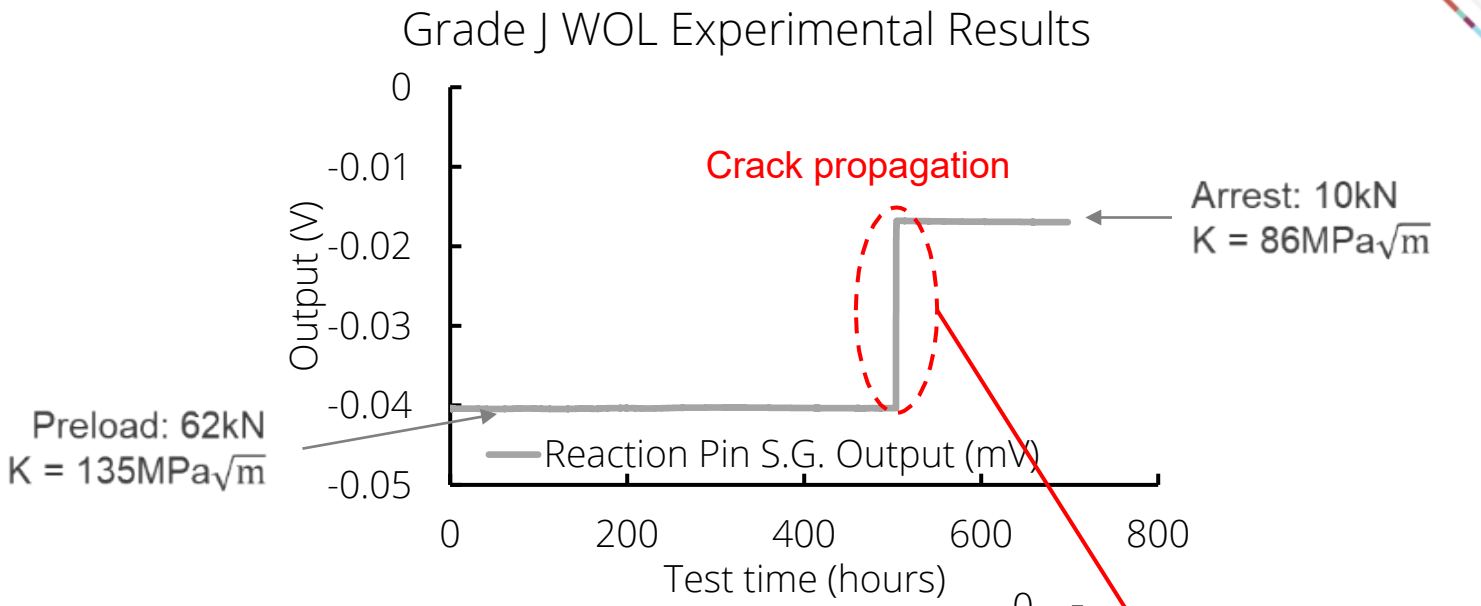


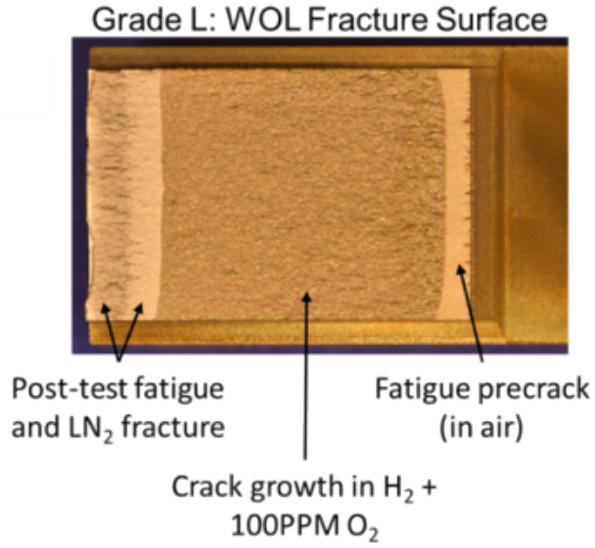


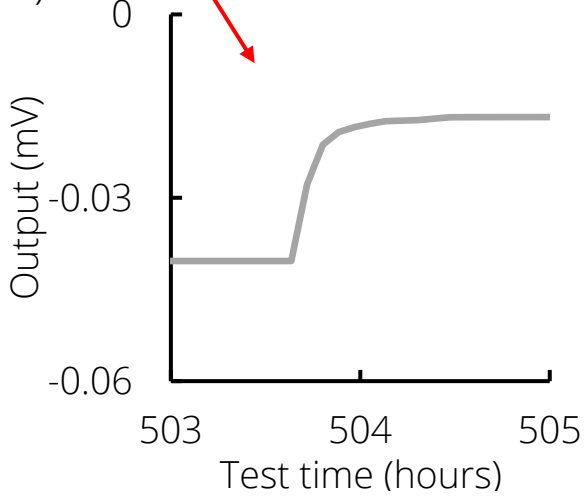
ASTM E1681 – Threshold Stress Intensity Factor for Environment-Assisted Cracking

Crack initiation and growth rates were measured during constant displacement fracture experiments

- Instrumented reaction pins allow for determination of incubation time and crack growth rates
  - Continuous data collection throughout the duration of the experiments
- Time between the initial crack propagation and arrest can range between seconds to hours
  - With a constant displacement, the crack growth rates can be determined from the load on the reaction pin
- Post-test fatigue and heat tinting are used to mark fracture surfaces





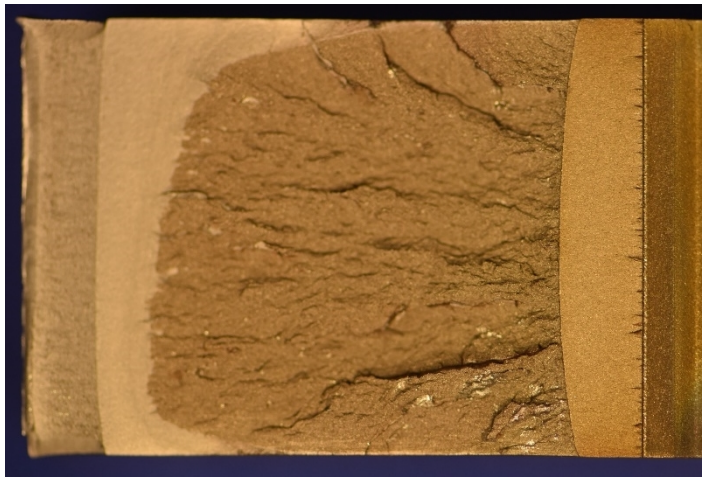


Material selection and fracture surfaces

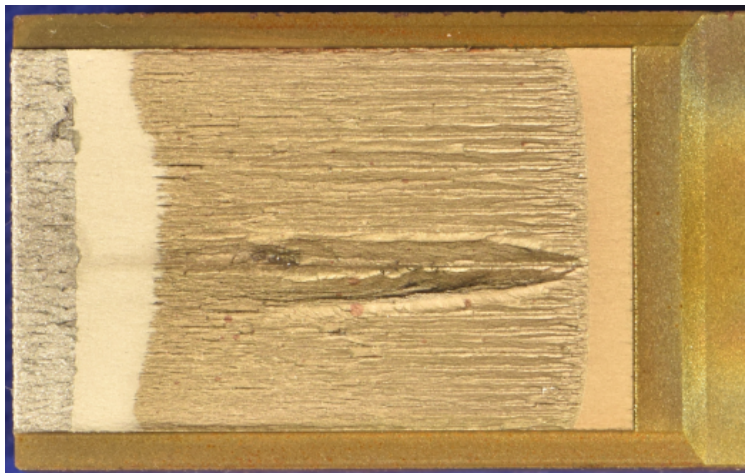
- SA372 Grade J steel
  - YS = 700 MPa
- SA372 Grade L steel
  - YS = 730 MPa
- Cr-Mo pressure vessel steel
  - YS = 750 MPa
- X100 pipeline steel
  - YS = 760 MPa
- Precipitation Hardened 13-8 stainless steel
  - YS = 1480 MPa

Grade L

Grade J

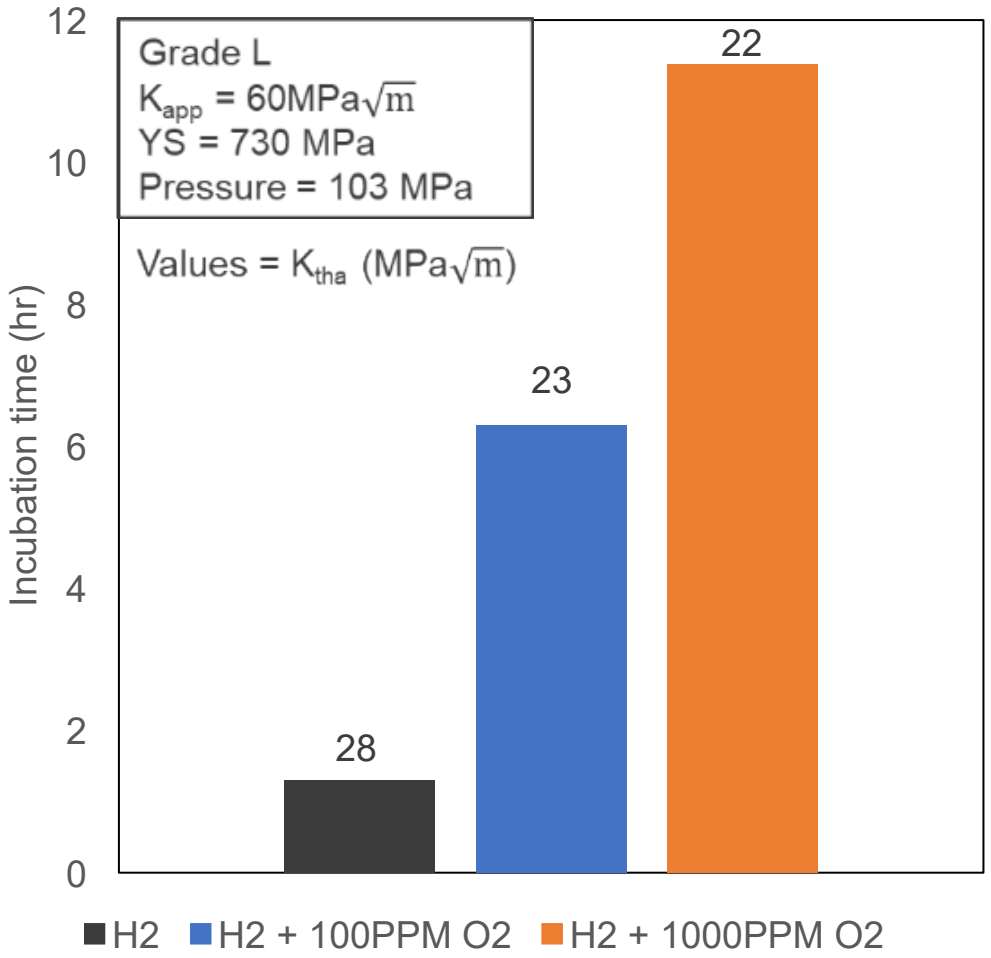


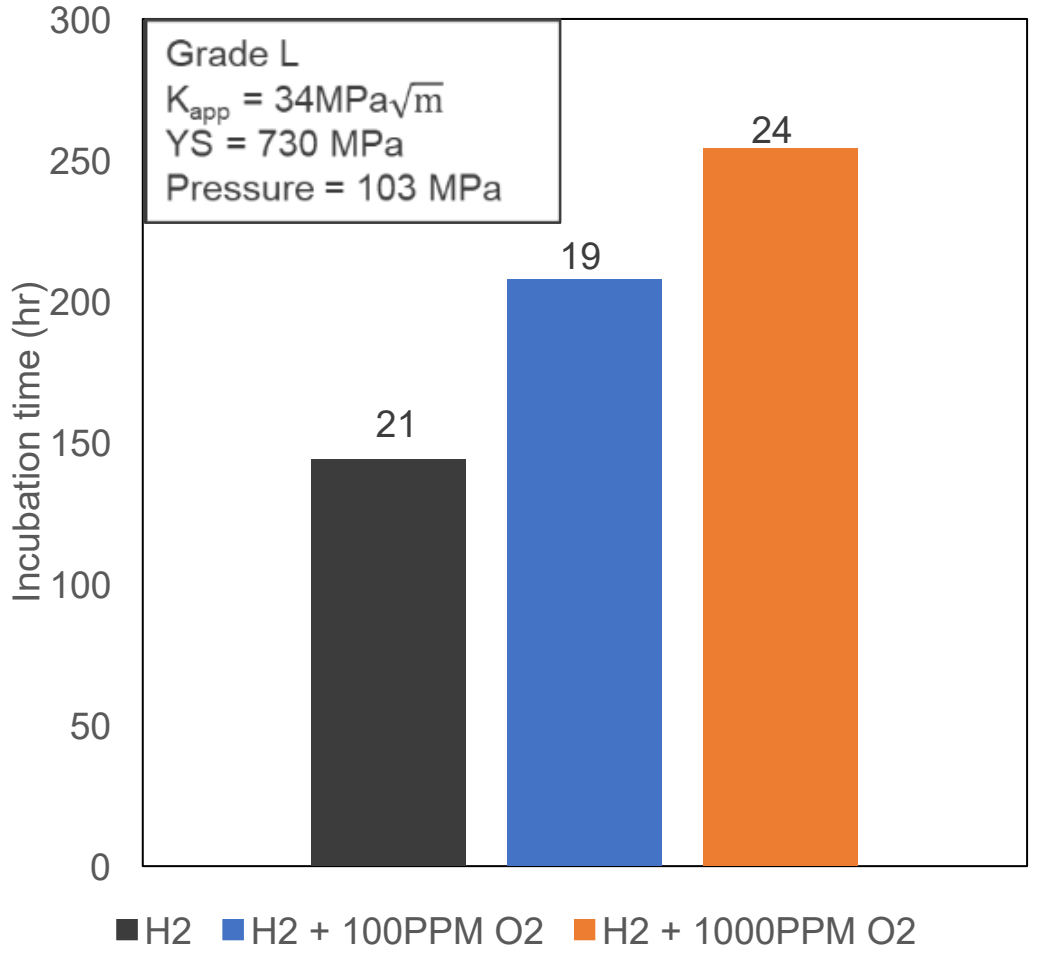
X100



13-8

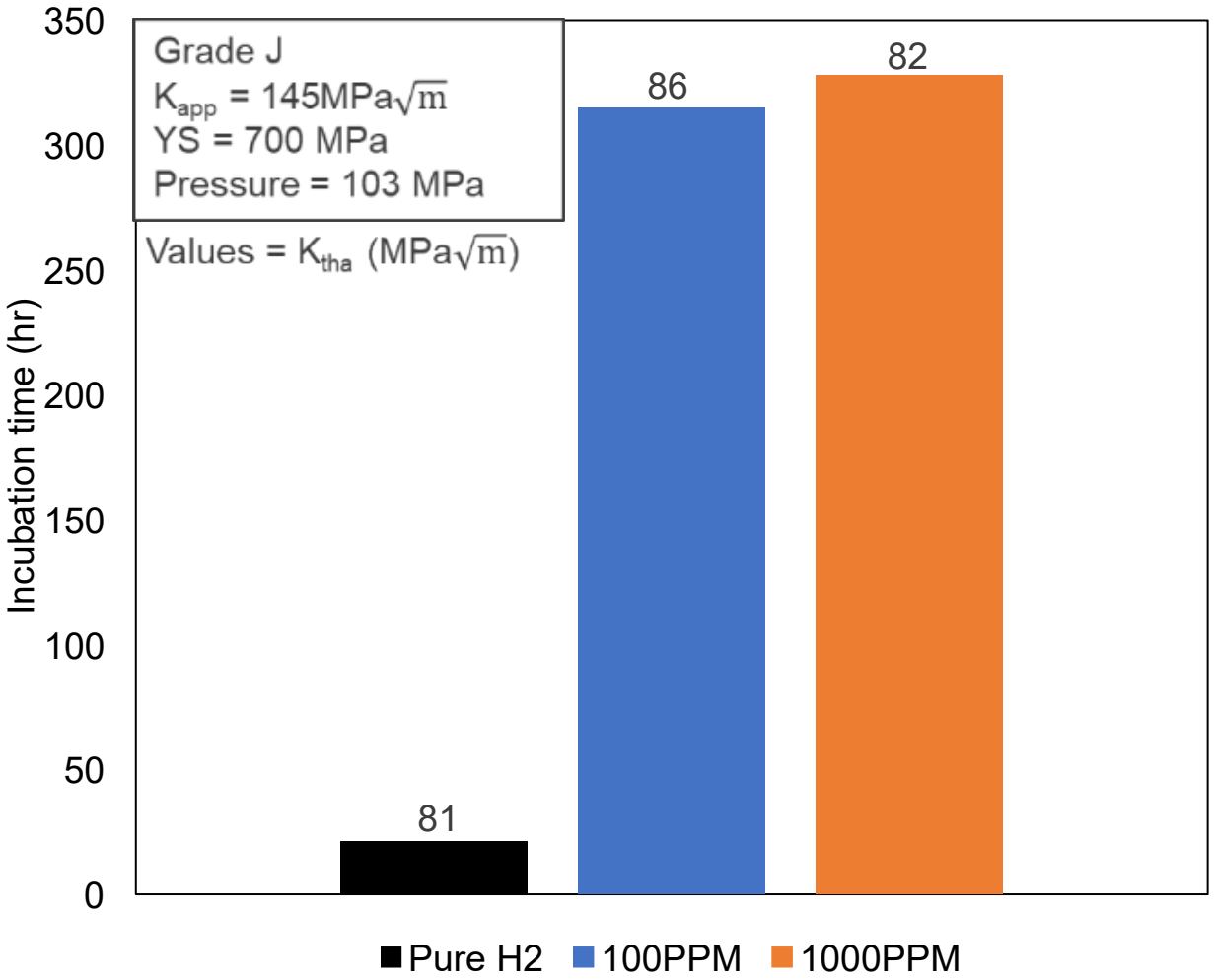
Grade L: 100PPM & 1000PPM O_2 delay incubation time

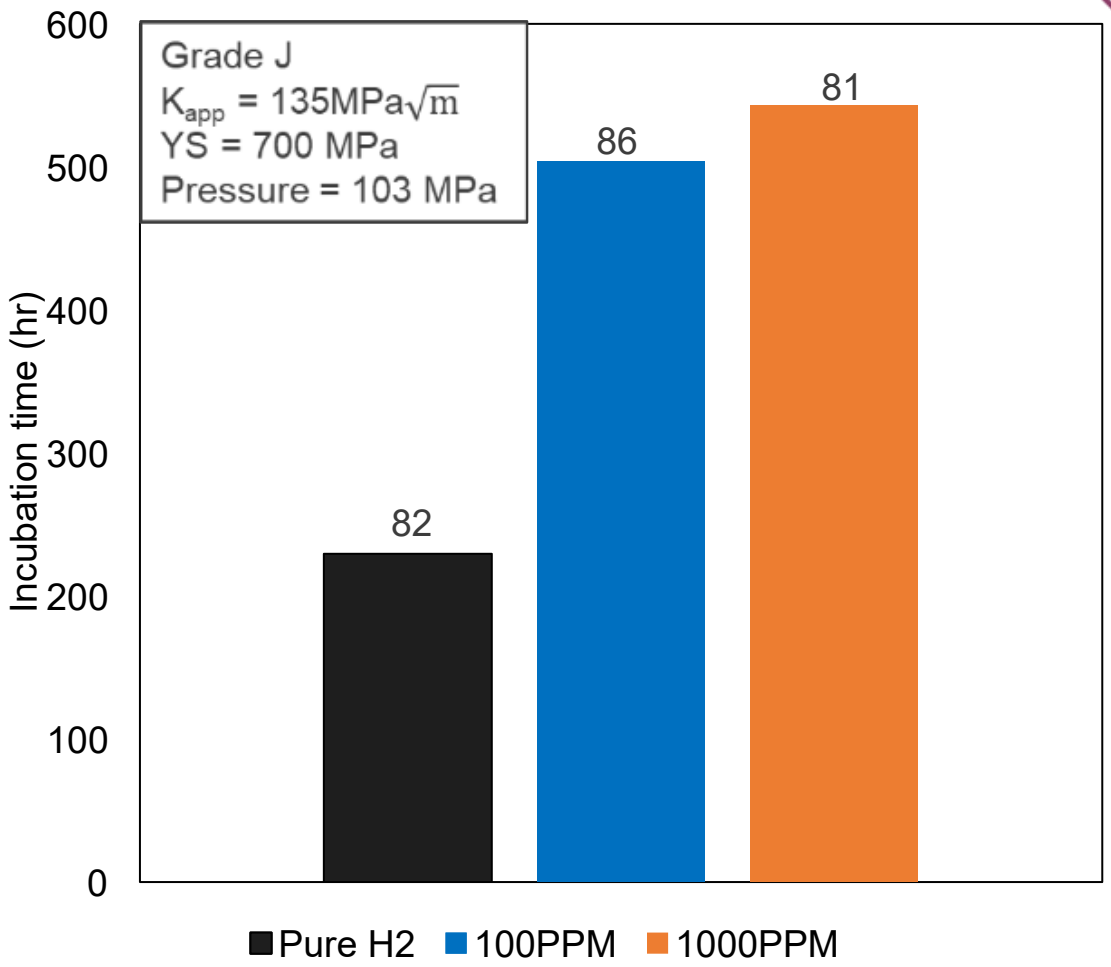




- Grade L shows a 5x delay at higher preload (K_{app} = 60 MPa$\sqrt{m}$) and a 1.5x delay at lower preload (K_{app} = 34 MPa$\sqrt{m}$)
- Similar crack arrest thresholds for all test conditions

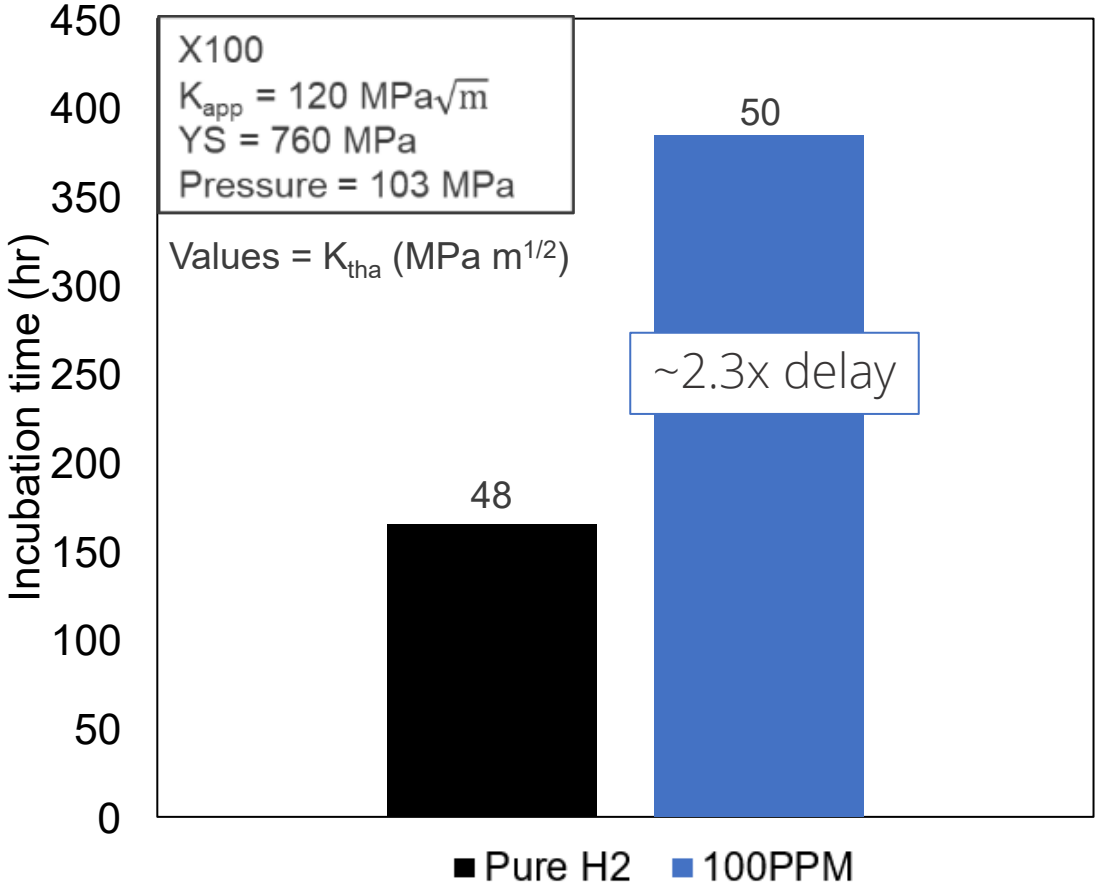
Grade J: 100PPM & 1000PPM O_2 delay incubation time

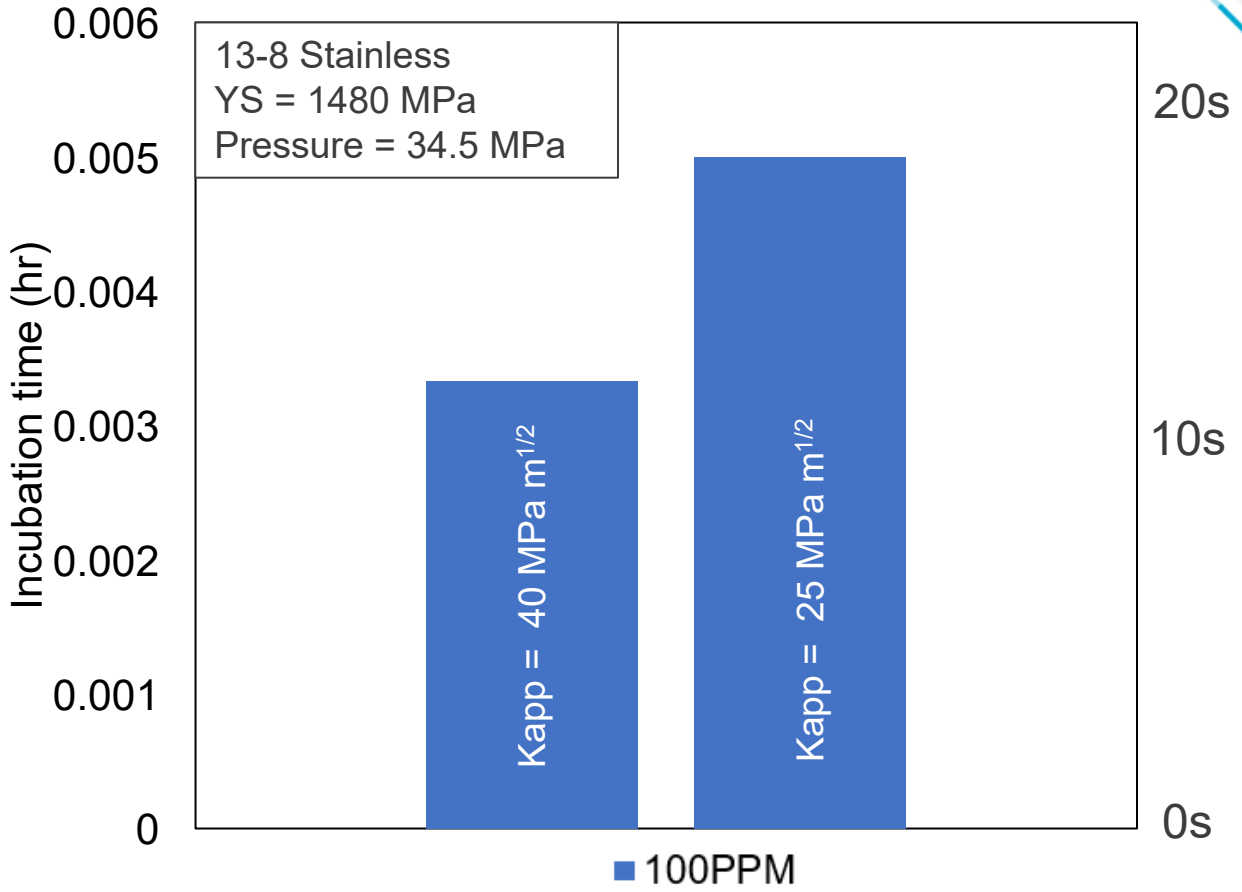




- Similarly, the Grade J material showed delays of 15x at a higher preload (Kapp = 145 MPa$\sqrt{m}$) and a 2.2x delay increase at a lower preload (Kapp = 135 MPa$\sqrt{m}$)
- K thresholds were within ±5 MPa$\sqrt{m}$ of average for both the pure and mixed gas conditions

100PPM O_2 delays incubation time for X100, but 13-8 fractured immediately

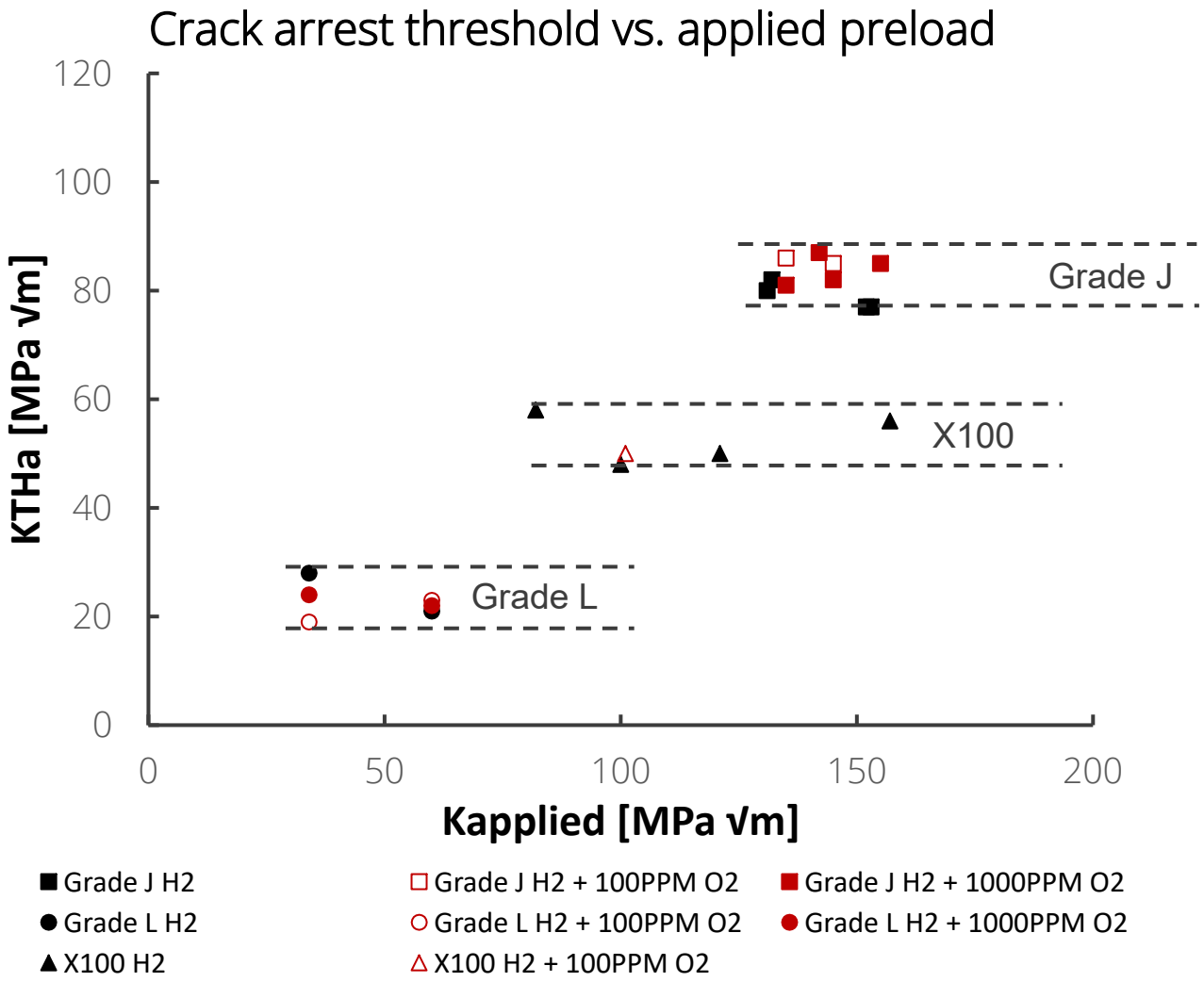




- X100 also saw a delay with the addition of 100PPM O_2
- Both 13-8 samples fractured (a/W > 97%) within seconds of exposure to H_2 + 100PPM O_2

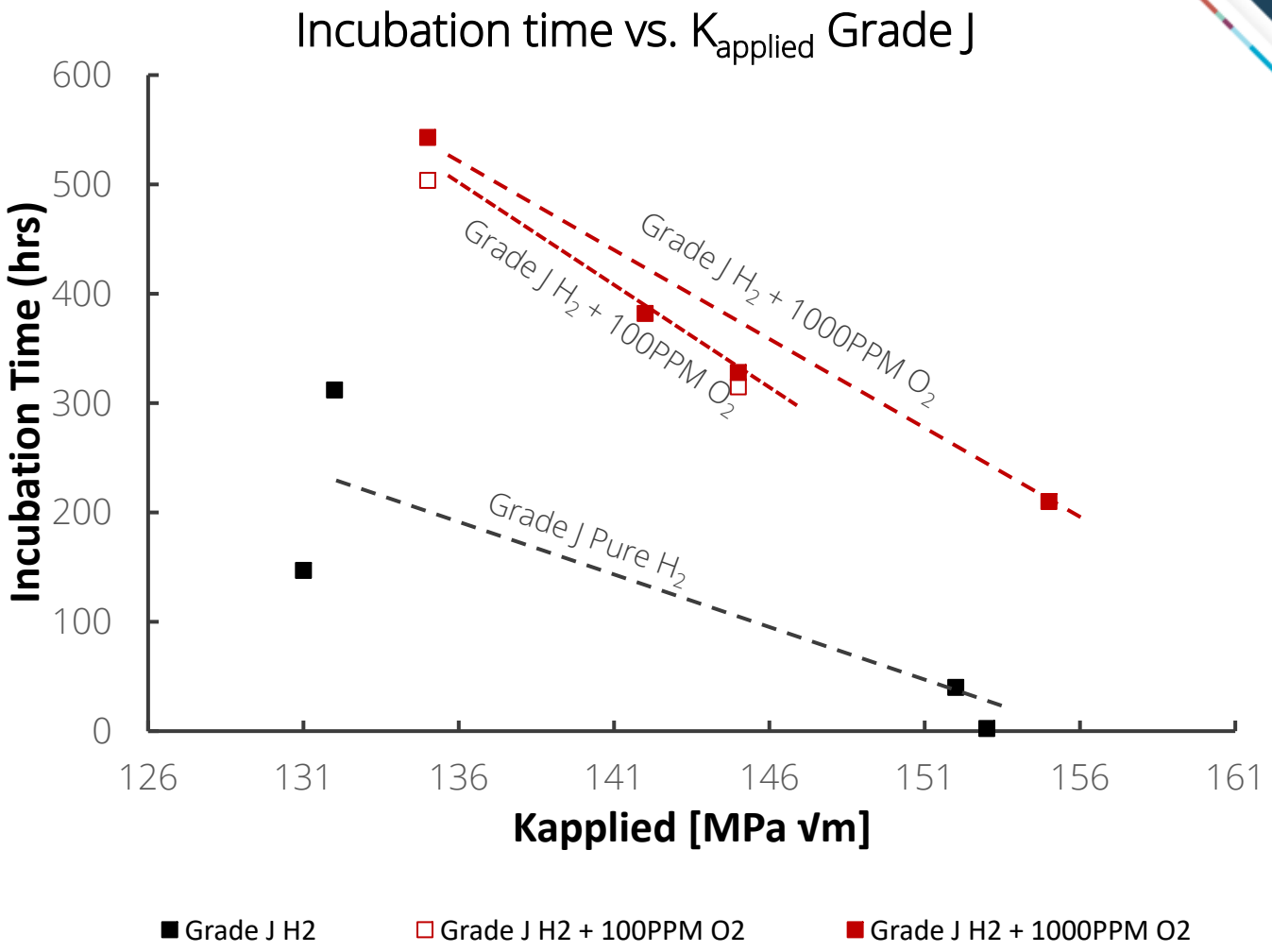
Summary and Conclusions

- Constant displacement fracture tests were carried out in pure hydrogen and mixed gas (100 and 1000PPM oxygen) environments at 103MPa (15ksi)
- K_{THa} appears to be independent from oxygen content
  - All tests with pure hydrogen and oxygen impurities fall within an apx. 10MPa$\sqrt{m}$ range



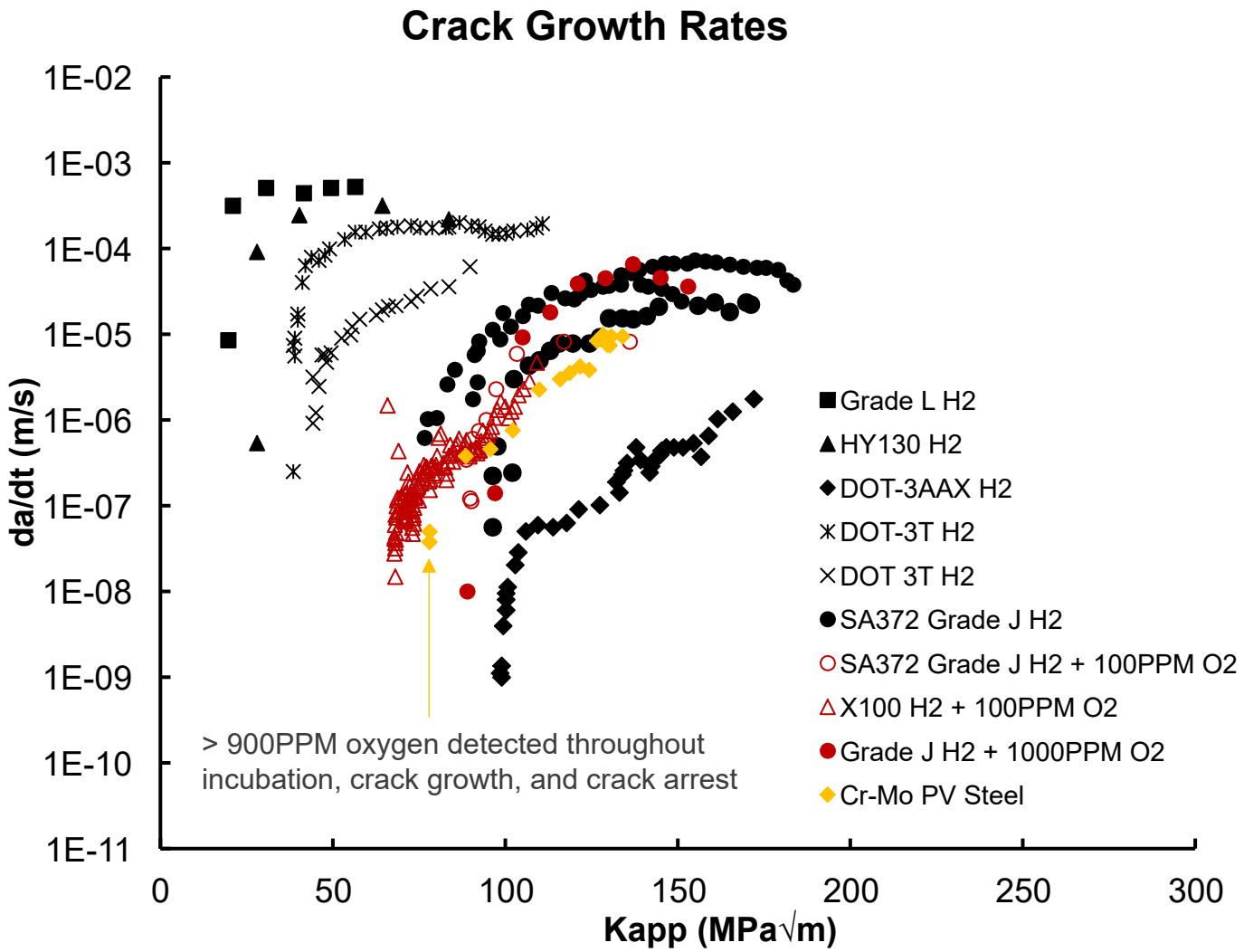
Summary and Conclusions

- Introducing 100PPM oxygen increased the incubation time by factors between 1.5x and 15x, but did not prevent crack propagation
- For the Grade L and Grade J, increasing the oxygen content from 100PPM to 1000PPM further delayed the incubation time, but had a smaller relative effect compared to the delay from pure hydrogen to hydrogen + 100PPM oxygen
- Based on this data, low oxygen impurities should not be relied upon for long-term mitigation of hydrogen embrittlement



Conclusions and future work

- Crack growth rates (da/dt) fall within the expected ranges from previous tests in pure hydrogen at similar pressures
- Future research is planned to determine the mechanisms behind the delay of hydrogen embrittlement in the presence of oxygen impurities
  - Surface effects that slow the hydrogen uptake and embrittlement process when oxygen impurities are present



Thank you for your attention!

- Rob Wheeler - rwheel@sandia.gov
- Chris San Marchi - cwsanma@sandia.gov
- Joe Ronevich - jaronev@sandia.gov

- We would like to acknowledge the Hydrogen Effects on Materials Laboratory (HEML)
  - James McNair
  - Brendan Davis
  - Keri McArthur
  - Tanner McDonnell
  - Jeff Campbell

Acknowledgement:

Sandia's Hydrogen Effects on Materials Laboratory (HEML) gratefully acknowledges sustained support from the Office of Energy Efficiency and Renewable Energy's (EERE) Hydrogen and Fuel Cell Technologies Office (HFTO) within the U.S. Department of Energy (DOE). Any subjective views or opinions that might be expressed in the paper do not necessarily represent the views of the U.S. Department of Energy or the United States Government.