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Subcritical Crack Growth in High-pressure Hydrogen and Hydrogen With Oxygen Impurity

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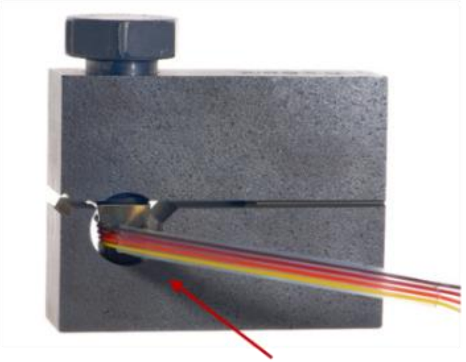
Overview

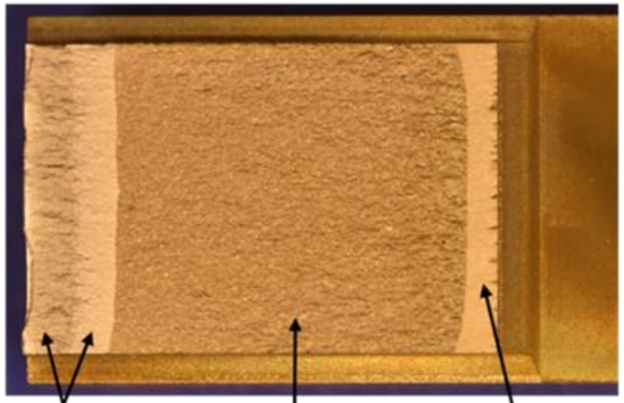
- Background and Motivation
- Experimental Methods
- Subcritical Crack Growth in High-pressure Hydrogen and Hydrogen With Oxygen Impurity
  - SA372 Grade J steel
  - SA372 Grade L steel
  - X100 pipeline steel
  - 13-8 stainless steel
- Summary and Conclusions

Wedge-opened Loaded (WOL) Sample



Reaction pin (load tup) with externally monitored strain gauge

Grade L: WOL Fracture Surface

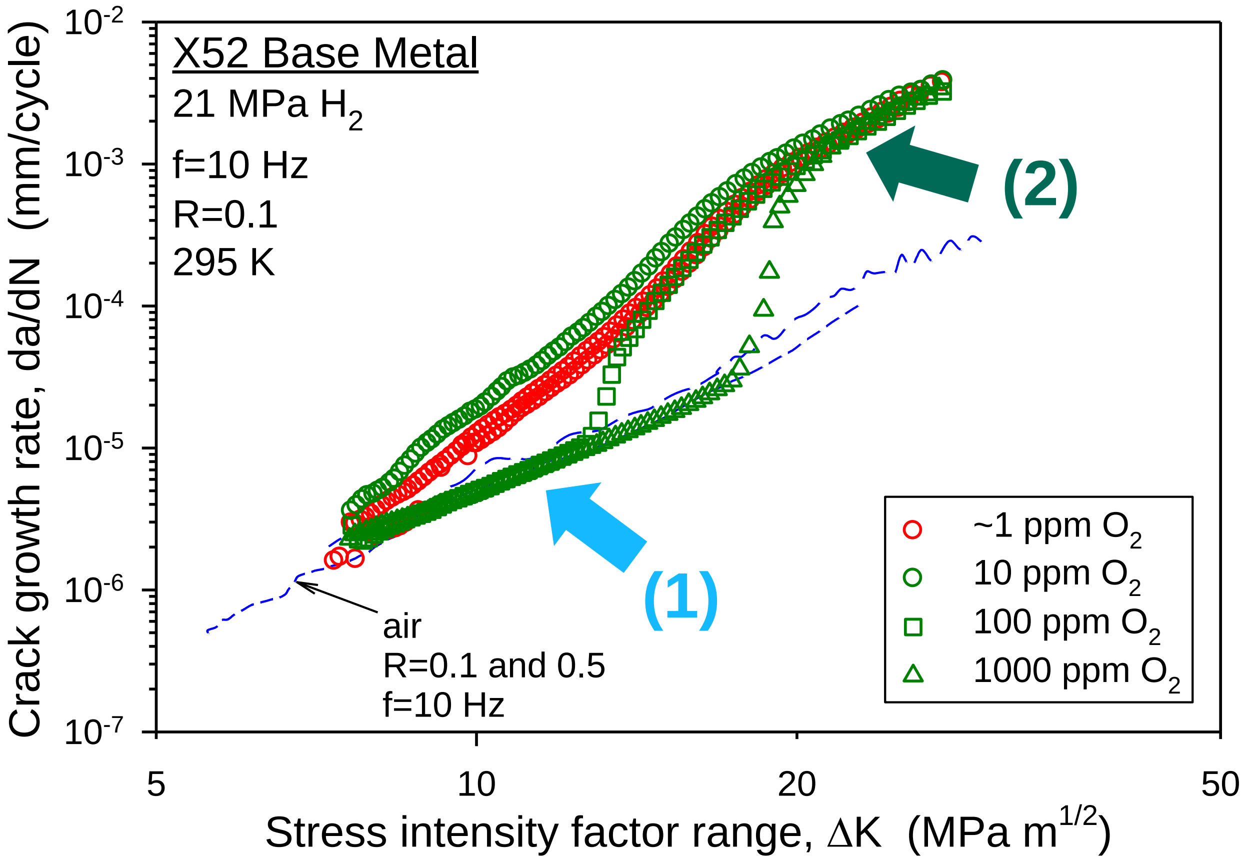


Post-test fatigue and LN_2 fracture

Fatigue precrack (in air)

Crack growth in H_2 + 100PPM O_2

Oxygen is known to affect measurements of fatigue and fracture



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

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

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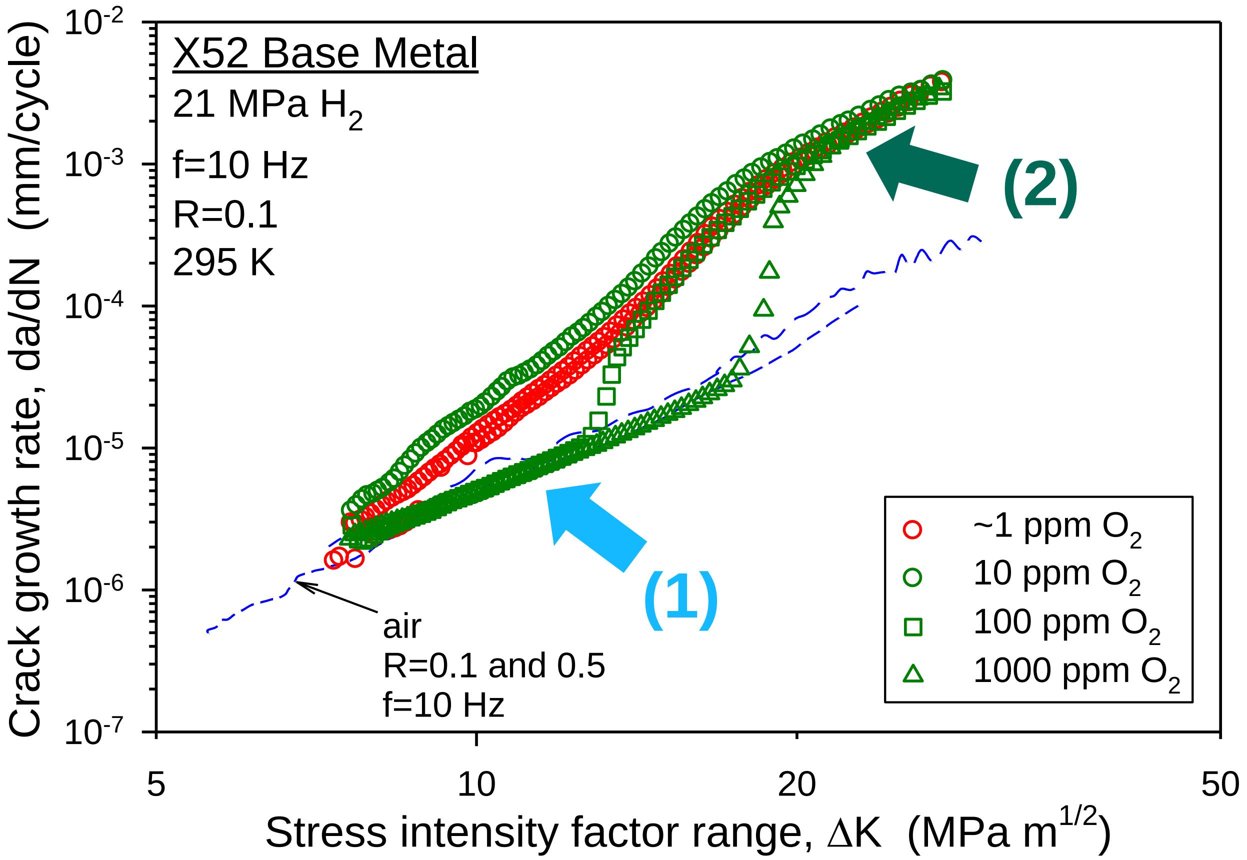


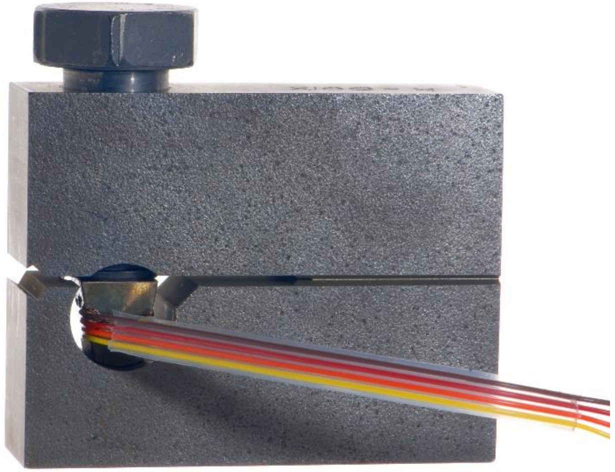
- Fatigue crack growth tests are typically performed at 1 Hz (±decade)
  - $da/dN = 10^{-5}$ mm/cycle
  - Time for Δa = 1mm: ~1 day
  - 1 day = 0.02% of 10 year life
- Are the time scales of a typical laboratory fatigue test sufficient to demonstrate kinetics over decades?
  - More accurately simulate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

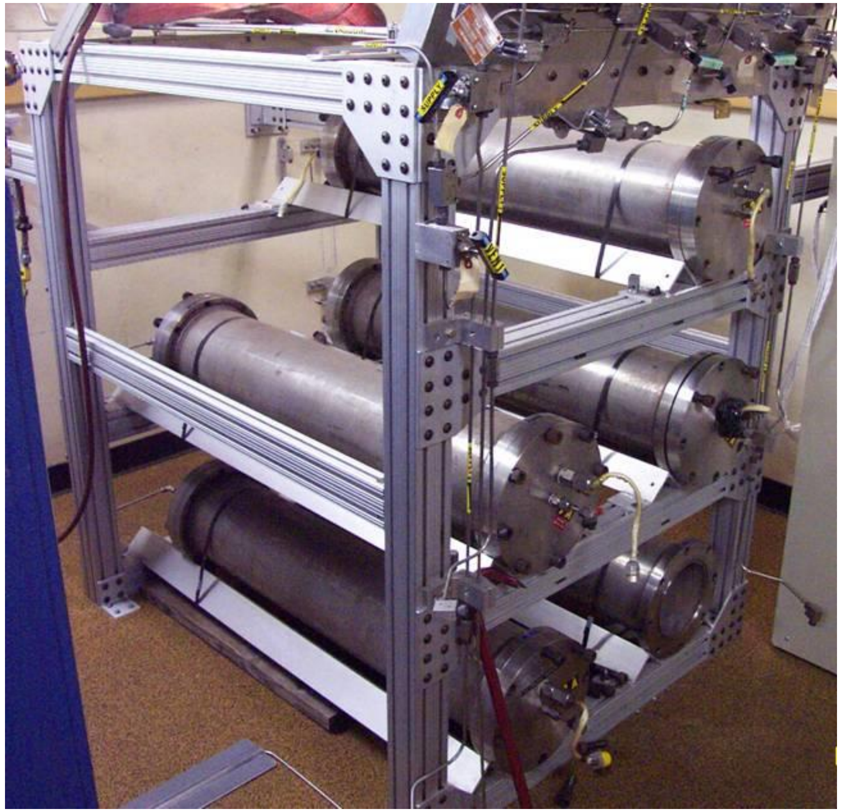
Ref.: Somerday et al, *Acta Mater* 61 (2013) 6153.
Ref.: Nibur et al, SAND2010-4633 (2010).

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
- Test durations can range from days to years
- Instrumented reaction pins allows us to determine incubation time
- Directly compare subcritical crack growth in hydrogen and mixed gas environments

Wedge-opened loaded (WOL)

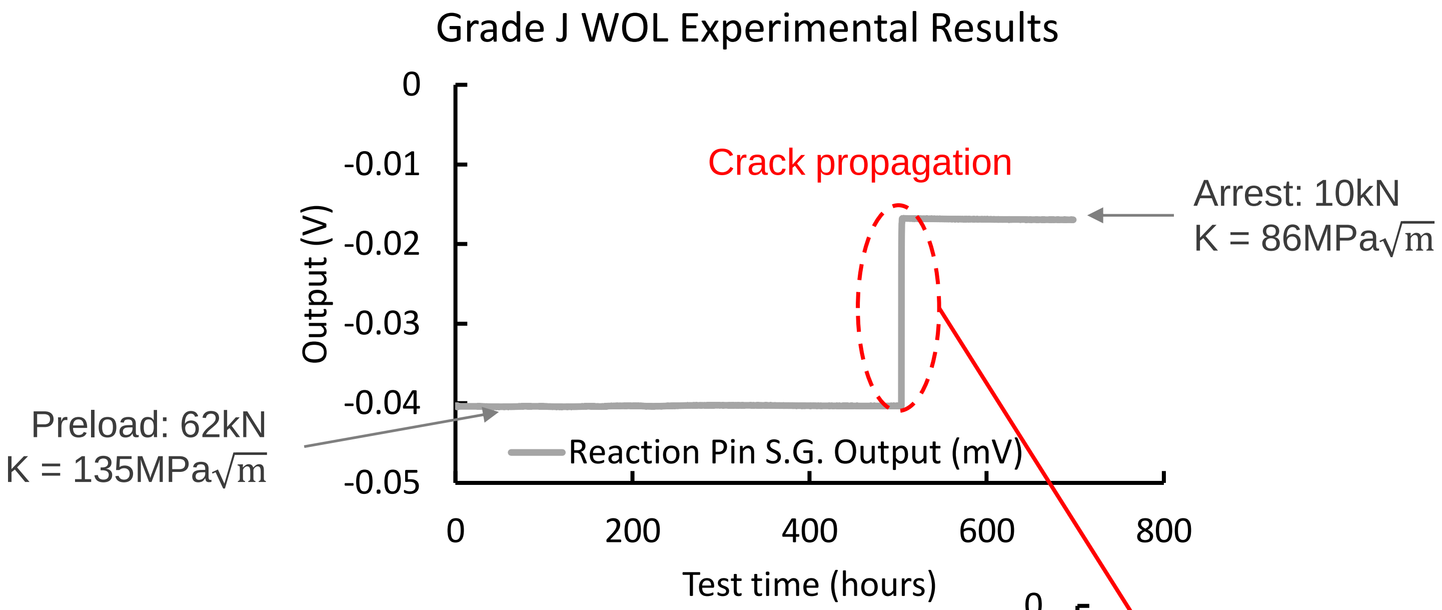


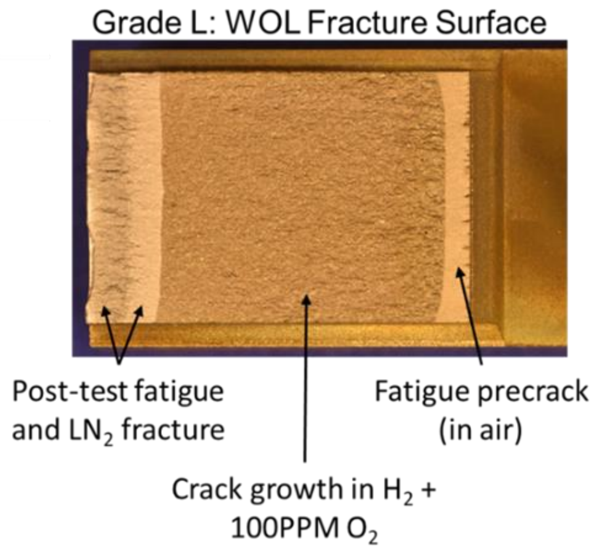


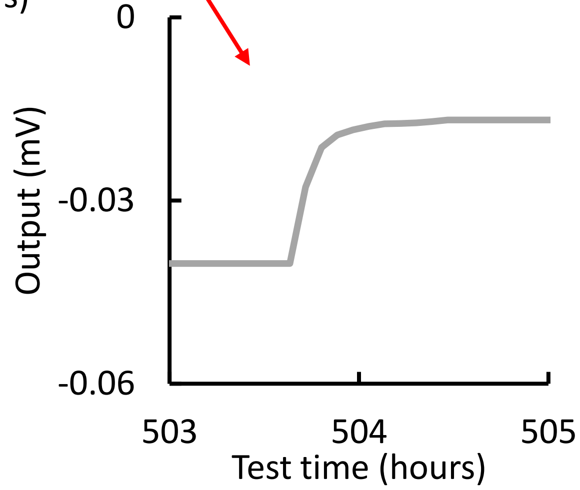
ASTM E1681 – Threshold Stress Intensity Factor for Environment-Assisted Cracking

Crack initiation and growth rates can be 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

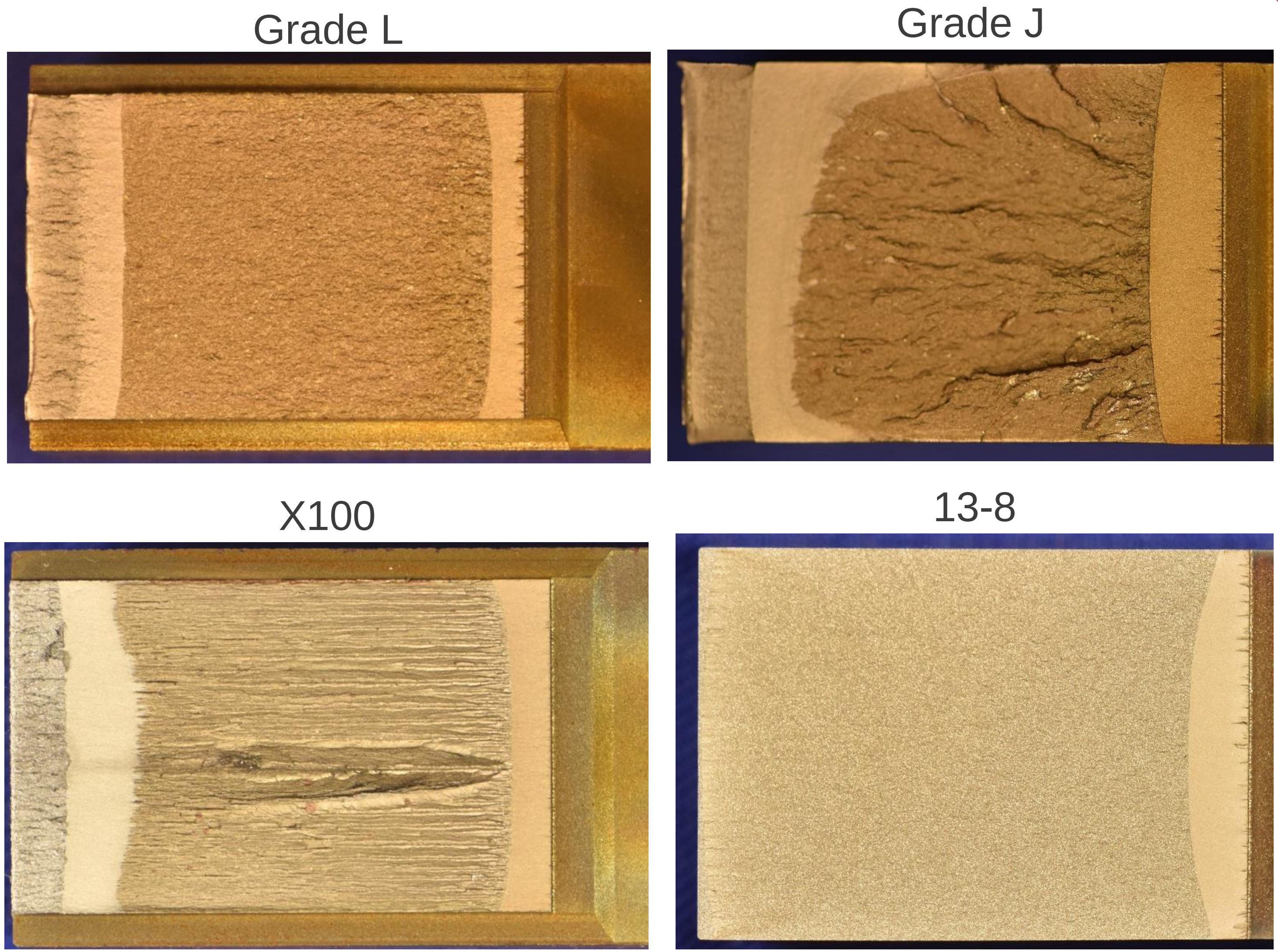




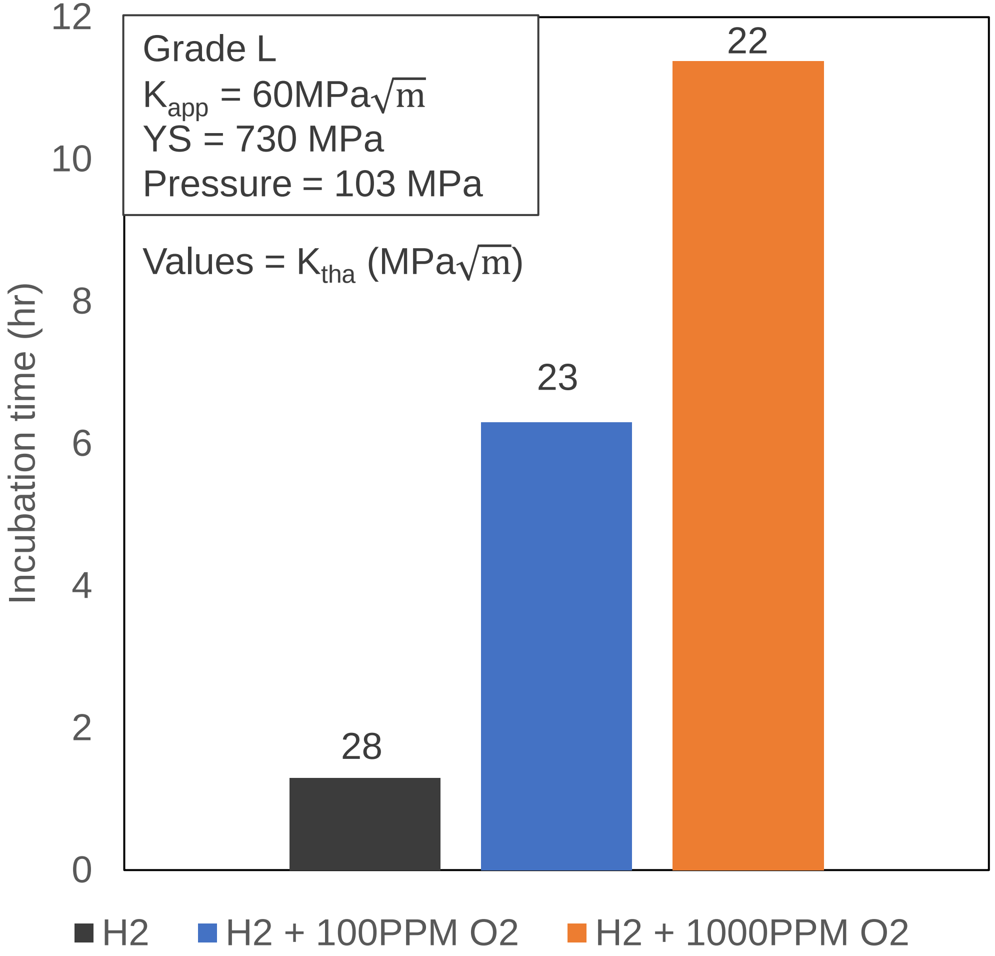


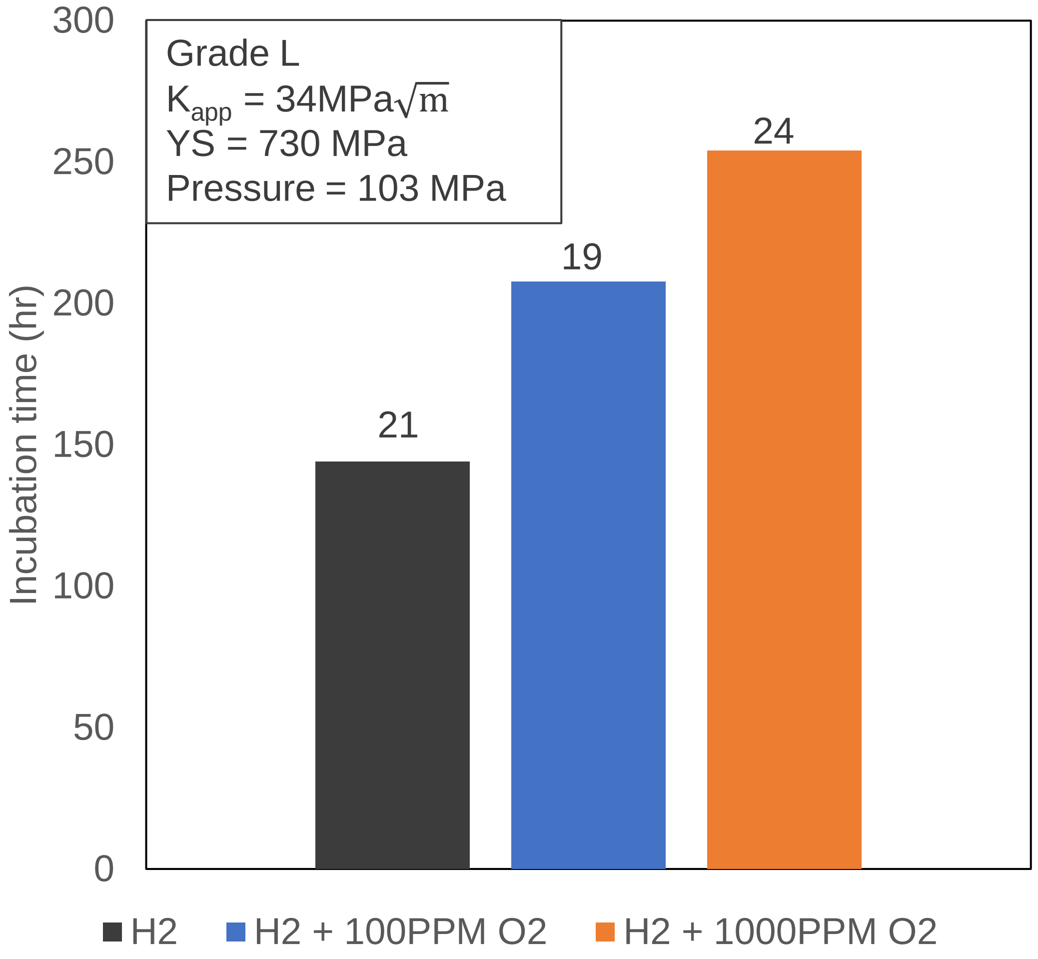
Material selection and fracture surfaces

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



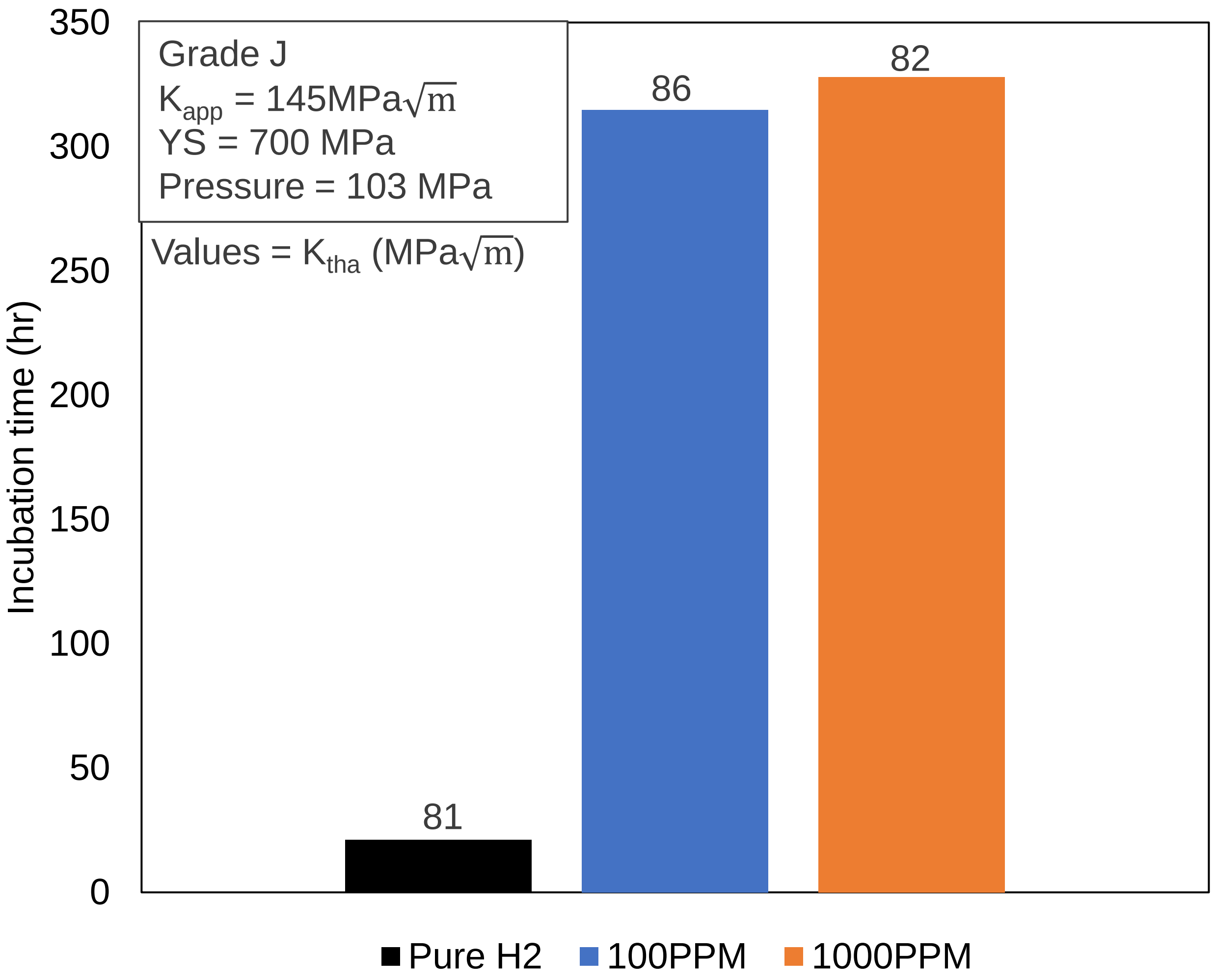
100PPM & 1000PPM O_2 delay incubation time

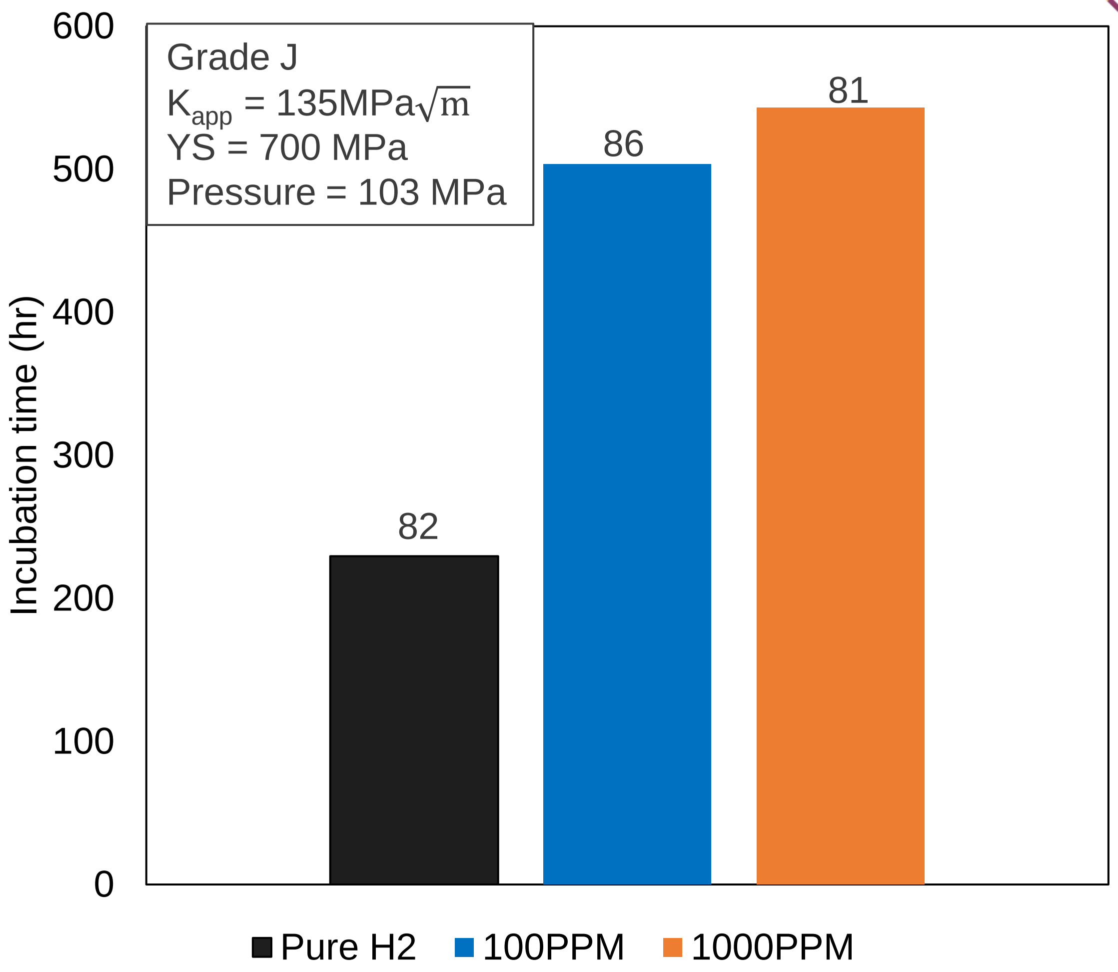




- Grade L shows a 5x delay at higher preload (K_{app} = 60 MPa$\sqrt{m}$) and a 1.5x delay at lower preload (K_{app} = 34 MPa$\sqrt{m}$)
- Similar crack arrest thresholds for all test conditions

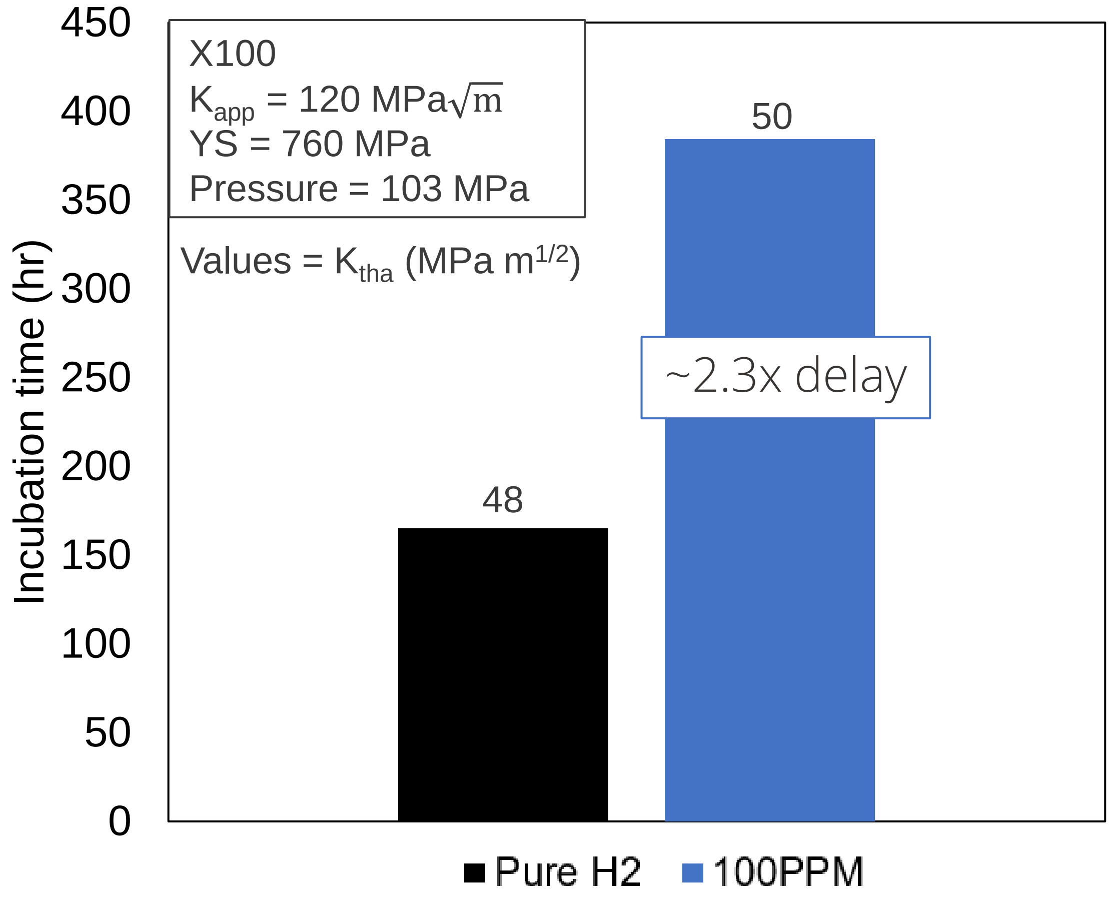
100PPM & 1000PPM O_2 delay incubation time

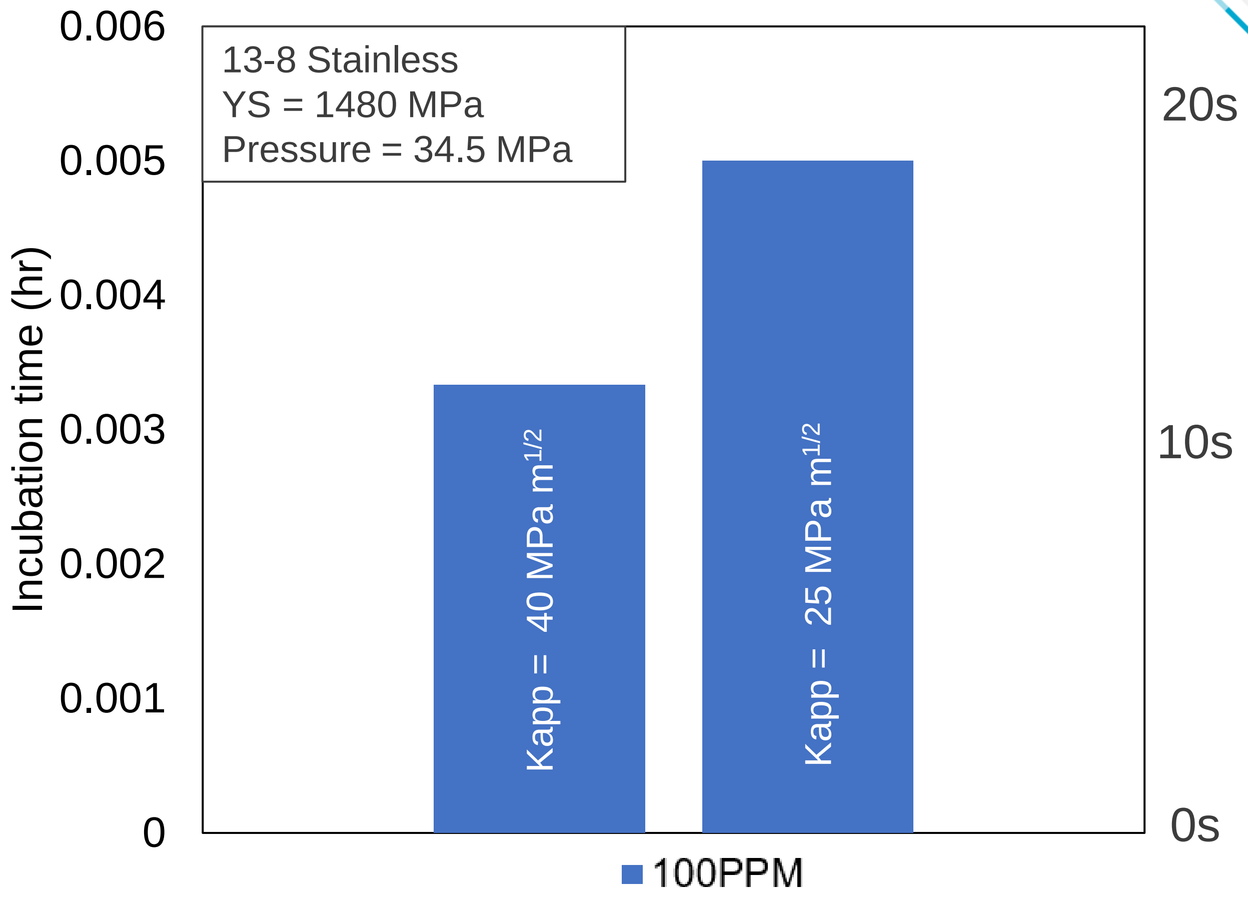




- Similarly, the Grade J material showed delays of 15x at a higher preload (Kapp = 145 MPa$\sqrt{m}$) and a 2.2x delay increase at a lower preload (Kapp = 135 MPa$\sqrt{m}$)
- K thresholds were within ±5 MPa$\sqrt{m}$ of average for both the pure and mixed gas conditions

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

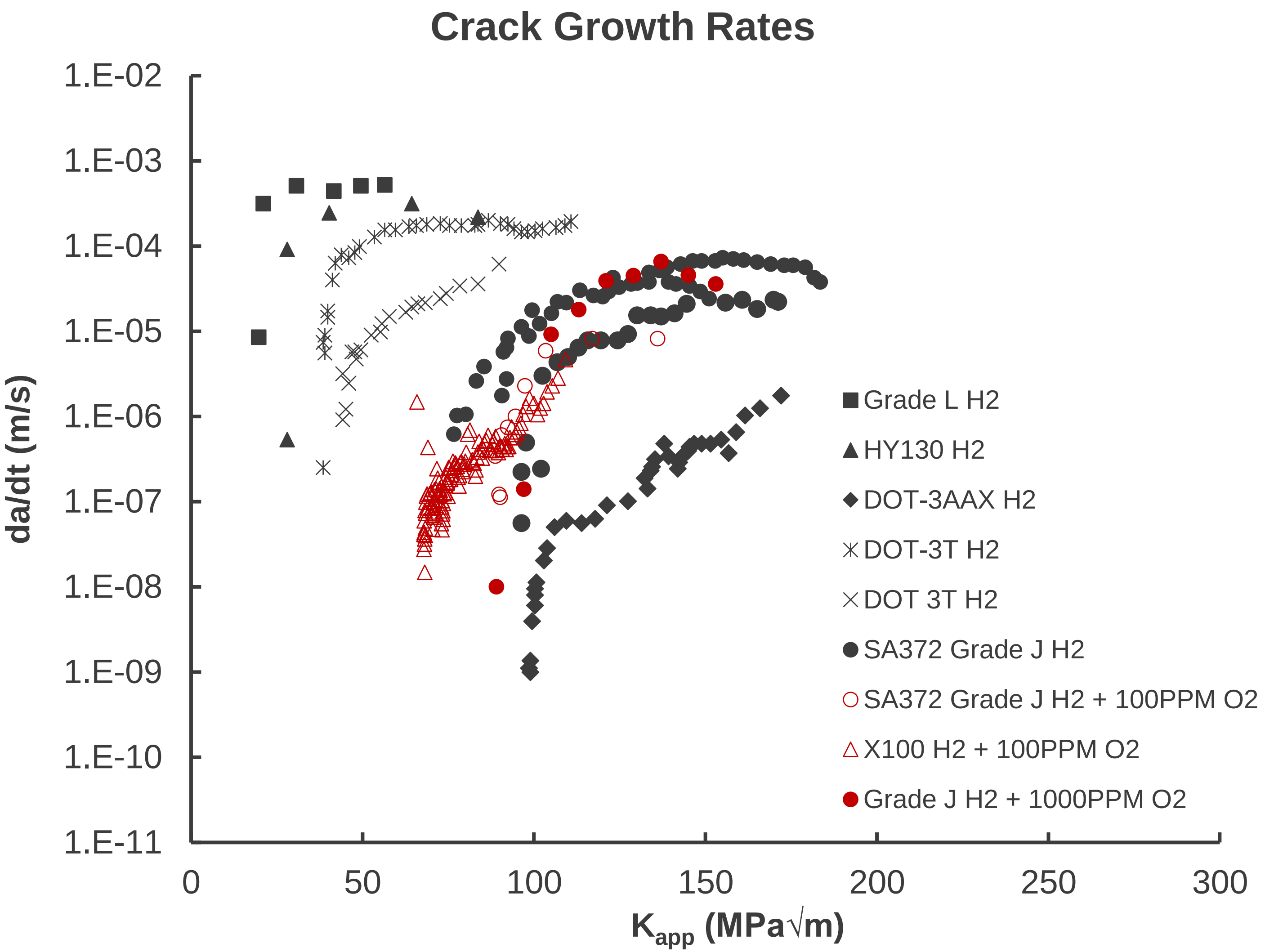




- X100 also saw a delay with the addition of 100PPM O_2
- Both 13-8 samples fractured (a/W > 97%) within seconds of exposure to H_2 + 100PPM O_2

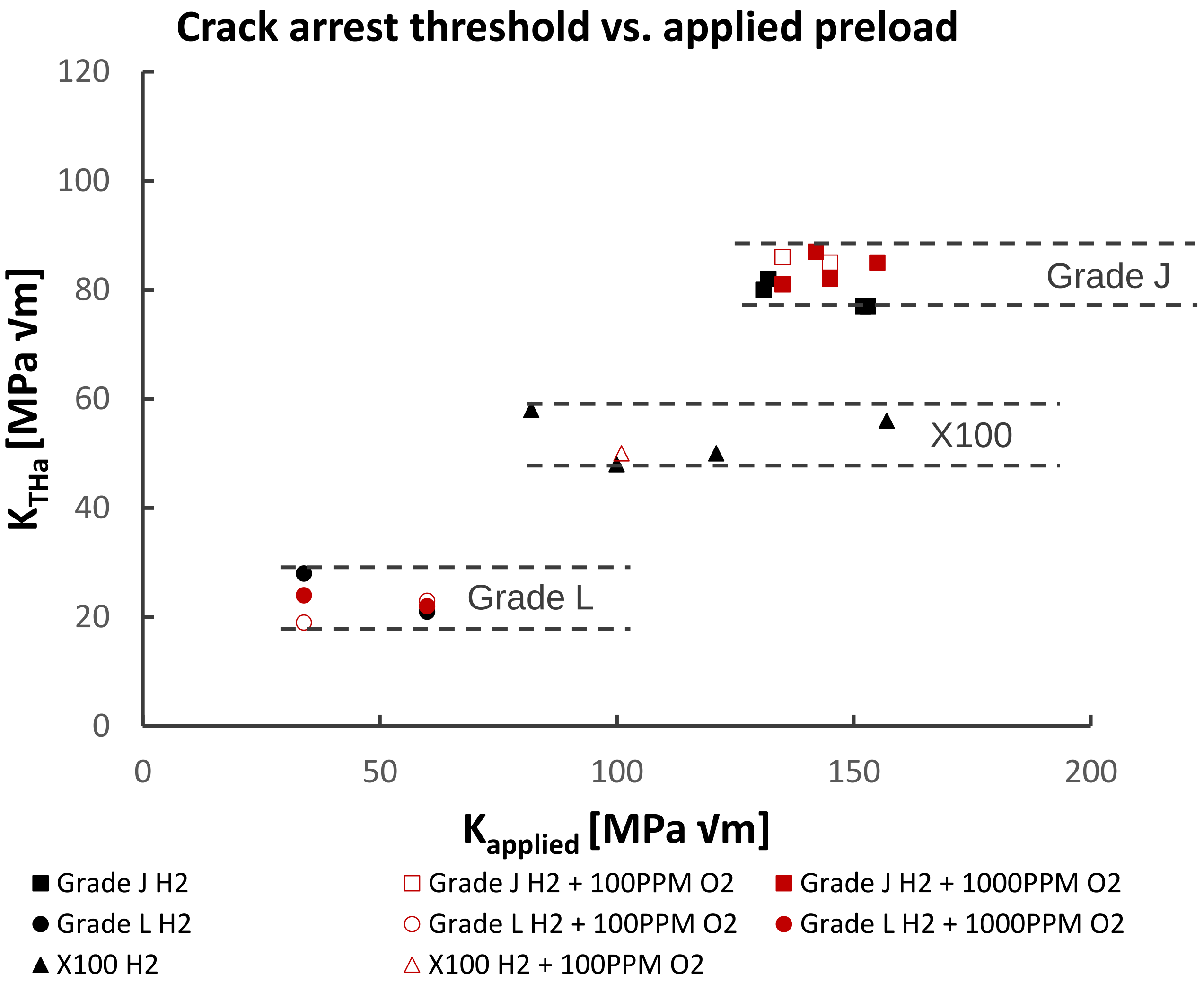
Similar crack growth rates and arrest thresholds for both H_2 and mixed gas environment

- Crack growth rates (da/dt) fall within the expected ranges from previous tests in pure hydrogen at similar pressures
- Adding oxygen to a high pressure hydrogen environment (103MPa) was shown to increase the time it took for subcritical cracks to propagate
  - The addition of oxygen impurities did not prevent crack propagation or effect crack arrest thresholds



Summary

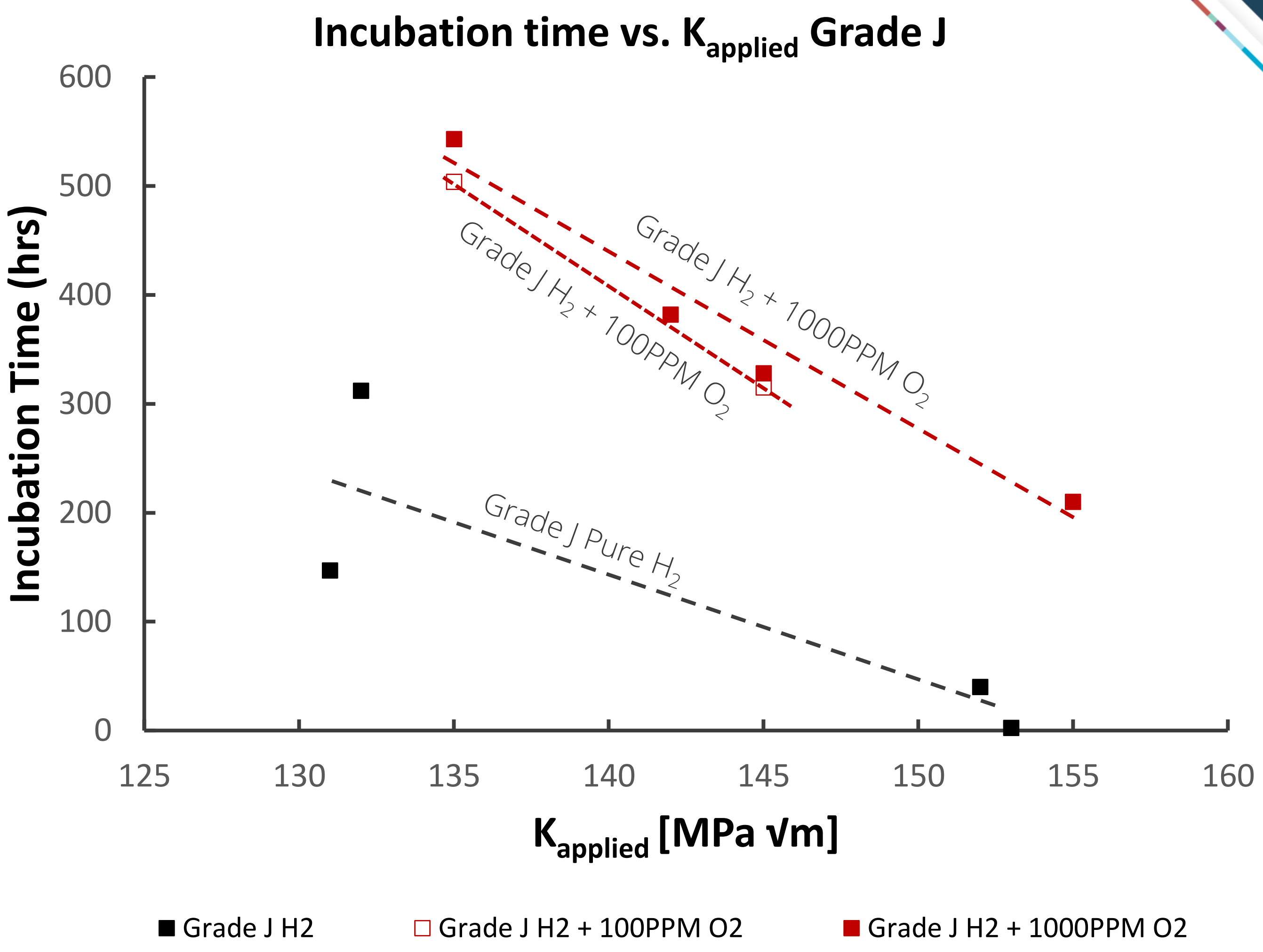
- The effects of low oxygen impurities in hydrogen gas on subcritical crack growth in high pressure hydrogen environment was studied
  - Constant displacement fracture tests were carried out in pure hydrogen and mixed gas (100 and 1000PPM oxygen) environments at 103MPa (15ksi)
- K_{THa} appears to be independent from oxygen content
  - All tests with pure hydrogen and oxygen impurities fall within an apx. 10MPa$\sqrt{m}$ range
- The 13-8 material fractured nearly through the width of the sample during initial pressurization



Summary

- Introducing 100PPM oxygen increased the incubation time by factors between 1.5x and 15x, but did not prevent crack propagation
- Increasing the oxygen content from 100PPM to 1000PPM further delayed the incubation time, but had a smaller relative effect than going from pure hydrogen to hydrogen + 100PPM oxygen
  - The relative increase in incubation time when moving from 100 to 1000PPM oxygen was minor for the Grade J material
- Based on this data, low oxygen impurities should not be relied upon for long-term mitigation of hydrogen embrittlement

- We would like to acknowledge James McNair, Jeff Campbell, and Brendan Davis for their assistance with the experimental setup



Thank you for your attention!

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