

Study Group on Materials Testing and Qualification for Hydrogen Service

Chastain E (room) - Friday, July 21, 2023

Peachtree Westin, Atlanta GA

8:00am – 5:00pm (local time)

8:15	Opening Remarks and Introductions
Standards for hydrogen transmission	
	<i>Current state of standards, Taylor Shie</i>
	<i>Welding, hot tapping and plugging in-service hydrogen lines, Otto Jan Huising</i>
	<i>ANSI/CSA CHMC1, Chris San Marchi</i>
9:45	Break
Component testing and design methods	
	<i>Full-scale pipeline testing update, Bostjan Bezensek</i>
	<i>Subscale testing to simulate full-scale pipe testing, Chris San Marchi</i>
	<i>Fatigue-based versus crack growth-based design methods for hydrogen components – pros and cons?, Carl Fischer</i>
12:00	Lunch (no host)
Test methods for low strength materials	
	<i>Best practices for fracture testing in gaseous hydrogen, Joe Ronevich</i>
	<i>Lessons learned from sour service testing, Robin Gordon</i>
	<i>Advanced practices for fatigue testing in gaseous hydrogen, Chris San Marchi</i>
15:00	Break
Trends of hydrogen-assisted fatigue and fracture	
	<i>Hydrogen-assisted fatigue and fracture test results from HyBlend, Milan Agnani</i>
	<i>Effect of impurities on hydrogen-assisted fatigue and fracture, Rob Wheeler</i>
	<i>Effect of fatigue pre-crack condition for fatigue crack growth test, Un-Bong Baek</i>
	<i>Characterization of hydrogen embrittlement of line pipe steels using sub-size specimens, Yazid Madi</i>
Free Discussion/Wrap-up	
17:00	Adjourn

Study Group on Materials Testing & Qualification for Hydrogen Service

CURRENT STATUS OF STANDARDS

TAYLOR SHIE, FASME (SHELL)

Taylor Shie, FASME (Shell)

Senior H2 Supply Chain Engineer

- » Nine years at DNV and ten years at Shell
 - Pipeline integrity management as primary technical area
- » Member of B31.4 Code Committee Since 2006
- » Past Chair ASME Pipeline Systems Division
- » Chair of ASME Energy Sources and Processing Technology Group
- » 2022 International Pipeline Conference Technical Chair
- » Co-Author of Pipeline Integrity Management: A Practical Approach (ASME Press)

Agenda

- » ASME Code Structure
- » How to Make Changes to ASME Codes
- » Problem Statement for Hydrogen Pipelines
- » Competing Hydrogen Pipeline Standards
- » Potential Solutions
- » Recommended Path Forward
- » What Happens Next
- » How to Get Involved

Backup Slides

- » Other Options Considered
- » Key Areas for B31.12 Improvement
- » PRCI and EFI Descriptions
- » EPRG White Paper Update

ASME Codes Structure



ASME Codes And Standards Are:

Consensus Standards
Standing Committees
Volunteer Driven



ASME Strives To Have A Balance Of Interested Groups:

Manufacturer	User
Pipeline Operator/Owner	Constructor
Designer	Regulatory
Insurance/Inspect	



ASME Code Reporting Structure:

Board of Pressure Technology and Codes and Standards

↳ B31 Standards Committee

↳ B31 Code Committee (e.g., B31.3, B31.4, B31.8, etc.)

How do you Change ASME Codes?

1. A record is opened either by a committee member or from an external party ([link](#))
2. A project manager is assigned within the committee to work the record to resolution
3. The record is refined and balloted within the committee
4. The record is balloted to the B31 Standards Committee
5. The record is balloted to the Board of Pressure Technology and Codes and Standards
6. The change appears in next edition
 - B31.4 and B31.12 (3 years), B31.8 (2 years)

Current Text	Proposed Revision with Mark-up
403.2 Criteria for Pipe Wall Thickness and Allowances 403.2.1 Criteria. The nominal wall thickness of straight sections of steel pipe shall be equal to or greater than t_n determined in accordance with the following equation: $t_n \geq t + A$ where A = sum of allowances for threading, grooving, corrosion, and erosion as required in paras. 403.2.2 through 403.2.4, and increase in wall thickness if used as protective measure in para. 403.1 t_n = nominal wall thickness satisfying requirements for pressure and allowances t = pressure design wall thickness as calculated in inches (millimeters) in accordance with the following equations: (U.S. Customary Units) $t = \frac{P_i D}{2S}$ (SI Units) $t = \frac{P_i D}{203}$ where D = outside diameter of pipe, in. (mm) P_i = internal design gage pressure, psi (bar) S = applicable allowable stress value, psi (MPa), as determined by the following equation: $S = F \times E \times S_y$ specified minimum yield strength of the pipe, psi (MPa) where E = weld joint factor as specified in Table 403.2.1-1 F = design factor based on nominal wall thickness. The value of F used in this Code shall be not greater than 0.72. Where indicated by service or location, users of this Code may elect to use a design factor, F, less than 0.72. In setting design factor, due consideration has been given to and allowance has been made for the underthickness tolerance and maximum allowable depth of imperfections provided for in the specifications approved by the Code. The value of F used in this Code shall be not greater than 0.72. Where indicated by service or location, users of this Code may elect to use a design factor, F, less than 0.72.	403.2 Criteria for Pipe Wall Thickness and Allowances 403.2.1 Criteria. The nominal wall thickness of straight sections of steel pipe shall be equal to or greater than t_n determined in accordance with the following equation: $t_n \geq t + A$ where A = sum of allowances for threading, grooving, corrosion, and erosion as required in paras. 403.2.2 through 403.2.4, and increase in wall thickness if used as protective measure in para. 403.1 t_n = nominal wall thickness satisfying requirements for pressure and allowances t = pressure design wall thickness as calculated in inches (millimeters) in accordance with the following equations: (U.S. Customary Units) $t = \frac{P_i D}{2S}$ (SI Units) $t = \frac{P_i D}{203}$ where D = outside diameter of pipe, in. (mm) P_i = internal design gage pressure, psi (bar) S = applicable allowable stress value, psi (MPa), as determined by the following equation: $S = F \times E \times S_y$ specified minimum yield strength of the pipe, psi (MPa) where E = weld joint factor as specified in Table 403.2.1-1 F = design factor based on nominal wall thickness. The value of F used in this Code shall be not greater than 0.72. Where indicated by service or location, users of this Code may elect to use a design factor, F, less than 0.72. In setting design factor, due consideration has been given to and allowance has been made for the underthickness tolerance and maximum allowable depth of imperfections provided for in the specifications approved by the Code. The value of F used in this Code shall be not greater than 0.72. Where indicated by service or location, users of this Code may elect to use a design factor, F, less than 0.72.

Supporting info

Excerpt from ASME B31.8-2016 that shows how this formula is presented in another code

Problem Statement for Hydrogen Pipelines

- » There is a large movement in the pipeline industry to update and improve ASME code language as they relate to hydrogen pipelines
- » The current guidelines in B31.12 for pipelines are not always technically justified and lead to excessive conservatism by the users of the code
- » B31.12 has had 40+ visitors for three meetings in a row that are mainly pipeline participants
 - This shows enthusiasm for the code and the need for action with respect to the pipeline chapter.
 - These visitors need a mechanism to be engaged in the process and retained as volunteers in the code development process.
 - The B31.12 committee has struggled to achieve quorum at recent meetings. It appears that the majority of the work lands on the shoulders of a few.

Competing Hydrogen Pipeline Standards

- » AS 2885 update for hydrogen (Australia)
- » Z662 update for hydrogen (Canada)
- » DNV-OS-F101 / RP-F104 (Norway)
- » ISO 13623 (Europe)
- » EPRG Hydrogen Pipelines Integrity Management and Repurposing Guidelines
- » IGEM/TD/1 Edition 6 Supplement 2

Note: Many operating companies, service providers, and standards bodies have embarked on the journey to create hydrogen standards and guidance

Potential Solutions for Pipelines Options Considered by B31 Leaders

- » **Option 1** - Development of B31.8 chapter for hydrogen pipelines ahead of 2024 or 2026 edition and removal from B31.12
- » **Option 2** - Update of B31.12 pipeline chapter ahead of 2026 edition
- » **Option 3** - Update of both B31.8 and B31.12 with clear delineations of when to apply each code
- » **Option 4** - Do nothing

Recommended Solution for Pipelines

- » **Option 1** - Development of B31.8 chapter for hydrogen pipelines ahead of 2024 or 2026 edition and removal from B31.12
 - Sour service can be used as a template for the update
 - Other gases (e.g. N₂, O₂, etc.) may need similar chapters
 - ASME B31.8 is the code of record for gas transmission and distribution
 - Sections of B31.8 are already incorporated in 49CFR192
 - PRCI and other international organizations are looking to bolster ASME hydrogen pipeline code and need a location
 - The 40 visitors from B31.12 would likely move to this effort

Recommended Solutions Added During Discussion

- » Members of the B31 Standards Committee recommended that the Process Piping sections be removed from B31.12 and put into B31.3
- » Members of the Board of Pressure Technology Codes & Standards requested that the Power Piping sections be removed from B31.12 and put into B31.1

Implementation Plan for Pipelines

- » PRCI EFI will develop and fund a project to write the new chapter of ASME B31.8 on ASME's behalf.
 - Work will include national lab and other international research groups work in the space
 - PRCI EFI will engage the 40 visitors of B31.12 and ASME B31.8 committee during the project to ensure industry consensus is achieved.
 - ASME and PRCI EFI will jointly discuss the pending changes with PHMSA
- » Work product will be provided to ASME B31.8 with sufficient time for review and approval ahead of the 2024 or 2026 edition's publication
 - All revisions must be approved by the B31 Standards Committee by April 15th of the publication year

How Does This Effort Mesh With Competing Hydrogen Standards

- » AS 2885 update for hydrogen (Australia)
- » Z662 update for hydrogen (Canada)
- » DNV-OS-F101 / RP-F104 (Norway)
- » ISO 13623 (Europe)
- » EPRG Hydrogen Pipelines Integrity Management and Repurposing Guidelines
- » IGEM/TD/1 Edition 6 Supplement 2

Note: Many operating companies, service providers, and standards bodies have embarked on the journey to create hydrogen standards and guidance

Approved Path Forward

- » Move Power Piping from B31.12 to B31.1
- » Move Process Piping from B31.12 to B31.3
- » Move Pipelines from B31.12 to B31.8
- » Retire B31.12 once moves are complete
 - B31.12 will continue to improve the code as these changes are being made

What Happens Next?

Task Groups Are Being Formed

» B31.1 Power Piping

- Lead: Kristi A. Vilminot - Kristi.Vilminot@cai-engr.com

» B31.3 Process Piping

- Lead: Don Frikken - frikkend@fidmail.com

» B31.8 Gas Transmission and Distribution Pipelines

- Lead: Taylor Shie – Taylor.Shie@shell.com

Note: Contact ASME Staff Secretary as well (see next slide)

How to Get Involved In Codes and Standards

www.ASME.org

← → ↻ asme.org

Shell Related Benefits Technical Personal

ASME
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ASME CODES & STANDARDS

- Council on Standards and Certification
- Board Level CSC Training Committee
- Board on Conformity Assessment (BCA)
- Board on Council Operations
- Board on Hearings and Appeals
- Board on Level Training Committee
- Board on Nuclear Codes and Standards
- Board on Pressure Technology Codes and Standards
- Board on Safety Codes and Standards
- Board on Standardization and Testing
- Technical and Strategic Advisory Board

Contact Committees

Contacting ASME Standards Development Committees

Standards development committees meet regularly to consider revisions based on technological advances, new data, and changing environmental, industry and mechanical engineering needs. All meetings are free and open to the general public.

Check the [ASME Calendar of Events](#) for a schedule of upcoming meetings.

Each [Supervisory Board and Standards Development Committee](#) has a web page. By expanding the list you can find all existing committees, the charters defining the scope of each group's activities, information about future meetings and contact information for staff responsible.

Standards & Certification Committees welcome contact from the standards users as well as the general public in connection with:

ASME will only issue cases or interpretations of official ASME codes or standards. Translations, derivative works, or products not created in their entirety by ASME will not be addressed. ASME cases or interpretations apply only to official ASME codes or standards.

- Requests for Interpretation**
Many ASME standards development committees will respond to requests to clarify the application of an existing requirement. To request an interpretation access the [Interpretation Submission Form](#). To review previously issued interpretation access the [Interpretation Database](#).
- Requests for Revision**

Thank you



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Backup Slides

Options Considered

Option 1 - Development of B31.8 Chapter Strengths and Weaknesses

» Strengths

- There is an established format in the code for this process
 - See Chapter IX on sour service
- Immediate savings to the industry to remove incorrect and onerous requirements in B31.12

» Weakness

- Would likely require funding for an external consultant
- Would likely require updates to B31.8S
- Requires approval from Board of Pressure Technology Codes & Standards for scope change
 - Pure hydrogen is a scope change for the codes

Option 2 – Update of B31.12

Strengths and Weaknesses

» Strengths

- Current code of record for hydrogen pipelines globally
- No approval of scope change required from Board of Pressure Technology Codes & Standards
- 40+ potential new volunteers already

» Weakness

- Committee does not appear to understand sense of urgency within industry or risk to ASME of inaction
- The Interest Categories for existing B31.12 members is not transparent
- Conflict with status quo

Option 3 – Update of Both B31.8 & B31.12

Strengths and Weaknesses

» Strengths

- Allows users to select the code that is most appropriate for their application
 - Blend or pure stream
 - Transmission or distribution
- More complete solution for ASME standards
- Robust integration of evolving research
 - Supported by PRCI

» Weakness

- Potential for codes to become divergent over time
- Potential for spreading volunteers too thin
- Requires approval from Board of Pressure Technology Codes & Standards for scope change to B31.8

Option 4 – Do Nothing

Strengths and Weaknesses

» Strengths

- None

» Weakness

- Loss of ASME's standing as the code of record on hydrogen pipelines
- Other organizations will develop competing and potentially conflicting codes
- Confirmation of ASME as a dated organization

Key Areas of B31.12 Improvement

Areas for B31.12 Improvement

- Design Pressure

- » The design Pressure section of B31.12 (PL-3.7.1) includes a Materials Performance Factor (H_f) not included in B31.8 Sour Service
- » This results in overdesign of the entire pipeline system: i.e. increased pipeline wall thickness with no demonstrable reduction in risk
- » Cost implication of >\$10M for pipelines >100 miles

Areas for B31.12 Improvement

- Hardness Control

- » B31.12 GR-3.10 required HV 235 in weld metal, HAZ and BM during welding procedure qualification, which is more conservative than sour service requirements in NACE MR0175, and yet, it is well known that the H flux into the metal is much less in H2 service than in sour service
- » B31.12 PL-3.19.8 required hardness testing of production welds, which measures material properties where it matters the least – the weld cap. Another example of increase in cost, without a reduction on risk
- » These hardness control requirements will drive operators to use sour service grade line pipe, which often comes with a 25% cost premium. The hardness requirements will also add cost to welding qualification and construction, as well as increasing execution risk, all of which will increase construction costs >\$10M for pipelines >100 miles

Areas for B31.12 Improvement

- Repurposing Existing Pipelines

- » Over prescriptive sampling and testing requirements that are not risk or system based.
- » A pipeline dig every one mile to extract samples for destructive testing is extremely costly, particularly for large pipelines systems and networks, without a demonstrable decrease in risk
- » This may be the biggest challenge to the broader adoption of H2 as a fuel source in the H2 economy.

What is the Emerging Fuels Institute?

- » The Emerging Fuels Institute (EFI) was started by Pipeline Research Council International (PRCI) and has 21 corporate members
- » PRCI/EFI performed a state-of-the-art study related to emerging fuels, specifically hydrogen, in 2020 to identify gaps for closure with subsequent research.
- » Target areas are: 1) Transmission and distribution pipeline integrity, 2) Safety, 3) End-use, 4) Metering & gas quality, 5) Network management & compression, 6) Maintenance & inspection, 7) Hydrogen-natural gas separation, and 8) Underground gas storage.
 - EFI Summaries by topic: <https://www.prci.org/Research/JEFI/222296/EFI-00-01/239815/248383.aspx>
- » PRCI/EFI expects to spend ~\$9MM between 2022 and 2025 to close the gaps identified in the 2019 report
 - EFI is also leveraging work performed by national labs and joint industry projects
- » PRCI/EFI desires to imbed these learnings in ASME B31.8/B31.12 or equivalent industry standard.

What is the Relationship Between PRCI/EFI and ASME?

- » The Executive Director of ASME (Tom Costabile) and the President of PRCI (Cliff Johnson) have had a series of discussions on ways of working between the organization and how to formalize them.
- » PRCI would like to provide the results of its research for inclusion in the improvement of ASME Codes and Standards. ASME would then provide access to conference proceedings for use in PRCI research.
 - This requires ASME Codes and Standards and Conferences and Publication groups to both agree
- » EFI has targeted the B31 codes as the location to provide the results of its research to in order to improve the code over time.
- » PRCI makes significant content contributions to ASME events

Current Status

- Existing hydrogen pipelines are generally designed and operated in accordance with hydrogen specific codes
- Code landscape is currently fluid – various codes either recently issued or in development
- Most current codes either explicitly or implicitly refer back to ASME B31.12
- Research is currently moving very quickly, standards are struggling to keep up
- Practical application of ASME B31.12 is challenging, especially in terms of repurposing
- There is a lack of detailed guidance on integrity management in hydrogen service

ASME B31.12-2019
(Revision of ASME B31.12-2014)

Hydrogen Piping and Pipelines

ASME Code for Pressure Piping, B31

AN AMERICAN NATIONAL STANDARD



EPRG Position

- Significant H2 pipeline research programme launched
- Ultimate aims are to develop a Guideline for repurposing existing pipelines to H2 service, and a Guideline for new H2 pipelines
- The Guidelines will be designed to support existing codes, with the aim of being incorporated into or referenced by future editions of hydrogen pipeline codes
- In the interim a Repurposing Guideline white paper has been developed and is now before EPRG plenary for review

Executive Summary

EPRG Hydrogen Pipelines Integrity Management and Repurposing Guideline White Paper – Final Draft

Executive Summary

EPRG has launched a significant H₂ pipeline research programme with the ultimate aim of developing a Guideline for repurposing existing onshore/offshore pipelines to H₂ service and a Guideline for new onshore/offshore H₂ pipelines.

There are many existing industry design codes which reference hydrogen, the most detailed being ASME B31.12:2019 with other standards taking this code as a basis. B31.12 is challenging to apply practically to repurposing pipelines, particularly accounting for historic defects and damage and does not incorporate the latest research knowledge with specific application to Europe.

In the interim, due to the industry and government requirements to repurpose existing pipelines to H₂ service at an accelerated pace, the rapid evolving research developments in this area, and conscious that standards development cannot keep pace, EPRG has decided to develop a repurposing Guideline white paper that:

- Provides practical interim guidance on several requirements and input parameters (identified as key aspects in EPRG) in ASME B31.12 Option A and B using a three level approach depending primarily on the operating envelope and existing condition of the pipeline. The general philosophy behind these three levels is shown below, and further details may be found in the relevant appendices:
 - Level 1 Screening assessment aligned to ASME B31.12 Option A
 - Level 2 Standard assessment aligned to ASME B31.12 Option B but with minimal fatigue pressure/longitudinal loading or pipelines assessed according to experimentally derived S-N curves
 - Level 3 Detailed assessment aligned to ASME B31.12 Option B with possibly higher fatigue pressure/longitudinal loading and/or potentially defects outside ASME B31.12 limits and/or higher static loading in the longitudinal direction.
- Guidance for these key aspects is given with background commentary and identification of gaps either to be closed or requiring a new research programme.
- Identification of gaps that are being closed by ongoing research and gaps that are outstanding.

This Guideline is intended to augment existing standards with non-mandatory clarification guidance. Where aspects/requirements are not covered, it is deemed that ASME B31.12 is clear and requires no further clarification. This Guideline should not be used as a design code. In some instances, the requirements in ASME B31.12 may have been superseded by recent advances in research since the last publication of B31.12, and in these cases, it is recommended that the specific requirement be discussed with the jurisdictional authorities to determine if this can be revisited, recognising that the jurisdictional authorities are the ultimate authority in these matters.

The Guideline is aimed at repurposing pipelines for effectively 100 % H₂ service and can be applied for

Repurposing White Paper Aims

- Provide practical interim guidance on key aspects (requirements and input parameters) identified in ASME B31.12 Option A and Option B
- 3 level approach adopted – based on operating envelope and existing condition of pipeline
 - Level 1 – screening assessment aligned to B31.12 Option A (lower utilisation factor / operating stress)
 - Level 2 – Standard assessment aligned to B31.12 Option B but with minimal pressure / longitudinal fatigue loading – assessed according to crack growth calculations or S-N curves
 - Level 3 – Detailed assessment aligned to B31.12 Option B – full ECA approach for higher loading or larger potential defects

Repurposing White Paper Structure

- Each level has guidance related to the following three categories
 - Degradation and defects
 - Loading and operation in hydrogen service
 - Material requirements and restrictions
- For each topic, the level of confidence associated with the guidance is stated
- Specific recommendations for revisiting or replacing requirements within ASME B31.12 (e.g. the onerous hardness restrictions) are made where the data is felt sufficient to support this
- In addition to the levels Gaps are outlined which are considered to exist in current codes and assessment methods
- Gaps are classified according to whether they are currently being addressed by EPRG or other industry research, or whether they remain open
- The list of Gaps and current work reflects EPRG's best current knowledge, but is unlikely to be exhaustive

HyTap

In-service welding, hot tapping and stoppling
on a 20"(DN500) 66 bar(g) hydrogen line

O.J.C. Huising



Background



Content

- In-service welding JIP
- Welding parameters
- HyTap – welding
- HyTap – tapping
- HyTap – stoppling
- HyTap – completion plug
- Conclusion



DNV Research

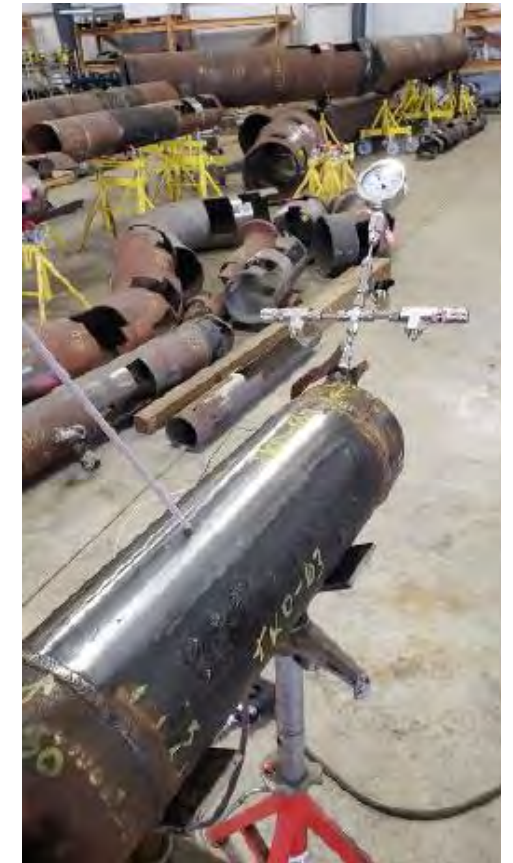
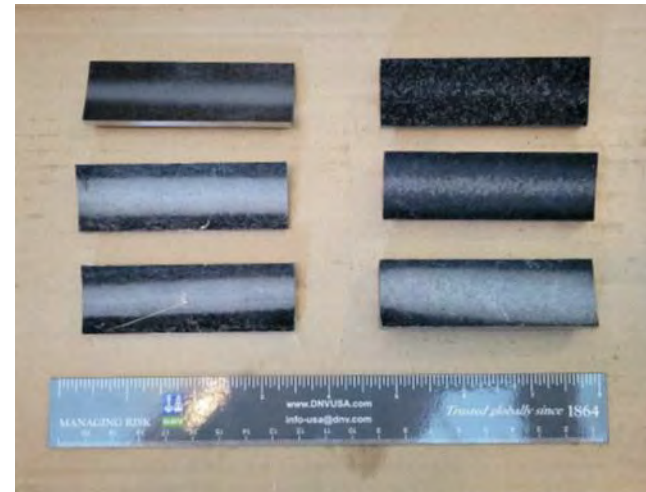
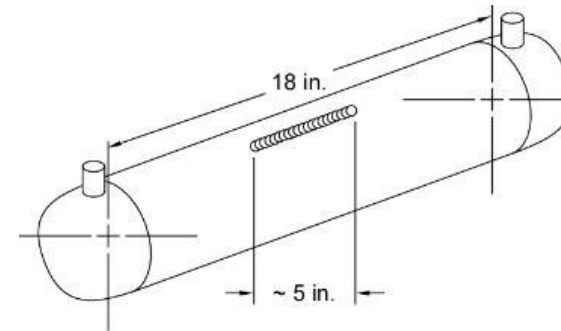
DNV JIP- In-Service Welding onto Hydrogen and Methane/Hydrogen Mixture Pipelines

Scope

- Determine the extent to which steel line pipe can become charged with hydrogen
 - Literature survey, evaluation of Hawaii Gas data/experience, etc.
 - Relevant experimental results from other projects (e.g., HyReady)
- Determine the extent to which elevated weld hydrogen levels can result from:
- Welding onto pipe pressurized with pure hydrogen and blends of hydrogen and methane
- Welding onto steel charged with hydrogen
- For cases that result in elevated weld hydrogen levels, develop measures to mitigate the increased risk of hydrogen cracking

Experimental procedure 1-3

- Prepare weld area
 - Remove mill scale/varnish/coating
 - Scrub with acetone and steel wool
 - Wipe with acetone immediately prior to welding
- Purge and pressurize vessels
 - Four purge cycles with nitrogen to 100 psi (0.69 MPa)
 - One purge cycle with methane/hydrogen or hydrogen to 400 psi (2.76 MPa)
 - Pressurize with test gas to test pressure plus 40 psi (0.28 MPa)
 - Bleed off vent line to test pressure (400 or 800 psi [2.76 or 5.52 mPa])
- Prepare for welding
 - Open foil pouch of E7018-H4R electrodes, transfer to holding oven, remove for use one at a time.
 - Set electrode angle and spacing and place small ball of steel wool for arc initiation



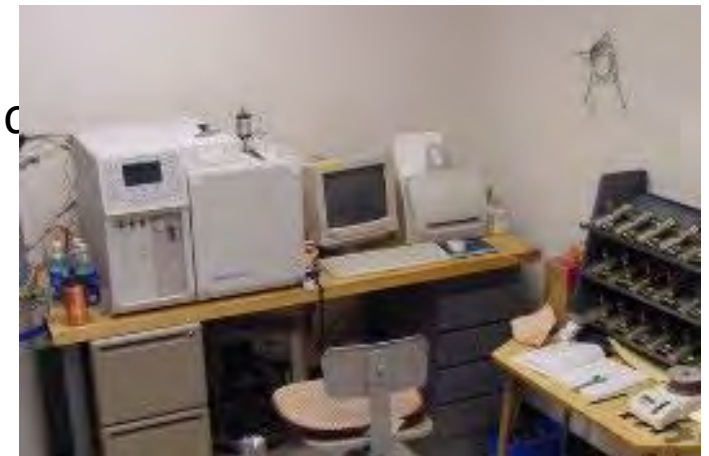
Experimental procedure 2-3

- Make weld
 - Vacate area and initiate weld by remote control
 - Control weld length by arc time
- Depressurize and quench
 - Measure weld length while vessel depressurizes
 - After 15 seconds, transfer to ice water bath
 - After 10 additional seconds, transfer to tub for application of liquid nitrogen
 - Swab with liquid nitrogen until temperature is well below 0°C (32°F)
 - Transfer to freezer at -30°C (-22°F)
- Transfer vessels to machine shop in dryice the following day
 - Cool specimens to liquid nitrogen temperature
 - Remove specimens by cryogenic machining



Experimental procedure 3-3

- Transfer specimens to laboratory in dry ice
 - Follow AWS A4.3 procedure for warming to ambient temperature/cleaning/drying specimens
 - Place in canisters and purge with argon for hydrogen bake out
- Perform analysis using gas chromatograph
 - Receive raw data the following day
 - Retrieve specimens for deposited weld metal weight determination
 - Weigh specimens with welds
 - Determine pre-weld weight based on dimensions of specimens and density of steel
- Calculate diffusible hydrogen in ml/100 g of deposited weld metal



Results on hydrogen at 800 psi (5.52 MPa) pipes

For thicker materials (0.280 in. [7.1 mm] thick):

- Moderate increase in weld hydrogen level
 - ~2.6 –3.3 ml/100 g increase

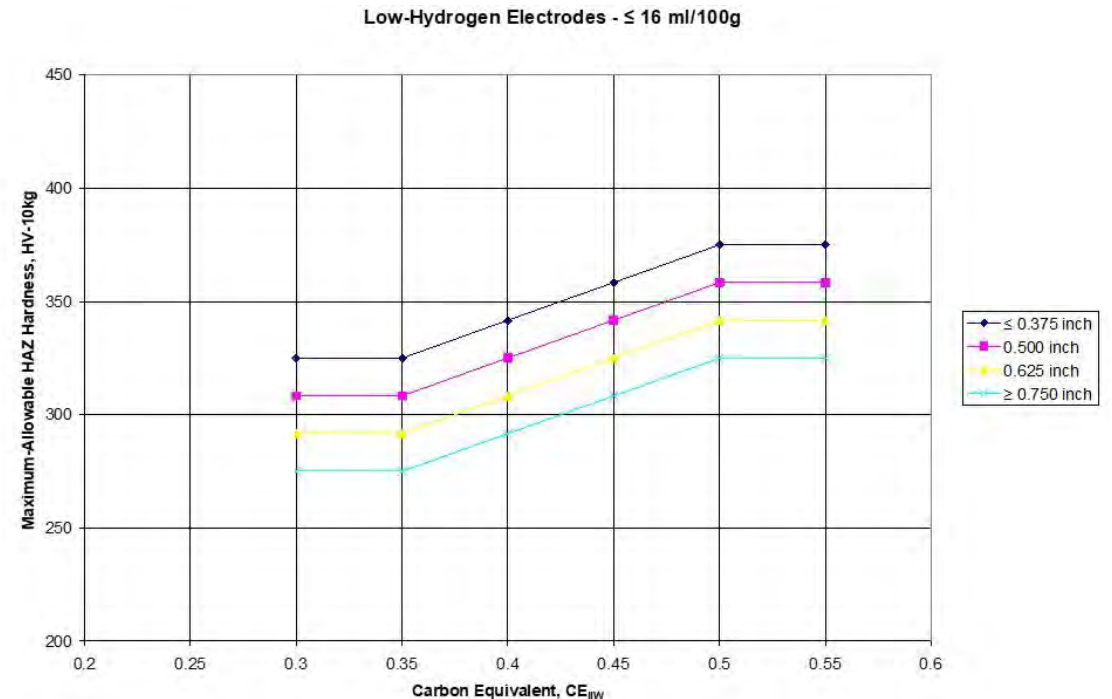
For thinner materials (~0.154 in. [3.9 mm] thick):

- Significant increase in weld hydrogen level at 15 kJ/in. (0.59 kJ/mm)
 - 8.0 ml/100 g average increase
- Slightly smaller increase at 12 kJ/in. (0.47 kJ/mm)



Mitigative measures

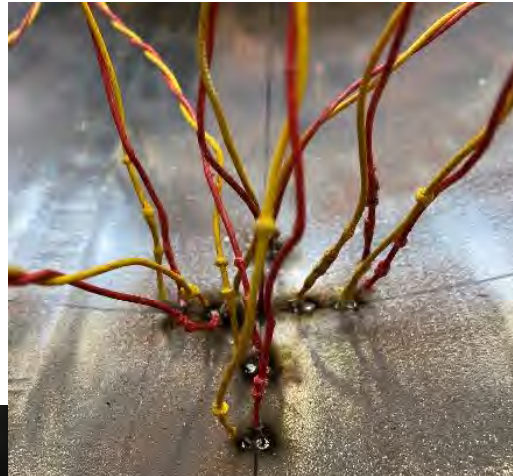
- For cases that result in elevated weld hydrogen levels, develop measures to mitigate the increased risk of hydrogen cracking
- Primary mitigative strategy is to limit inside surface temperature to below that for which significant increases in weld hydrogen levels occur
Seems to coincide with the austenite transformation temperature of steel or 1333 °F (723 °C)
- Not restrictive for thicker wall pipelines, but may be a challenge for thinner wall lines
- For welds on thinner wall lines where it is not possible to limit inside surface temperature, mitigative strategy involves accounting for the additional hydrogen in the weld by further limiting the maximum allowable HAZ hardness



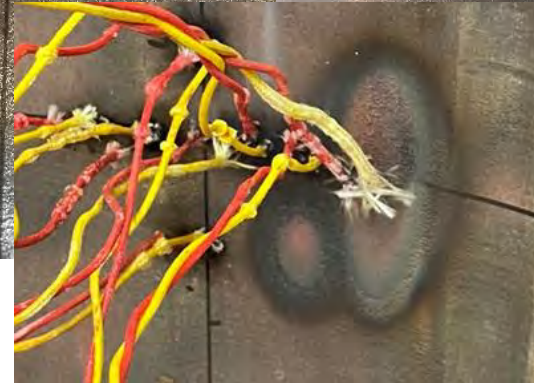
Conclusion On Hydrogen Weld Testing

- During tests no hydrogen induced cracking despite relatively fast cooldown and the absorbed amount of hydrogen from the pipe contents.
- Proposed on the research performed is to keep the internal wall temperature below 723°C/ 1333°F .
- If this cannot be met, use hardness limits as given in HAZ limits for in-service welding

Inside temperature during HyTap



3.5 2.5 X X 3 7.6 1 2 3 4 5 1/2 → 2.5 4/2 → 2.5			
1			ROOT.
2	40°C	570°C	55mm HP
3	60°C	760°C	100mm Fill 1
4	35°C	512°C	55- Fill 2
5	60°C	670°C	Fill 3
6	50°C	NUT	60mm Fill 4
7	25°C	NUT	70 Fill 5
8	32-25°C	NUT	70 Fill 6
9	32-25°C	NUT	70 Fill 7



maximum heat input of 1.0 kJ/mm
maximum interpass temperature 60°C(140°F).

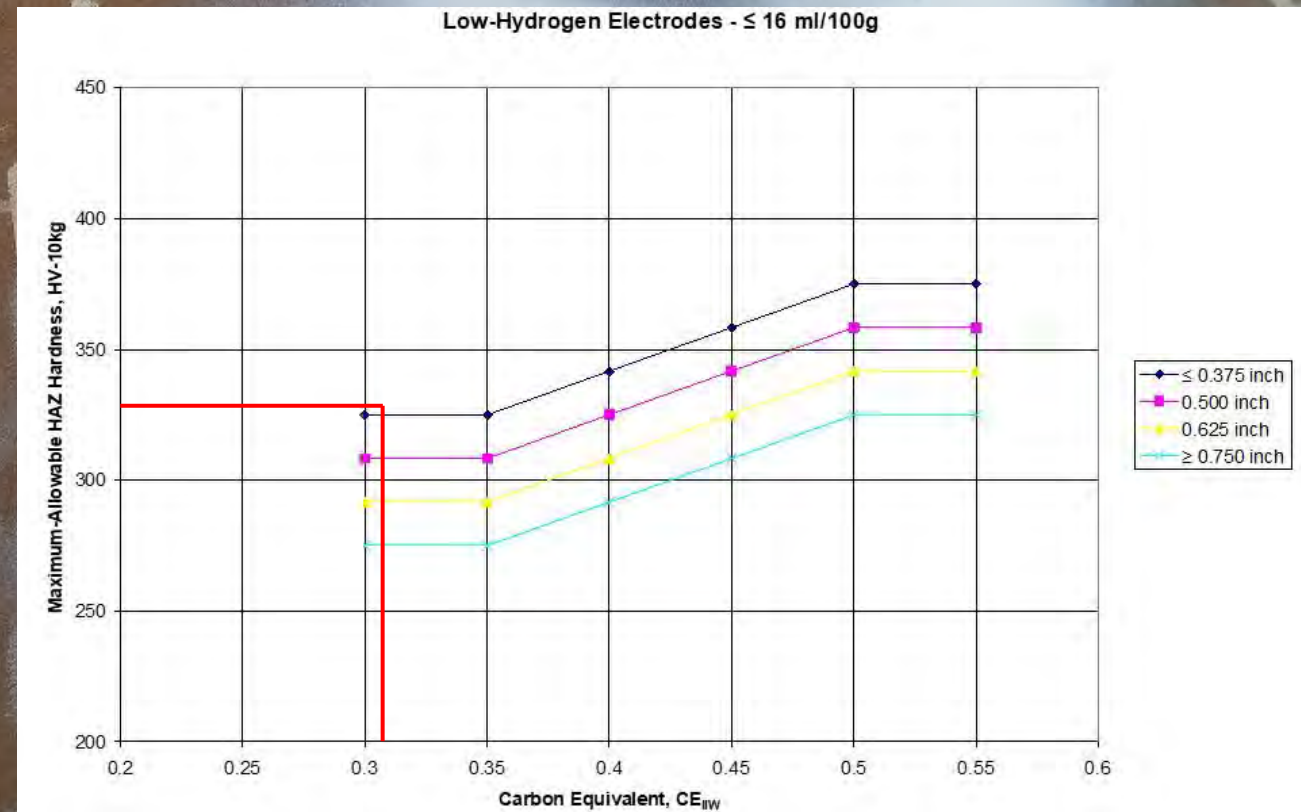
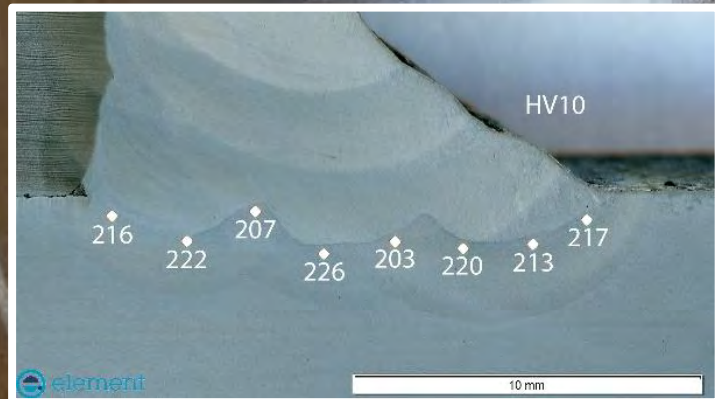
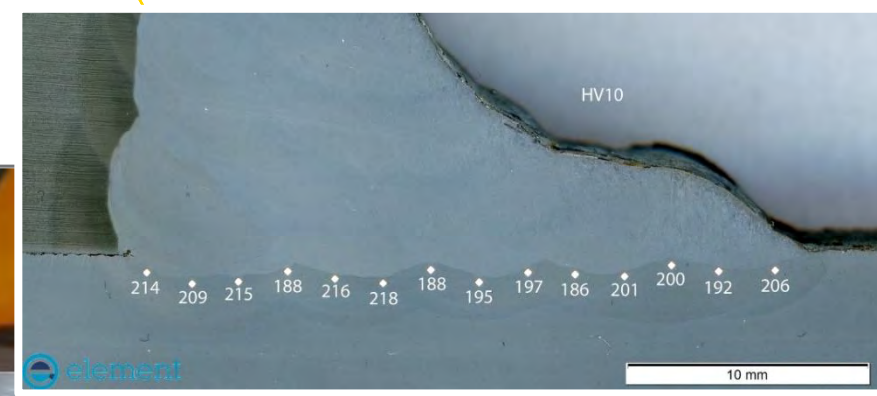
HyTap – Welding Setup



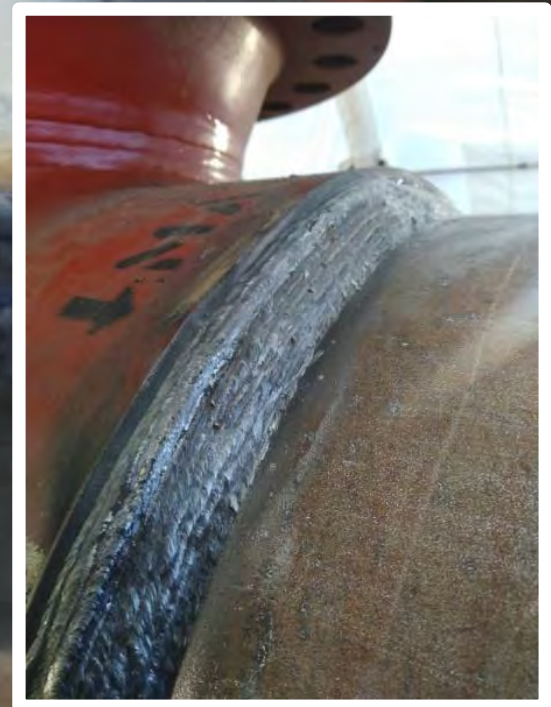
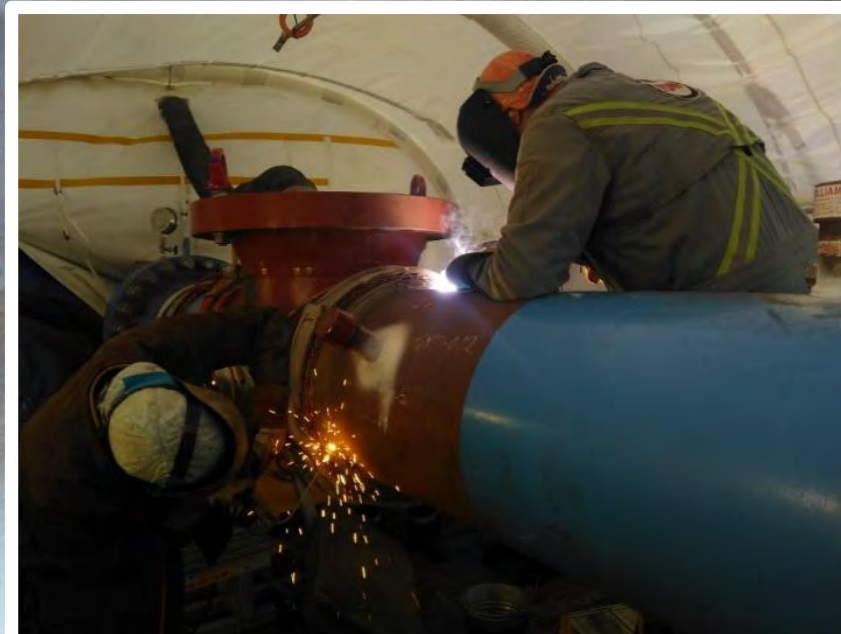
HyTap – Welding 1/3



HyTap – Welding 2/3



HyTap – Welding 3/3



HyTap – welding completed



72 hours after weld completion
Magnetic particle Testing

- No indications

Non Destructive Tests

- all acceptable

HyTap – Tapping



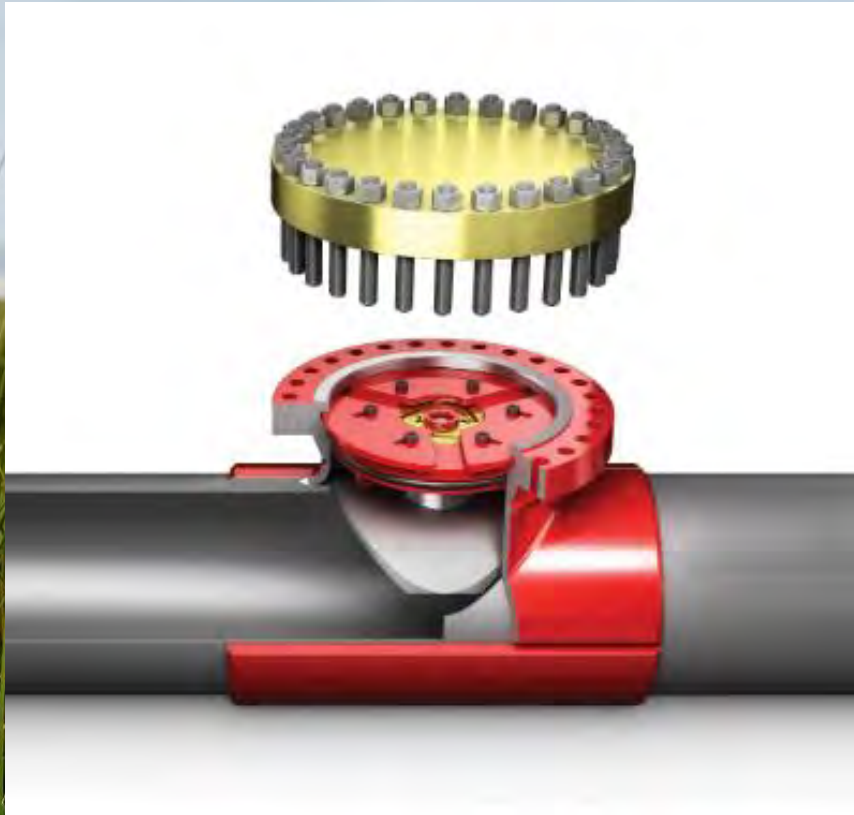
HyTap – stoppling 1/2



Hy – Tap stopping 2/2



Hy – Tap plug setting



Hytap-conclusions

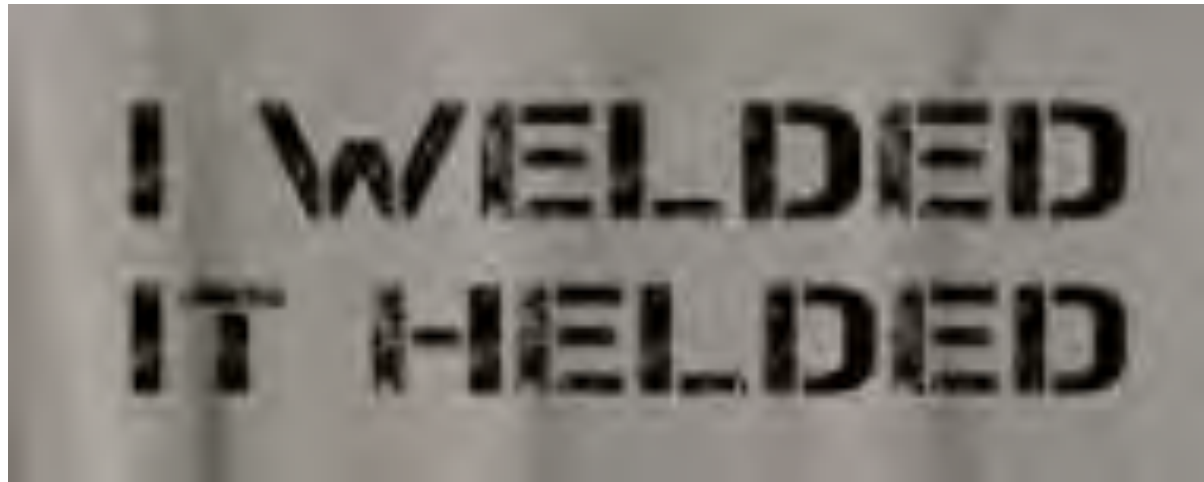
- Slightly adapted welding procedures
- Assessment of equipment
- Revised way of working (compared to NG)

ENABLES:

**SAFE GASEOUS HYDROGEN
WELDING-HOT TAPPING-STOPPLING**



Questions?





Exceptional service in the national interest

ANSI/CSA CHMC1

Chris San Marchi

Study Group on Materials Testing and Qualification for Hydrogen Service

Atlanta, GA

July 21st, 2023

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.





ANSI/CSA CHMC1 – Test methods for evaluating materials compatibility in compressed hydrogen applications - Metals

- **Section 4 – General requirements**
 - 4.1 Hydrogen gas purity
 - 4.2 Measurement and instrumentation
 - 4.3 Test temperature
 - 4.4 H₂ test pressure
- **Section 5 – Test methods**
 - 5.1 Apparatus
 - 5.4 Slow strain rate tensile testing
 - 5.5 Hydrogen-assisted cracking threshold stress intensity factor
 - 5.6 Fatigue crack growth rate (FCGR)
 - 5.7 Fatigue life tests



ANSI/CSA CHMC1 – Section 4.1: Hydrogen gas purity

- Source gas purity should be consistent with CGA G-5.3
- Purging processes is important
- Purity of the test gas “shall not be assumed to equal to that of source gas”
- Gas purity “shall be determined by analysis of gas samples”
 - Oxygen ≤ 2 vppm
 - Moisture/water ≤ 10 vppm
- Test gas must be sampled from test volume at conclusion of each test
 - If three consecutive test samples meet requirements, then verification testing can be performed annually
 - System or process changes require re-evaluation of test gas

Test gas purity is critically important and importance should not be underestimated



ANSI/CSA CHMC1 – Section 4.2: Measurement and instrumentation

- Transducers – both internal and external transducers can be acceptable with proper characterization
 - Internal transducers
 - consider accuracy, resolution, drift, and stability
 - External transducers
 - Force measurement methods must be documented with consideration of : (!) seal friction as a function of pressure (and rates); (2) force on sample due to pressure
 - Displacement measurements (such as load-train compliance) must be documented (may depend on pressure, fixturing, alignment and other factors)

***Editorial note:* Internal measurements are preferred and robust methods exist for both internal load and displacement (on the sample) measurements**



ANSI/CSA CHMC1 – Temperature (4.3) and pressure (4.4)

- Testing should consider the service conditions with respect to temperature and pressure
- Temperature of maximum embrittlement may not be room temperature for ambient applications
- Hydrogen effects depend on pressure: “testing results are valid for all pressures equal to or less than the qualification test pressure”

Editorial note: Room (laboratory) temperature is generally considered ‘worst-case’ for fatigue life testing



ANSI/CSA CHMC1 – Section 5.1 Apparatus

- "...test specimen [must] be immersed in the gaseous hydrogen environment"
- Reference to ASTM G142

Note:

- Surrogate environments are not acceptable
- **HOWEVER**
 - Surface effects can mediate some test results, especially Slow Strain Rate Tensile (SSRT) testing
 - Example: A286
 - ASTM G142 references A286 as a materials that is 'immune' to hydrogen
 - Fatigue and fracture tests demonstrate very strong effects of hydrogen on A286 (internal hydrogen also show >50% loss of ductility)



ANSI/CSA CHMC1 – Section 5.4 Slow stain rate tensile testing

- **Reference to ASTM G142**
- **Nominal strain rate: 10^{-5} s^{-1} for smooth specimens**
 - **Notched specimens should be tested 10x slower**
- **Editorial note:**
 - **Surface effects can mediate some test results, especially Slow Strain Rate Tensile (SSRT) testing**
 - **Example: A286**
 - **ASTM G142 references A286 as a materials that is 'immune' to hydrogen**
 - **Fatigue and fracture tests demonstrate very strong effects of hydrogen on A286 (internal hydrogen also show >50% loss of ductility)**



ANSI/CSA CHMC1 – Section 5.5 Hydrogen-assisted cracking threshold intensity factor

- Method should be consistent with ASTM E1820
 - Compact tension or single-edge bend specimens
 - Testing and analysis should follow standard methods– but tests must meet the requirements of ASTM E1820
 - K (linear elastic)
 - CTOD (crack tip opening displacement) or
 - J (elastic plastic)
 - No restriction on methods for crack monitoring
 - Precracking in air or in hydrogen
 - Displacement control between 0.1 to 1 MPa m^{1/2} /minute (in elastic region)
 - No mention of soak time



ANSI/CSA CHMC1 – Section 5.6 Fatigue crack growth rate (FCGR)

- Method should be consistent with ASTM E647
 - Must use constant force amplitude method
 - K-decreasing methods can be used for $da/dN < 10^{-8}$ m/cycle
 - Test parameters:
 - $R = 0.1$
 - Frequency = 1 Hz
 - Waveform can be either triangle wave or sine wave
 - Recommended range of $\Delta K = 8$ to 20 MPa $m^{1/2}$
 - No mention of soak time

***Editorial note:* Other load control methods, other R, and other frequencies have been demonstrated to improve test efficiency and access to broader FCGR ranges**



ANSI/CSA CHMC1 – Section 5.7 Fatigue life tests

- References to ASTM documents:
 - E1012 – load train alignment
 - E466 – force-controlled testing
 - E606 – strain-controlled testing
 - G142 – notched specimen geometry (K_t shall be ≥ 3)
 - E739 - analysis
- Additional specifications
 - *Force-controlled*
 - Constant R
 - For notched specimens: $R = 0.1$
 - Frequency ≤ 1 Hz for low-cycle testing ($< 100,000$ cycles) and ≤ 20 Hz for high-cycle ($> 100,000$ cycles)
 - *Strain-controlled*
 - Same basic requirements



Closing thoughts

- CHMC1 is the best available test method description
 - Captures important aspects of testing (e.g., gas quality and transducers)
 - Generally provides flexibility in test hardware and transducers (perhaps too much)
- CHMC1 does not mention ASTM E1681 – constant load/displacement fracture methods
- As noted, no requirements for soak time
- Fatigue crack growth method in CHCM1 is arguably over-specified
 - Need flexibility on R, frequency and control methods
 - Design methods must consider the effect of R
 - Frequency dependencies are related to FCGR (eg, da/dN), therefore both higher and lower frequency are needed for conservative and efficient testing



EPRG Pj 231: Preparations of the full scale H₂ fatigue crack growth test - updates July 2023

SNL Hydrogen Study Group meeting at ASME PVP conference

21st July 2023

Scope of this presentation

- Introduction to the EPRG Hydrogen programme
- Updates for the preparation of the test on the modern pipe:
 - Material selection
 - Notching
 - End cap welding
 - Crack growth monitoring plans
 - Crack initiation (nucleation) study
 - Test plant preparations progress

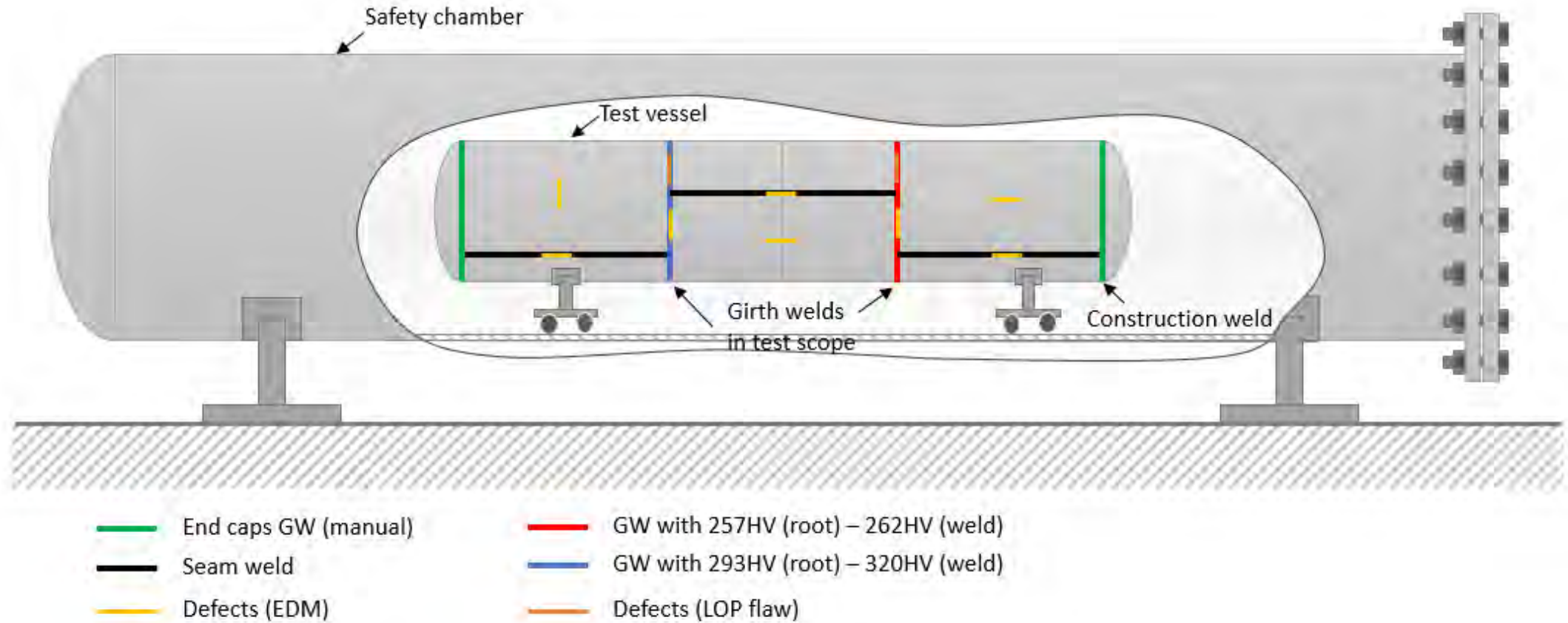
Overview - EPRG Hydrogen projects

- Pj 221 : SOTA review + Fitness for service primer (completed)
- Pj 231 – Full scale fatigue crack growth tests in hydrogen gas
 - On a modern pipe material (this presentation)
 - On a vintage pipe
- Pj 232 – Small scale fatigue crack growth and fracture toughness testing in hydrogen gas
 - On a modern pipe and modern girth welds
 - On a vintage 1970' material and vintage cellulosic girth weld

Aims of the EPRG programme

- Challenge the hardness limitations to 235 HV / 248 HV of B31.12
- Compare Material performance of modern vs vintage materials vis-a-vis ASME B 31.12's 1 dig/mile limitations
- Compare material performance w.r.t fatigue crack growth measured on small scale lab test versus full pipe test
- Provide guidance on small scale fracture toughness testing (which test method) by correlation with the full scale pipe behaviour

Full scale test setup

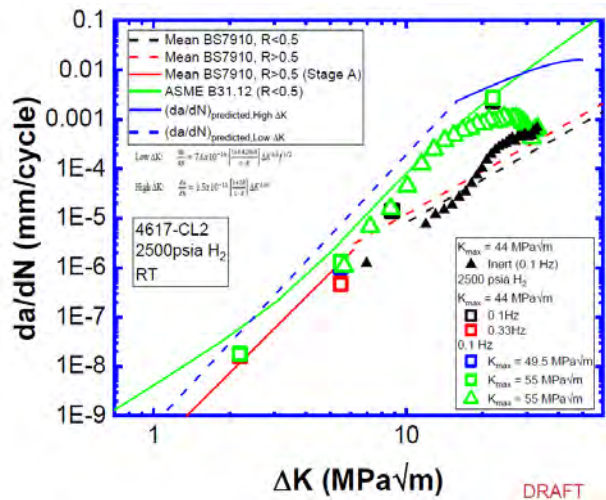


EPRG modern pipe

Parameter		Modern pipe
Pipe	Diameter	28"/DN 700
	Wall thickness	17,5 mm
	Grade	API 5L X70/L485M
	Seam weld	DSAW
	Production Date / Manufacturer	2021 / Europipe
	Microstructure	Per ISO 3183/App A : Ferrite – pearlite with low %C
Girth welds	Welding process	Mechanised GMAW: normal hardness 250HV and high hardness 300 HV

EDM Notch sizing

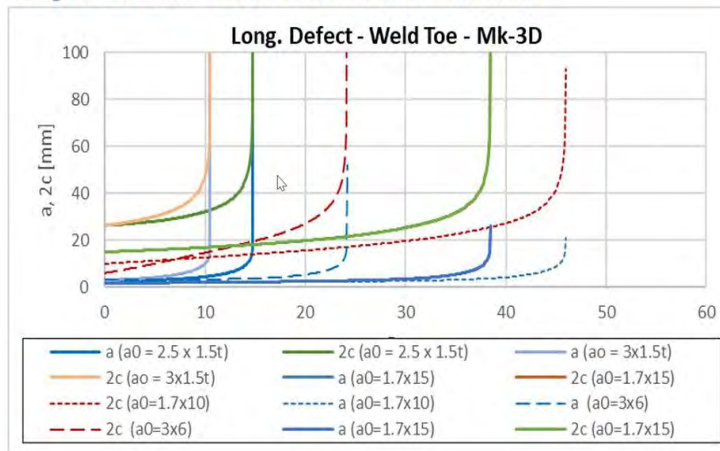
- 1st group; workmanship criteria (N10 for L or EPRG Tier 2 for C)
- 2nd group: ECA derived to fail in a pre-defined time of ~30 days:



Results



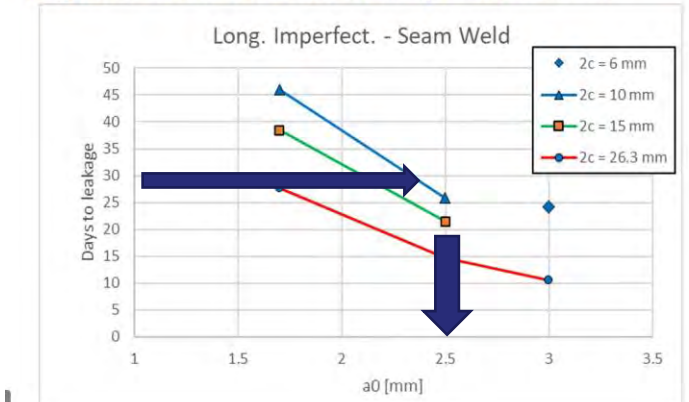
Long. Defect – Notch in Seam Weld – Recent Calculations



Results



Long. Defect – Notch in Seam Weld – Summary various calculations

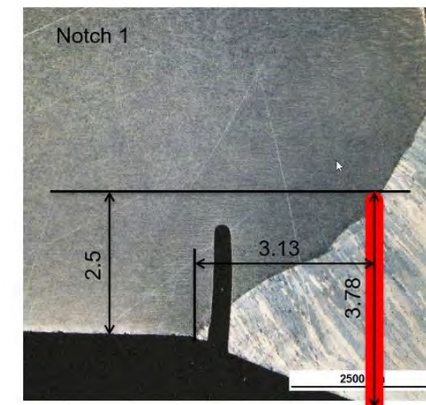
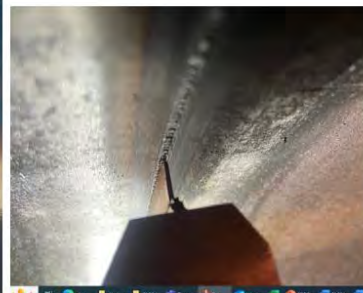
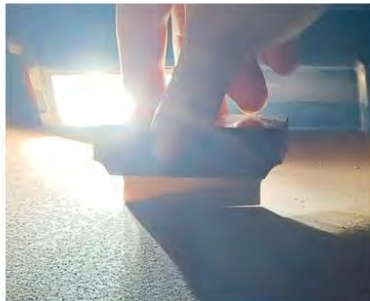


Test vessel notching

Notch orientation	Method	Length	Depth
Longitudinal	ECA calculated	15 mm	2.5 mm
Longitudinal	Workmanship	50 mm	1.7 mm
Circumferential	EPRG workmanship	52 mm	5 mm
Circumferential LORP	EPRG workmanship	120 mm	3 mm



Longitudinal notches (weld toe)
New Positioning in Seamweld



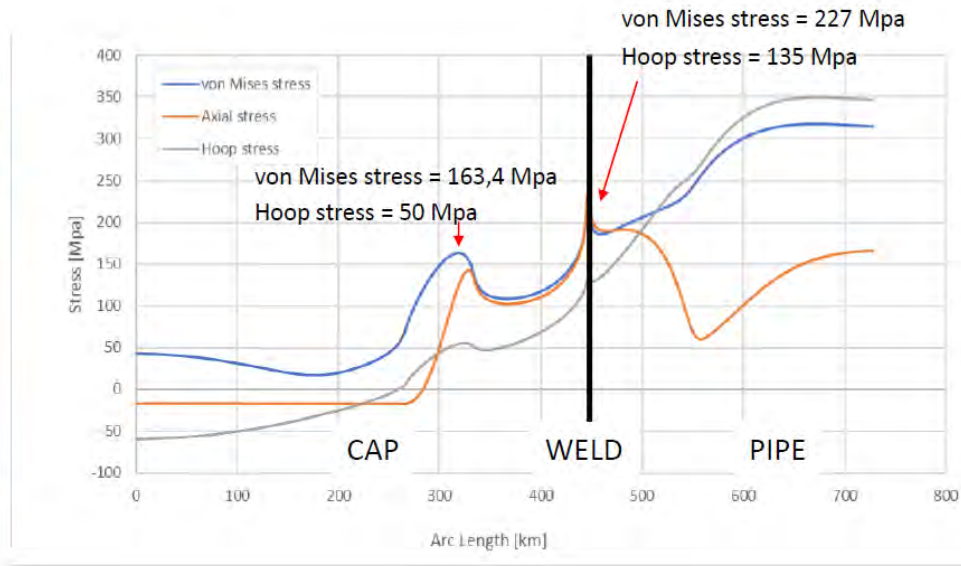
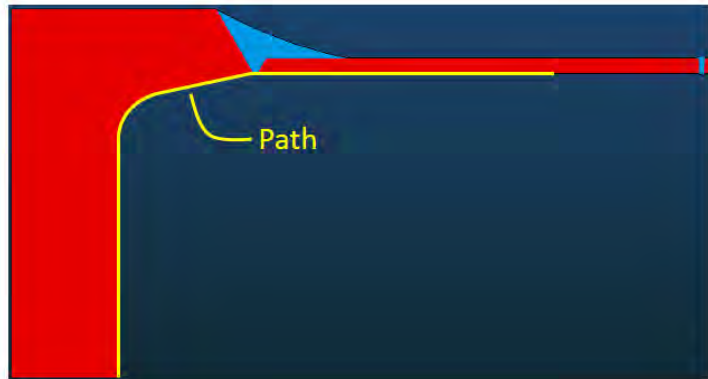
Girth welds

- Mechanized PGMAW process with CMT root
- Standard setup for 250 HV weld
- Dedicated setup for 300 HV weld with interpass cooling
- LORP flaws also introduced in both welds



End caps

- Optimised for cyclic fatigue test



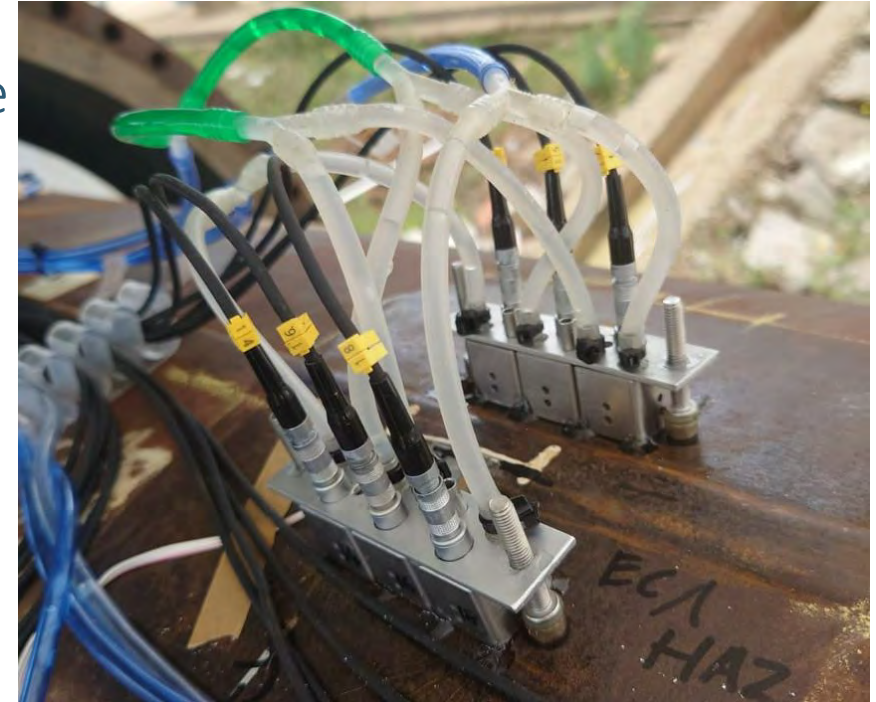
Test vessel fabrication

- Precision machined Aluminium filler bars used to reduce the internal volume



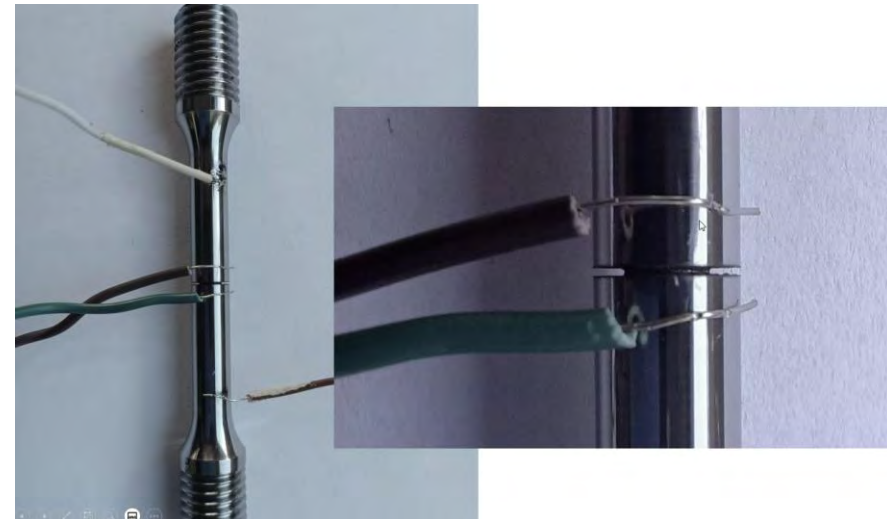
Monitoring of crack growth

- Longitudinal notches (5 off):
 - Phase Array UT sensors permanently mounted on pipe
 - Semi-continuous monitoring
 - Flexibility
 - Essential to control the test
- Circumferential notches
 - Expect low crack growth
 - 2x stoppages & manual UT planned



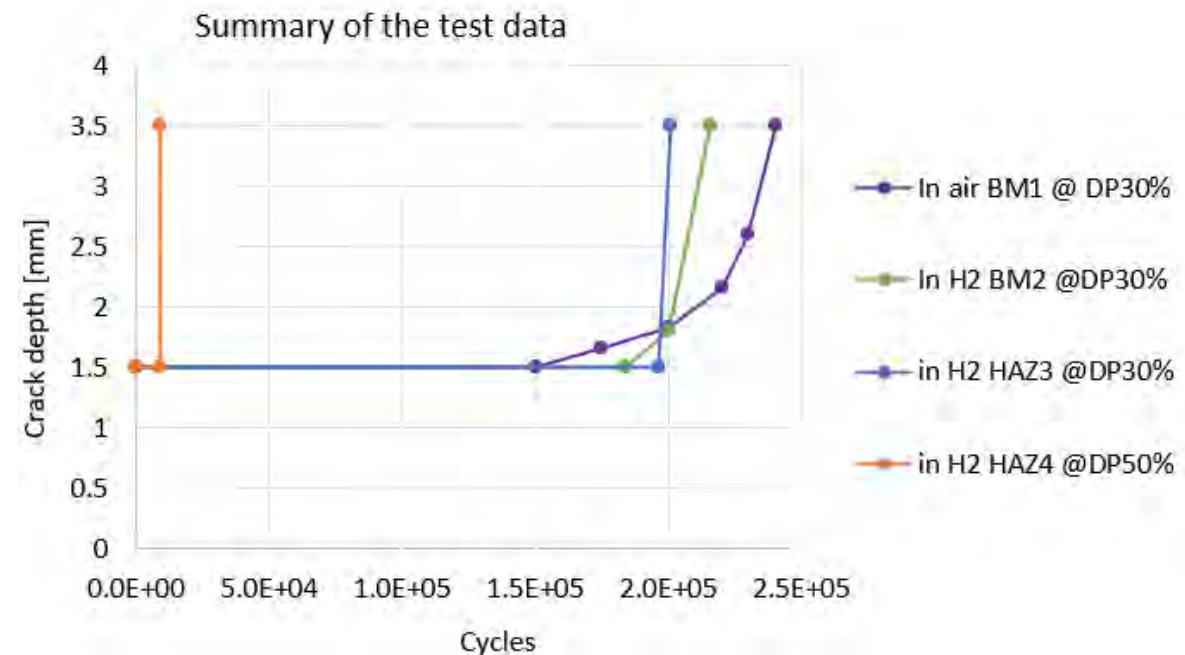
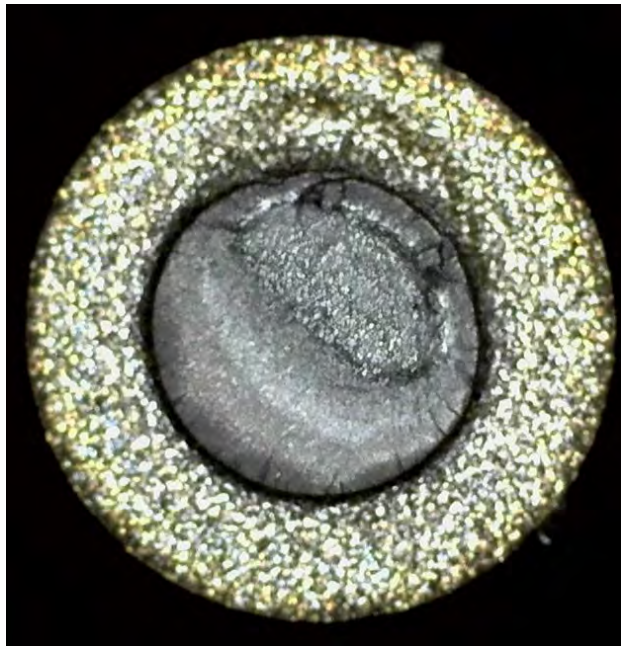
Crack nucleation study

- Round notched tensile bar setup
- Same EDM notch width & tip as in full pipe (same contractor)
- Fatigue cycle testing in the same conditions (172 barg, pure H₂)
- Same stress profile as for the full scale pipe
- FEA checks done to show comparability of the sample to full pipe → Good



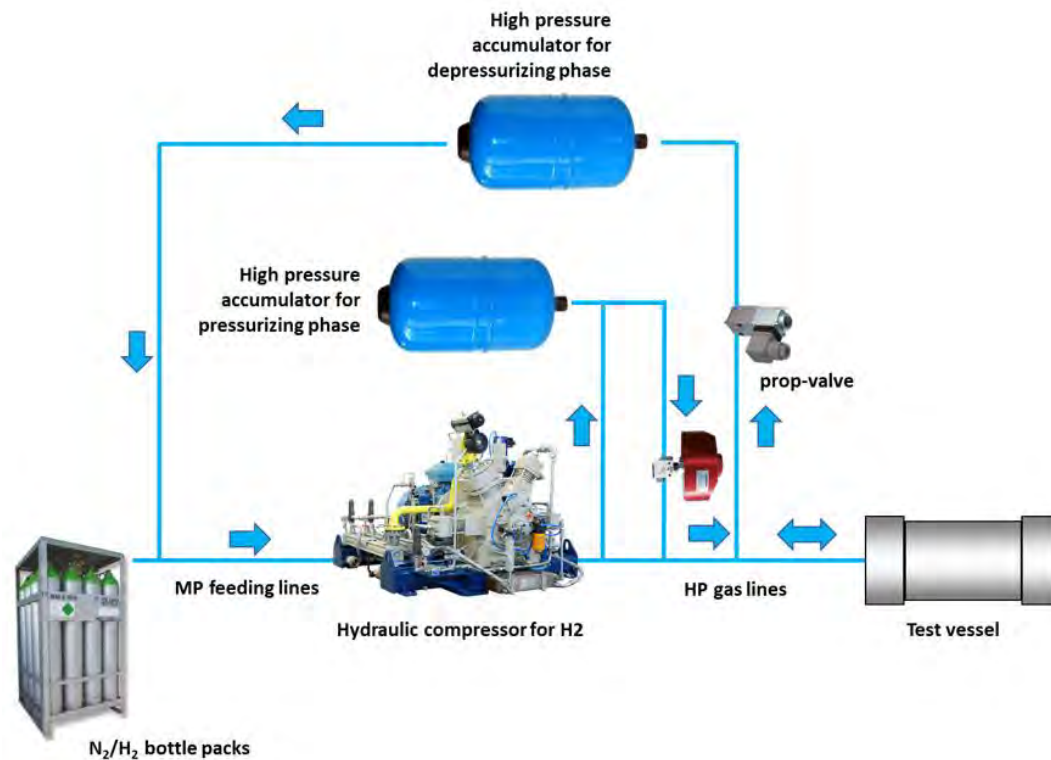
Crack nucleation study Results

- At low Delta pressure ($R=0.7$) small effect of H_2 gas on the initiation
- At larger Delta pressure ($R=0.5$) notable acceleration in the initiation of a fatigue crack → order of magnitude

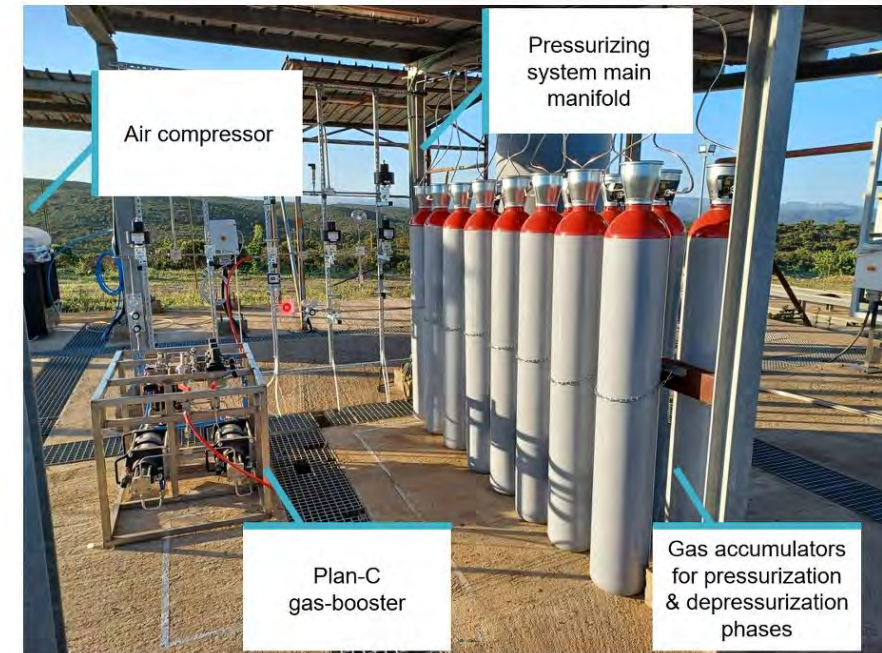


Test plant

- Cycling hydrogen gas
- Two banks of accumulators + Booster pump



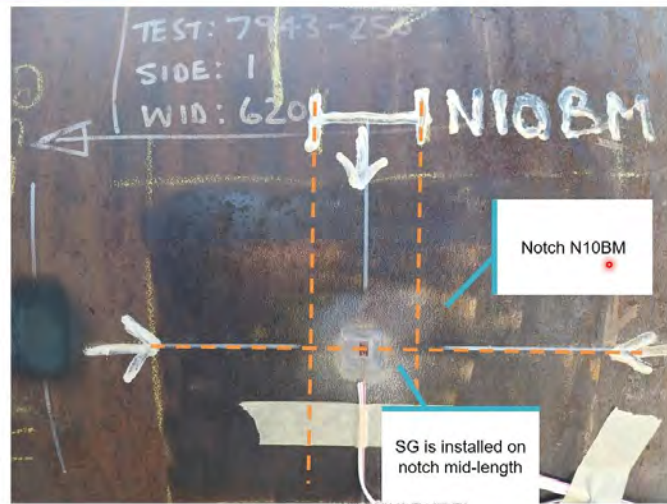
Test rig preparation status



Test sample preparation

- Ready !
- Strain gauges and UT probes installed at each longitudinal notch

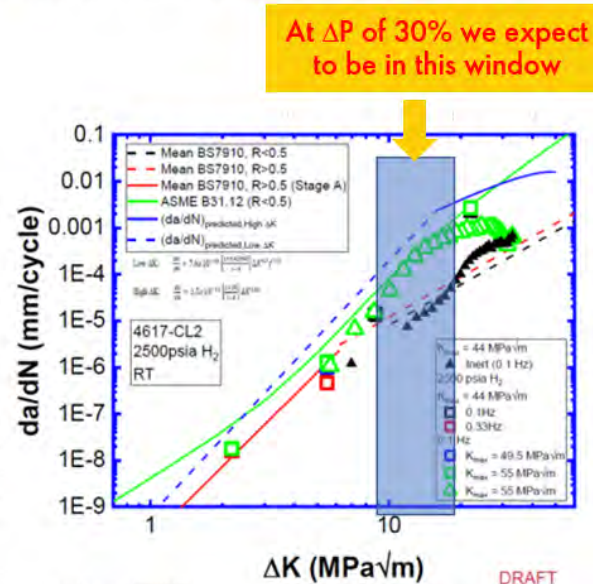
Sample #1 - SG instrumentation



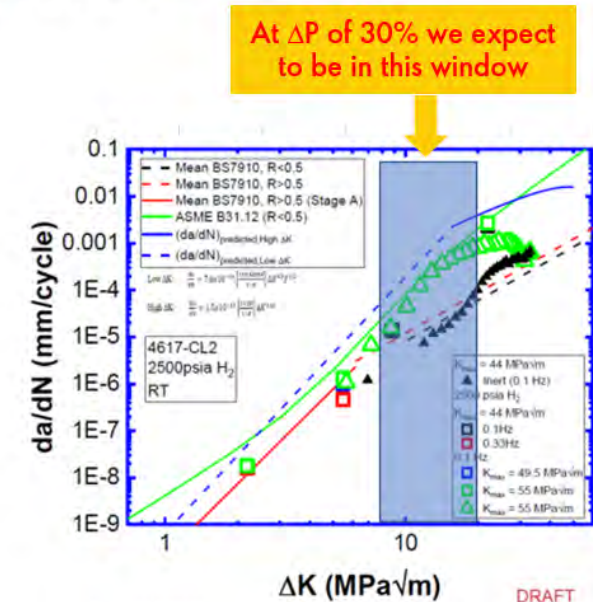
Test protocol

- Start test at ΔP of 50% of the MAOP to nucleate a growing fatigue crack
- Rapid transition from ΔP 50% to ΔP 30%
- Aim to study FCGR in the ΔK of 10 ~30 MPa $\sqrt{m}$ window by running the test at a constant ΔP of 30% of MAOP

■ 2.5 x 15 mm nominal flaw size :



■ 1.7 x 50 mm nominal flaw size :



Test environment

- Target to keep O_2 as low as practical ($\ll 10$ ppm)
 - O_2 is one of key elements affecting fatigue crack growth
 - EPRG test aiming at O_2 'free' FCGR test
- Target to keep $H_2O < 5$ ppm
 - Excessive H_2O may interact with H_2 in a similar way as O_2 (+ hydrate concerns) hence controls put in place
- Plan:
 1. Extensive purging with N_2
 2. Fuel cell ISO 14687 compliant H_2 source
 3. Periodic measurement of O_2 levels at low pressure using a vent line → a sensor was found

Oxytrans II
Oxygen Transmitter

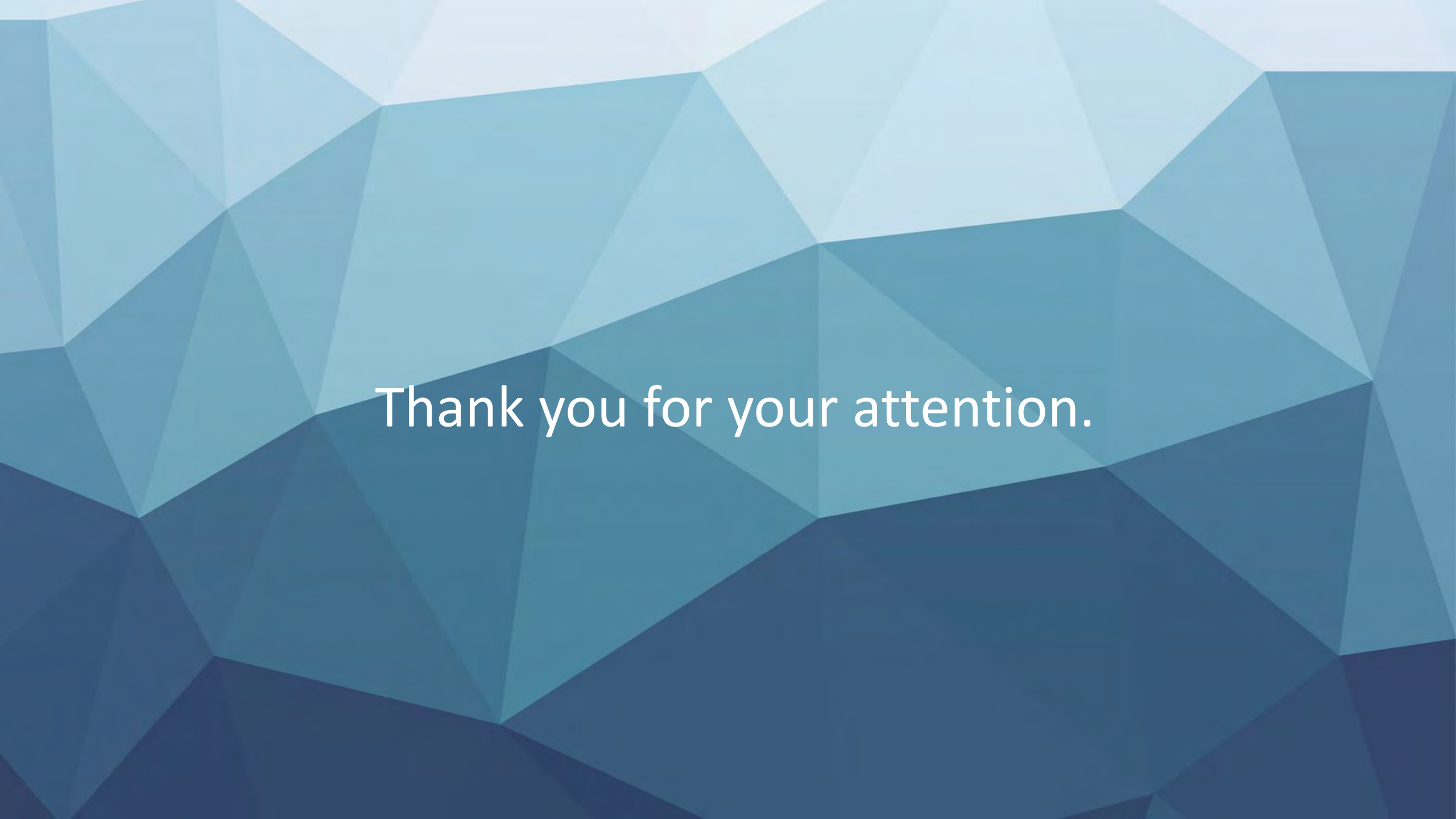


Figure 1: Oxytrans II

Resolution	0.1 ppm < 0-100 ppm 1 ppm at 0- 1000, 0-10000ppm 0.1% for 25%
Digital Output	HART Communication
Load	Typical 470 ohm
Pressure	Max 10bar (flow through)

Conclusions

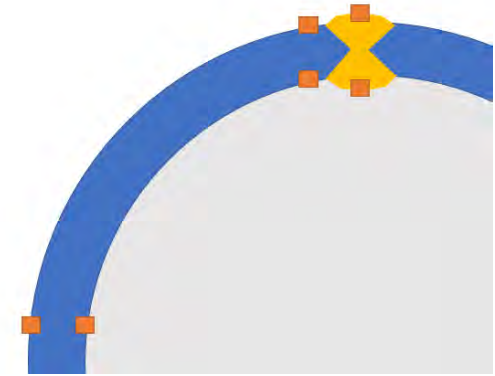
- EPRG is the first to perform the full scale fatigue crack growth test using a modern X70 material with seeded defects in pure hydrogen gas
- Normal and high (300 HV) hardness girth welds in scope of the study
- Aim to confirm small scale FCGR reported data in the ΔK of 10 ~30 MPa $\sqrt{m}$ window
- Latest status - test sample setup complete, Test plant undergoing the final commissioning checks. Expect to start testing in late July.

The background is an abstract geometric pattern composed of numerous triangles in various shades of blue and teal. The triangles are arranged in a way that creates a sense of depth and movement, with some triangles being larger and more prominent than others. The colors range from light, almost white, blues to deep, dark blues and teals.

Thank you for your attention.

Residual stress effects

- Some data received from literature → WRS in the order of 100 MPa ~150 MPa
- Measurements performed with
 - Deep hole drilling through the WCL → WRS ~150 MPa
 - Shallow hole drilling at HAZ & WCL → WRS 200 MPa at ID HAZ and 400 MPa on ID weld cap surface
- Also plan to do the same measurement on the small scale CT sample coupons
- Important to know as this feeds into post test analyses of findings





Exceptional service in the national interest

Subscale testing to simulate full-scale pipe testing

Chris San Marchi, Joe Ronevich, Brendan Davis

Study Group on Materials Testing and Qualification for Hydrogen Service

Atlanta, GA

July 21st, 2023

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.





Motivation for small-scale testing platforms

Full-scale testing is challenging

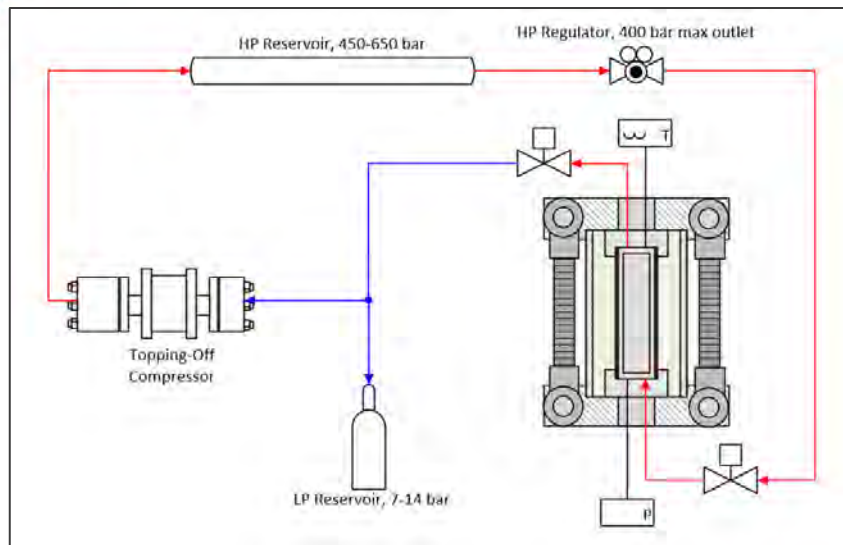
- Large-volume applications, such as transmission pipe and pressure vessels require expensive infrastructure, including safe 'failure management'
- Construction, execution, and turn-around requires time

Subscale testing

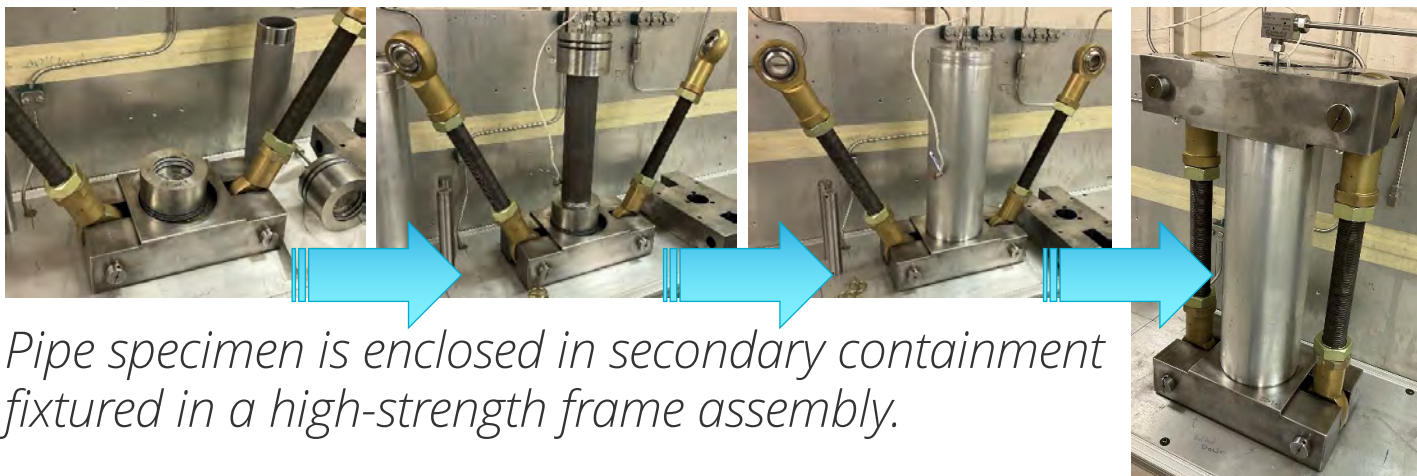
- Significantly less infrastructure and cost
- Safety is easier to manage
- Quick execution and turn-around (e.g., multiple fixtures)
- Easier to control boundary conditions to address research questions
 - For example: role of oxides and surface condition



Sub-scale component, hydrogen test system



Simplified P&ID of Capability



Pipe specimen is enclosed in secondary containment fixtured in a high-strength frame assembly.

Test Specimen

- Standard 2-in steel pipe
- Schedule 40
- ASTM A53

Pipe flaw

- 2x notches cut with plunge EDM
- Semi-elliptical notch specifications
 - Length: 40 mm
 - Depth: 1 mm

Initial Test Parameters

$P_{\max} = 4,000$ psi ($\sim 56\%$ SMYS)

$P_{\min} = 2,000$ psi

Freq: 1 cycle/min

Gas Mixture: N_2 with 3% H_2

Predicted failure

650 cycles (after crack initiates)



Initial test demonstration

Test Stage 1:

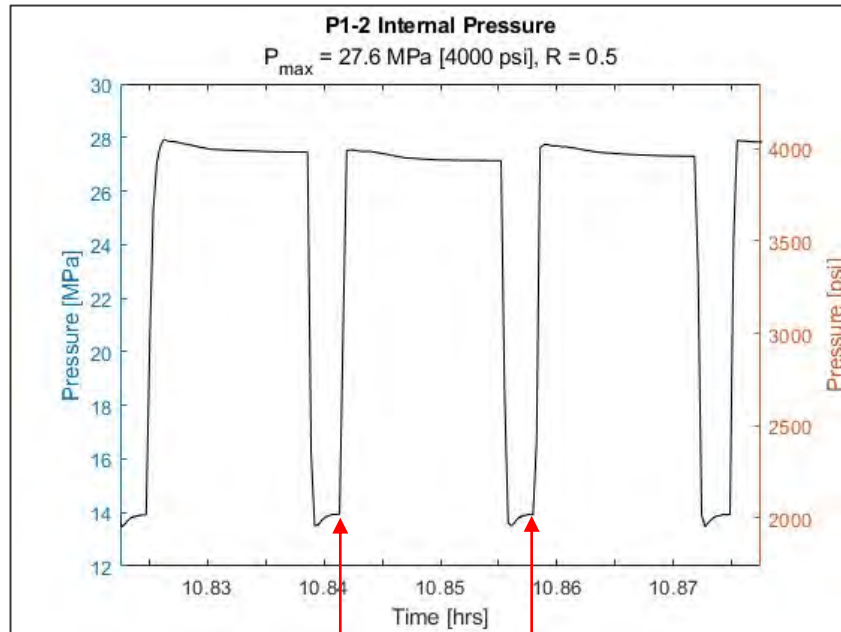
- $P_{\max} = 4,000$ psi
- $P_{\min} = 2,000$ psi
- Cycles Completed: 1,900
- No pipe failure

Test Stage 2:

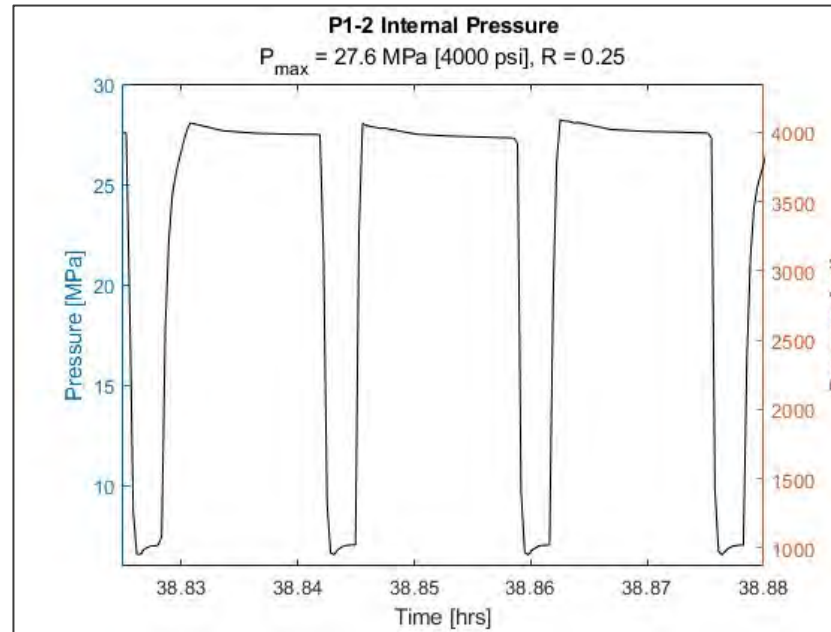
- $P_{\max} = 4,000$ psi
- $P_{\min} = 1,000$ psi
- Cycles Completed: 406
- No pipe failure

Test Stage 3:

- $P_{\max} = 5,000$ psi
 - 69% SMYS
- Cycles Completed: 0
- *Pipe failure*



Cycle Period:
1 minute

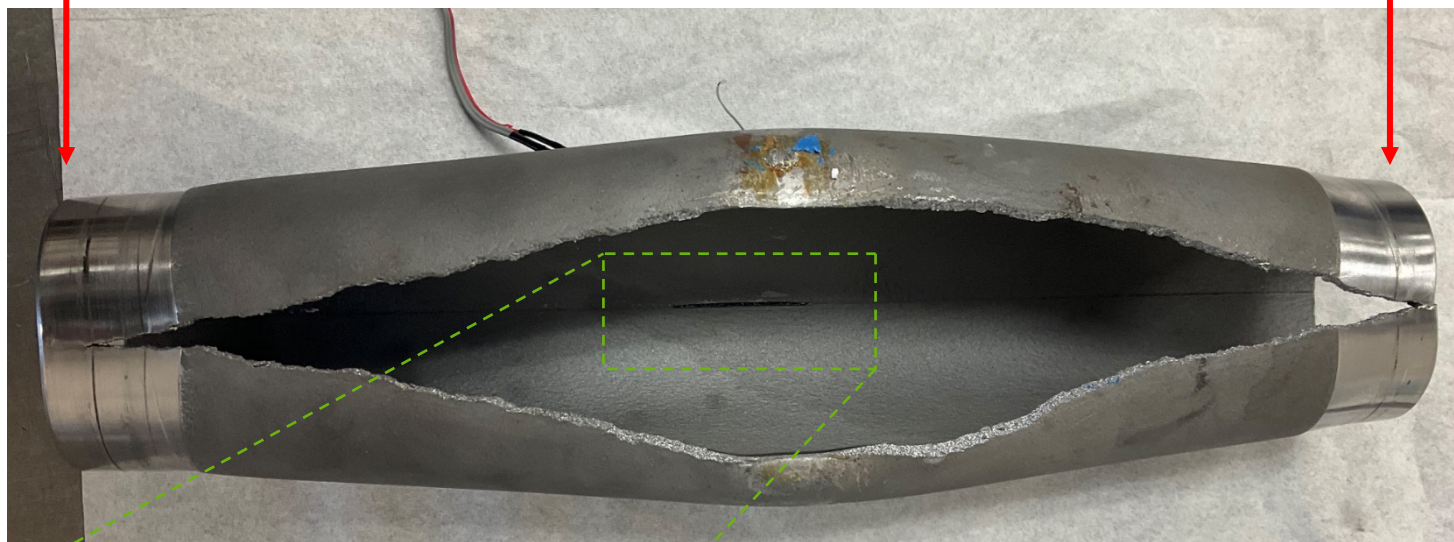


Snapshot of pressure cycles near the end of the test operation (approx. 2300 cycles)

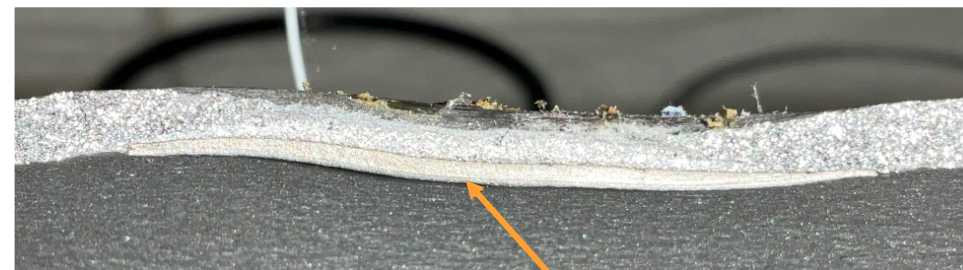


Post-test observations

30.5 cm
[12 in]

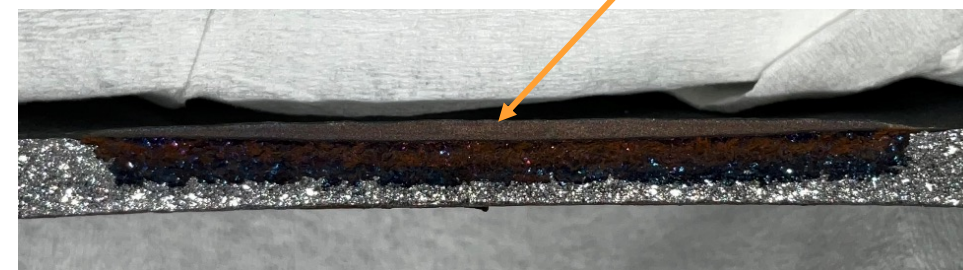


Weld Seam Notch

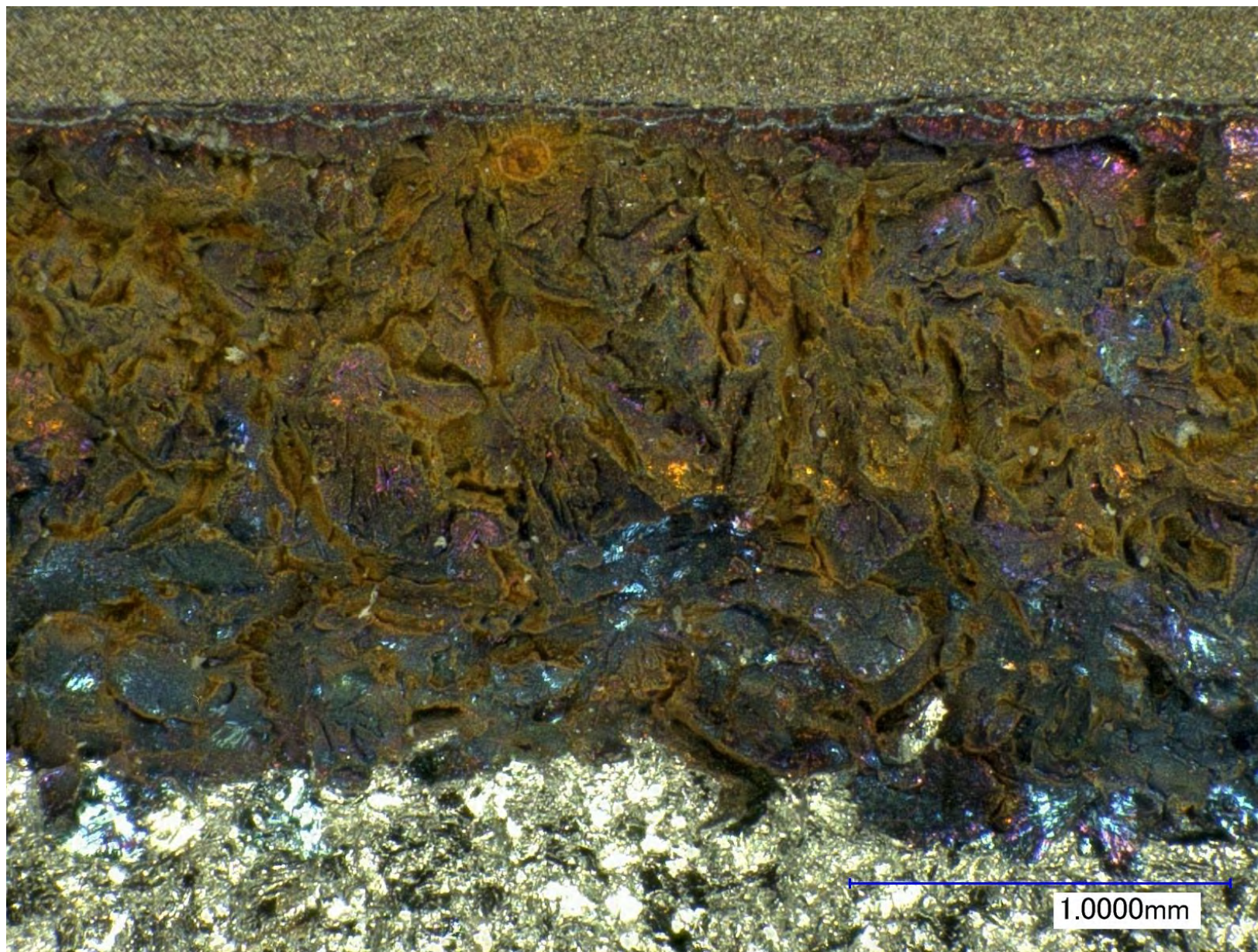


Notch 1 - 'Failed' notch

Original
EDM
notches



Notch 2 (weld seam), heat-tinted





Next steps

1. Tune/tweak system to improve operation and pressure cycle response
2. Develop method to identify crack initiation during operation
3. Add logic to stop test upon failure and enable continuous (overnight) operation
4. Proceed with next test operation
 - Design test for single load cycle and extend patience in testing
 - All consider 3% hydrogen

Longer term (months)

1. Work toward use of 100% H₂
2. Develop methods to control internal surfaces
3. Design and manufacture external flaws – evaluate role of oxides on H₂ uptake
4. Manufacture additional ‘fixtures’ for parallel operation
5. Develop strategy for multitude of simulated flaws

Fatigue-based versus crack growth-based design methods for hydrogen components – pros and cons?

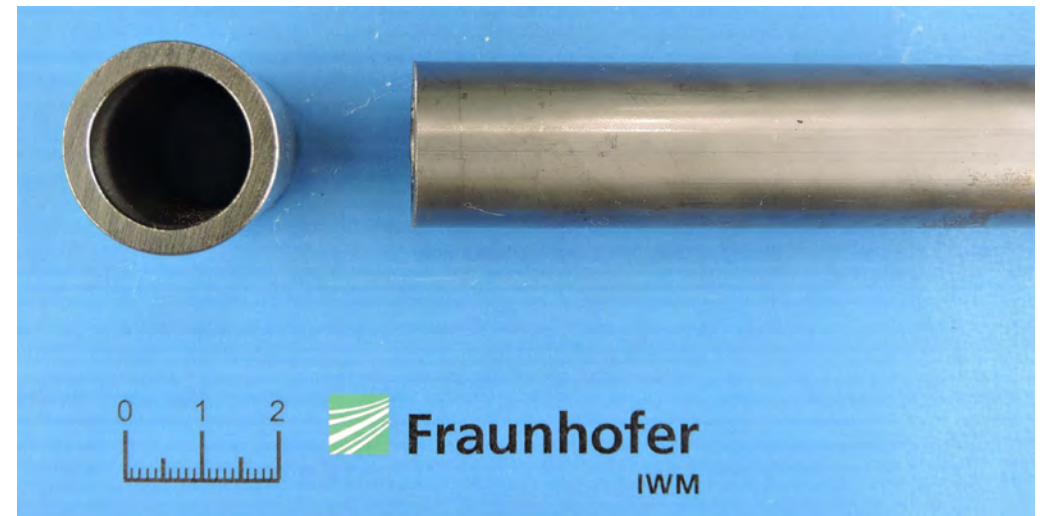
Carl Fischer – Fraunhofer Institute for Mechanics of Materials IWM, Germany
Study Group on Materials Testing and Qualification for Hydrogen Service
Friday, July 21, 2023, Atlanta, Georgia

Problem statement

Lifetime assessment for hydrogen-pressurized small tubes

Hydrogen tubes with small dimensions

- Used in FCEV or in distribution lines in hydrogen gas stations
 - Example shows low-alloy carbon X52 steel
 - Outer diameter in the range of 20 mm
 - Wall thicknesses in the range of 2.5 mm
- How to apply a fracture mechanics assessment route for small tube dimensions?
- Or can a crack-based criterion be used?
 - LCF or HCF models typically assume defect free component
 - Requirement of a defect-free surface until which defect size? 100 μm ?



Thinking beyond pipelines, there is a need to design components for hydrogen service with walls of only a few millimeters. Below some wall thickness a crack initiation approach will have to be used.
Where/how do we draw a line?

Crack growth-based design methods

Example: ASME B31.12

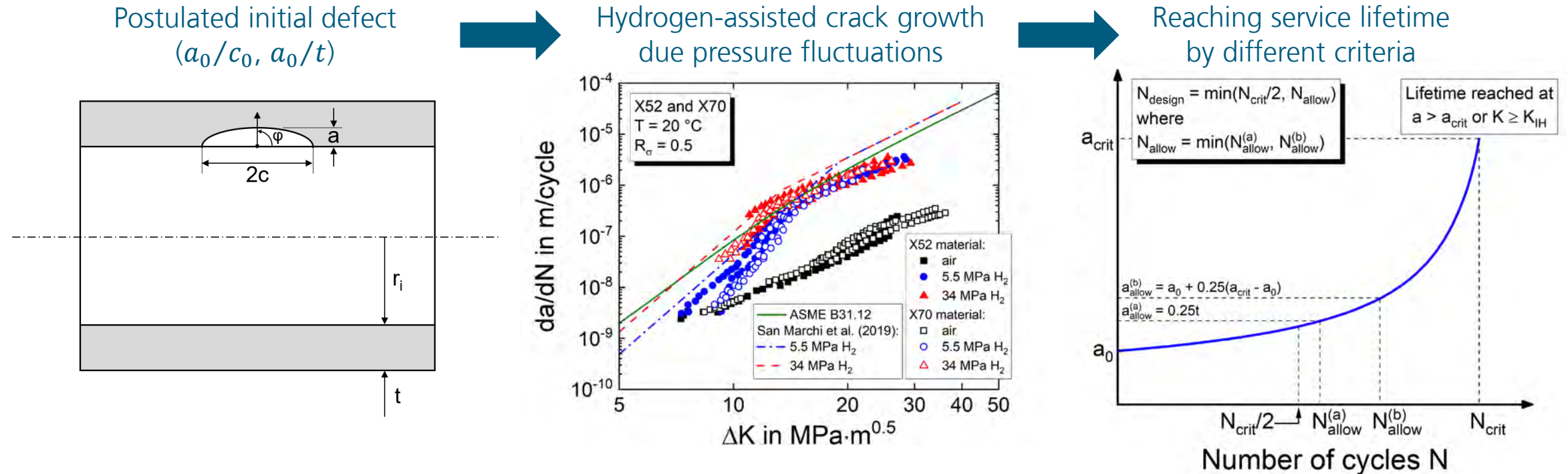
Literature data in middle figure:

X52: A.J. Slifka et al. (2012). Proc. 2012 Int. Hydrog. Conf. Hydrog. Mater. Interact. 9-12 Sept 2012, ASME Press, Jackson Lake Lodge, WY, USA, 2012: pp. 299–308.

X70: E.S. Drexler et al. (2014). Fatigue Fract. Eng. Mater. Struct. 37 (2014) 517–525.

<https://doi.org/10.1111/ffe.12133>

Design curve from C. San Marchi et al. (2019). Proc. ASME 2019 Press. Vessel. Pip. Conf. (PVP2019), 14-19 July 2019, ASME Press, San Antonio, TX, USA, 2019: PVP 2019-93907



- + Well established
- + Good experimental data base in particular for low alloy steels
- +/- Assumption of existing defect is usually (over-)conservative
 - Extracting fracture mechanics specimen from components with high curvature/thin walls is difficult

Fatigue-based design methods

Example: German AD 2000 bulletin S2 (Analysis for cyclic loading)

Conservatively assumed S-N curves from experimental data

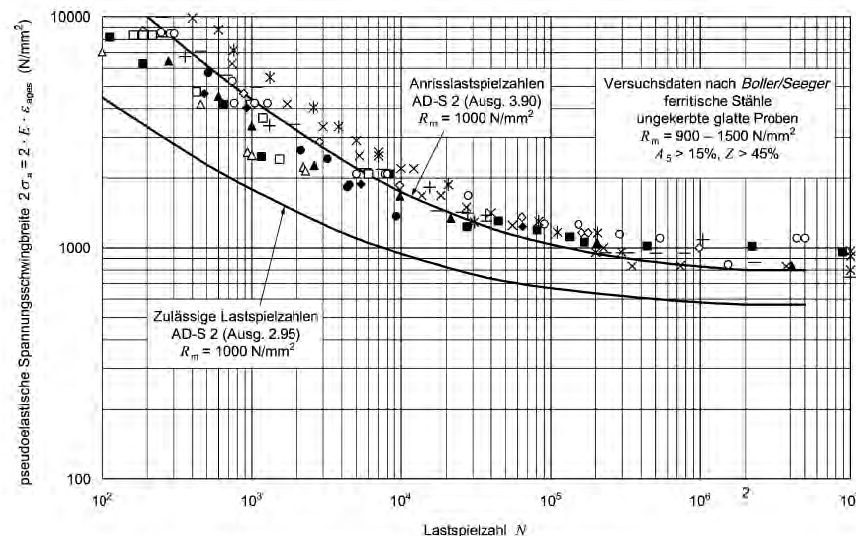
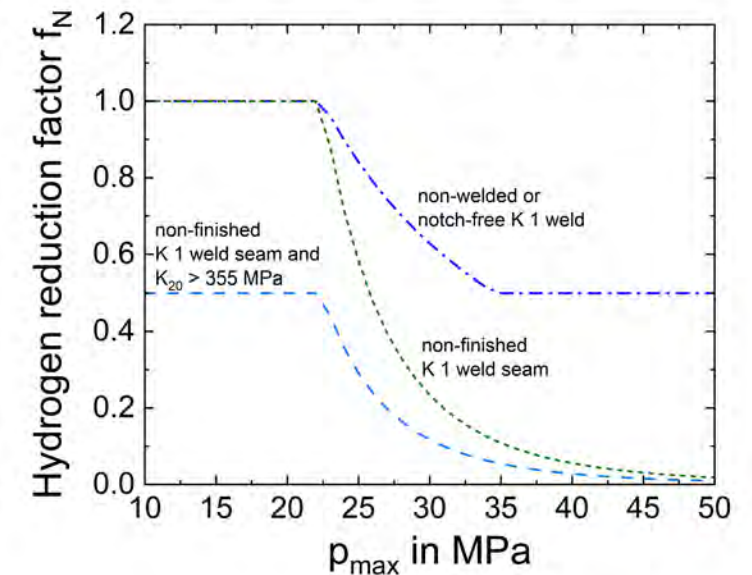


Bild A.8 — Vergleich zwischen Versuchsdaten und AD 2000-Merkblatt S 2

Reduction factors for temperature, surface roughness, mean stress, wall thickness, weld seam quality

Reduction factor for hydrogen influence depending on weld seam and material strength



- + Easy to apply
- +? Seem applicable for new designed hydrogen pipelines or storage tanks, where a defect-free surface might be assumed
- +/- Conservatism is in the hydrogen reduction factor and the safety factors
 - Only a few fatigue tests in gaseous hydrogen are published in literature

Discussion of pros and cons

Fatigue-based versus crack growth-based design methods

Discussion points

- Fatigue-based versus crack growth-based design methods: Where/how do we draw a line?
 - Size of component? Wall thickness?
 - Guaranteed surface quality?
 - Safety requirements?
 - ...
- Fatigue-based: What's a defect-free surface/component?
 - Guarantee of defect-free surface until which size?
 - Is in-air experience applicable here?
 - How can a manufacturer guarantee a defect-free component?
- For two standards: How to get them compatible to avoid totally different results in the transition zone (e.g., wall thickness) between two standards?

Contact

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Exceptional service in the national interest

Best practices for fracture testing in gaseous hydrogen

Joe Ronevich, Milan Agnani, Rob Wheeler, Chris San Marchi

Sandia National Labs

Study Group on Materials Testing and Qualification for Hydrogen Service

Atlanta, GA

July 21st, 2023

SAND2023-06706 PE

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.





Motivation

- **Fracture toughness testing is critical for assessing structural integrity of components**
 - Industry utilizes fracture toughness data when performing fracture mechanics assessment
 - However, fracture resistance is influenced by environment (pressure, testing rate, gas quality) and how the test is performed can influence the reported fracture values

How can we ensure fracture test data can be utilized without ambiguity?



Variables to consider

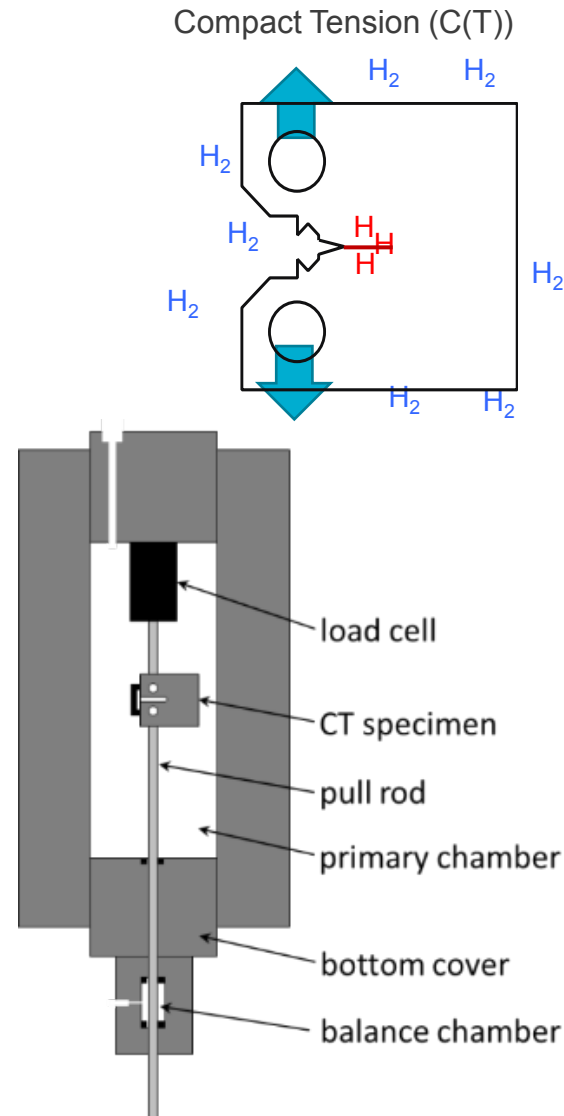
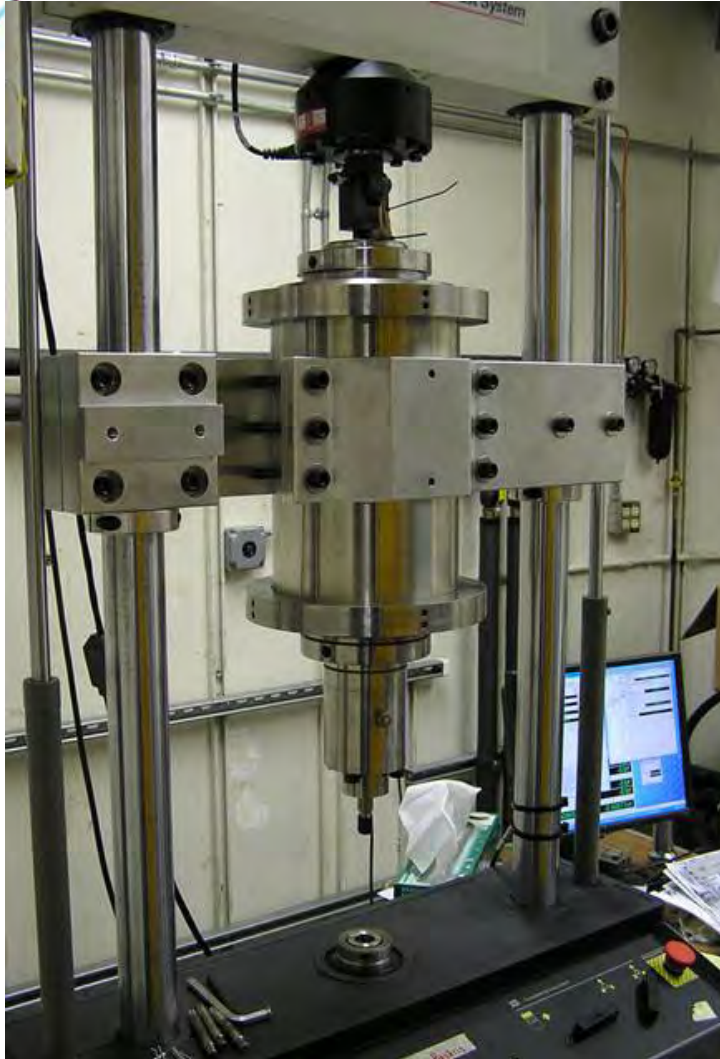
- Transducers
- Fracture test method
- Specimen size
- Crack monitoring
- Testing rate
 - J0, J0.2, J0.5 what should be reported
- Soak time (if needed)
- Gas quality

Testing conditions should be most representative of application (when possible)

→ Ensures most impactful data



Fatigue & Fracture Testing at SNL-CA



Instrumentation

- Internal Load cell
- Clip gauge
- Direct Current Potential Difference (DCPD)

Fatigue: ASTM E647

- Load ratios (R) 0.1 to 0.8
- Frequency: 0.01 → 10 Hz
- Constant load or K-control

Fracture: ASTM E1820 (Elastic-Plastic)

- Loading rates (0.1 → 1 MPa m^{1/2}/min)
- Crack length monitor (DCPD)

Environment

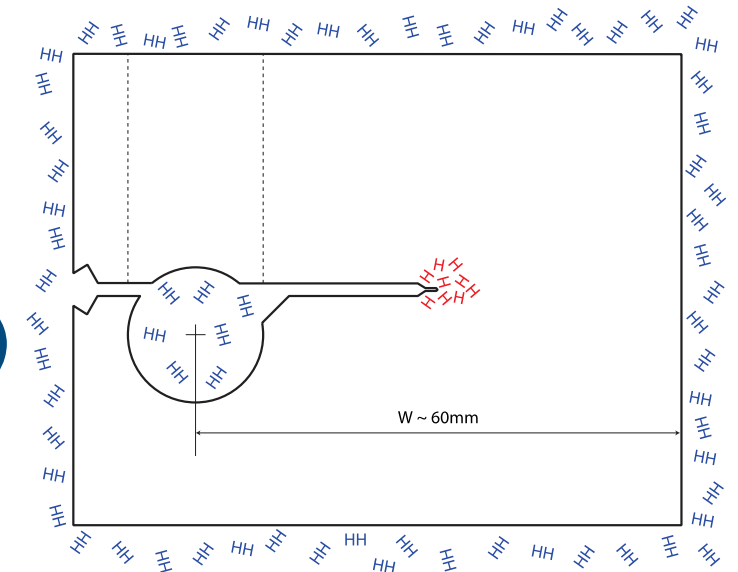
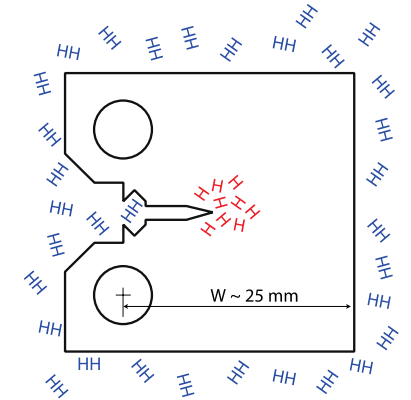
- Air
- Pure H₂
- Gas blends, e.g. N₂ – 3% H₂
- Gas impurity mixtures:
e.g. H₂ + 10-1000 ppm O₂



How is fracture measured in gaseous hydrogen?

- **Hydrogen pressure vessels**
 - ASME BPVC VIII.3 KD-10
 - ***Fatigue: ASTM E647***
 - Linear-elastic fracture mechanics method
 - Compact Tension specimen (CT)
 - ***Fracture: ASTM E1681***
 - Linear-elastic fracture mechanics method
 - Modified Bolt-Load Compact specimen
 - Compact specimen (similar to CT specimen)
- **Hydrogen pipelines**
 - ASME B31.12 - refers to BPVC VIII.3 KD-10

Compact Tension



Modified Bolt-Load Compact



What is the minimum specimen size?

Consider the case of low-strength, ductile metals, such as line pipe steel

- **ASME B31.12**

- Minimum $K_{IH} = 55 \text{ MPa m}^{1/2}$

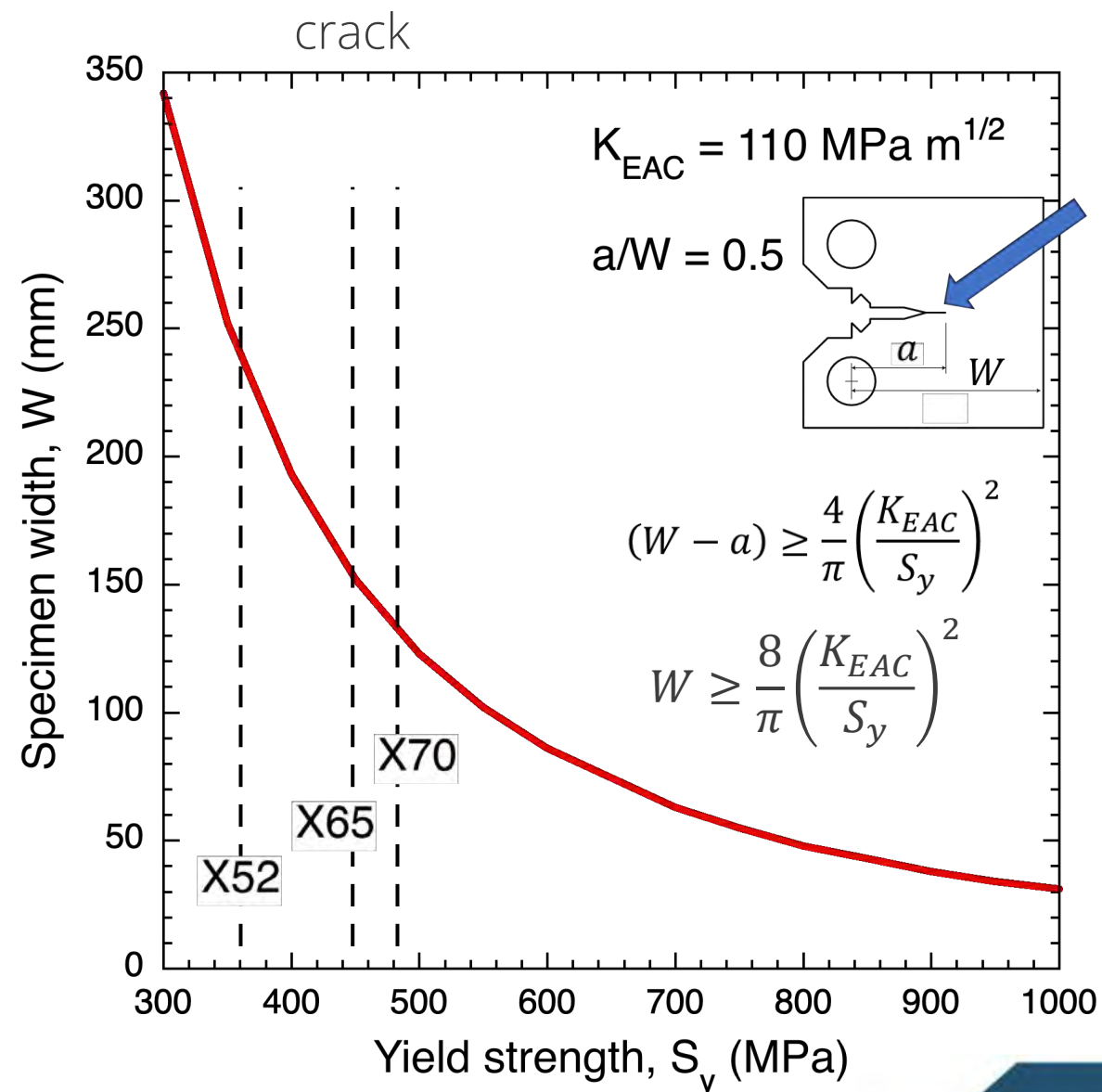
- **ASME test method: BPVC VIII.3 KD-10**

- $K_{IH} = \frac{1}{2} K_{EAC}$ (if no crack propagation)

- **Therefore, in practice, specimens are typically 'over loaded':**

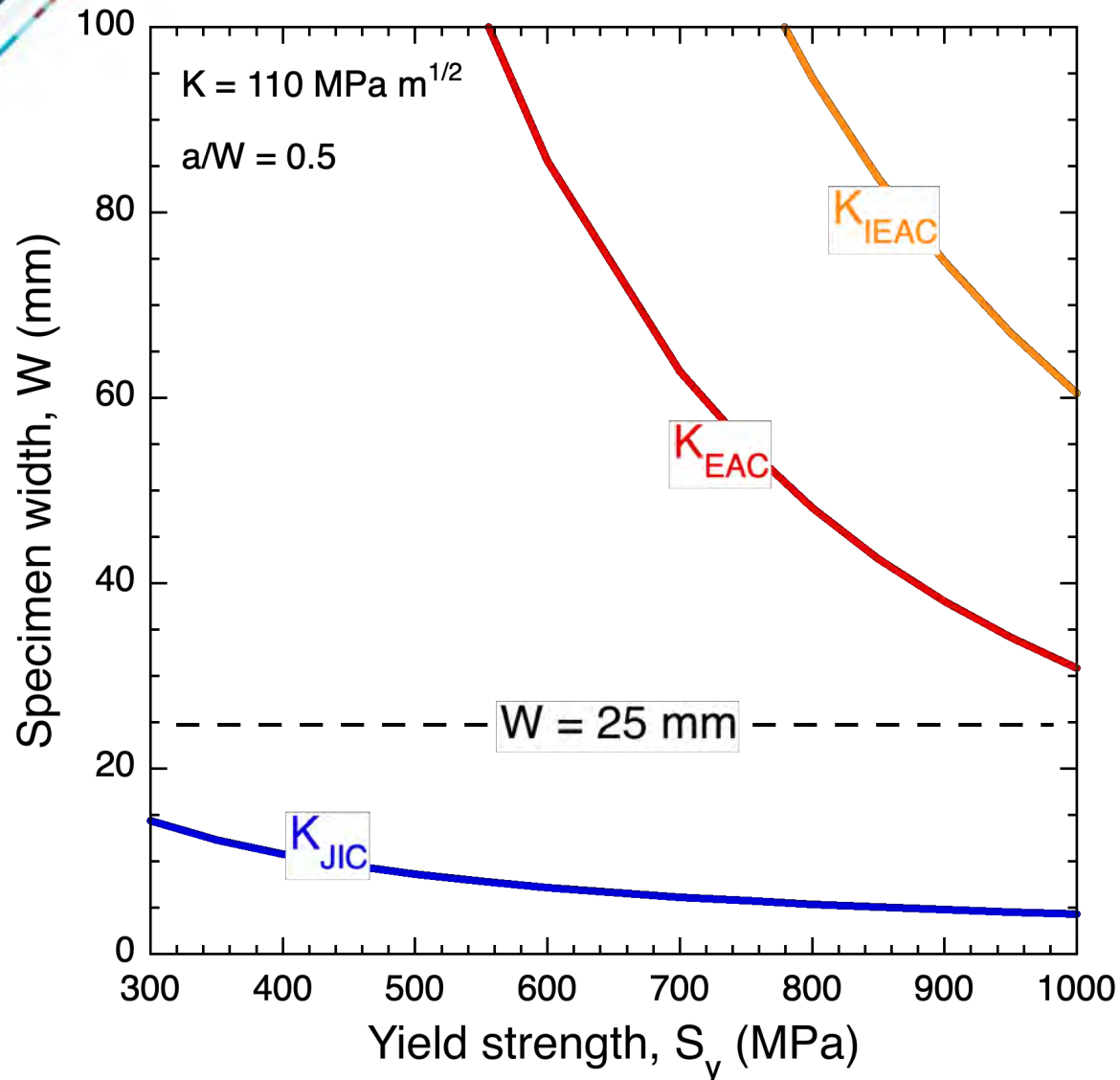
- $K_{EAC} \geq 110 \text{ MPa m}^{1/2}$

Specimen size requirements for line pipe steels cannot be satisfied





Comparison of specimen sizes



- **ASTM E1681 requires exceptionally large specimens for determining fracture thresholds of low-strength, high ductility steels**
- **ASTM E1820 elastic-plastic fracture can be measured using specimens that are ~100x smaller than ASTM E1681 specimens**
 - **Compact tension specimens with $W > 25$ mm are generally adequate to satisfy requirements of E1820**



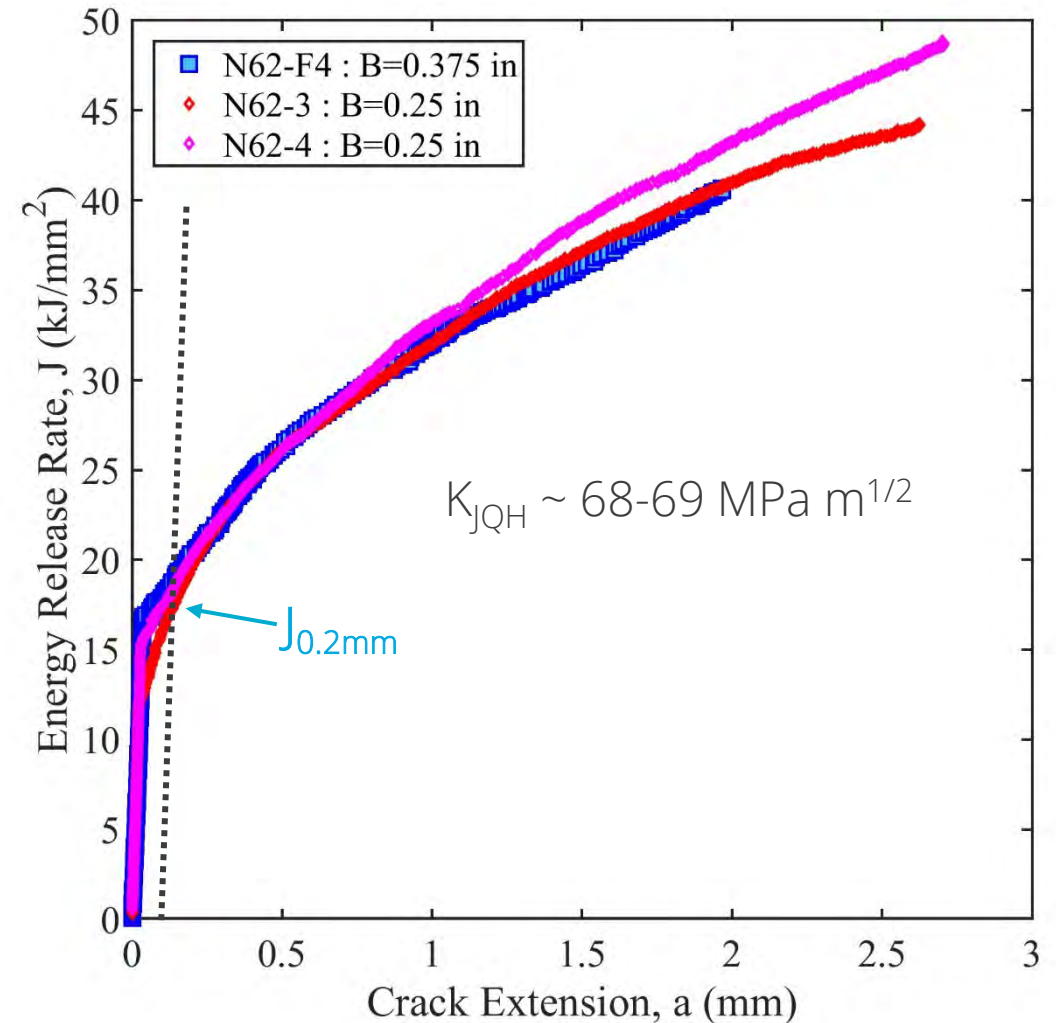
Hydrogen-Assisted Fracture : Thickness

N62 BM 210 Bar GH2

B = 0.25 in



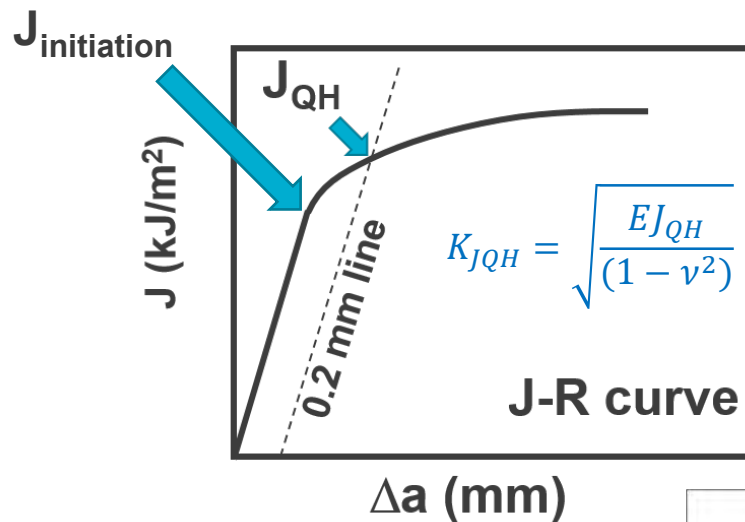
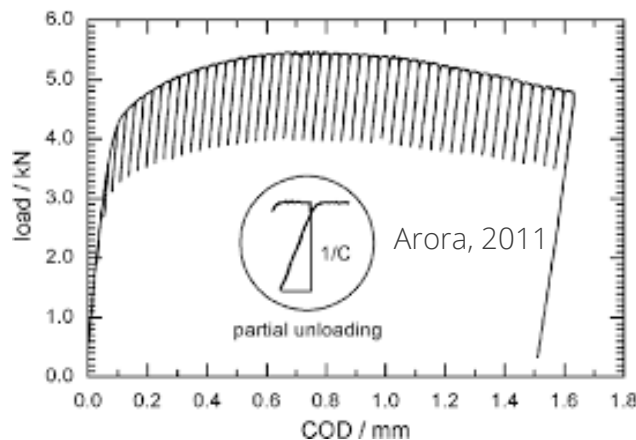
B = 0.375 in



How do delaminations influence K_{JQH} ?

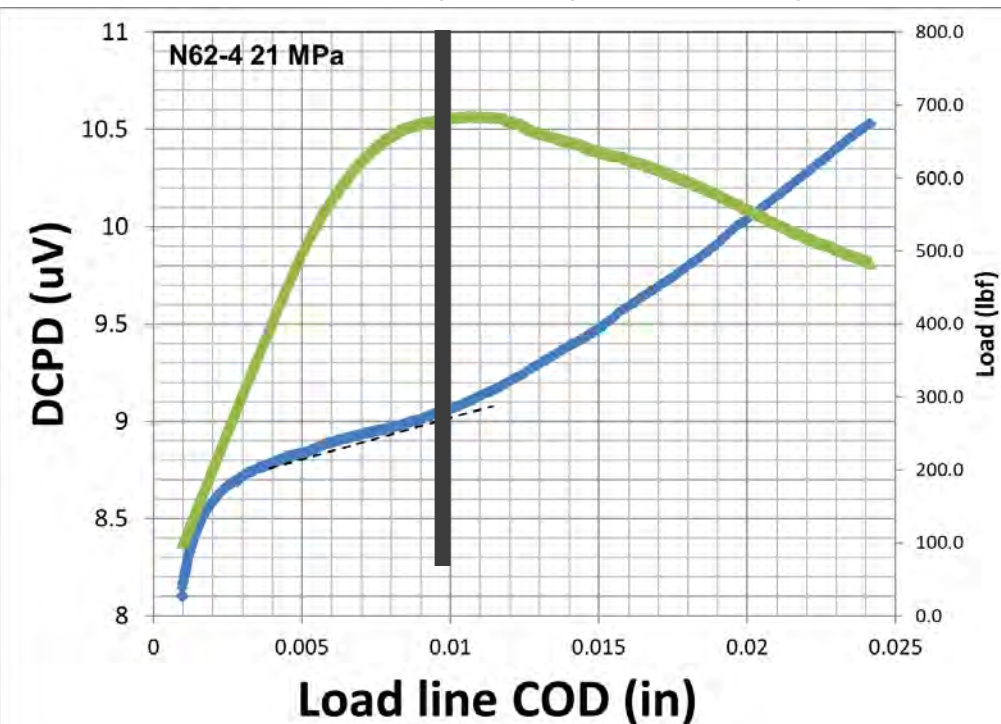
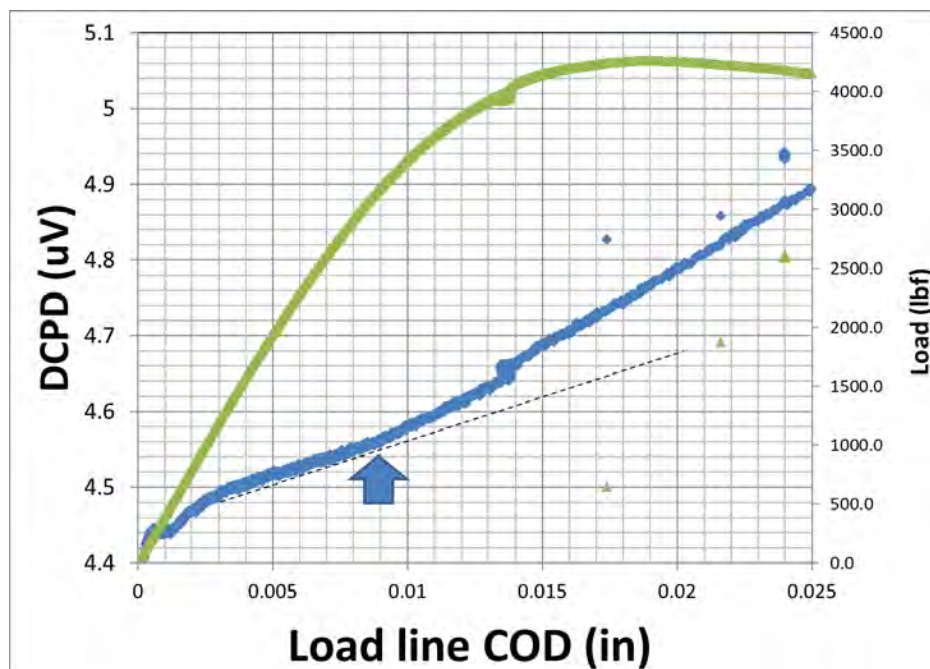
Direct Current Potential Difference (DCPD) can be used to identify crack initiation

Unloading
compliance

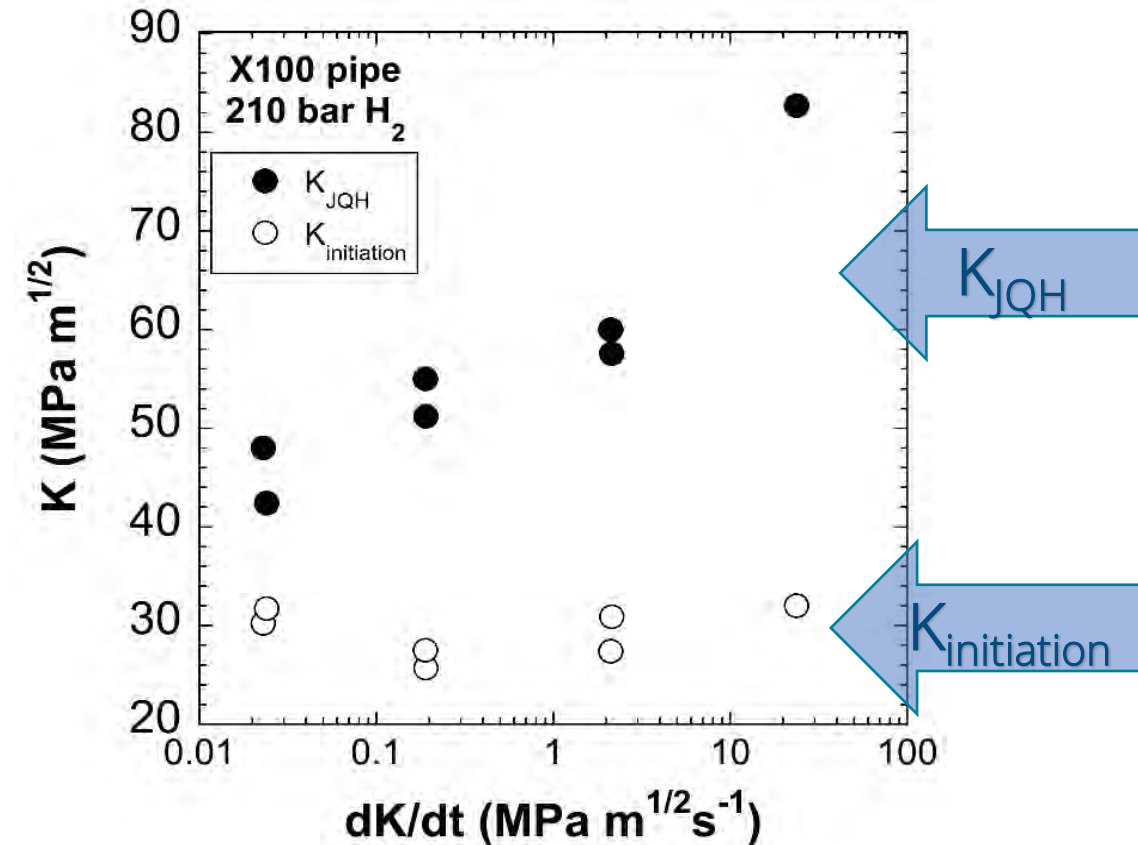
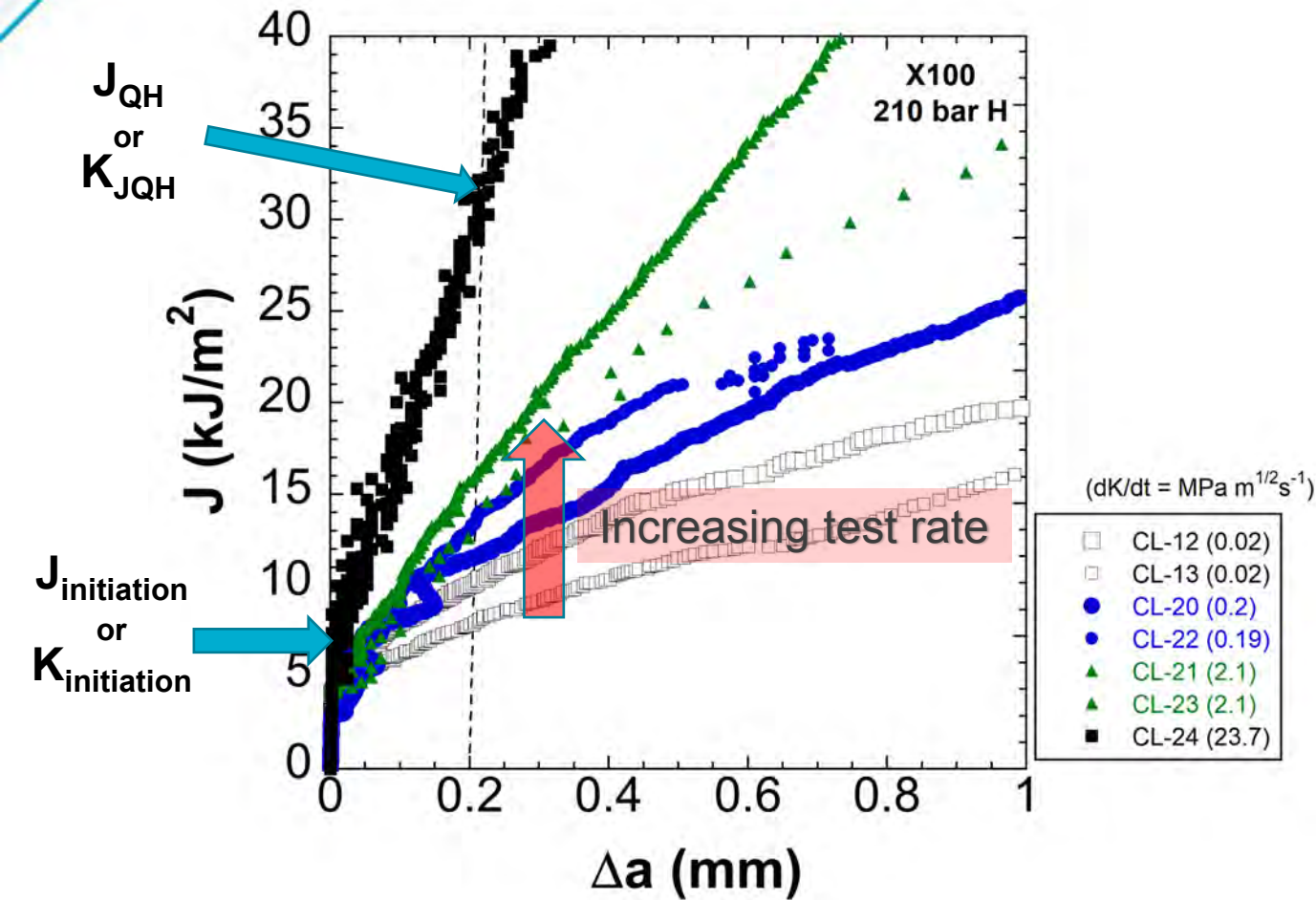


Not always easy to identify initiation

Continuous loading
DCPD



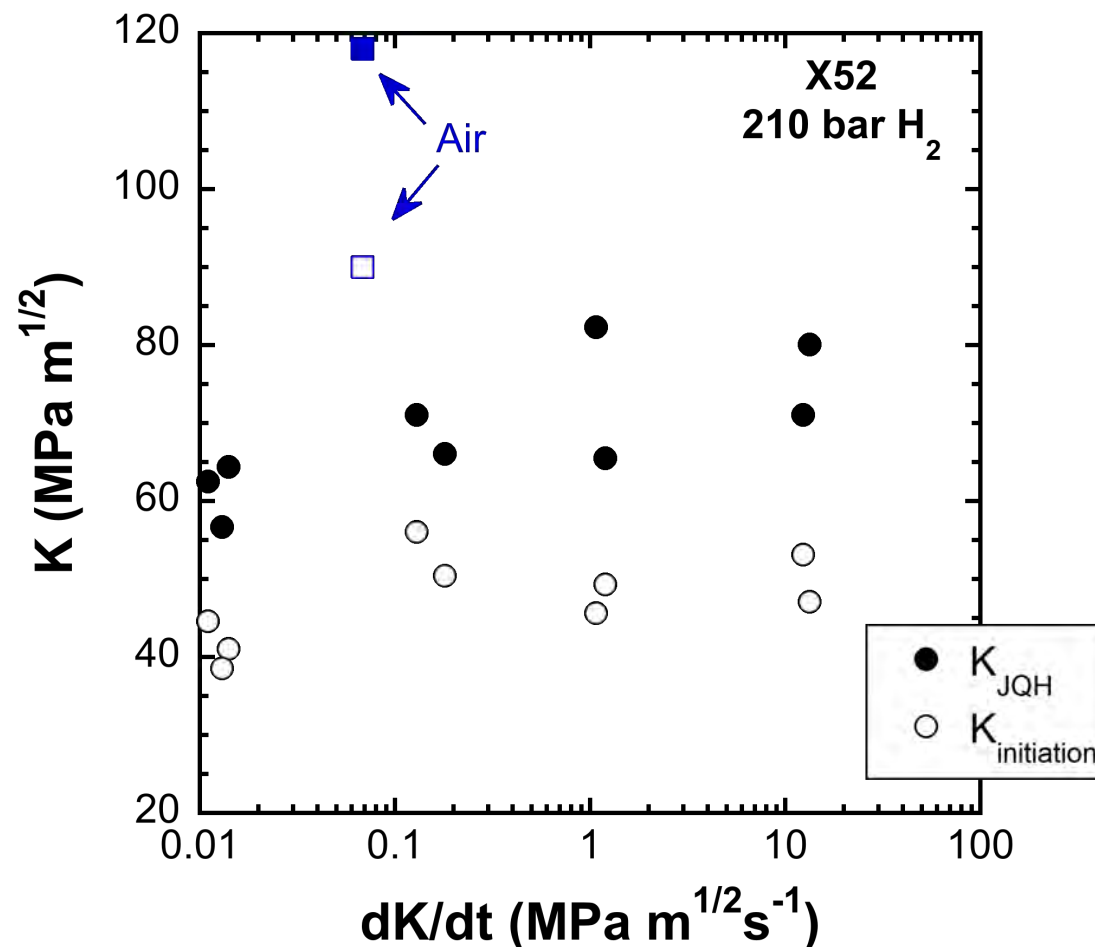
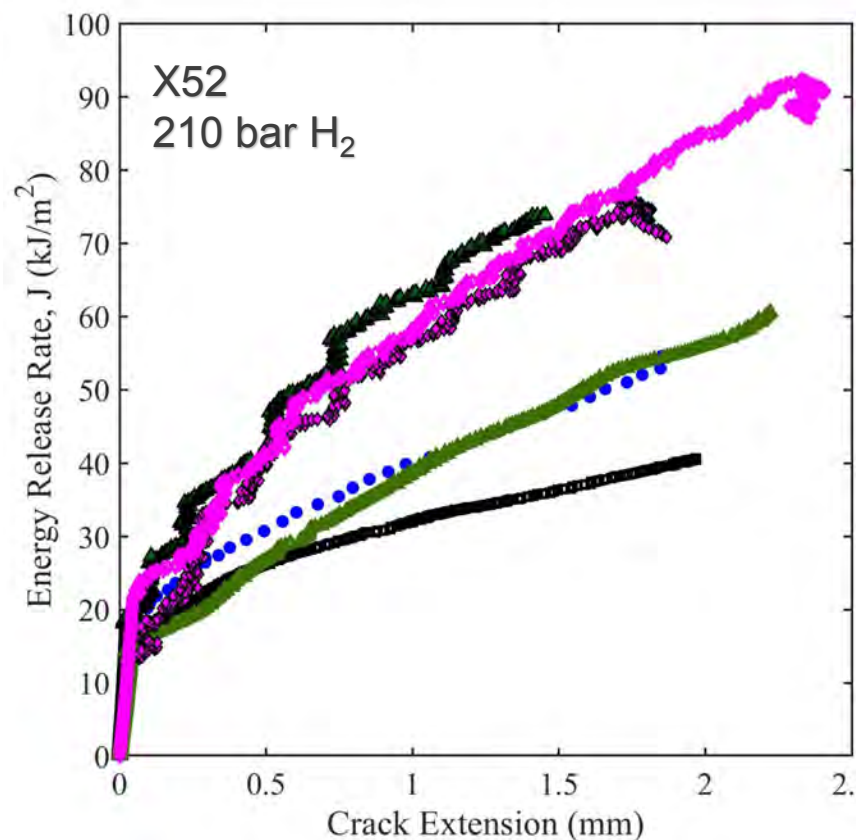
Slope of J-R curve is clearly influenced by testing rate



**X100 fracture resistance (K_{JQH}) was rate-dependent
 $\rightarrow K_{initiation}$ was less sensitive to rate**



X52 (vintage 1962) showed less influence of testing rate



- 0.005 mm/min
- 0.05 mm/min
- 0.5 mm/min
- 0.5 mm/min
- 5 mm/min
- 5 mm/min



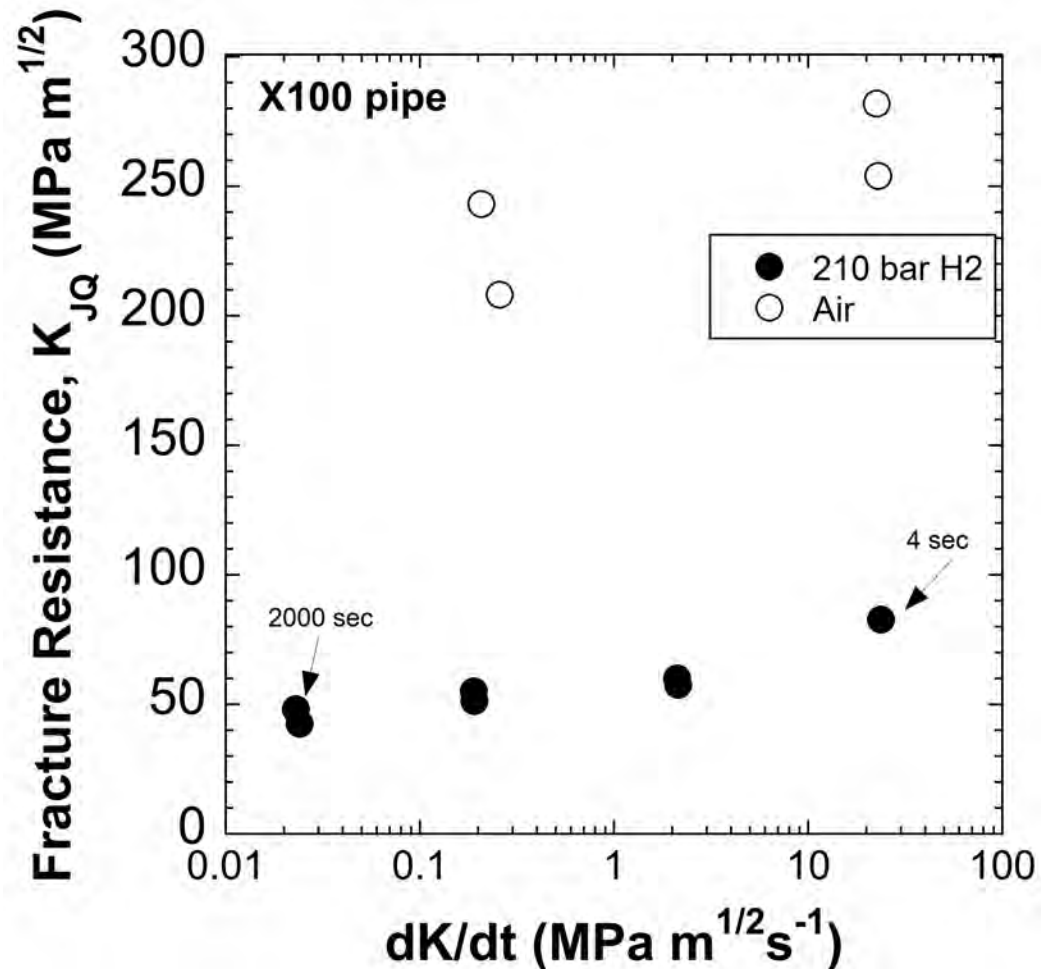
Should testing rate be even slower?

What should be reported J_0 , $J_{0.2}$, $J_{0.5}$?

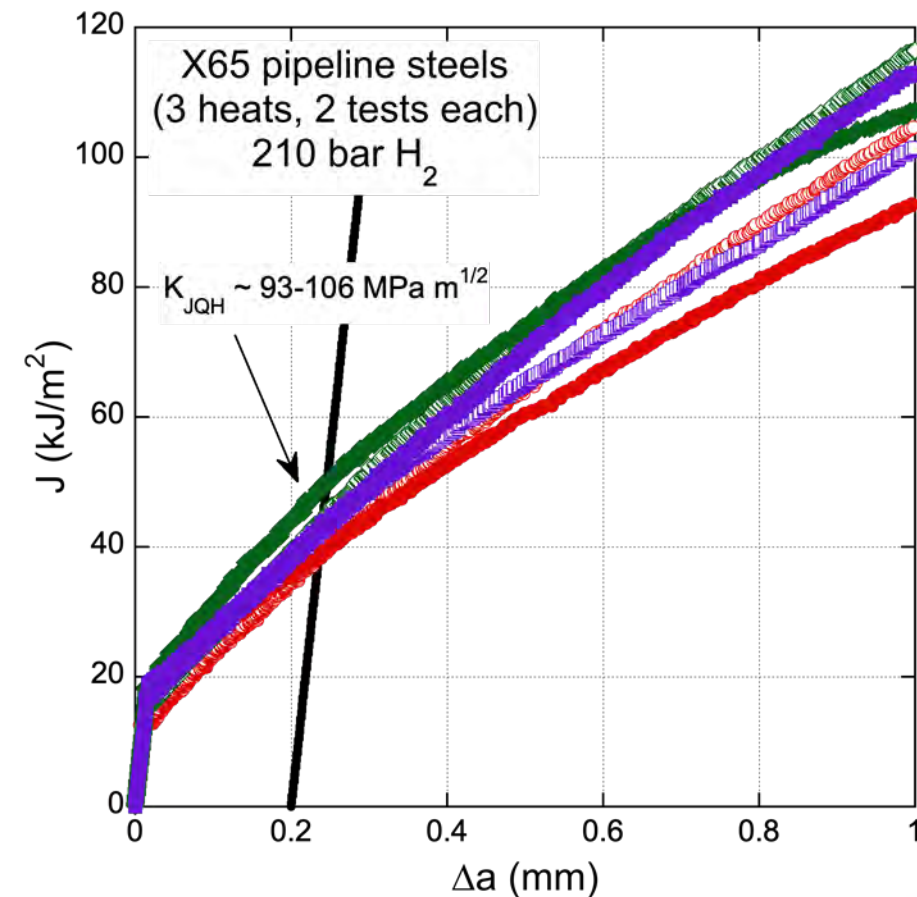
Is there a minimum required soak time in H₂?

- We have not systematically controlled this variable**

Fracture only tests experienced from
30 min-15 hr soak time → No clear trend

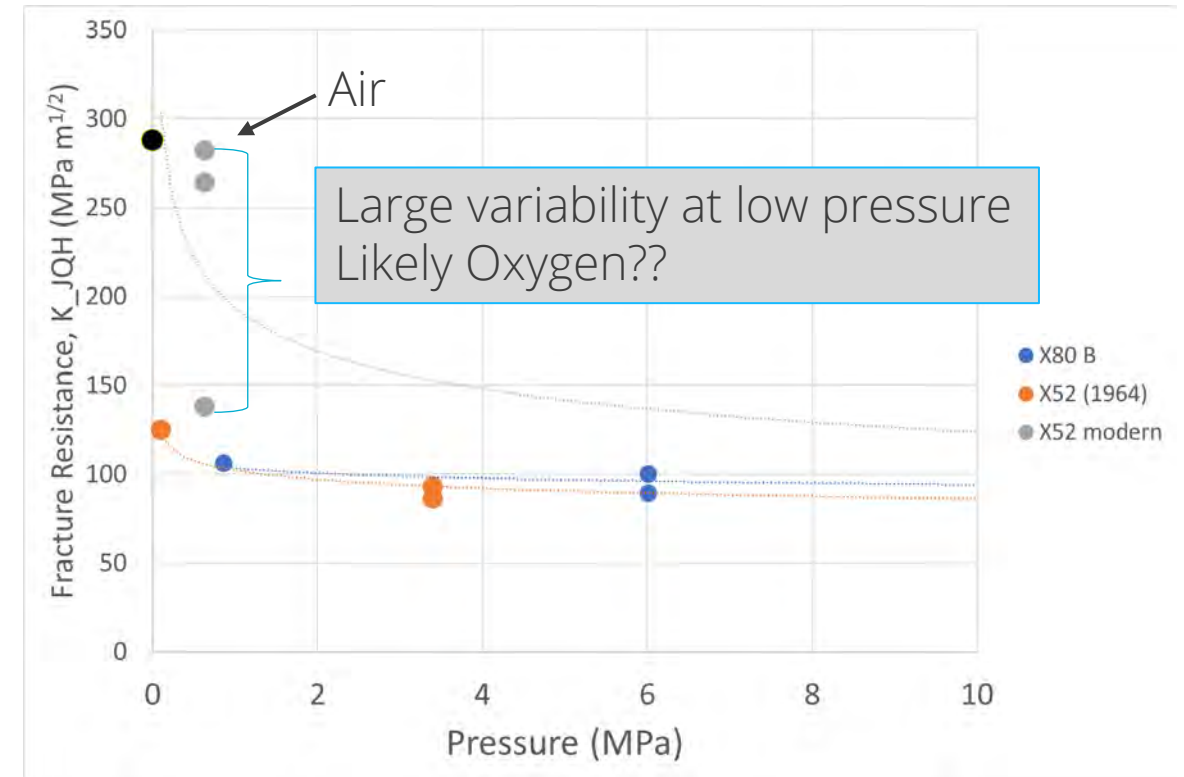
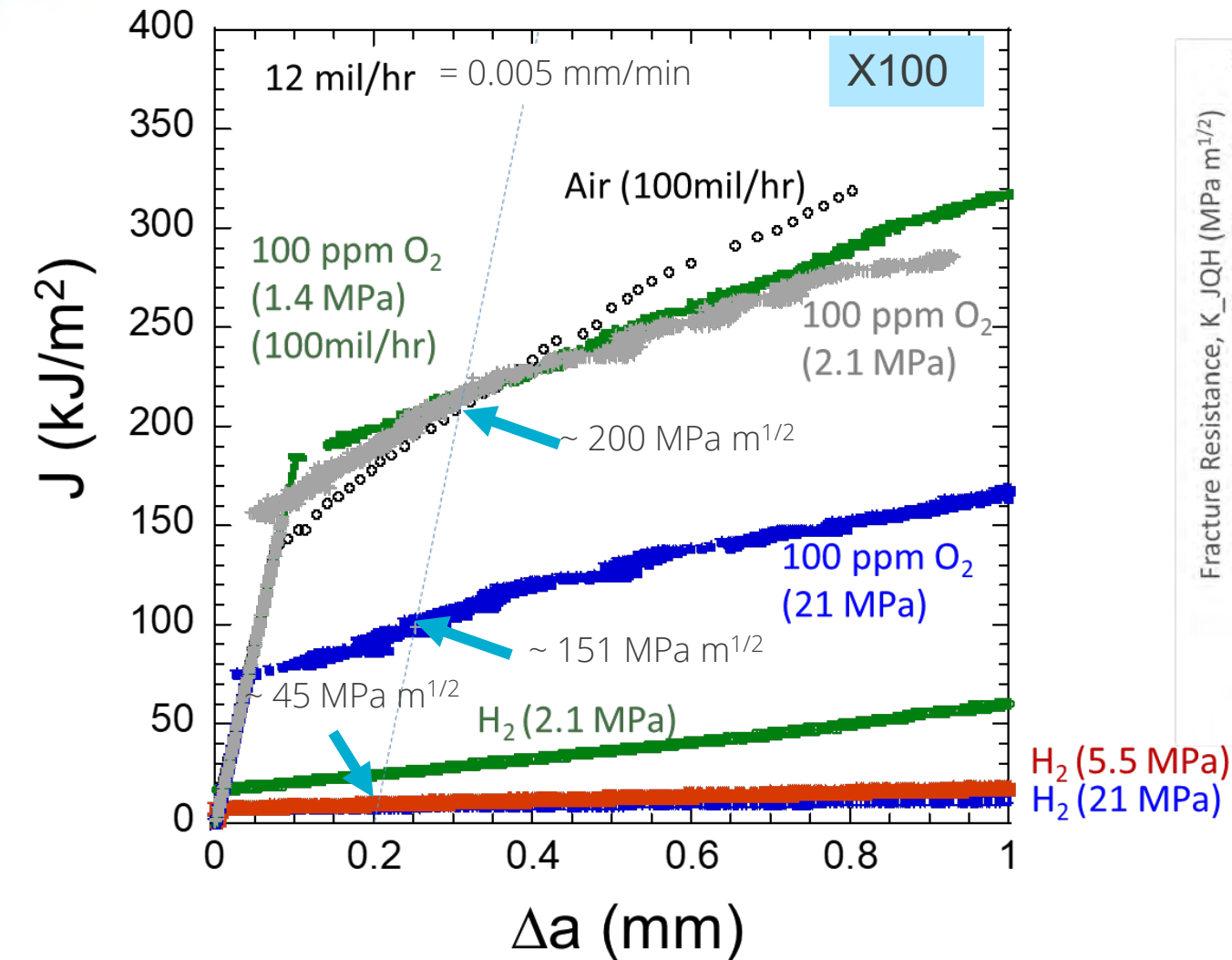


2-4 days of fatigue in GH2 performed prior
to fracture test



Small amounts of oxygen can influence fracture, particularly at low pressures

CHMC-1:

Water ~ 10 ppm, O₂ ~ 2 ppm



Summary

- Elastic-plastic fracture (ASTM E1820) necessary for pipelines
- Fracture resistance (K_{JQH}) is influenced by
 - Testing rate – K_{JQH} increases with increasing dK/dt
 - Gas pressure – decreases with increasing P (non-linear)
 - Gas quality – small amounts of impurities can have large influence at low pressures
- Influence of soak time is not been systematically studied
- DCPD is valuable at identifying initiation
 - Not always easy to pick initiation

We should provide industry with concise, clear, relevant results to eliminate ambiguity of fracture data

Fracture Toughness Tests for Hydrogen Pipelines

Robin Gordon Microalloying

**API 1104 Hydrogen Task Group
July 14, 2023**

Background

- One of the key challenges of transporting hydrogen in pipelines is the potential degradation in the fracture toughness and fatigue properties of the pipe material due to Hydrogen Embrittlement.
- There are approximately 1,600 miles of hydrogen pipelines currently being operating in the United States with an excellent safety record.
- Most of the existing hydrogen pipelines in the US share the following features:
 - 20" Diameter or less
 - Constructed using API 5L Grade X52 or lower
 - Operate at Design Factors of 0.50 or lower

Background contd ...

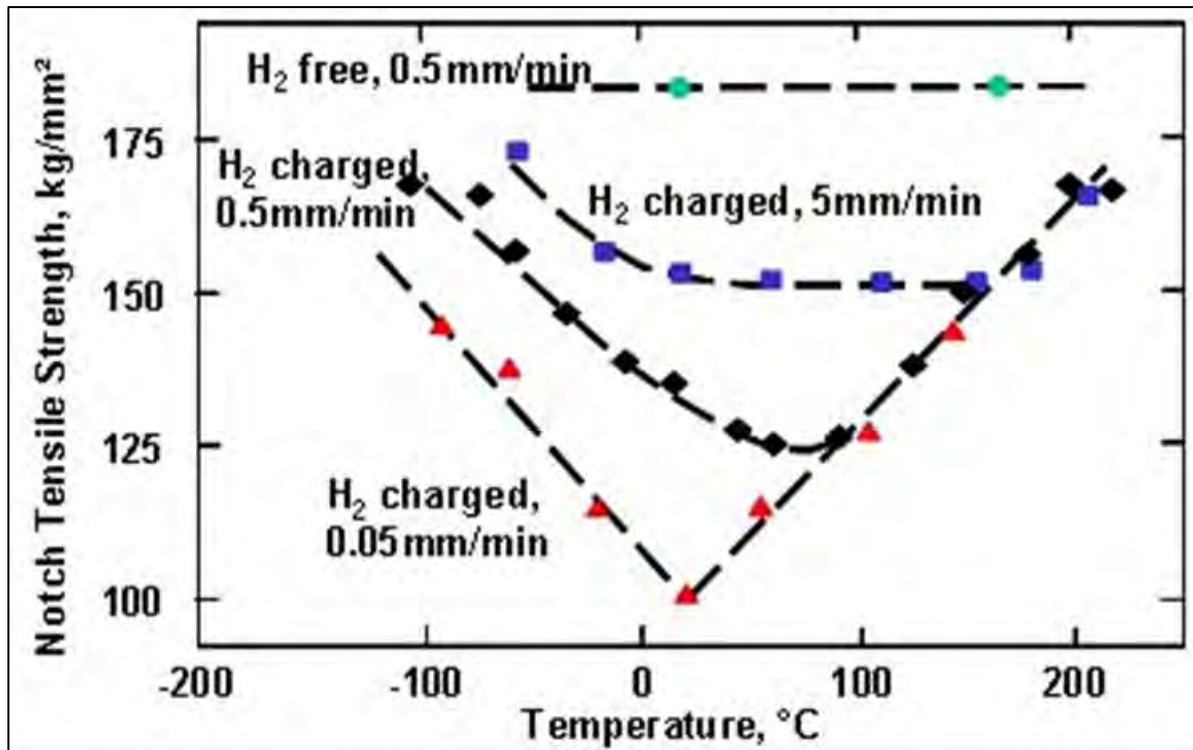
- Although ASME B31.12 has supported the design / construction of existing Hydrogen Pipelines there is increasing interest in extending hydrogen pipelines to higher grade linepipe, larger diameters and higher Design Factors.
- As Hydrogen Pipelines evolve it is important to ensure that:
 - The impact of hydrogen on both Fracture Toughness and Fatigue Crack Growth Properties is fully characterized.
 - Industry accepted Test Procedures are established particularly in cases where an Engineering Critical Assessment will be performed to develop Flaw Acceptance Criteria.

Hydrogen Embrittlement

- Pipelines that operate in Sour H₂S Service or Hydrogen Service are susceptible to Hydrogen Embrittlement.
 - Reduced Ductility
 - Reduced Material Toughness
 - Reduced Fatigue Performance
- Hydrogen embrittlement occurs when atomic hydrogen is absorbed into the steel.
- Hydrogen migrates to regions of high stress or local plastic deformation.
- Since Hydrogen diffusion is slow Tests in Hydrogen must be performed at slow loading rates.

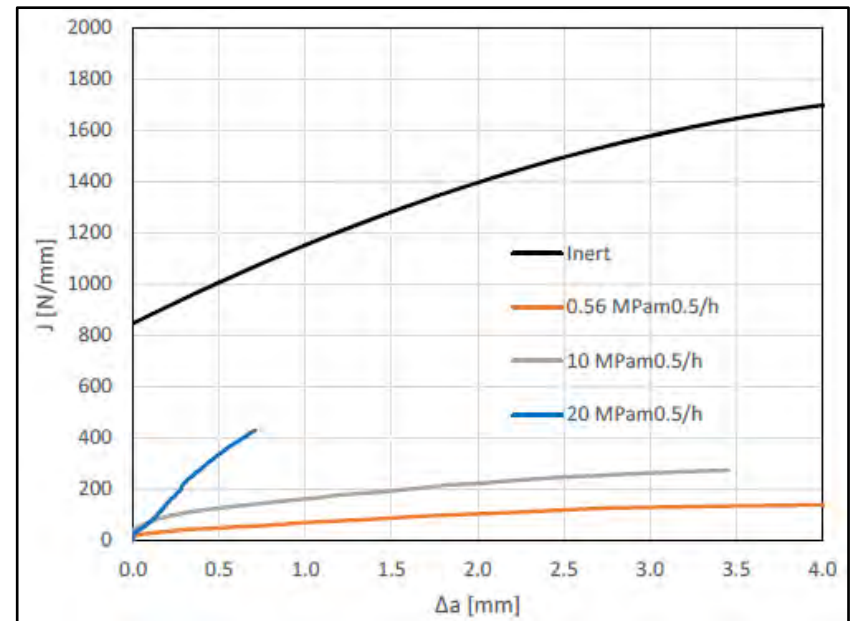
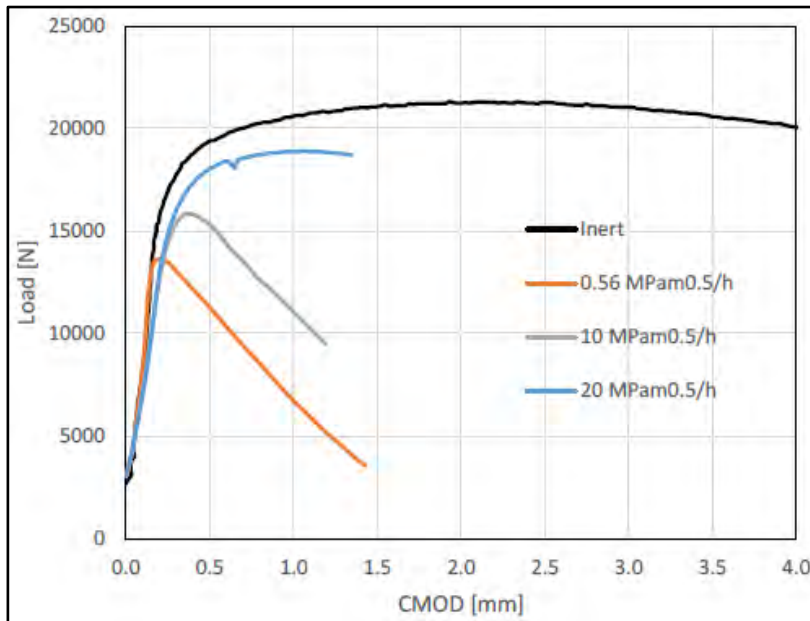
Hydrogen Embrittlement (HE)

- In addition to Hydrogen Concentration, HE is a Function of Test Temperature and Loading Rate as shown below by data generated by Graville (1967).



Hydrogen Embrittlement (DNV JIP)

- Fracture Toughness Tests performed in a Hydrogen Charged Environment also exhibit a Loading Rate dependence.



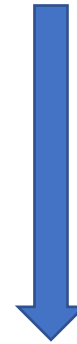
Fracture Toughness Test Methods

- Sour Service Fracture Toughness Test Procedures have evolved over the last 40 Years and Best Practice Guidelines have been established.
 - Consistent Results
 - Ability to Compare Results from Different Labs
- In comparison Fracture Toughness Test Procedures for Hydrogen Pipelines are still under development with different Test Labs using different Test Methods and procedures.
 - Inconsistent Results
 - Can't compare results from Different Test Labs
 - Variable Fracture Toughness Results for ECA

Sour Service Test Methods

Test Environments

- Hydrogen Charged Tests
- In Situ (Atmospheric Pressure) Tests
- Autoclave (Pressure > Atmospheric) Tests
- Autoclave (Fugacity Pressure) Tests



Evolution
over 40
Years

Test Procedures

- Slow Rising Load
- Step Load
- Constant Load (K1SSC)

Sour Service Test Methods

Slow Rising Load

- Perform Test at Slow Loading Rate and present results in the form of a J or CTOD R-Curve

Step Load Tests

- Load specimen in steps with Hold Periods (typically 12 – 24 hrs) between Load Steps.
- Identify the Load Step that results in Sustained Load Cracking

Constant Load Tests

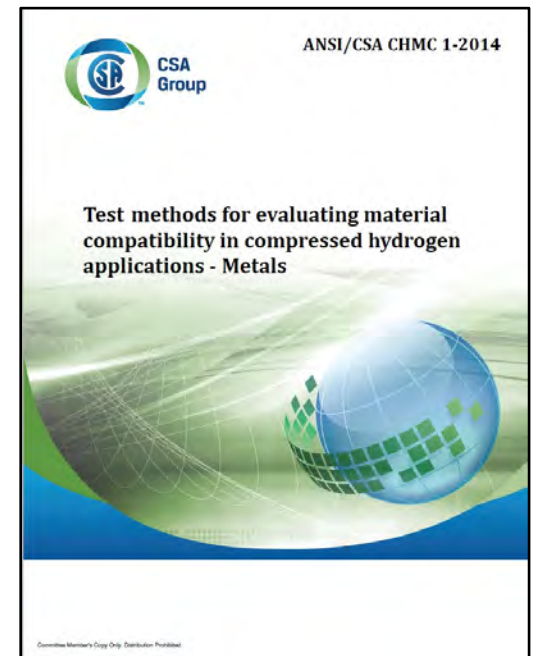
- Load Specimen to Target Load (Target K)
- Hold under Constant Load for 30 Days
- Break Specimen Open and look for indications of sour service Crack Growth.
- Identify the K_{1SSC} value where sour service crack growth is evident.

Sour Service Test Procedure (Best Practice)

- 1) Preferred Specimen Geometry (SENB or CT).
- 2) Machine and Fatigue Pre-Crack Specimens (through Thickness notch).
- 3) Coat all surfaces except surface with notch to represent single side exposure.
- 4) Soak Specimen in Test Environment for 4 – 5 Days to saturate specimen and allow stable scales to form.
- 5) Load Specimen under displacement control at a Loading Rate that produces a K rate in the elastic loading regime between:
 - 0.005 – 0.05 N.mm^{-3/2}/sec
 - 0.01 – 0.10 MPa√m/minute
- 6) Monitor crack growth during Test using Potential Drop (preferred) or Unloading Compliance (can introduce history effects & fatigue crack growth).
- 7) Present results as a J or CTOD R-Curve
- 8) Calculate J and CTOD values at:
 - Crack Initiation
 - 0.20 mm of Stable Crack Growth
 - Maximum Load

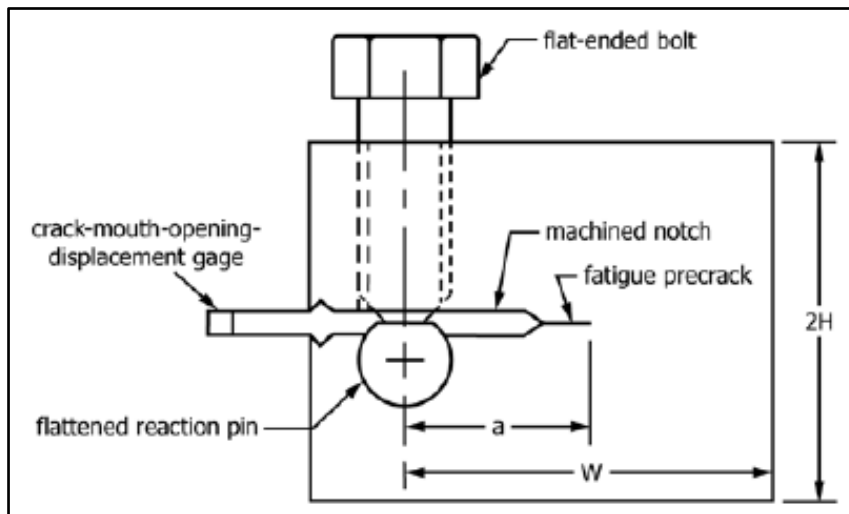
Hydrogen Fracture Toughness Test Methods

- 1) ASME B31.12 – 2019 “ Hydrogen Piping and Pipelines”
 - Constant Load or Constant Displacement Tests
- 2) ANSI / CSA CHMC 1-2014, “Test methods for Evaluating Material Compatibility in Compressed Hydrogen Applications – Metals”
 - Slow Rising Load Tests



Constant Displacement Fracture Tests

- ASTM E1681 / ASME B31.12 / ASME KD-1040



$$K_I = [V_m E / W^{1/2}] f(a/w)$$

$$f(a/W) = [1 - a/W]^{1/2} [0.654 - 1.88 (a/W) + 2.66 (a/W)^2 - 1.233 (a/W)^3]$$

This Equation implies that for specimens of the same size the applied K is the same for a given CMOD.

- Preload the CT Specimen and then expose the specimen to a Hydrogen Environment for a Fixed Duration (40 Days).
- Break the specimen open and examine for sub-critical crack growth

Constant Load Fracture Tests

- ASTM E1681 / ASME B31.12 / ASME KD-1040
 - SENB Specimens
 - Compact Tension (CT) Specimens
- Preload the Specimen and then hold the specimen under Constant Load in a Hydrogen Environment for a Fixed Duration (40 Days).
- Break the specimen open and examine for sub-critical crack growth .

$$K = \left[\frac{P}{BW^{\frac{3}{2}}} \right] f\left(\frac{a_o}{W}\right)$$

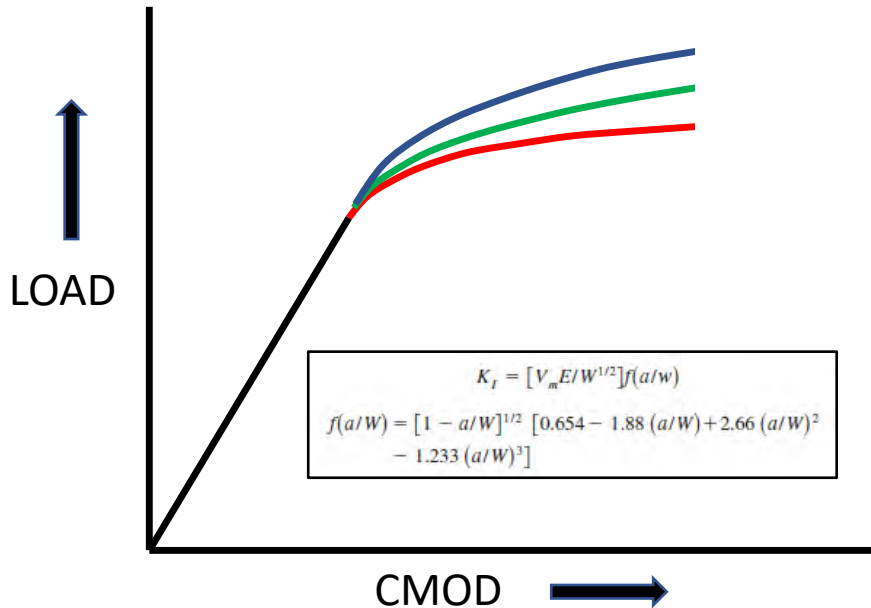
This Equation implies that for specimens of the same size the applied K is the same for a given Load.

Constant Load vs Displacement Fracture Tests

- If the subcritical crack growth < 0.25 mm the material is characterized as suitable for hydrogen service and a K_{1H} value can be determined.
- For tests conducted under Constant Displacement K_{IH} is equal to 50% K_{IAPP} .
- For tests conducted under Constant Load K_{IH} is equal to 100% K_{IAPP} .
- If the subcritical crack growth > 0.25 mm, then the procedure specified in ASTM E 1681 paras. 9.2.1 and 9.2.2 shall be used in establishing the K_{IH} value.

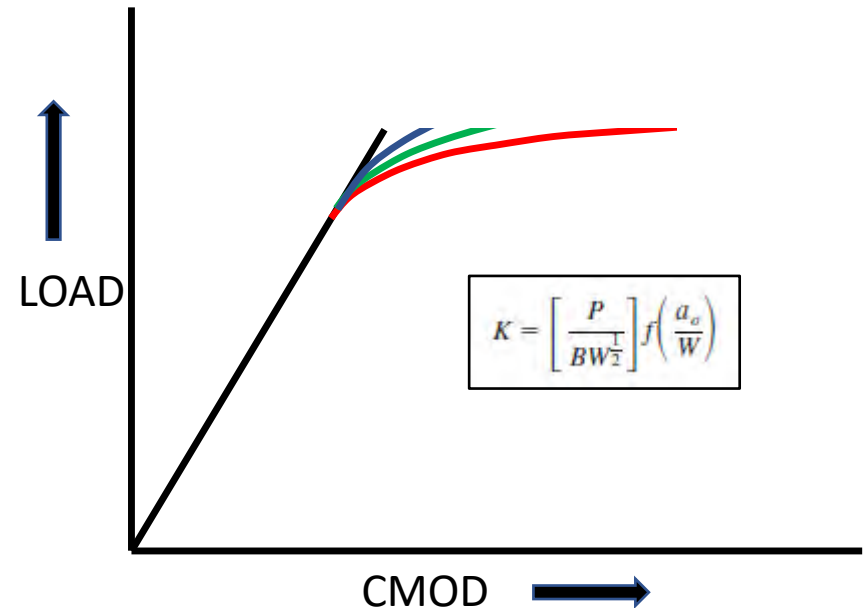
Load vs CMOD Traces

Loaded to a Fixed Displacement



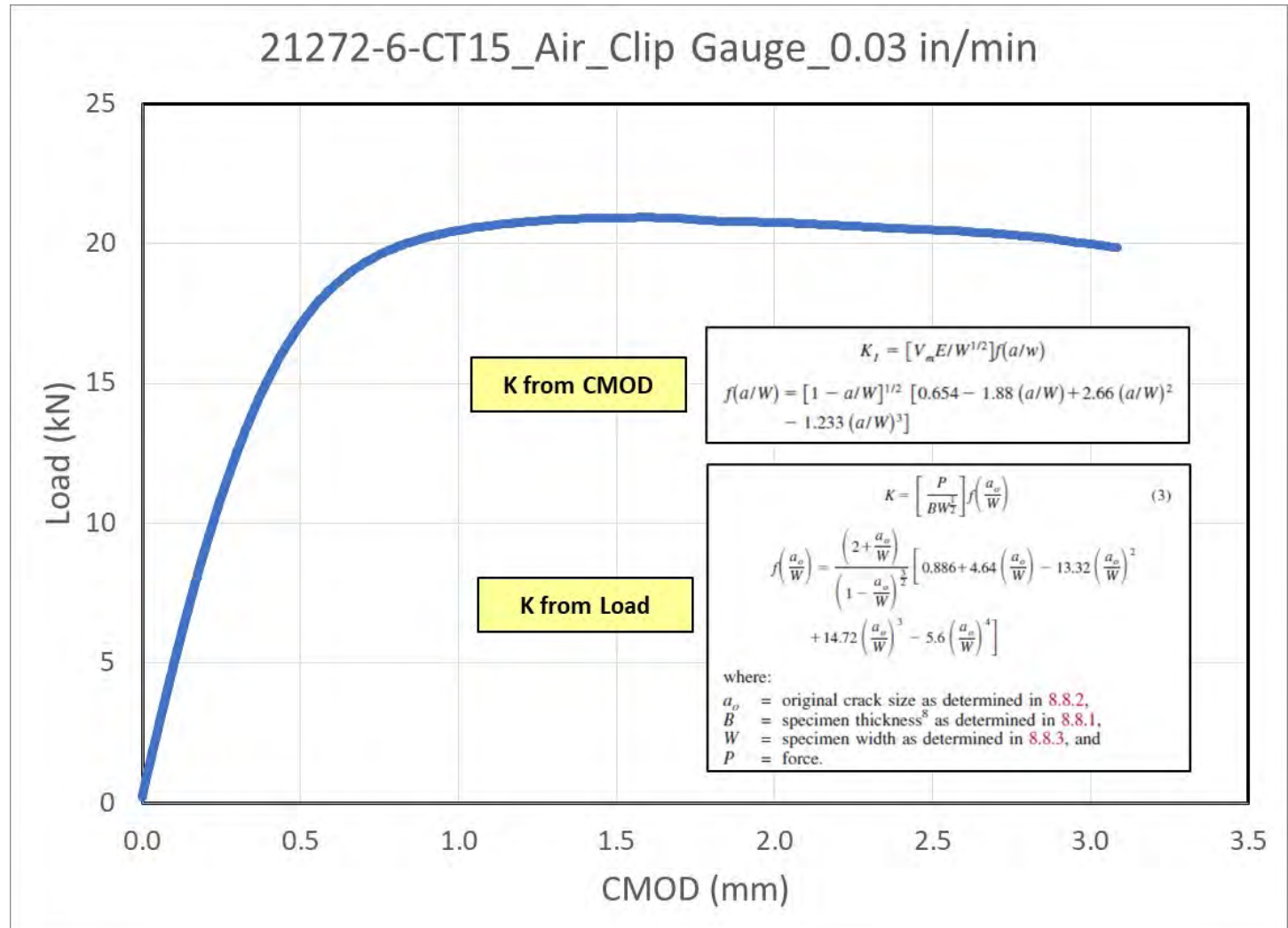
Same K using ASTM E1681 but
Different Applied J or CTOD

Loaded to a Fixed Load

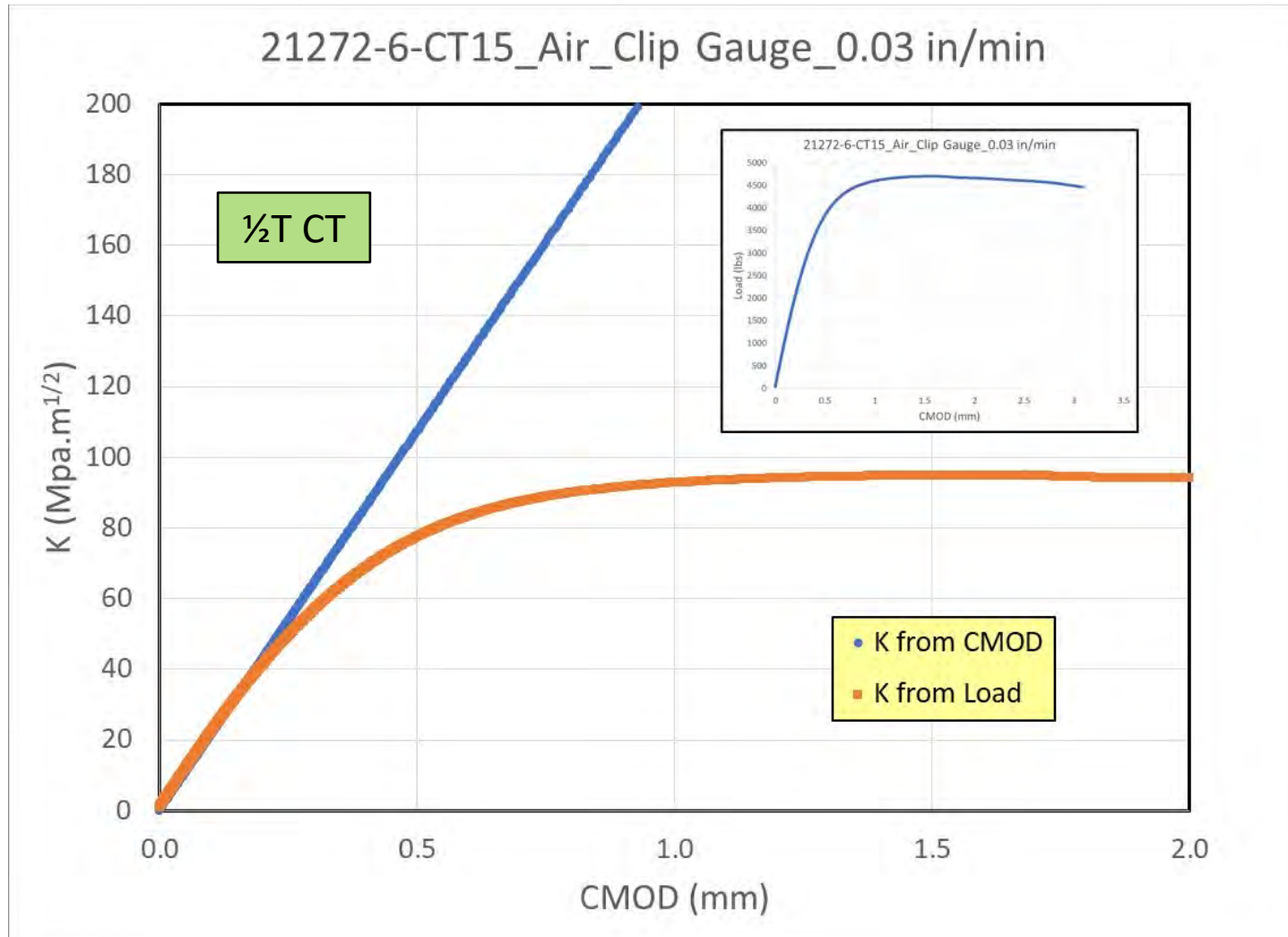


Same K using ASTM E1681 but
Different Applied J or CTOD

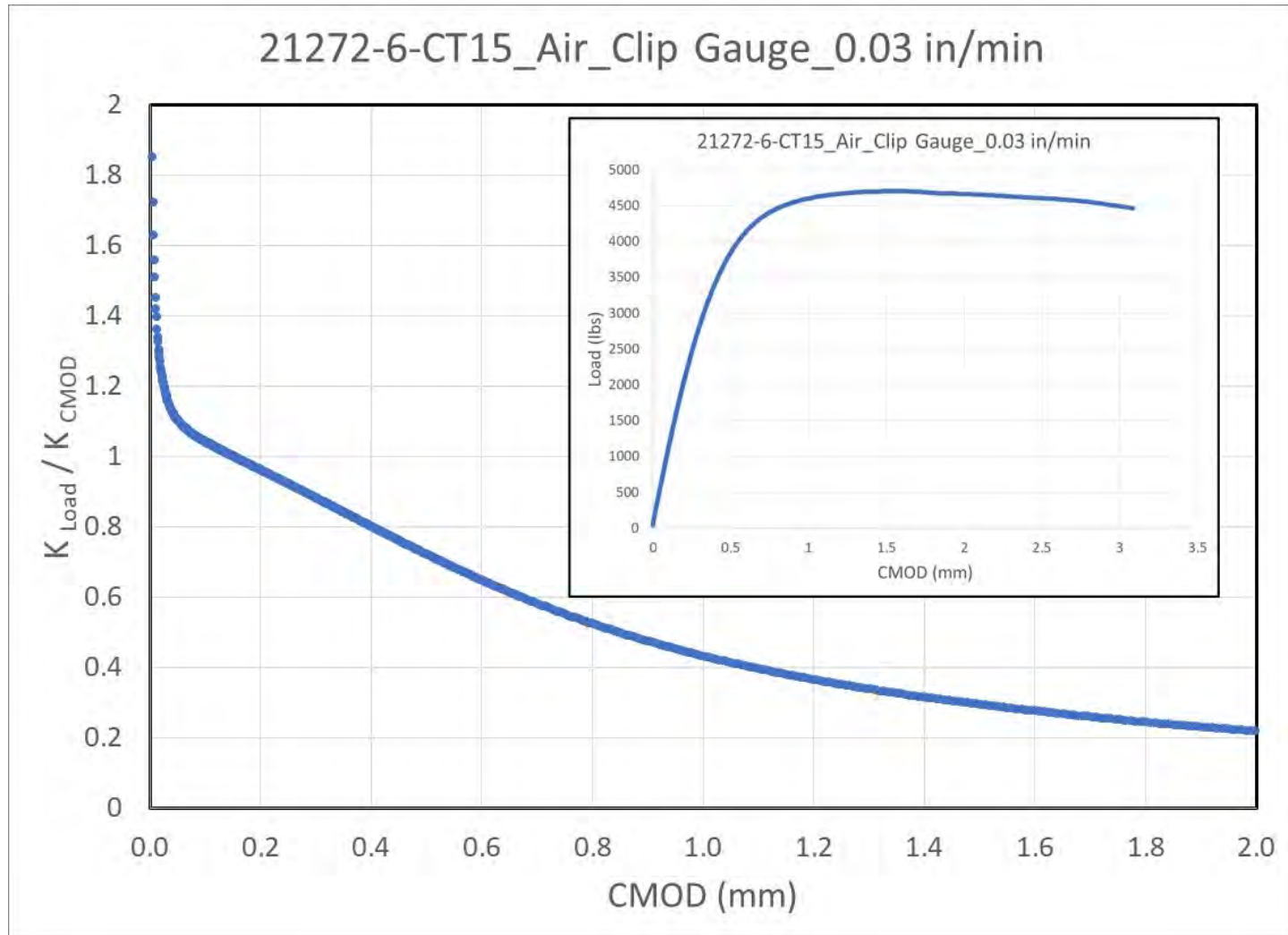
EWI JIP 1/2T CT (CPW)



K from CMOD vs K from Load (EWI)



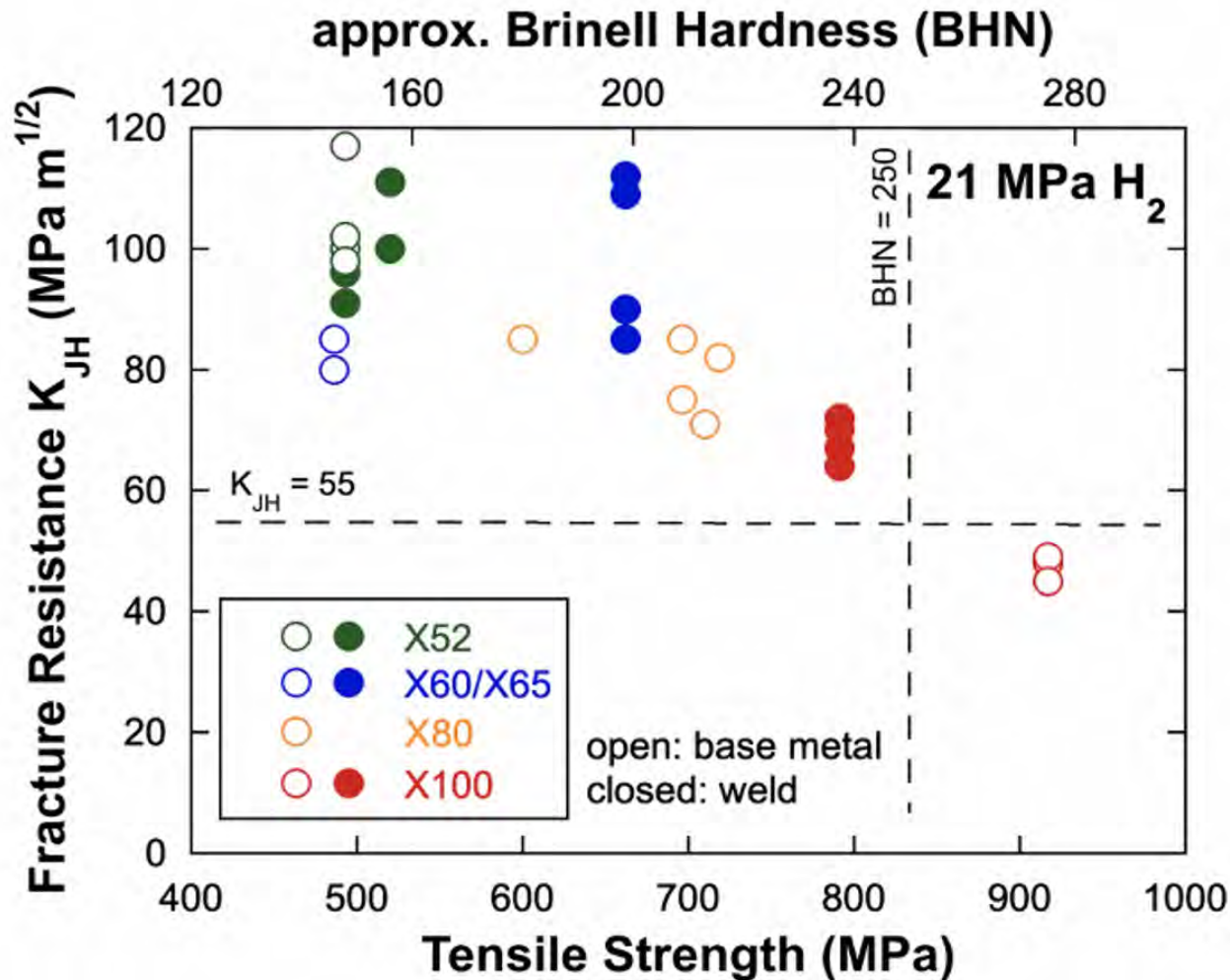
K from CMOD vs K from Load



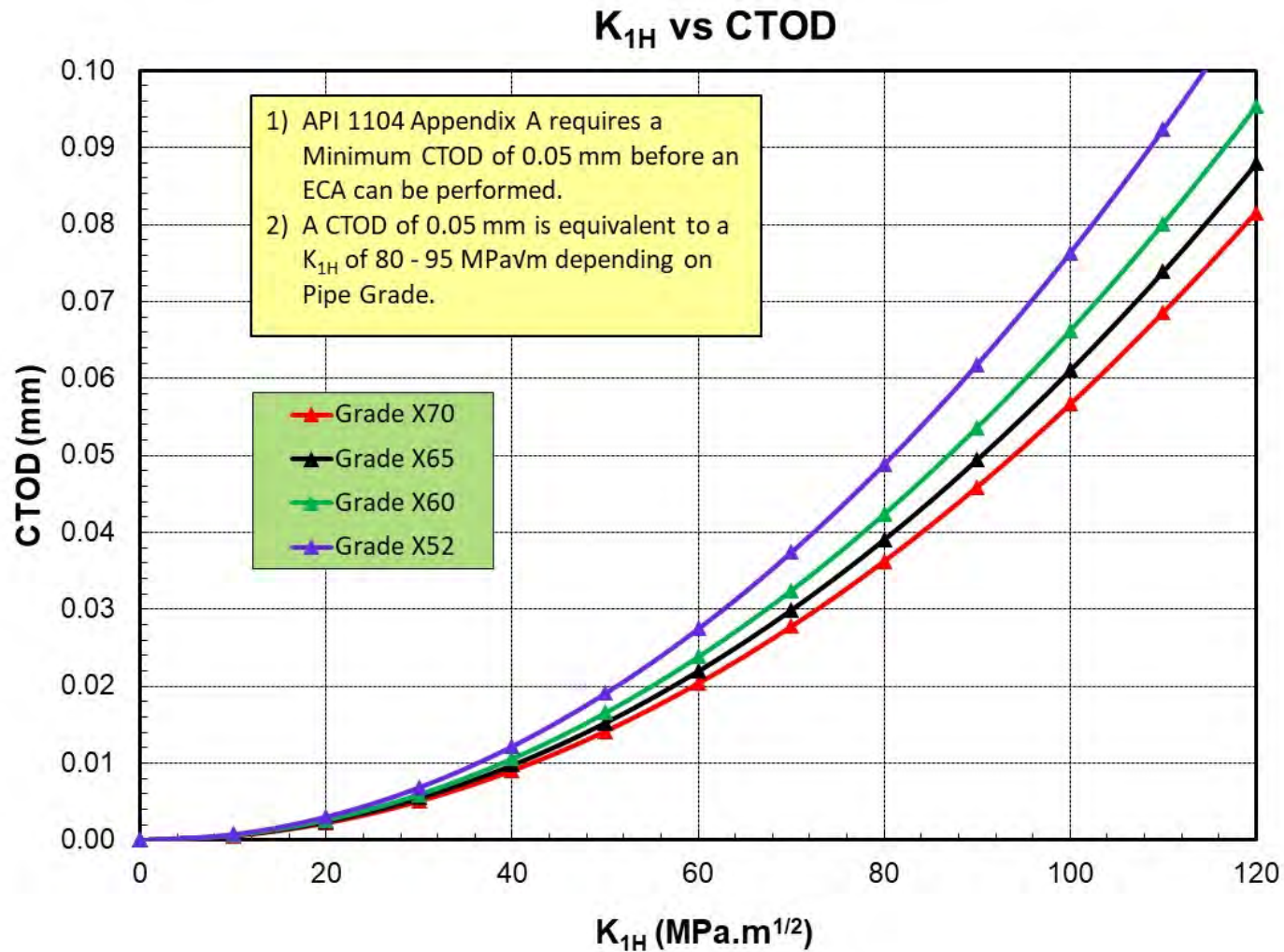
Constant Load & Displacement Fracture Tests

- Constant Displacement or Constant Load Fracture Tests only produce valid K results when the specimen behaves elastically, and the ASTM E1681 Specimen Size requirements are satisfied.
- Loading Specimens of the same size to the same displacement (Constant Displacement Tests) or the same Load (Constant Load Tests) does NOT guarantee a Constant Applied Crack Driving Force.
- The origin of the Reduction Factor of 2 for Constant Displacement Fracture Tests (Bolt Loaded Tests) but not Constant Load Fracture Tests is not clear.
- As a Specimen is Loaded under Displacement Control and held under constant displacement for an extended time the following will occur:
 - The Load will relax due to time dependent plasticity (creep).
 - As the Load relaxes the K at the crack tip will reduce.
 - As the Applied K reduces partial compressive residual stresses will form in the fracture process zone.

HyBlend Project : Slow Rising Load

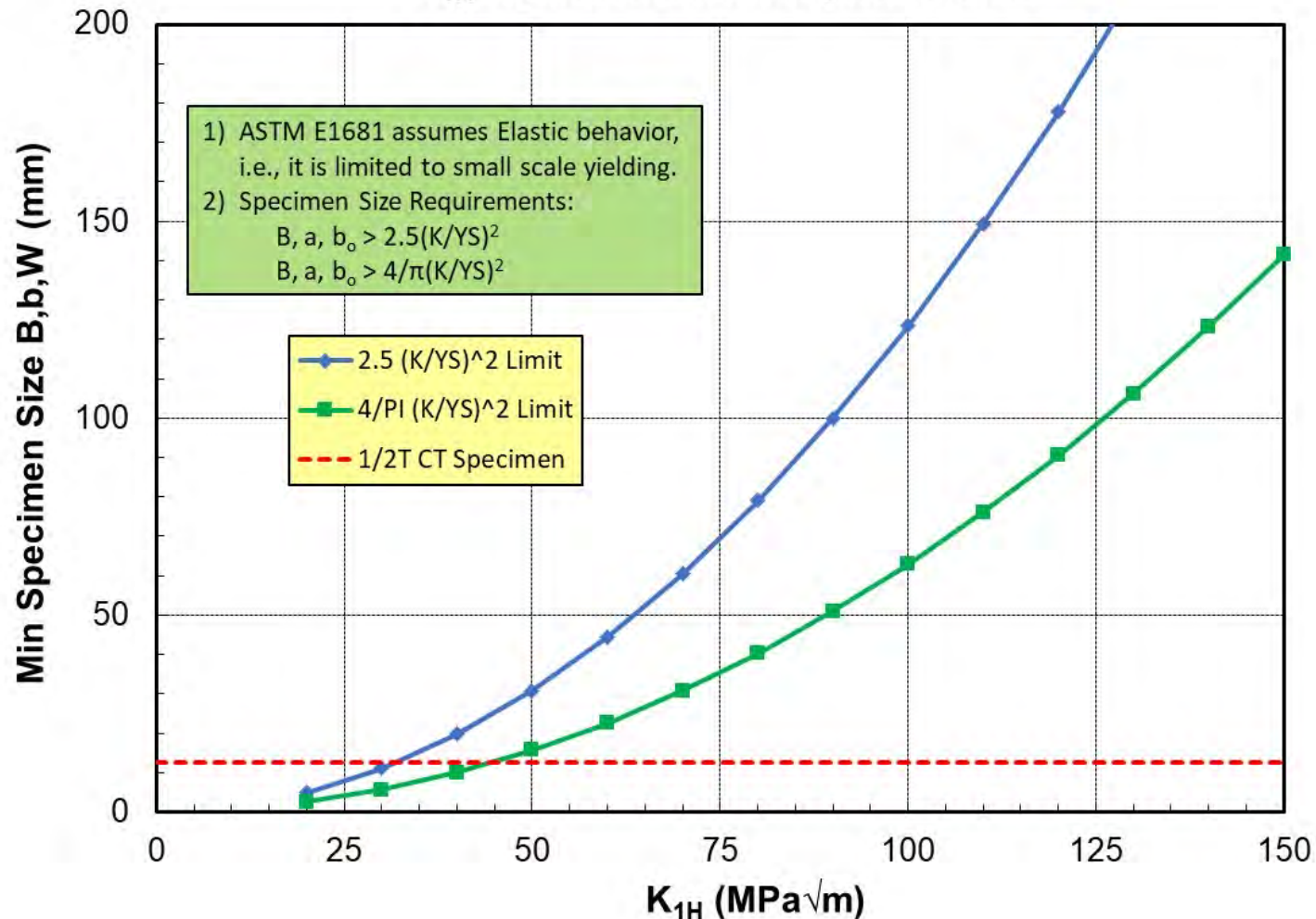


K_{1H} vs CTOD



K_{1H} Specimen Size Requirements

K_{1H} Specimen Size Requirements



ASME B31.12 – 2019 : Summary

- Although the Fracture Toughness Test Procedures recommended in ASME B31.12 may be well suited for Heavy Wall pressure vessel applications they are not appropriate for hydrogen pipelines (Thinner Wall).
- The ASME B31.12 Test Procedures can be used to rank materials and demonstrate hydrogen compatibility, but the values derived from these tests have limited applicability for hydrogen pipelines.
- The ASME B31.12 Fracture Toughness Guidelines need to be updated to reflect Best Practice.

ANSI / CSA CHMC 1-2014

- The Fracture Toughness Test Procedure in ANSI / CSA CHMC 1-2014 is much closer to Best Practice.
 - Slow Rising Load Tests (0.1 – 1.0 MPa√m/minute)
 - Underlying Standard ASTM E1820 (J / CTOD)
 - CT or SENB Specimens
 - Initiation Toughness defined as $J_{0.2/BL}$ or $CTOD_{0.2/BL}$
- ANSI / CSA CHMC 1-2014 also provides excellent guidance on:
 - Hydrogen Gas Purity Requirements
 - Purging Test Cell prior to start of Test
 - Fatigue Crack Growth Testing

ANSI / CSA CHMC 1-2014

- No recommendation on Soaking / Charging Specimens prior to Test.
- For Sour Service Tests soaking specimens is necessary to saturate the specimens with hydrogen and form stable films.
- For Hydrogen Tests soaking is less important since it is generally believed that hydrogen uptake in a hydrogen environment requires applied loading (strain) or creation of fresh material.
 - May impact initiation toughness
- One option would be to preload the specimens in the test cell to a specified K value (e.g., 20 MPa√m) followed by a 24 Hr hold prior to commencing the test.
- Alternatively, the specimens could be pre-conditioned by fatigue pre-cracking in hydrogen.

ANSI / CSA CHMC 1-2014

- The Loading Rates specified in ANSI / CSA CHMC 1-2014 are an order of magnitude faster than typical Loading Rates for Sour Service Tests:
 - 0.1 – 1.0 MPa√m/minute : (ANSI / CSA CHMC 1-2014)
 - 0.01 – 0.1 MPa√m/minute : (Sour Service)
- Reducing the Loading rate will produce a lower J or CTOD R-Curve and may have a small effect on Initiation Toughness.
- There is extensive sour service Test Data to support the Loading Rates used in Sour Service Tests.

ISO 13623 : Draft Amendment

DRAFT AMENDMENT ISO 13623:2017/DAM 1

ISO/TC 67/SC 2

Secretariat: UNI

Voting begins on:
2023-07-06

Voting terminates on:
2023-09-28

Petroleum and natural gas industries — Pipeline transportation systems

AMENDMENT 1: Complementary requirements for the transportation of fluids containing carbon dioxide or hydrogen

ICS: 75.200

H.5.1 Hoop stress basic design factors

The hoop-stress design factor f_h to be used in Formula (3) shall be determined in accordance with Formula (H.1):

$$f_h = f_{hH2} \times H_f \quad (\text{H.1})$$

where:

— f_{hH2} is the base hoop design factor for the transportation of fluids with hydrogen content exceeding the limit specified in H.2

— H_f is the performance factor depending on MAOP and steel grade.

f_{hH2} shall be:

— 0,55 for on-land and offshore pipelines

and, if Annex B is applicable:

— 0,55 for pipelines in location classes 1 to 4

— 0,45 for pipelines in location class 5,

H_f shall be obtained from Table H.1.

Table H.1 — Performance factors, H_f

Line pipe steel grade	MAOP [MPa] ^{a)}						
	6,89	13,79	15,17	16,55	17,93	19,31	20,68
L360	1,000	1,000	0,954	0,910	0,880	0,840	0,780
L415	0,874	0,874	0,834	0,796	0,770	0,734	0,682
L450	0,832	0,832	0,794	0,758	0,733	0,699	0,649
L485	0,776	0,776	0,742	0,706	0,684	0,652	0,606

a) Interpolation may be used to calculate H_f for intermediate values of MAOP.

1) X70 Pipeline : 1,440 psi:

2) DF = 0.55 x 0.832 = 0.46

Summary

- Fracture Toughness Tests performed per ASME B31.12 should be limited to ranking materials and assessing hydrogen compatibility.
- The Fracture Toughness Test Procedure in ANSI / CSA CHMC 1-2014 is much closer to Best Practice than ASME B31.12.
- Areas for Further Work / Discussion
 - Loading Rate Effects
 - Soaking / Charging Specimens prior to Test
 - This may impact initiation toughness since tests performed on samples that are not pre-conditioned may reach initiation before hydrogen charging has achieved equilibrium.
 - Coating Specimens to simulate single sided exposure
 - Correlation between Rising Load Fracture Results and Threshold Toughness for Sustained Load Cracking. This would provide a basis for specifying a rising load toughness for ECA.

Back-Up Slides

Hydrogen Embrittlement : Fracture Toughness

- Although the load displacement curves obtained from fracture toughness tests performed on hydrogen charged specimens will typically look fully ductile the fracture face will commonly have a quasi-cleavage morphology.
- The fracture mechanism involves small steps of subcritical crack growth, through the hydrogen-charged plastic zone at the crack tip, which advances with the growing crack.
- Following a step of crack growth, hydrogen will begin to accumulate in the new plastic zone until there is a sufficient hydrogen concentration to cause a further subcritical crack growth step.
- This explains the effect of strain rate on measured fracture toughness.

Sour Service Test Methods

- Hydrogen Charged Tests
 - Specimens charged in environment but Tested In-Air
 - Hydrogen is not replenished during Test
 - Hydrogen Diffuses out of Specimen as Test Progresses
 - Need to Test at Relatively High Loading Rates (< 1 day)
 - Simple, Low-Cost Test Procedure
 - Does not require sophisticated Test Cell
 - Does NOT produce Lower Bound Toughness Properties

Sour Service Test Methods

In-Situ Tests

- Specimens exposed to Test Environment throughout Test.
- Tests can be performed at Slow Loading Rates (7-10 Days)
- For H_2S Partial Pressure < Atmospheric Pressure the Tests can be performed under Atmospheric Pressure.
- For H_2S Partial Pressure > Atmospheric Pressure the Tests are performed in an Autoclave with Fugacity Corrected Pressure.
- In Situ Tests require specialized Test Cells
- In Situ Tests produce Lower Bound Toughness Properties

Sour Service Test Methods

K_{1SSC} Tests

- The K_{1SSC} Test is considered to be the most definitive Test Method since it defines the limiting condition under which sustained Load Cracking will occur.
- Takes 30 Days to perform and is a Go / No-Go Test requiring an Iterative Test Matrix.

Slow Rising Load Tests

- Test Durations typically 7 – 10 Days
- Tests can be performed in parallel
- Permits measurement of Different Toughness Parameters
 - J at Initiation (J_i)
 - J at 0.2 mm of Stable Crack Growth ($J_{0.2}$)
 - J at Max Load (J_{max})
- Reasonable correlation between K based on $J_{0.2}$ and K_{1SSC} .

ASTM E1681

- ASTM E1681 assumes Elastic behavior, i.e., it is limited to small scale yielding.
- Specimen Size Requirements:
 - $B, a, b_o > 2.5(K/Y_S)^2$
 - $B, a, b_o > 4/\pi(K/Y_S)^2$
- The K equations in ASTM E1681 are NOT valid if the sample is loaded well beyond the Yield Load.
- The K equation to determine K from CMOD was originally developed for heavy wall pressure vessels in Hydrogen Service.



Exceptional service in the national interest

Advanced practices for fatigue testing in gaseous hydrogen

Chris San Marchi

Study Group on Materials Testing and Qualification for Hydrogen Service

Atlanta, GA

July 21st, 2023

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.





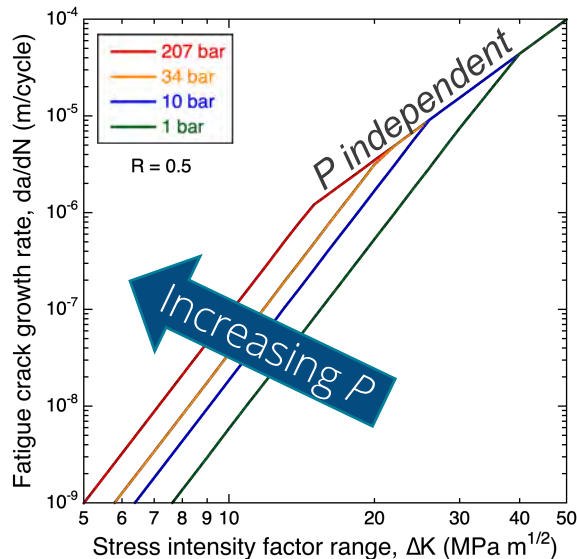
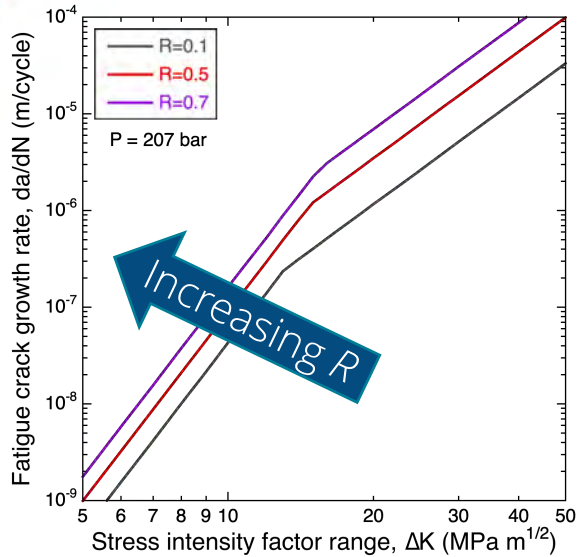
ANSI/CSA CHMC1 – Section 5.6 Fatigue crack growth rate (FCGR)

- Method should be consistent with ASTM E647
 - Must use constant force amplitude method
 - K-decreasing methods can be used for $da/dN < 10^{-8}$ m/cycle
 - Test parameters:
 - $R = 0.1$
 - Frequency = 1 Hz
 - Waveform can be either triangle wave or sine wave
 - Recommended range of $\Delta K = 8$ to 20 MPa $m^{1/2}$
 - No mention of soak time

***Editorial note:* Other load control methods, other R, and other frequencies have been demonstrated to improve test efficiency and access to broader FCGR ranges**



Master Design Curves (MDCs) were developed to bound fatigue crack growth behavior of steels



The effects of pressure and load ratio on fatigue crack growth are captured in conventional power law formulation :

- At low ΔK

$$\frac{da}{dN} = 3.5 \times 10^{-14} \left[\frac{1+0.4286R}{1-R} \right] \Delta K^{6.5} g(P)$$

- At high ΔK

$$\frac{da}{dN} = 1.5 \times 10^{-11} \left[\frac{1+2R}{1-R} \right] \Delta K^{3.66}$$

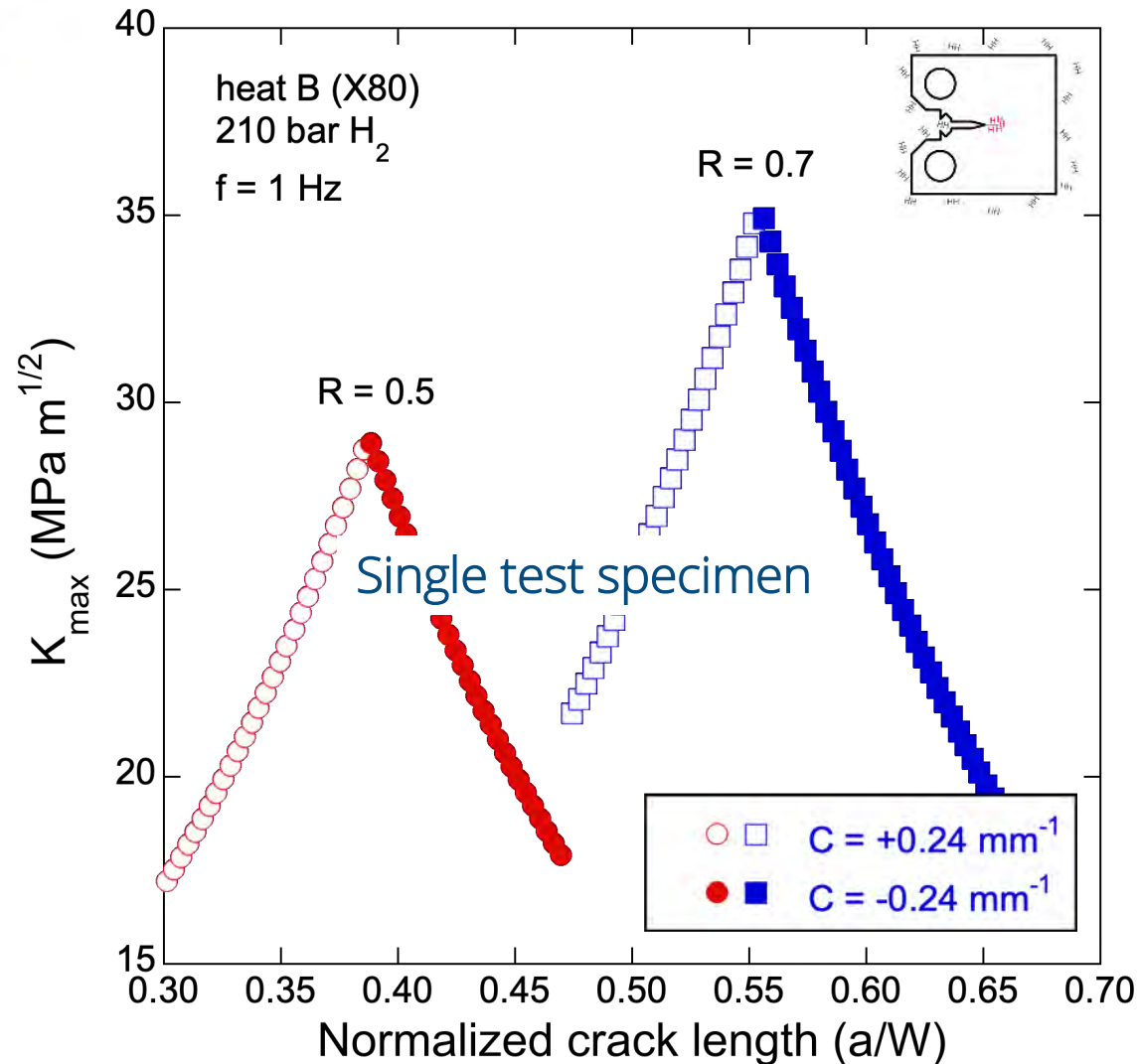
These Master Design Curves appear to be effective for a wide range of construction steels

Ref.: San Marchi et al., PVP2019-93907

Outcome:

- Master Design Curves provide a simple framework to bound the fatigue crack growth of steels in gaseous H_2

K-control fatigue crack growth tests enable efficiency



ASTM E647 fatigue crack growth methods using compact tension geometry

- C control, both K-increasing (C+) and K-decreasing (C-)
- Constant C test segments

$$C = \left(\frac{1}{K} \right) \left(\frac{dK}{da} \right)$$



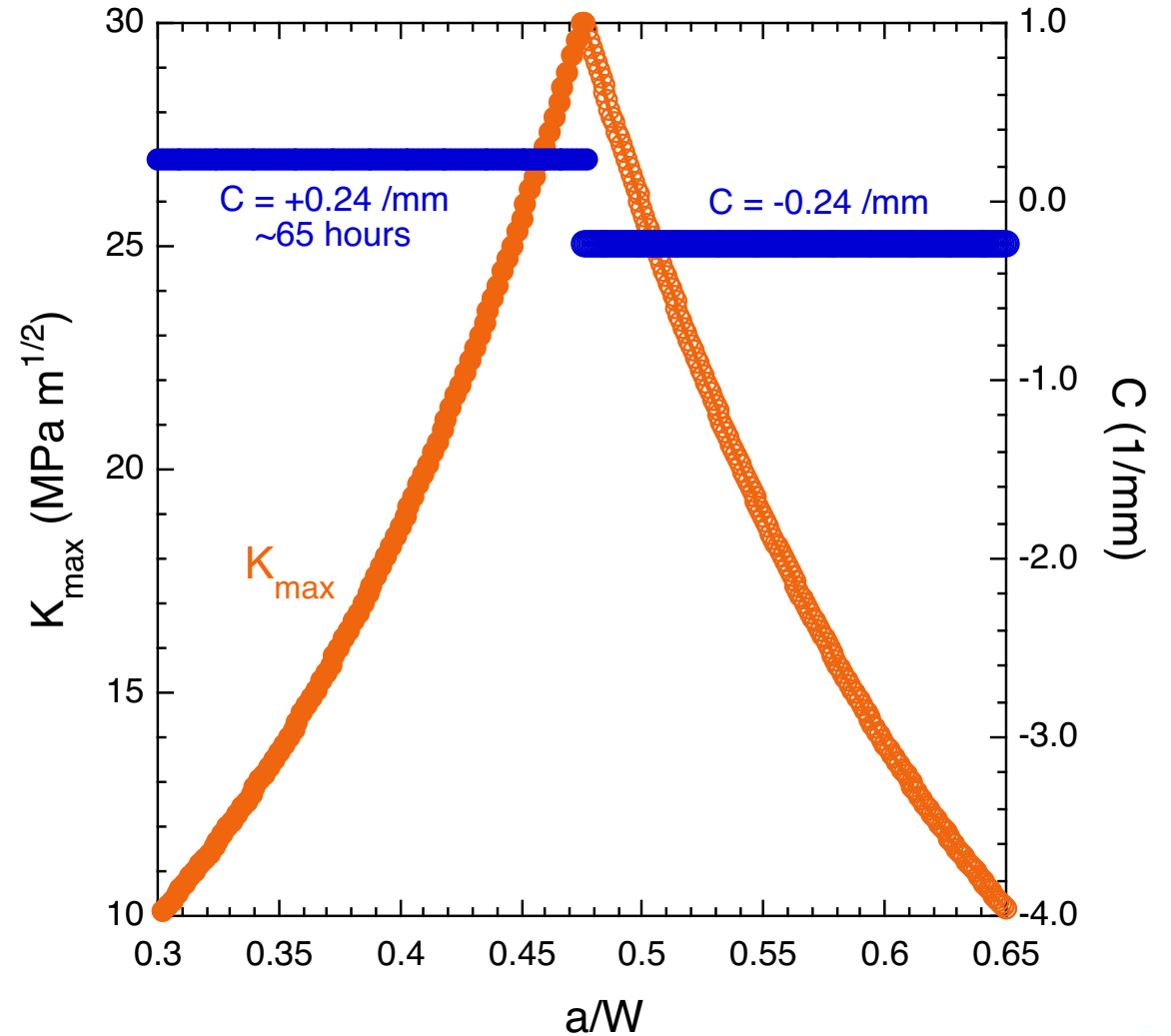
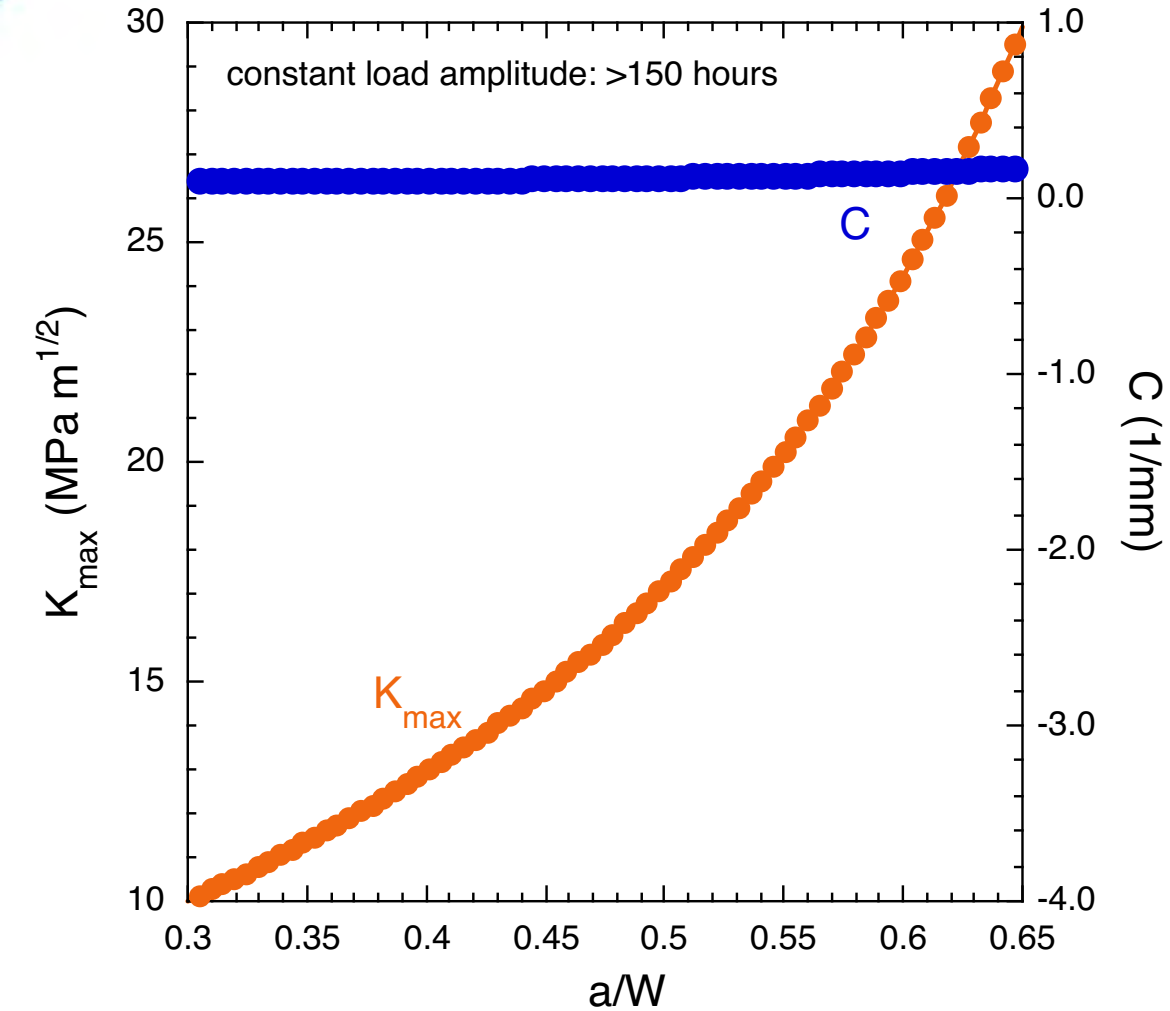
K-control fatigue crack growth tests enable efficiency

Normalize C by term proportional to plastic zone size and keep constant to accelerate K-decreasing fatigue crack growth tests

$$C_{norm} = C \left(\frac{K}{S_y} \right)^2 \sim \text{constant}$$

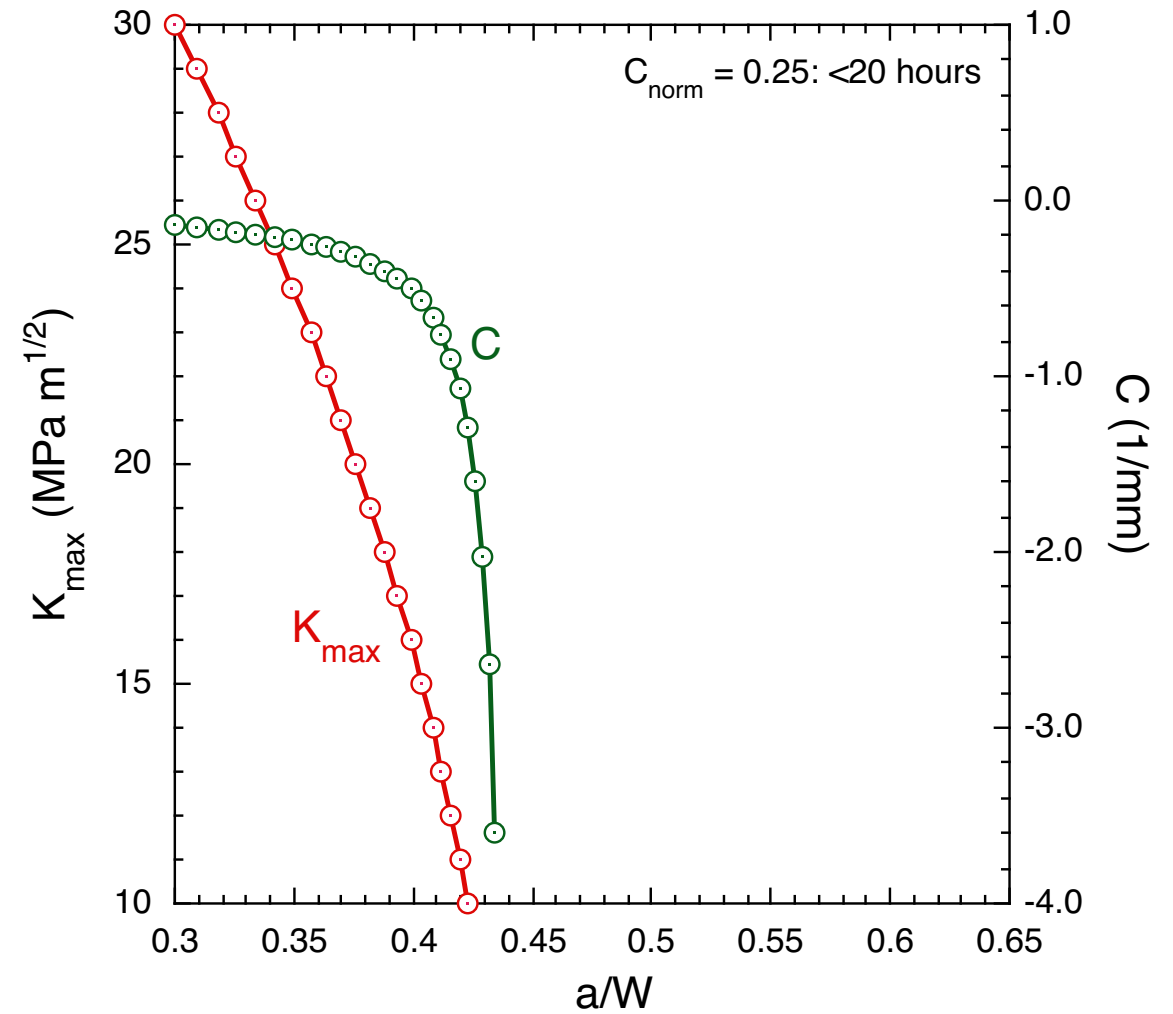
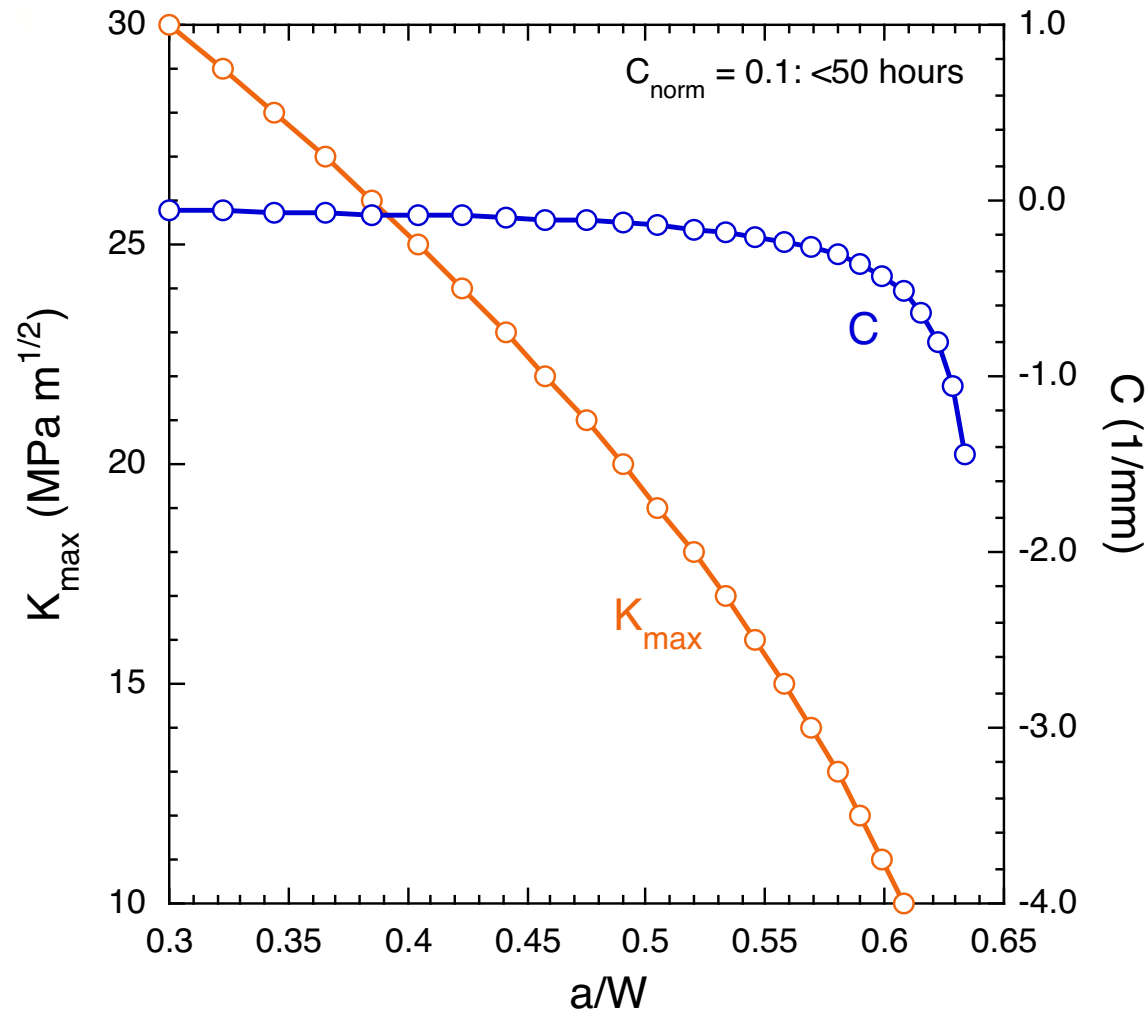
$K_{max} = 10 - 30 \text{ MPa m}^{1/2}$	
control	time
Constant load amplitude	154 hours
$C = +0.24$	~65
$C_{norm} = 0.1$	<50 hours
$C_{norm} = 0.25$	<20 hours

K-control fatigue crack growth tests enable efficiency





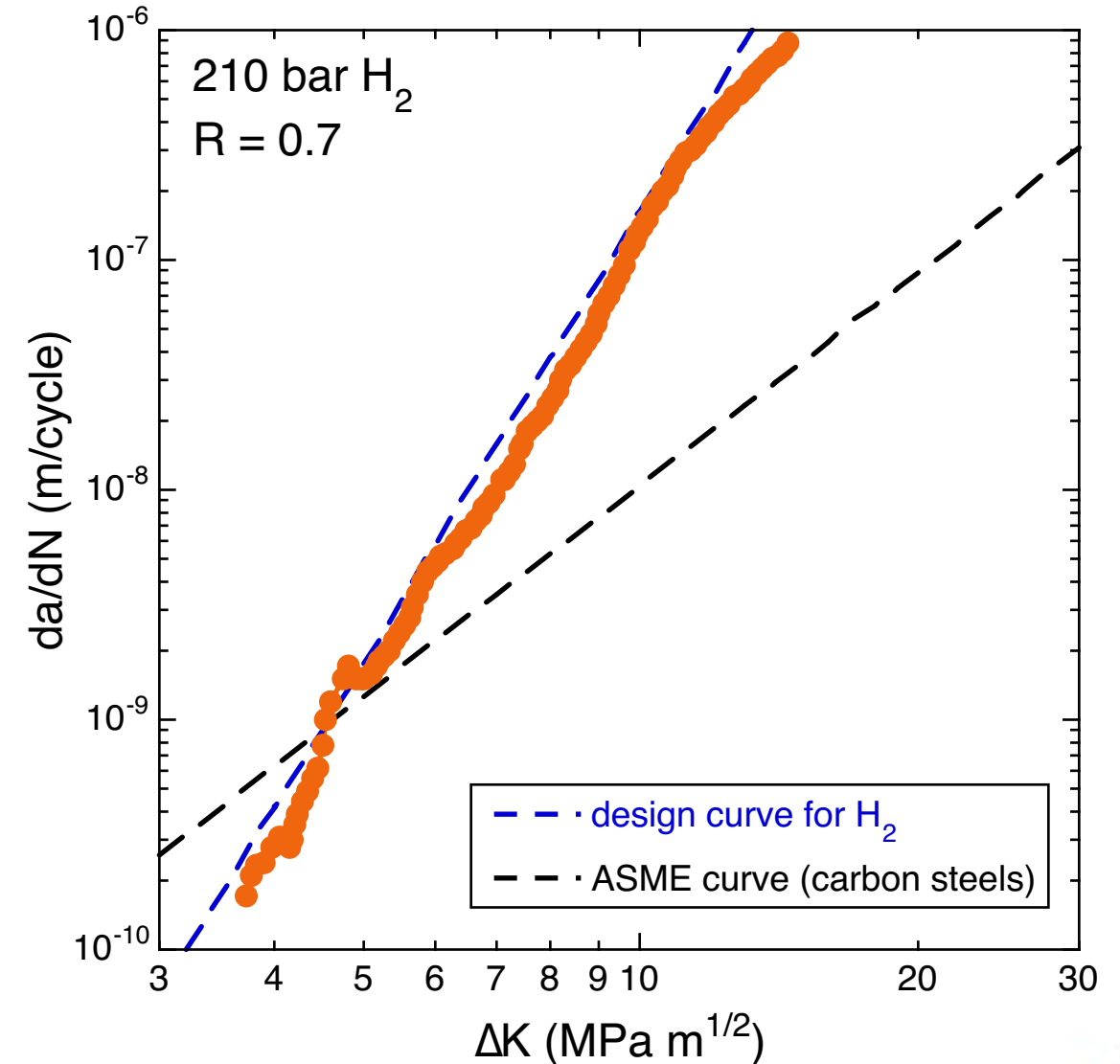
K-control fatigue crack growth tests enable efficiency





Demonstration of C_{norm}

R	C	ΔK (MPa m ^{1/2})
0.7	-0.24	15-5
0.7	-0.48	5-4
0.7	-0.72	<4





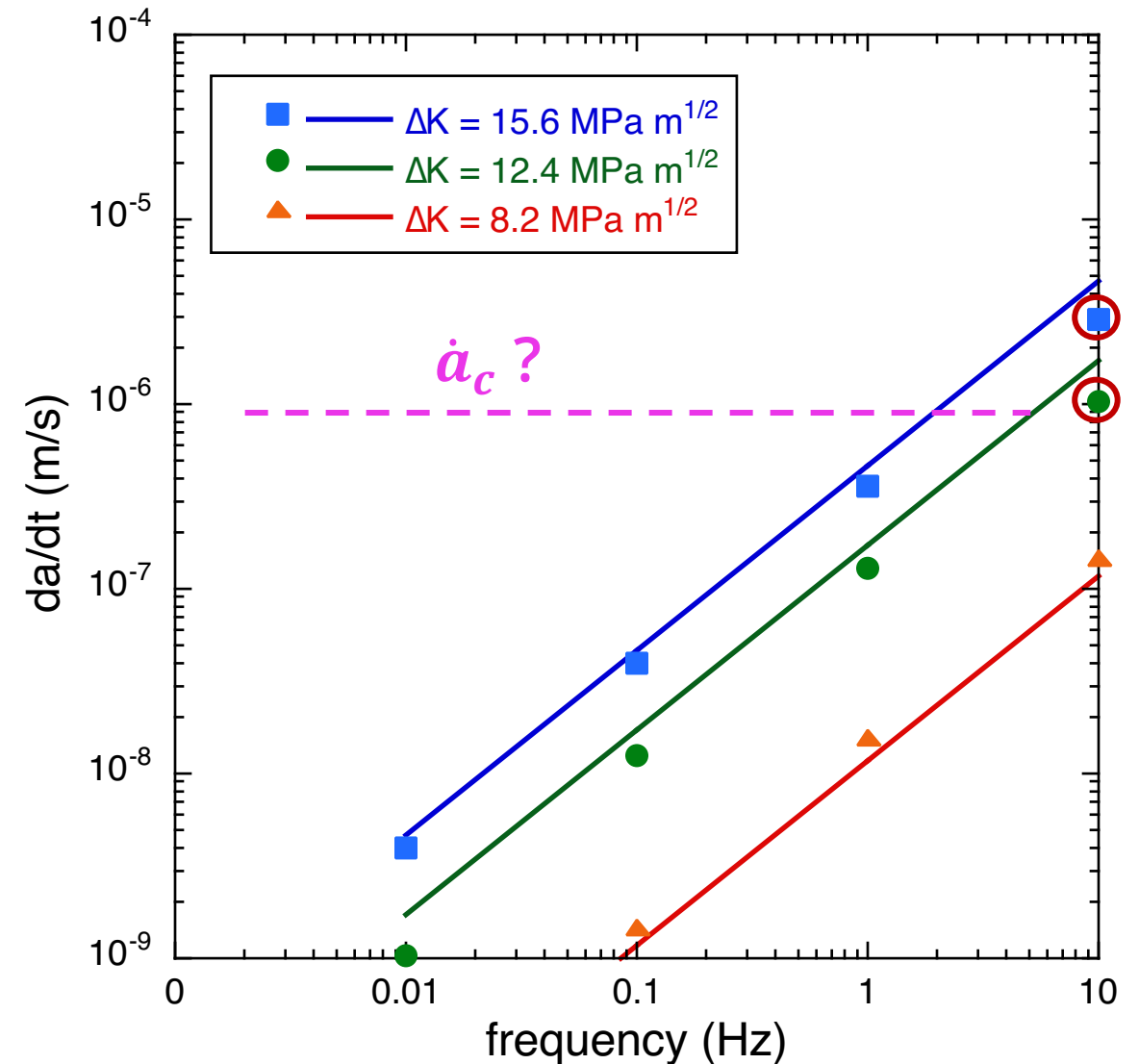
Frequency can be used to further accelerate testing

$$\frac{dx}{dt} = \frac{d}{dt}(\sqrt{4Dt}) \quad \text{Rate of hydrogen transport}$$

$$\frac{da}{dt} = f \frac{da}{dN} \quad \text{Rate of crack growth}$$

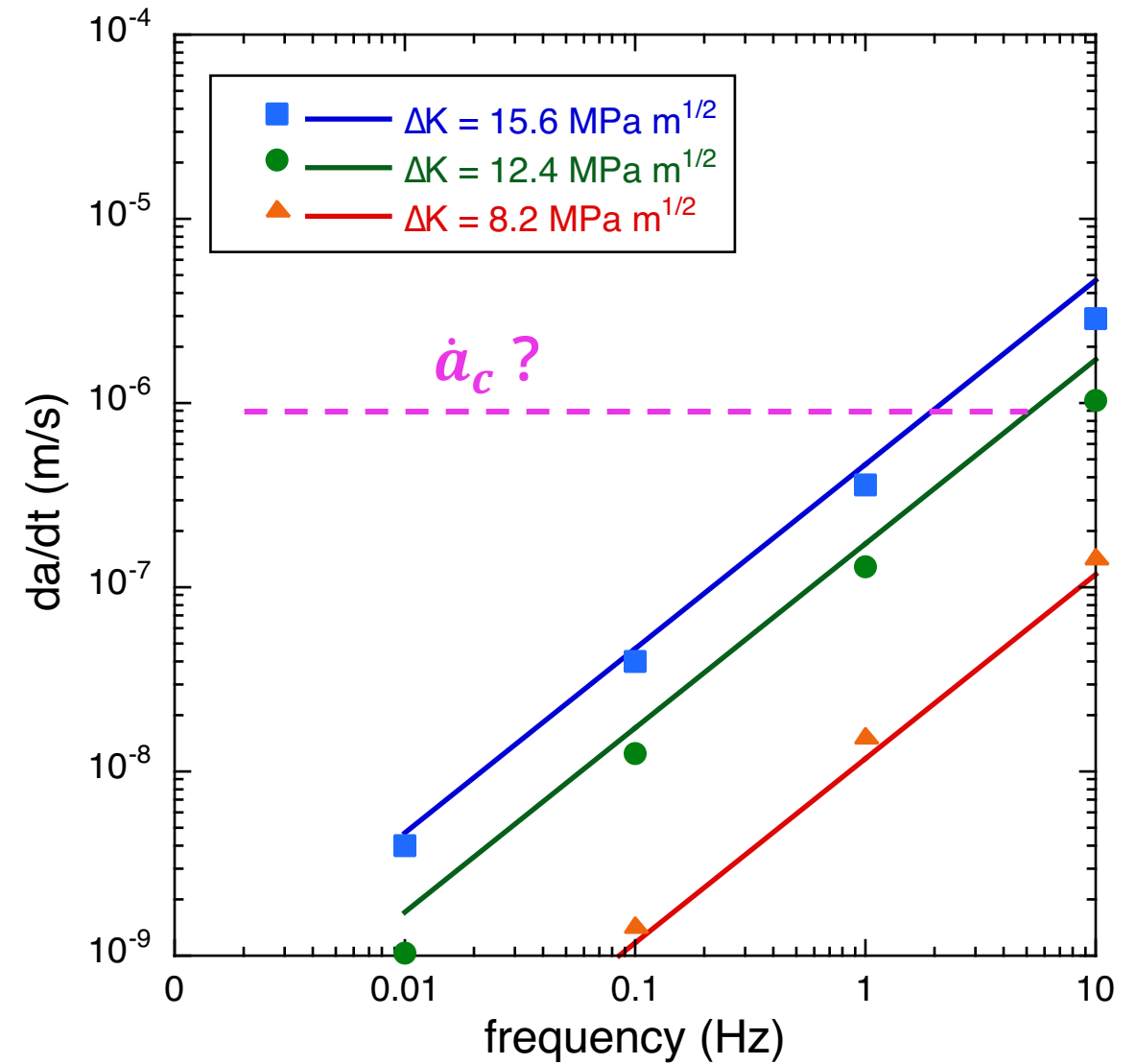
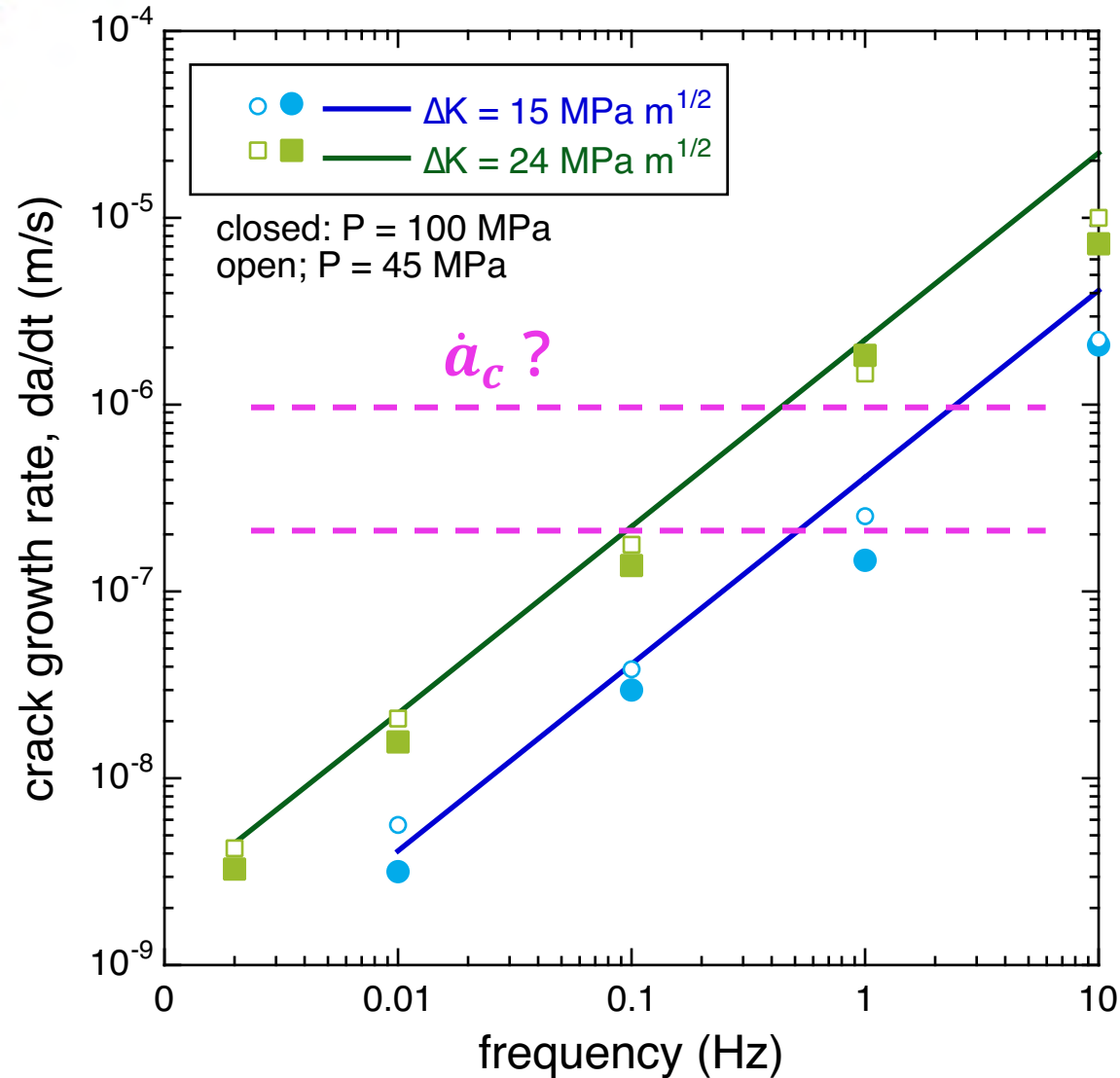
$$f = 1 \text{ Hz} \quad da/dN < \dot{a}_c$$

$$f = 10 \text{ Hz} \quad da/dN < \dot{a}_c / 10$$

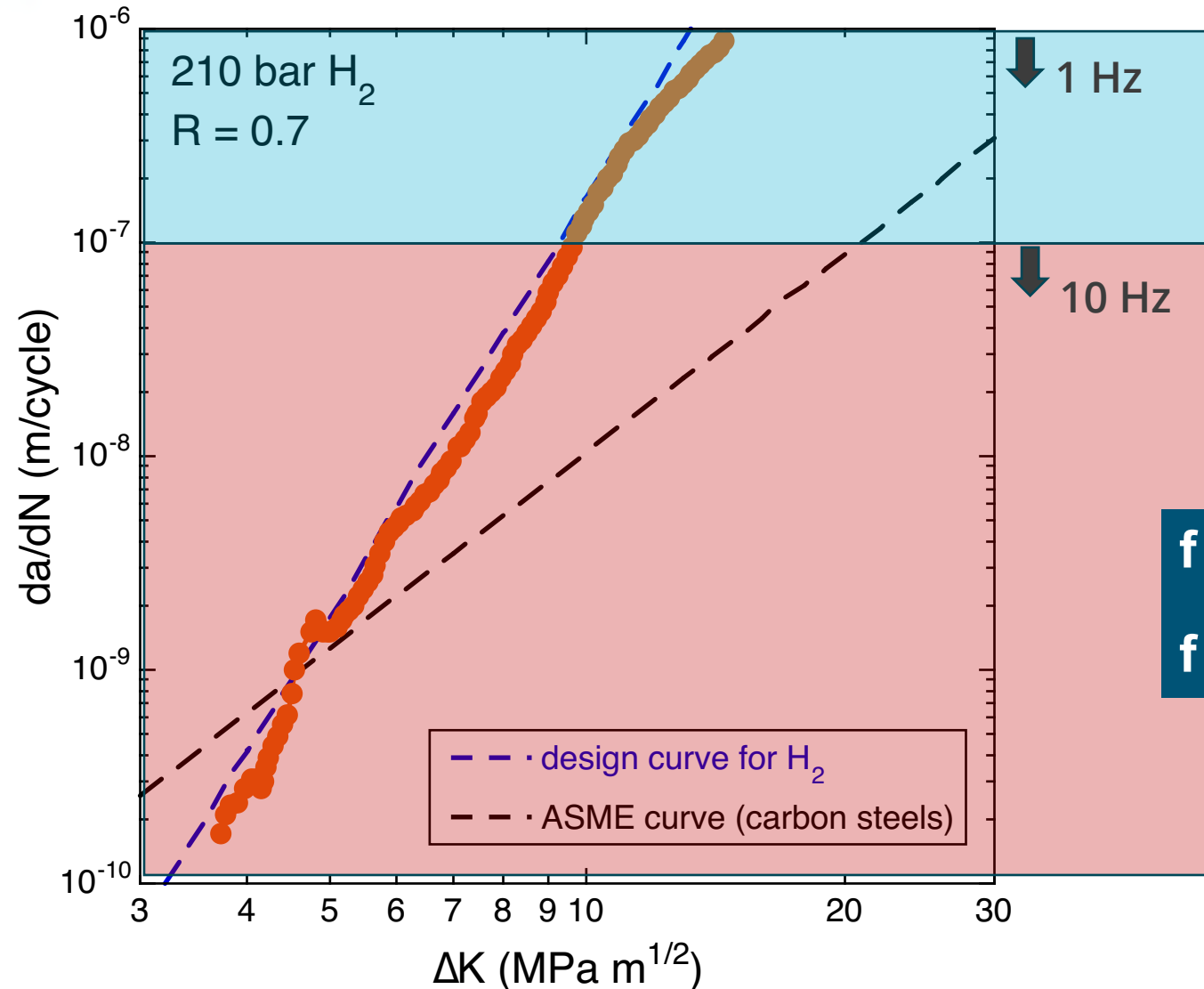




Frequency can be used to further accelerate testing



Frequency can be used to further accelerate testing

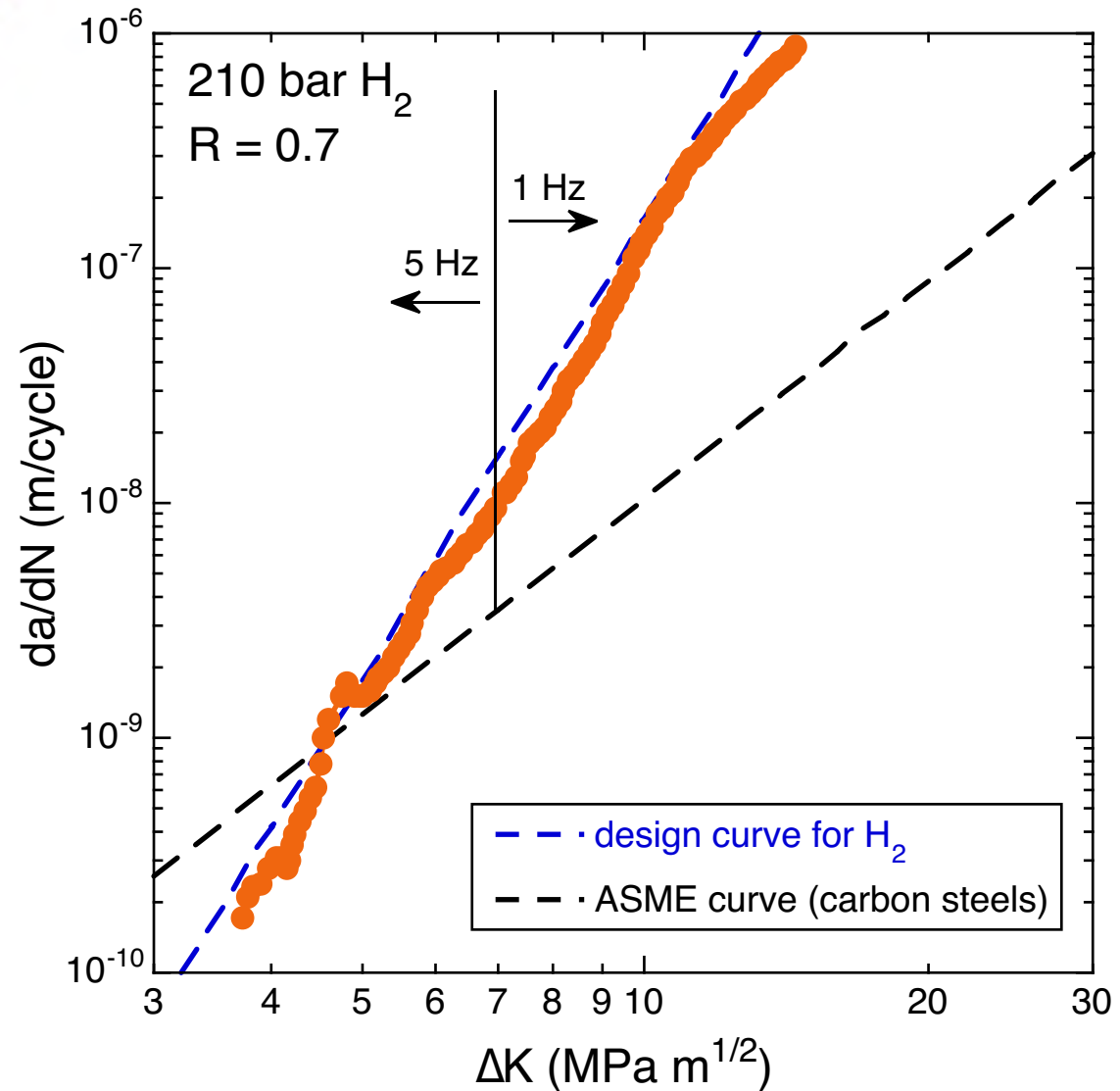


$$\dot{a}_c \sim 10^{-6} \text{ m/s}$$

$$f = 1 \text{ Hz} \quad da/dN < 10^{-6} \text{ m/cycle}$$

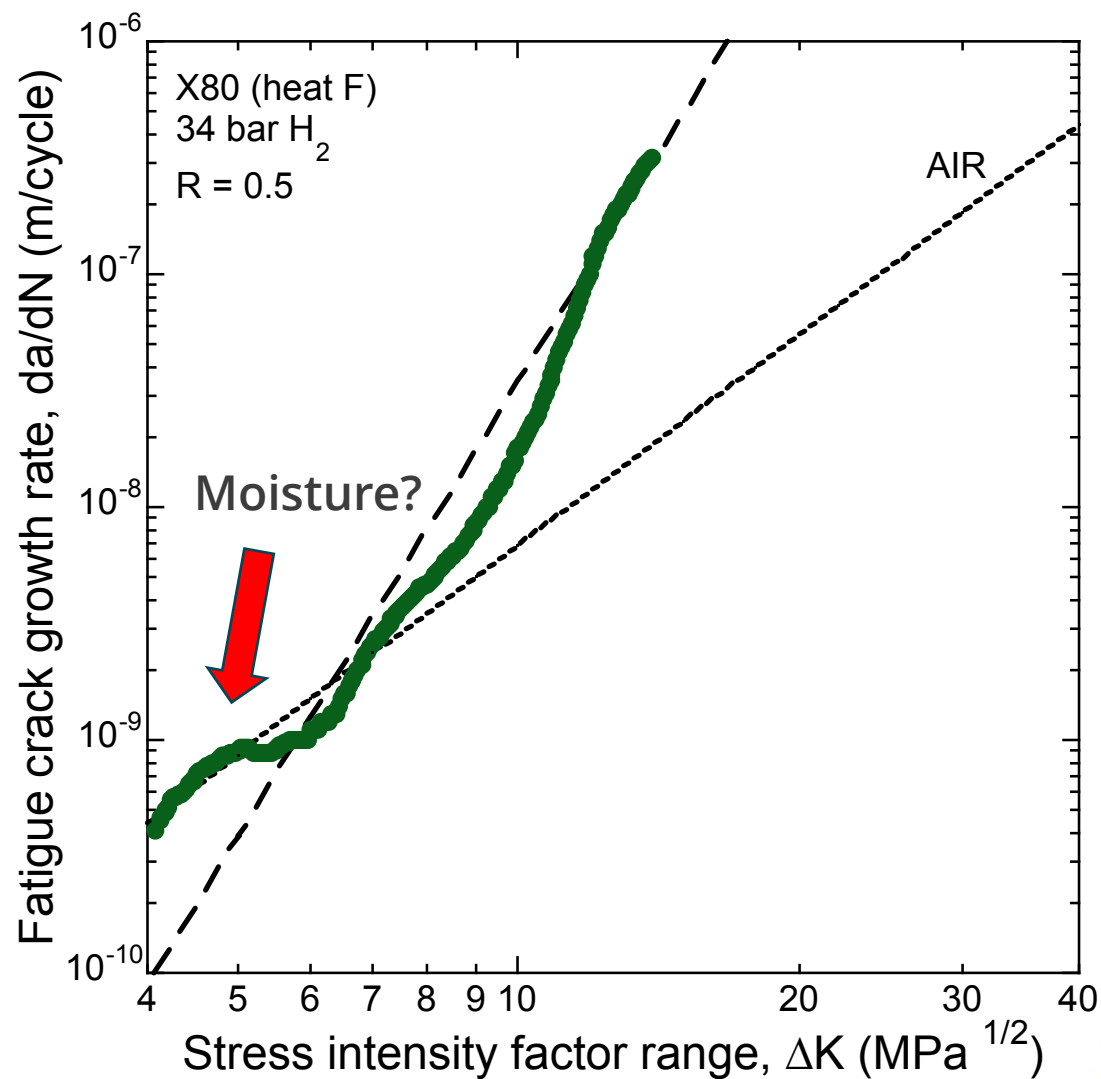
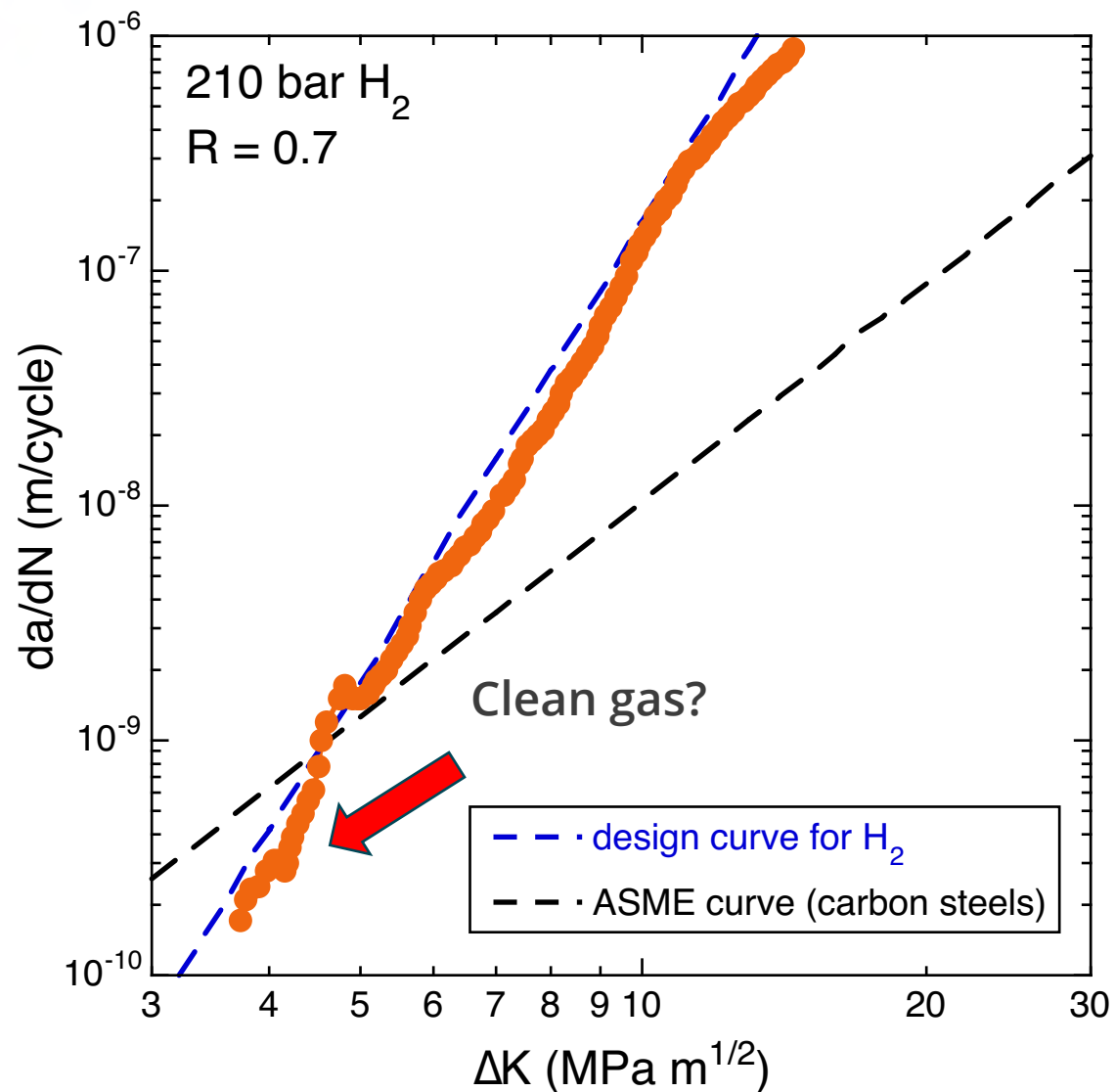
$$f = 10 \text{ Hz} \quad da/dN < 10^{-7} \text{ m/cycle}$$

Demonstration of C_{norm} and frequency





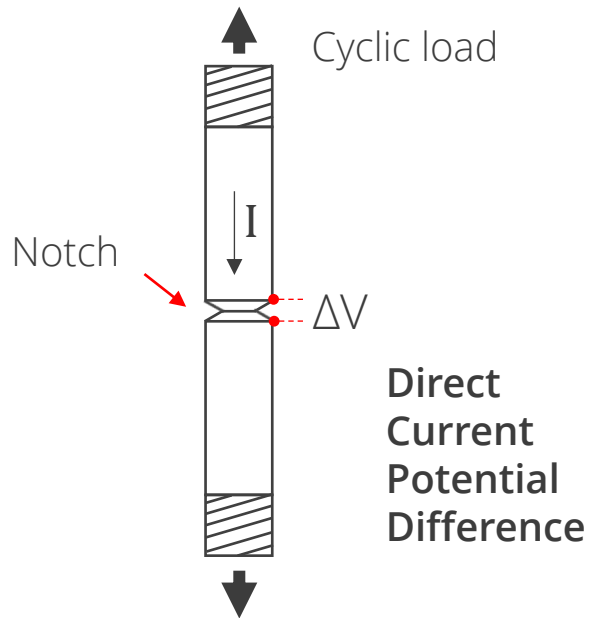
Role of impurities can be subtle



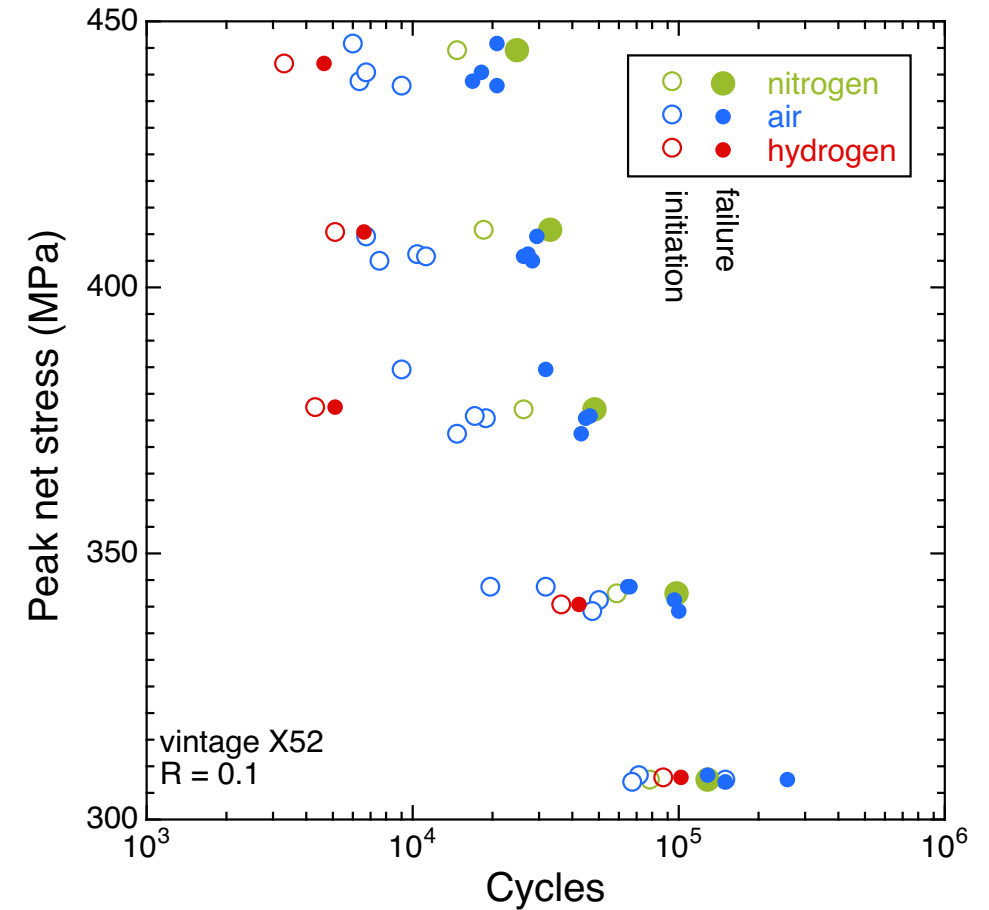
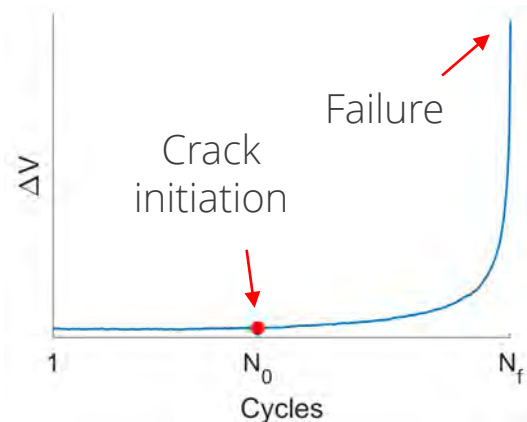


Effect of H pressure on crack initiation of X52 vintage steel

Circumferential notch tension (CNT) specimens



- N_2 (500 psi)
Baseline
- Air
Effects of O_2
- H_2 (3 ksi)
High partial pressure



Results:

- Air accelerates crack initiation,
- H_2 at high pressure accelerates BOTH crack initiation and growth.
- CNT tests can complement fracture mechanics approaches.



Closing thoughts and summary of HEML experience

- Fatigue crack growth in K-control (both K-increasing and K-decreasing) allows
 - Spanning ΔK ranges will less Δa
 - Multiple R with a single specimen
- Constant C_{norm} in K-decreasing enables further test efficiency
- High frequency (>1 Hz) can be used at low $da/dN \rightarrow$ efficiency near threshold
 - Critical $da/dt \rightarrow 10^{-6}$ m/s
- Impurities !?!
 - Special consideration for impurities needed near convergence with air da/dN
 - Otherwise ANSI/CSA guidance on gas quality recommended
- Fatigue life (S-N curves) affected at stress
- Convergence with air near fatigue limit



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<https://h-mat.org/>

<https://www.sandia.gov/matlsTechRef/>

<https://granta-mi.sandia.gov/>

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James McNair
Keri McArthur
Tanner McDonnell
Nalini Menon

And the many present and former
contributors, collaborators and friends



Exceptional service in the national interest

Hydrogen-assisted fatigue and fracture test results from HyBlend

Milan Agnani*, Joseph Ronevich, Chris San Marchi

ASME PVP 2023, Atlanta, GA

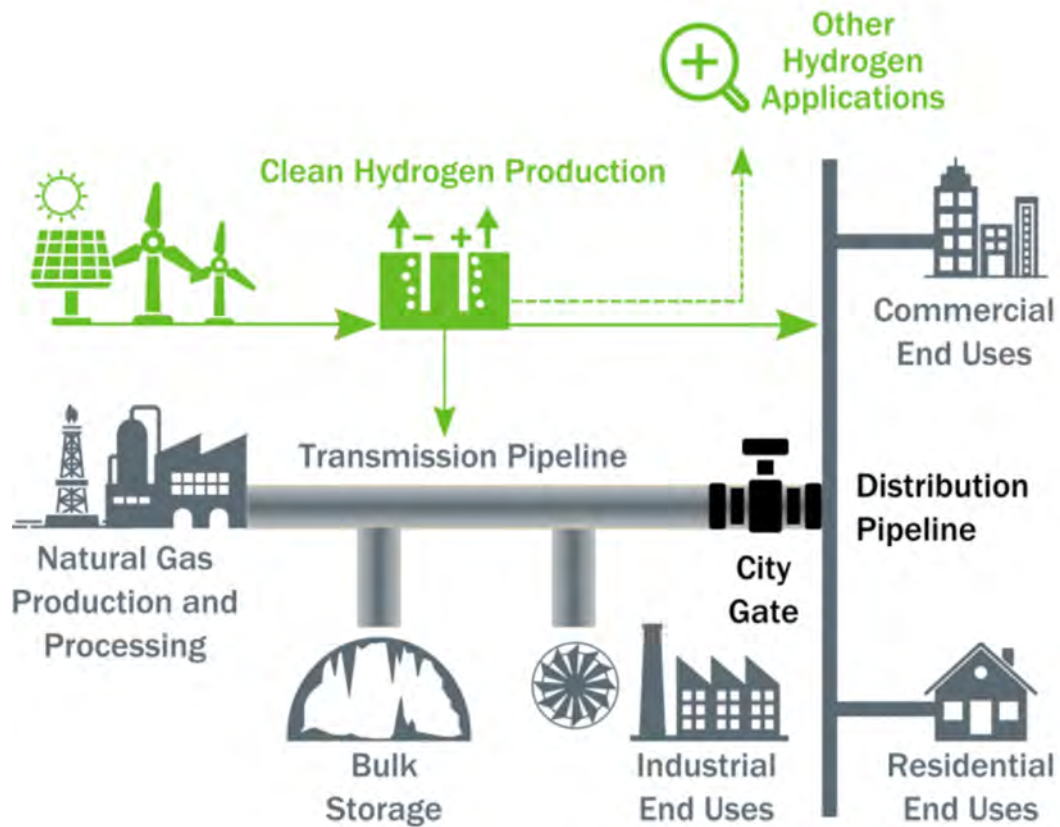
July 21st, 2023

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Overview – HyBlend™

Re-purposing Existing NG Pipeline Network for Transporting Blends of NG and H₂



Materials

- 15 + Vintage Steel (X52 and lower)
- 10 + Modern Steels (X52/65/70/80)
- 4 Vintage Seam Welds

Test Conditions

- GH2 Pressure
- Fatigue R-ratios and Frequency
- Fracture Displacement Rate



Outline

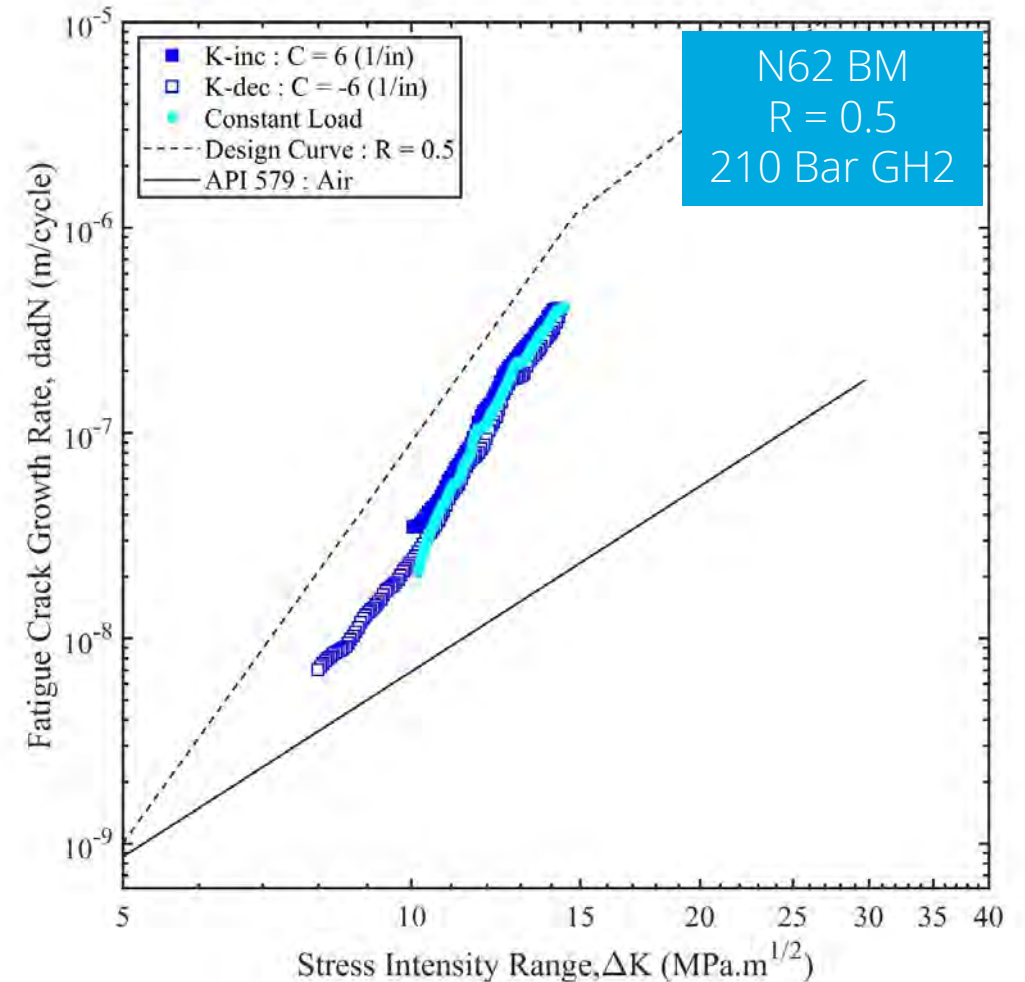
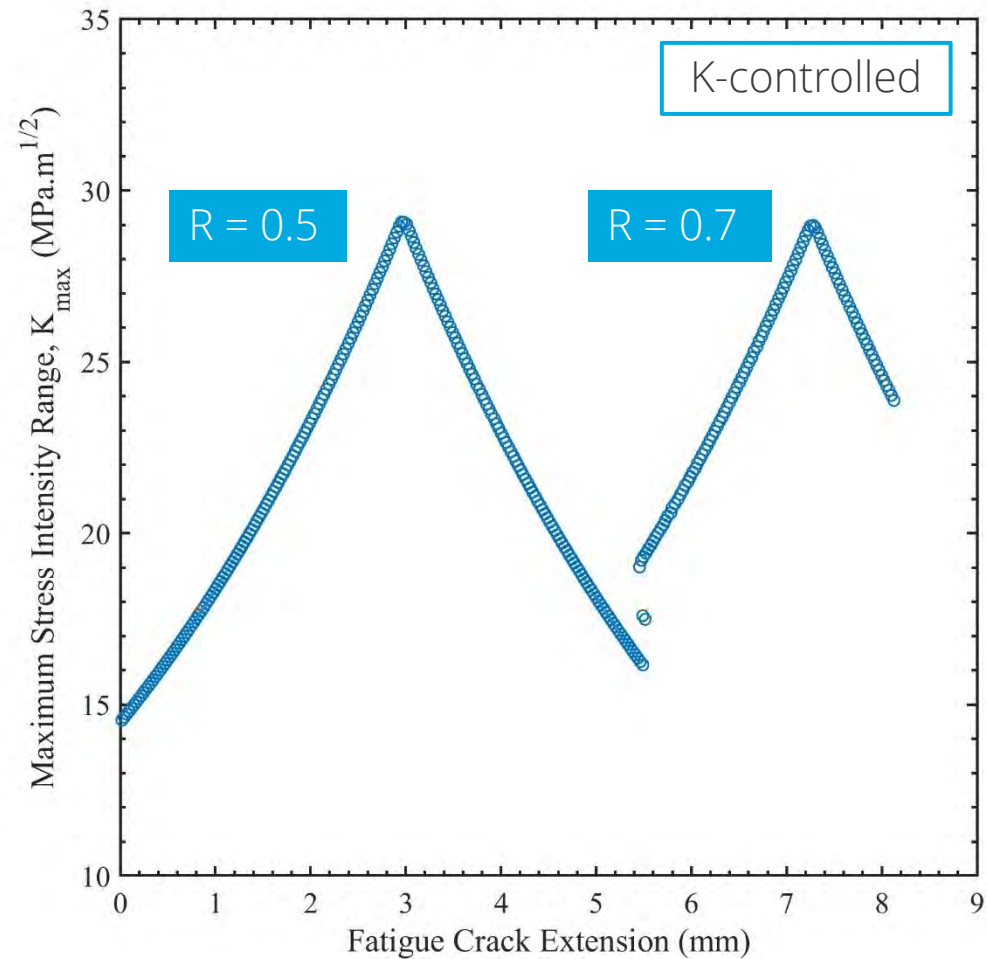
- Hydrogen Assisted Fatigue Crack Propagation (HA-FCG)
 - Test Methodology
 - Loading History
 - Microstructure Effects
 - H₂ Gas Pressure
 - Low ΔK Regime
- Hydrogen Assisted Fracture
 - Test Method and Materials Response
 - J-R Curves
 - Trends in Fracture Resistance
 - Microstructural Heterogeneities
 - Inclusion Content

The logo features a central dark blue diamond with the text 'HA-FCG' in white. This diamond is surrounded by a light gray diamond border. Two diagonal lines, each composed of several colored segments (cyan, orange, green, red, purple, and light blue), cross the background behind the central diamond.

HA-FCG



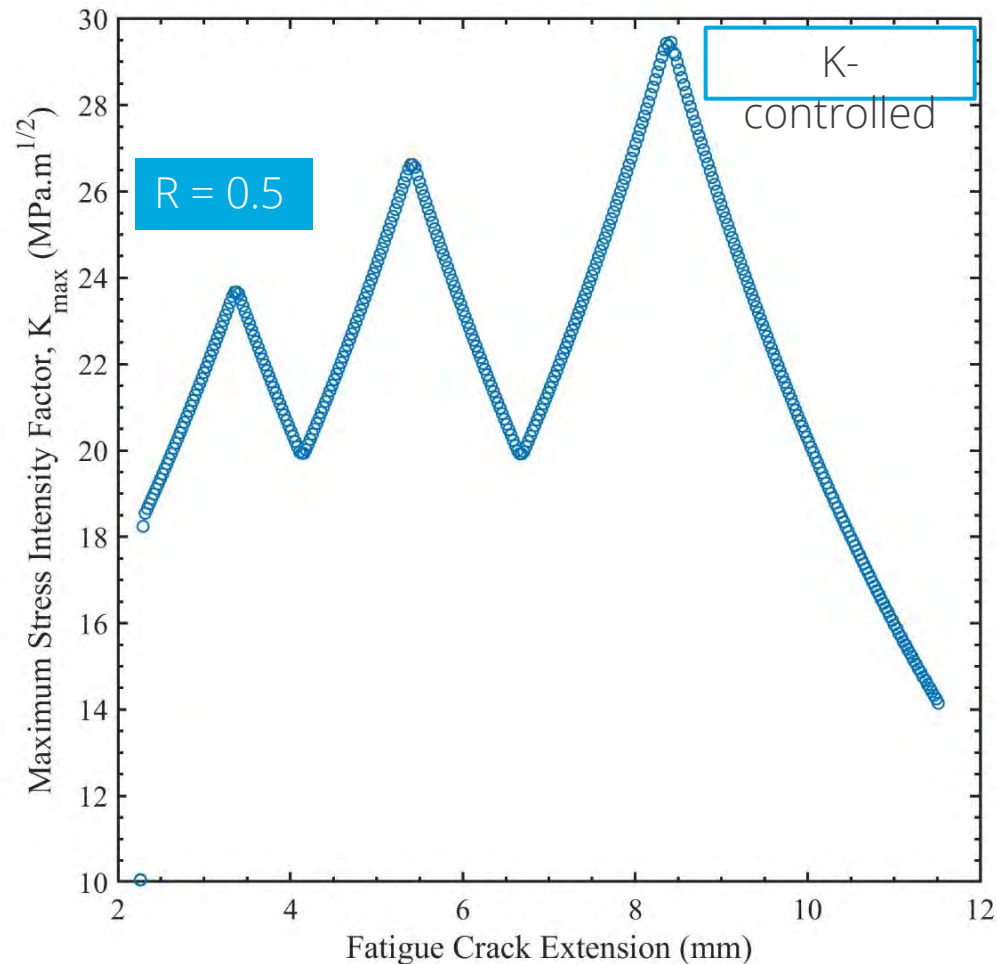
HA-FCG : Test Methodology



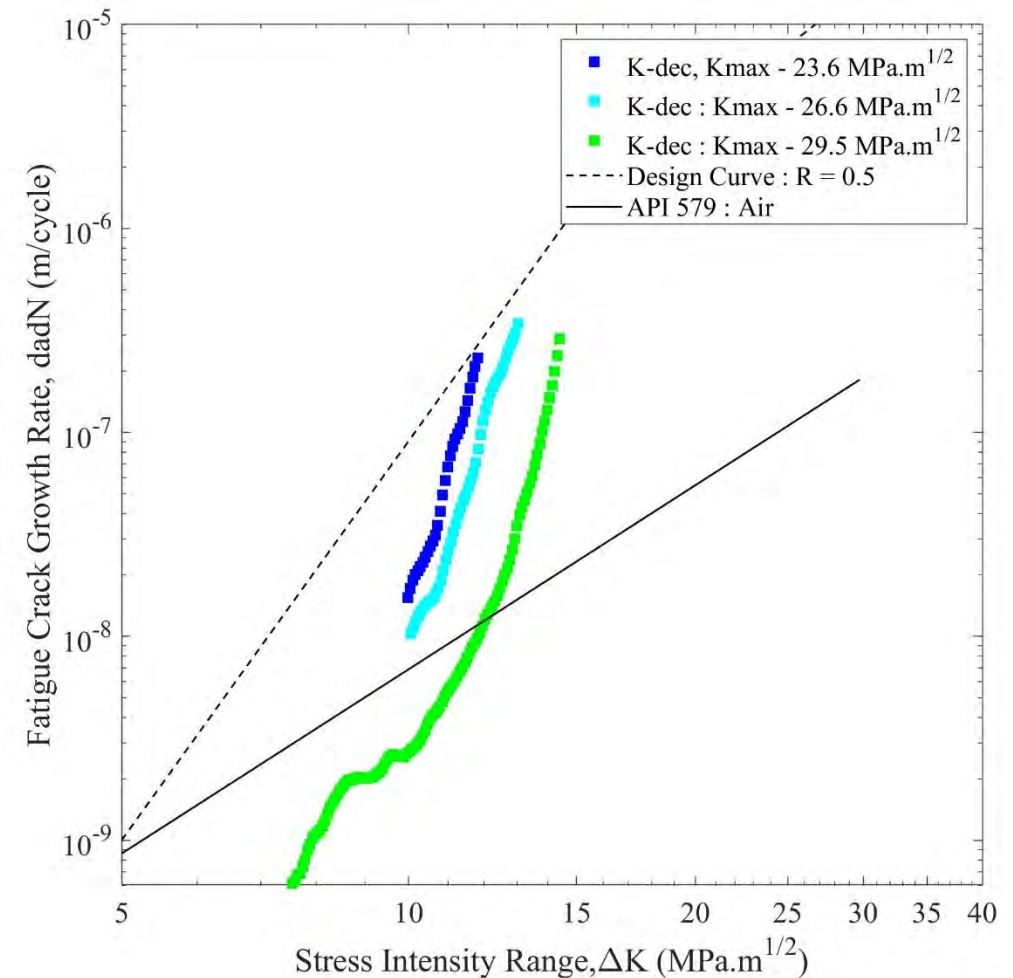


HA-FCG : Loading History

HG8 BM in 210 Bar GH2
HRA ~ 42

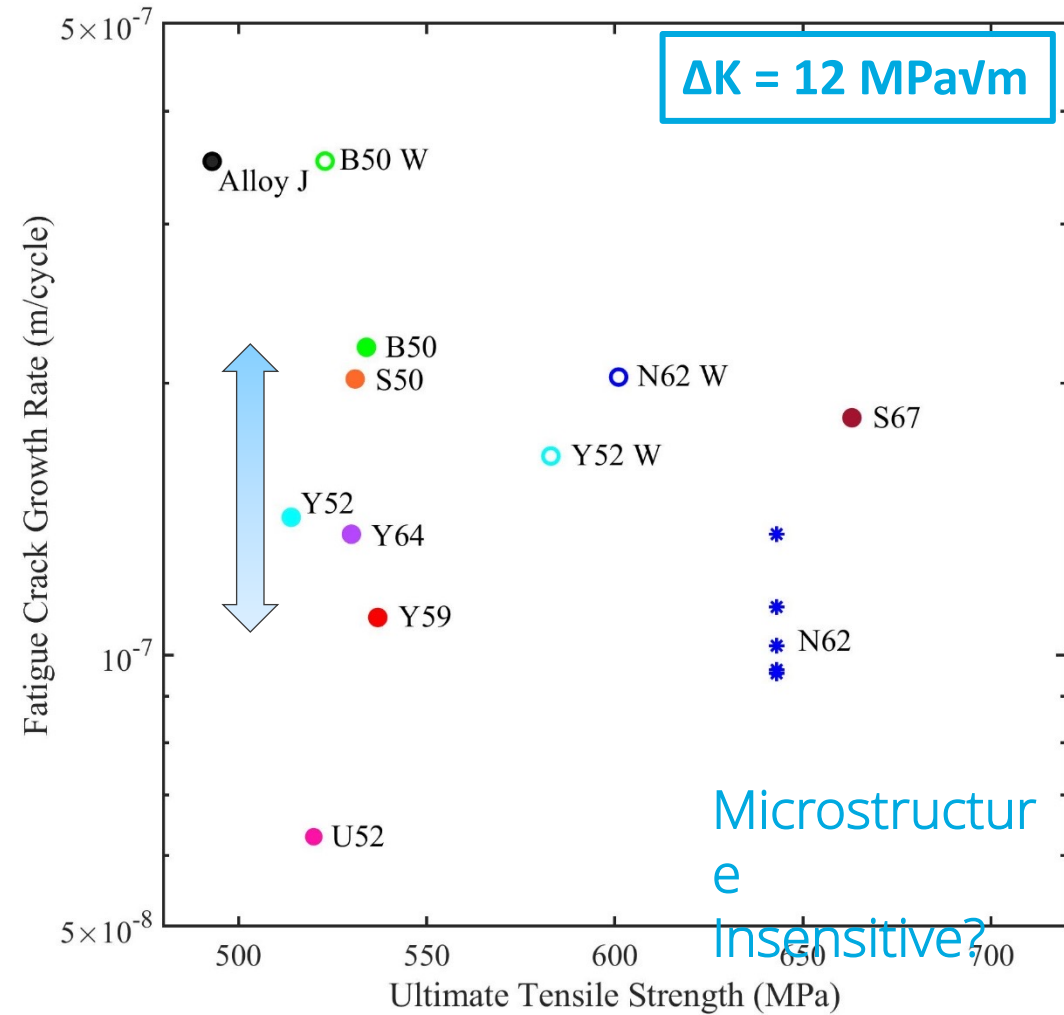
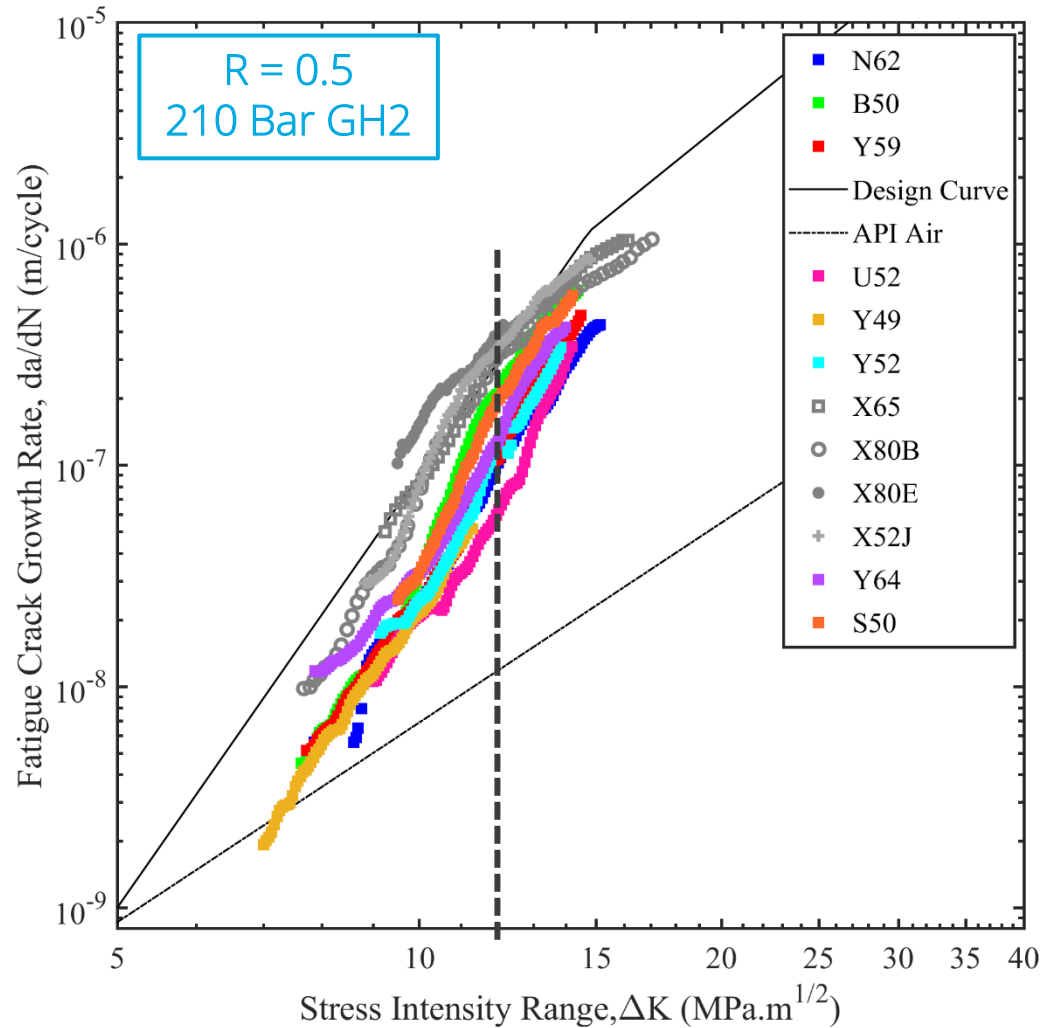


K-Decreasing Data



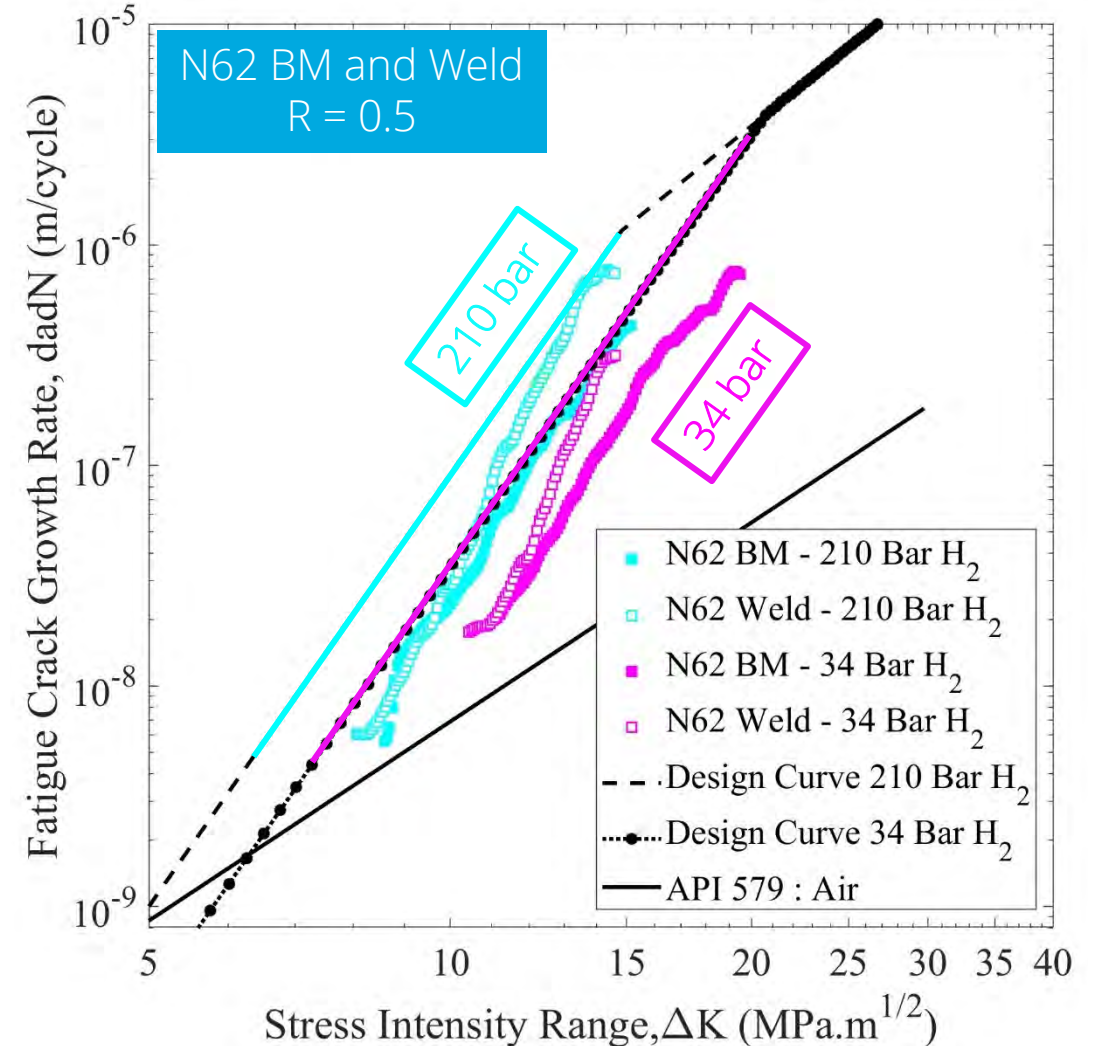
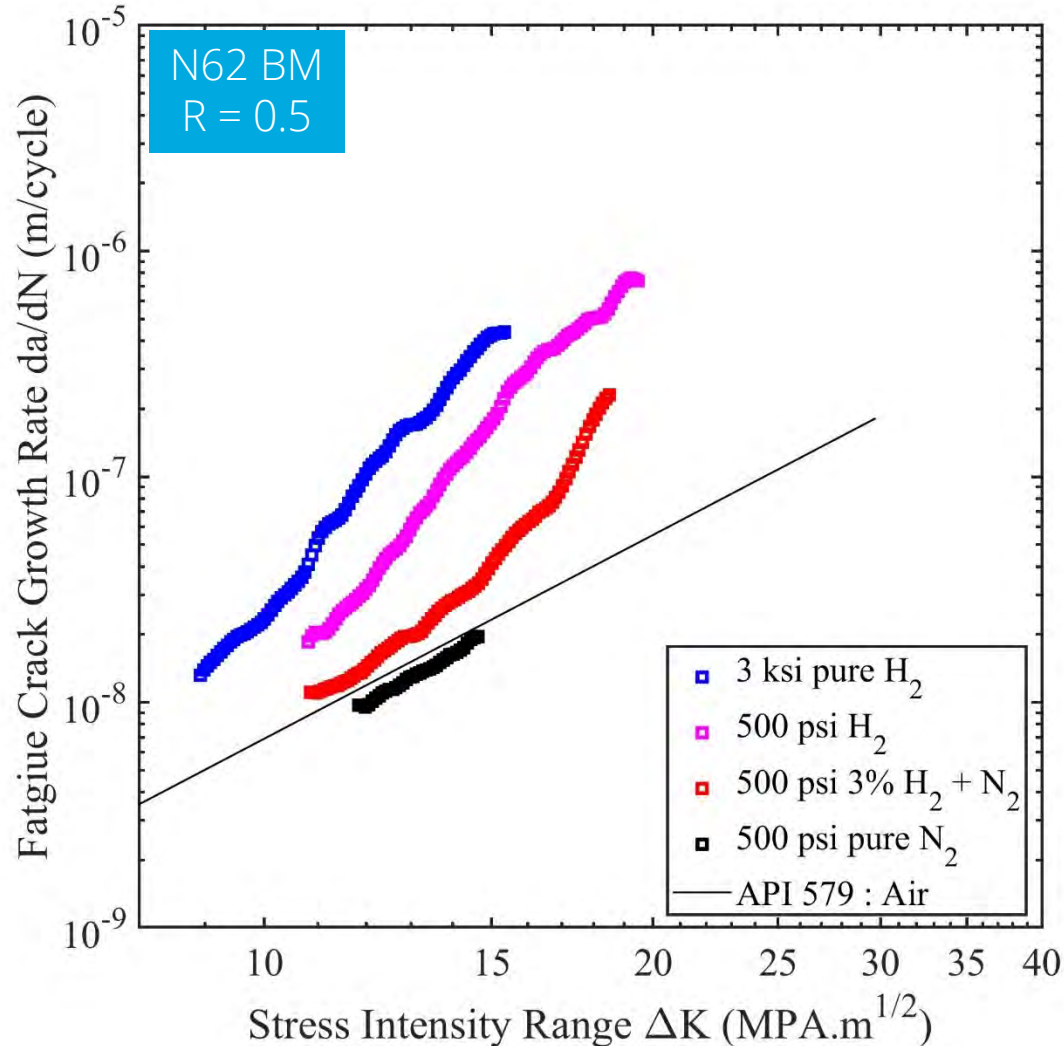
How do we account for the effects of load history on measured FGCR in GH2?

HAFCG – Microstructure Effects



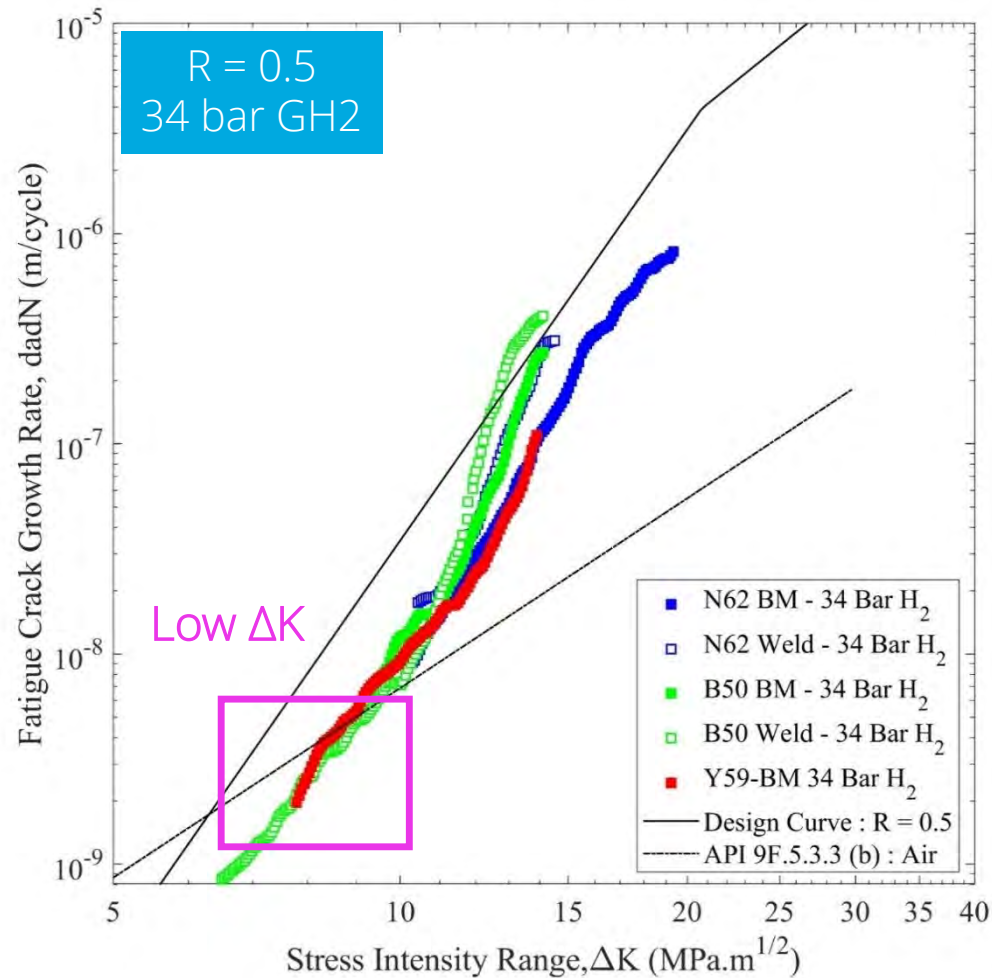


HAFCG – Pressure Effects



Trends at low 'P' can be extrapolated to high 'P'

HAFCG – Low ΔK Regime



HA-FCGR in Hydrogen lower than FCGR in Air

- Environment – dry GH_2 vs moist air
- Materials Response
- Crack Closure
- Residual Stresses

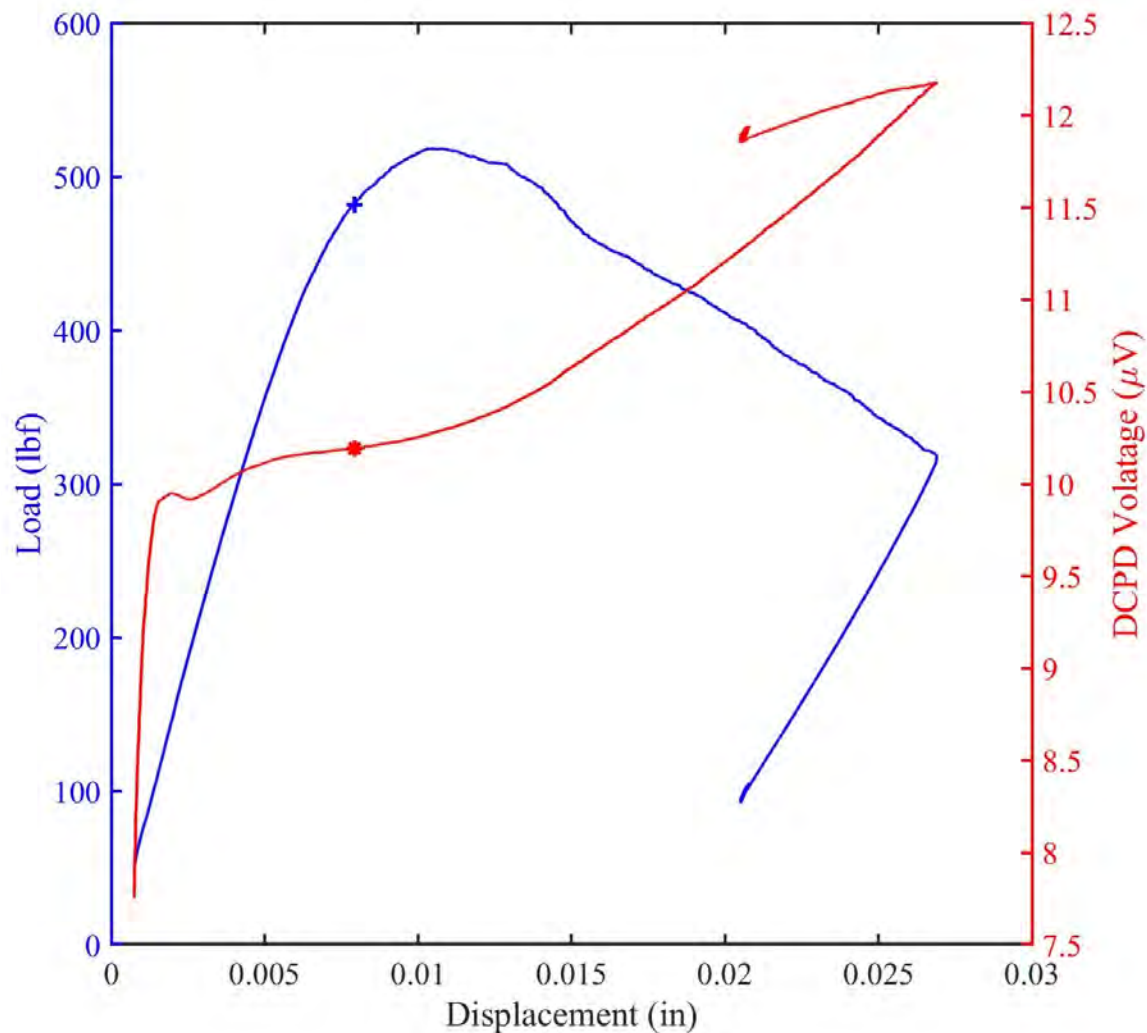
The image features a central dark blue diamond shape with the text "HA-Fracture" in white. This diamond is surrounded by a light gray diamond border. Two diagonal lines, each composed of several colored segments (blue, orange, green, red, purple, and light blue), cross the background behind the central diamond.

HA-Fracture



HA-Fracture : Methodology

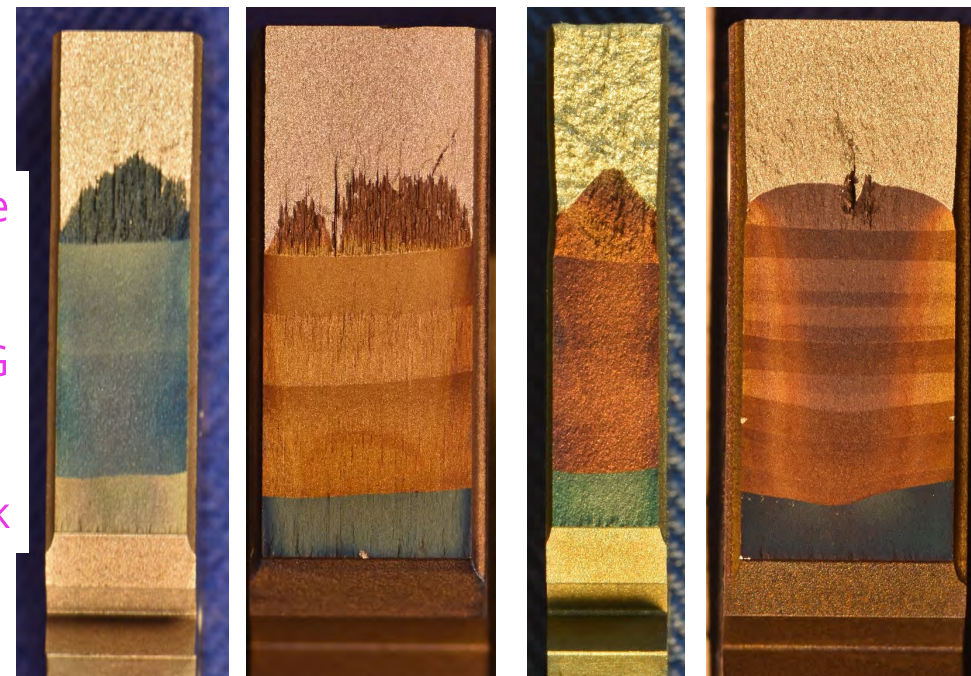
N62 BM 210 Bar GH2



HA-Fracture

HA-FCG

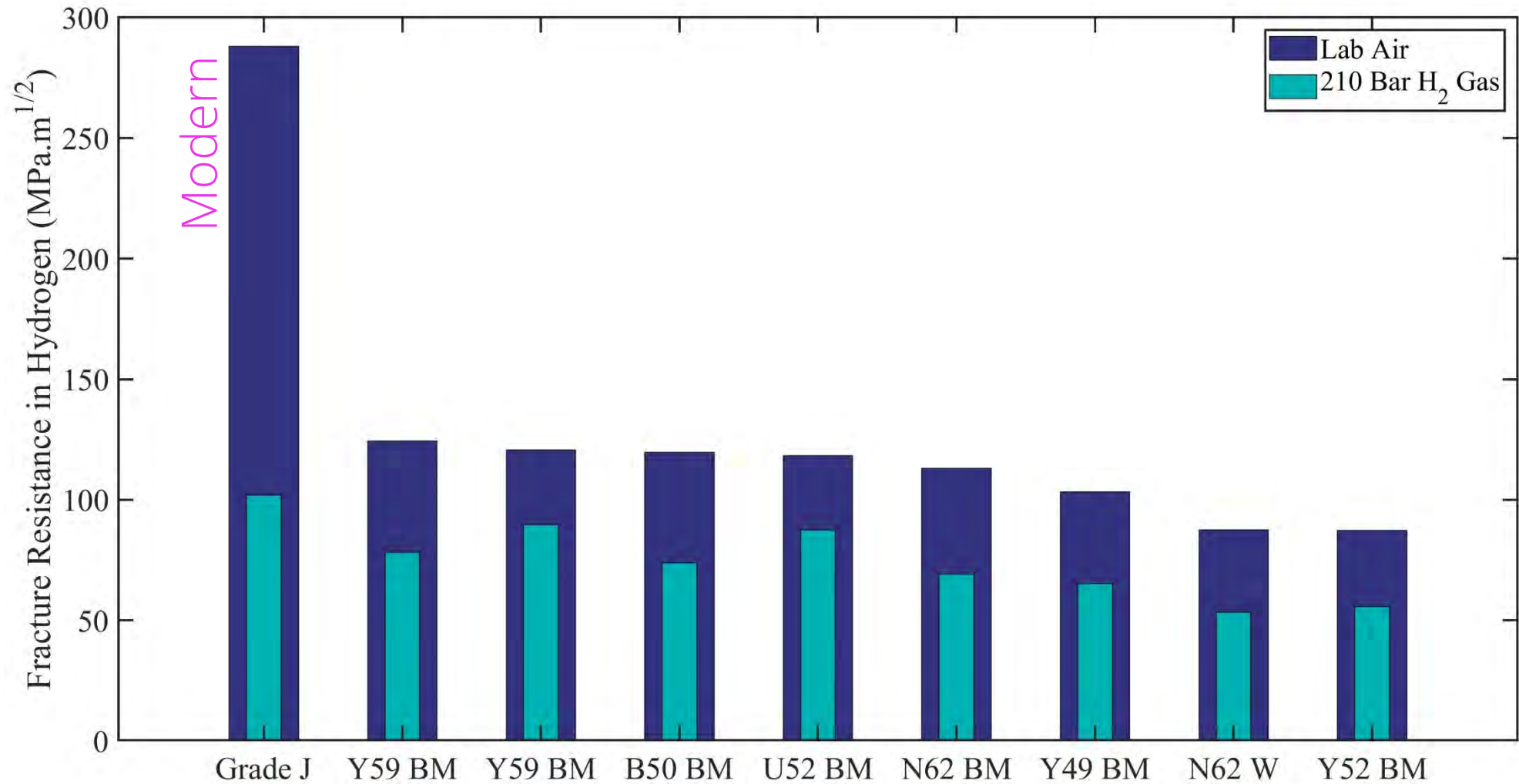
Air Pre-Crack



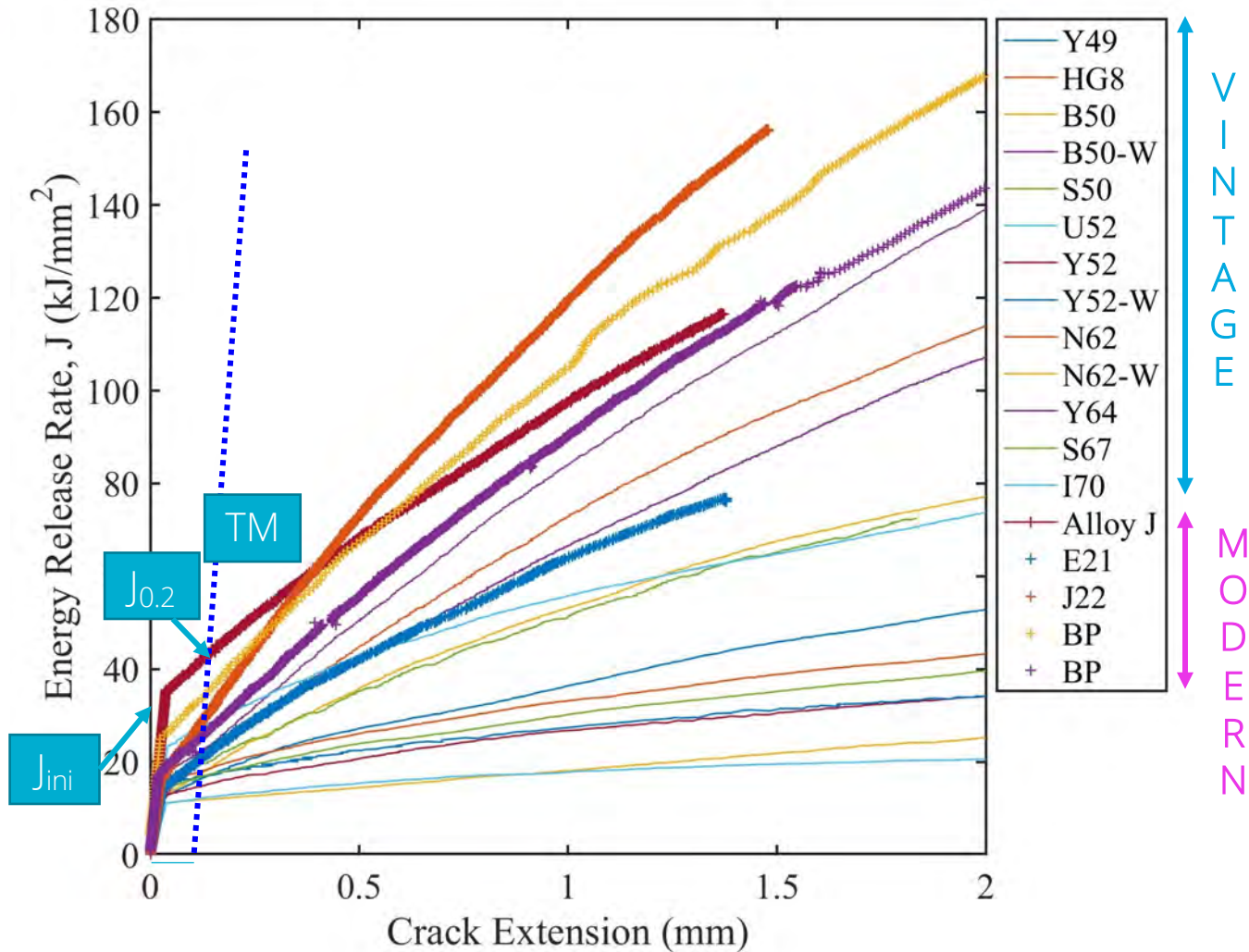
- Variable Sample Thickness
- Micro and Macro-Scale Delamination
- Crack Tunneling



HA-Fracture : Air v/s 3 ksi H₂



HA-Fracture : Variability in J-R Curves



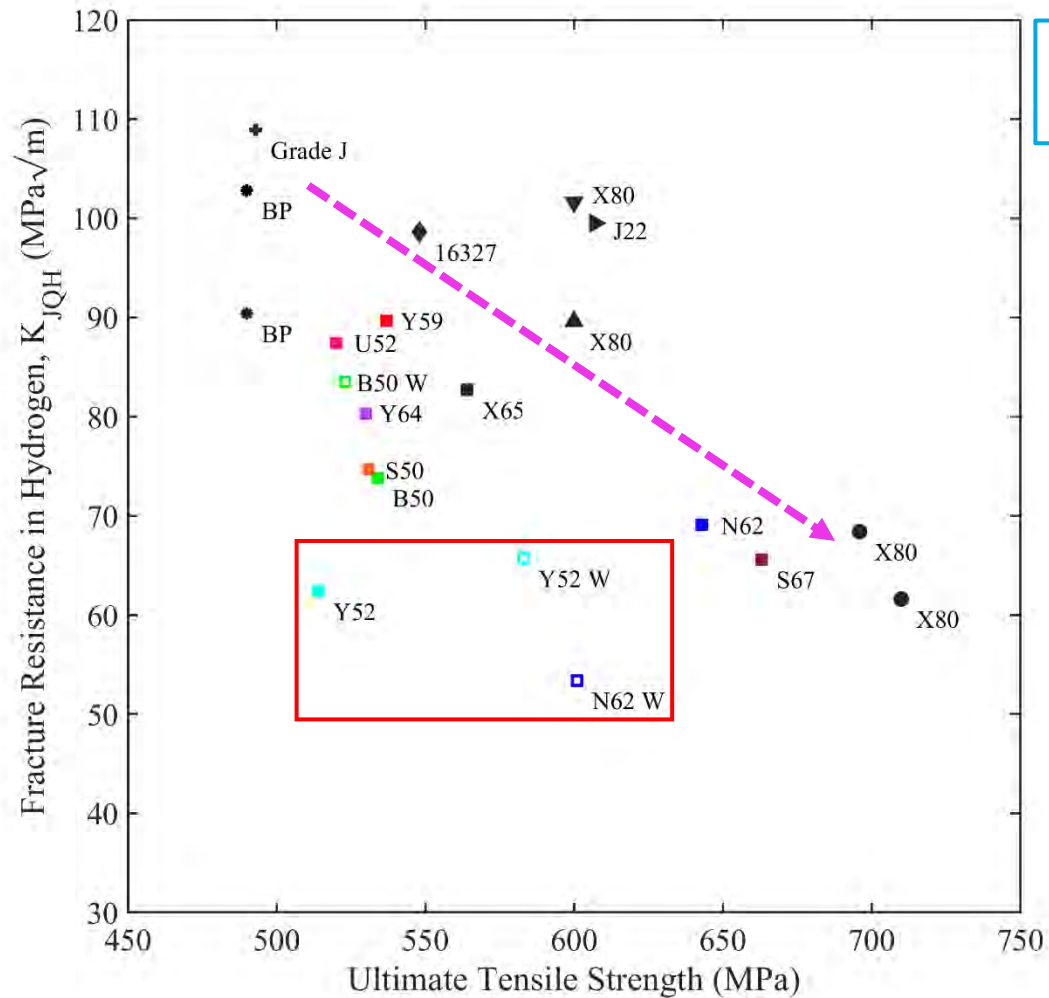
J-R Curve is Material Dependent

Large Variation in Tearing Modulus

Slope of the Tangent of the J-R curve at $J_{0.2mm}$



HA-Fracture : Strength Effects



Fracture resistance in hydrogen decreases with strength of the steel

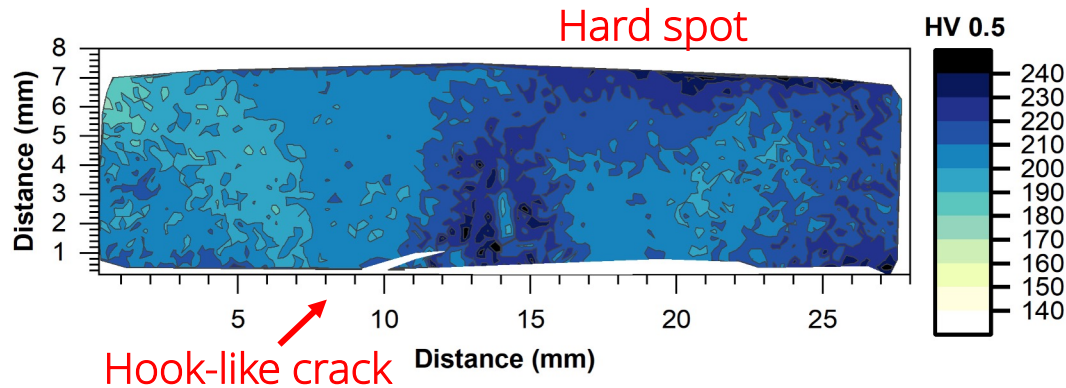
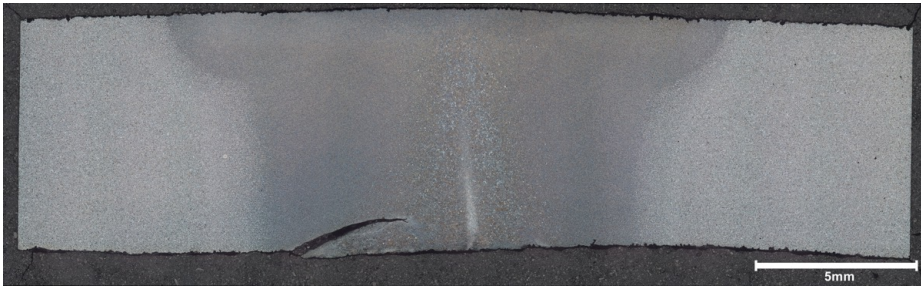
Outliers will be further investigated

HRA/B measurements will be performed



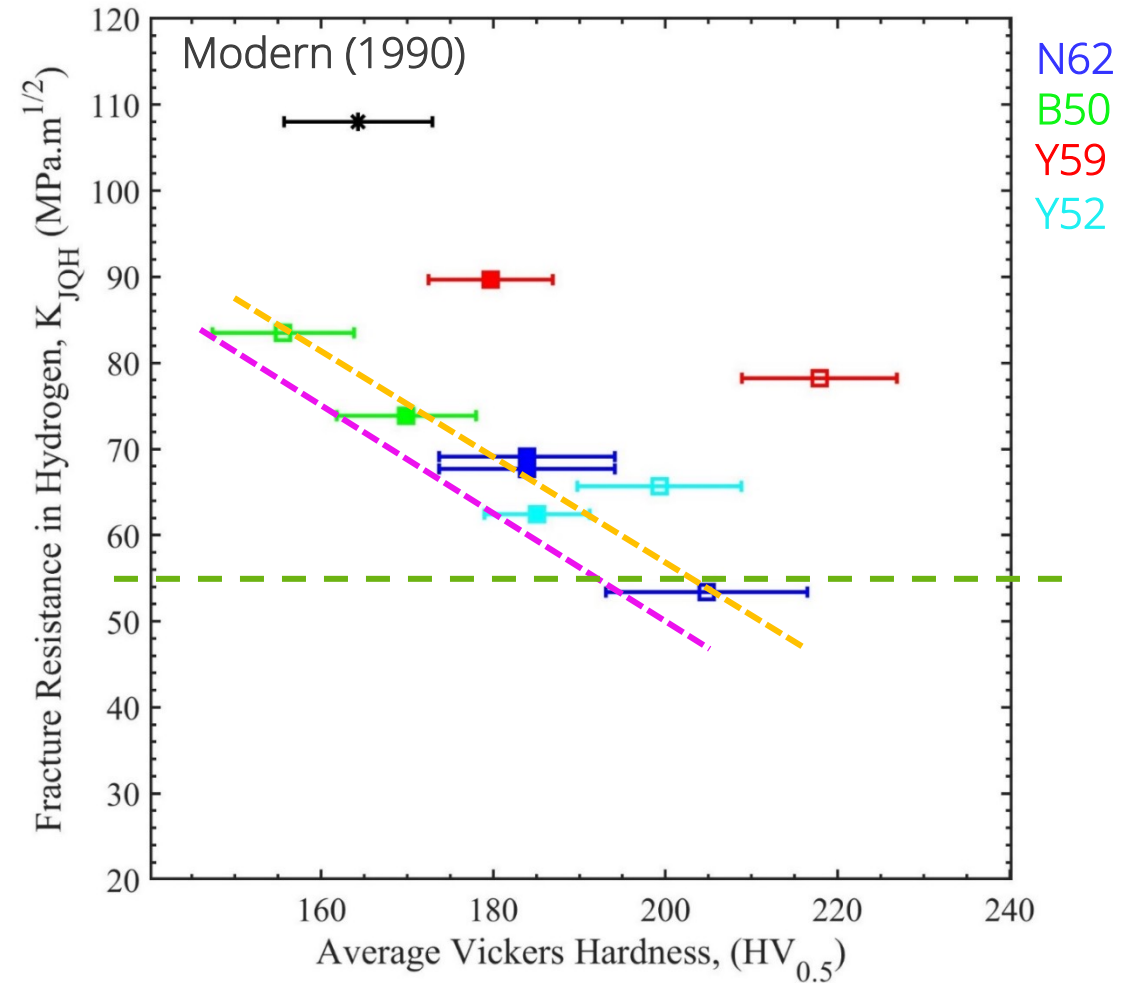
HA-Fracture : Microstructural Heterogeneity

Electric Resistance Weld (ERW) in a Vintage X52 Pipeline Section from 1959



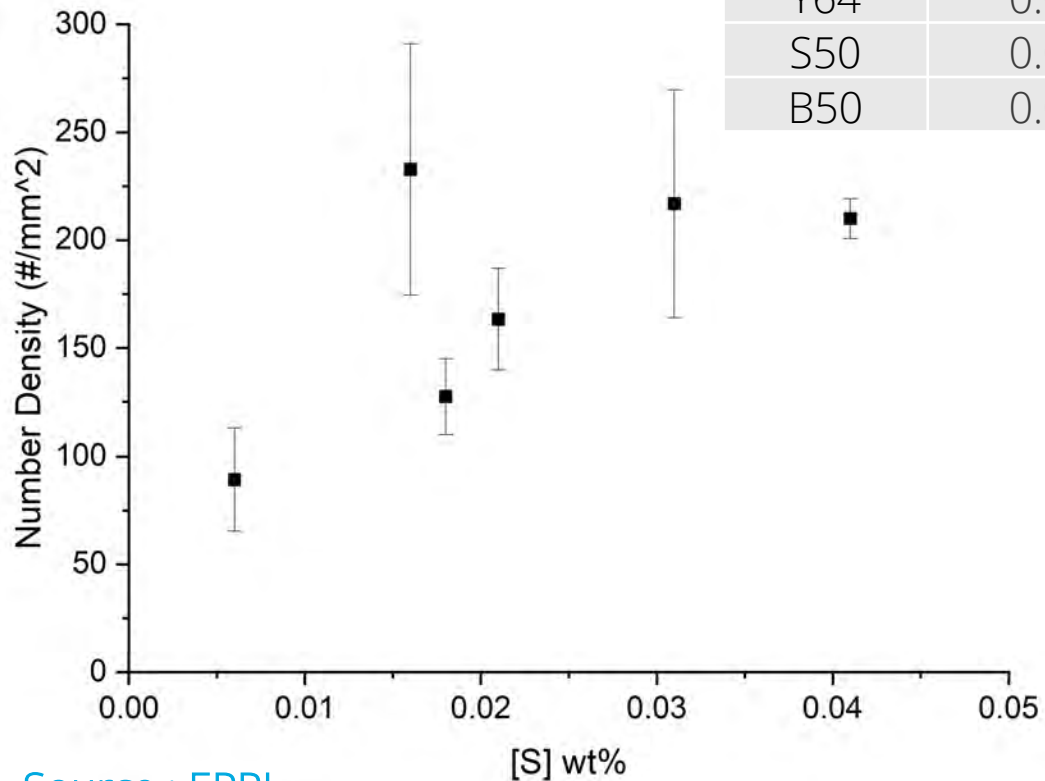
Source : EPRI

Fracture Resistance in 210 Bar GH2



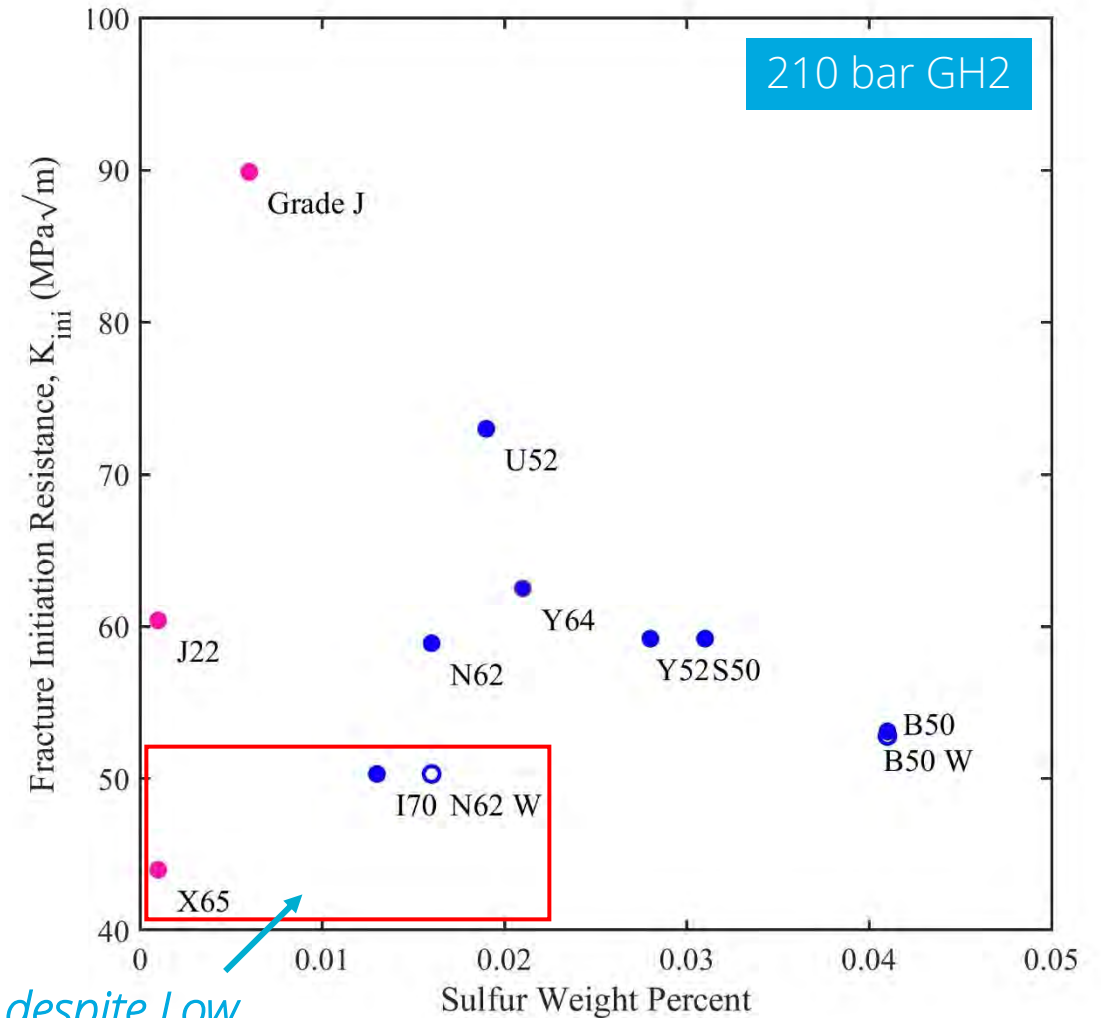


HA-Fracture : Inclusion Content



Source : EPRI

Sample	[S] wt%
X52J	0.006
N62	0.016
Y59	0.018
Y64	0.021
S50	0.031
B50	0.041



Low K_{ini} despite Low S content



Summary

- Accelerated fatigue crack growth was observed for modern and vintage steels in 3ksi H₂.
- Trends in HA-FCG rates were microstructurally insensitive, modern steels exhibited higher HA-FCG rates
- HA-FCG was observed for H₂ pressures as low as 1 bar. Low 'P' data can be extrapolated to high 'P'.
- Effects of prior load history, residual stress, crack closure and environmental impurities are unclear.
- Fracture resistance of pipeline steels is reduced in gaseous H₂. Larger reduction for modern steels
- Materials dependent J-R behavior observed. Large variations in the tearing modulus.
- Fracture resistance in hydrogen decreases with strength of the steel.
- Role of microstructural heterogeneities and inclusions on fracture resistance needs further investigation.

Thank you !

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Exceptional service in the national interest

Fatigue and Fracture in Hydrogen with Impurities

Subcritical Crack Growth in High-pressure Hydrogen and Hydrogen with Oxygen Impurities

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

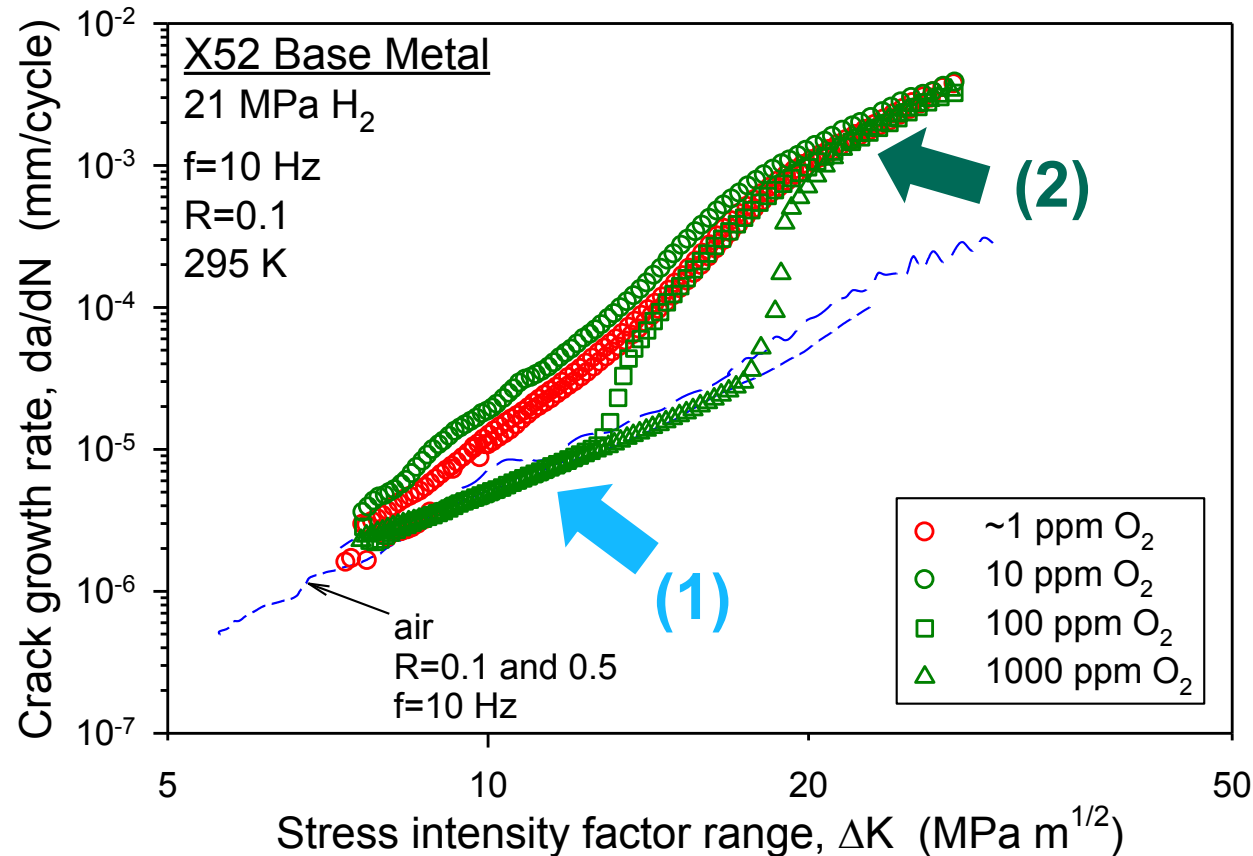
Hydrogen and Materials Science Department, Sandia
National Laboratories Livermore, CA, USA

PVP 2023 Hydrogen Study Group

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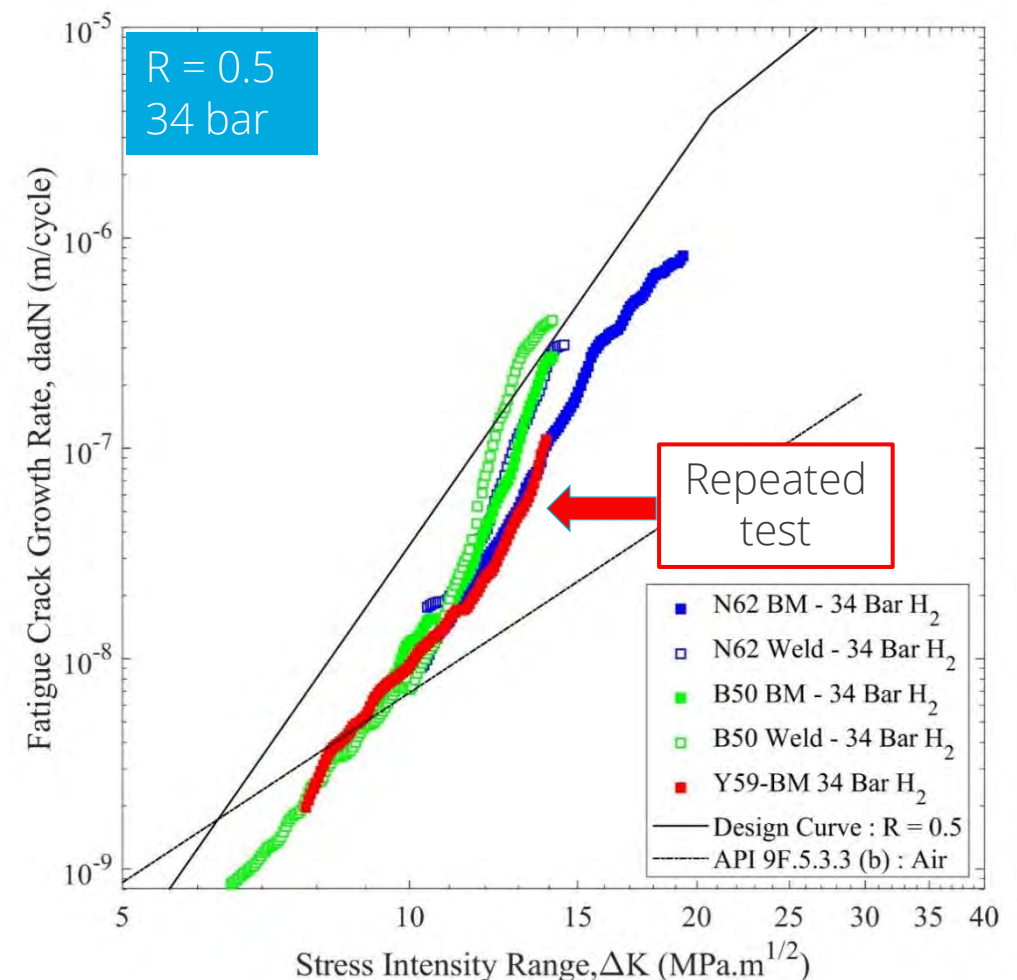
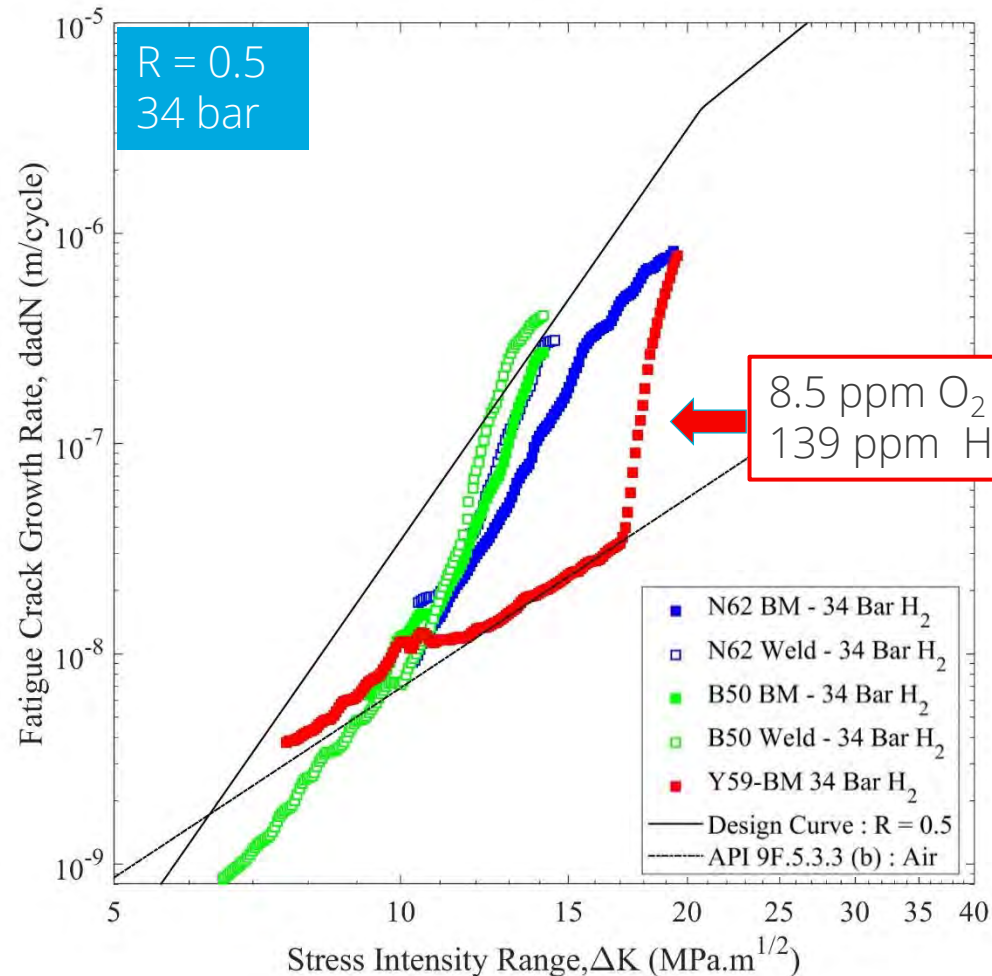


Oxygen and other impurities are known to affect measurements of fatigue and fracture



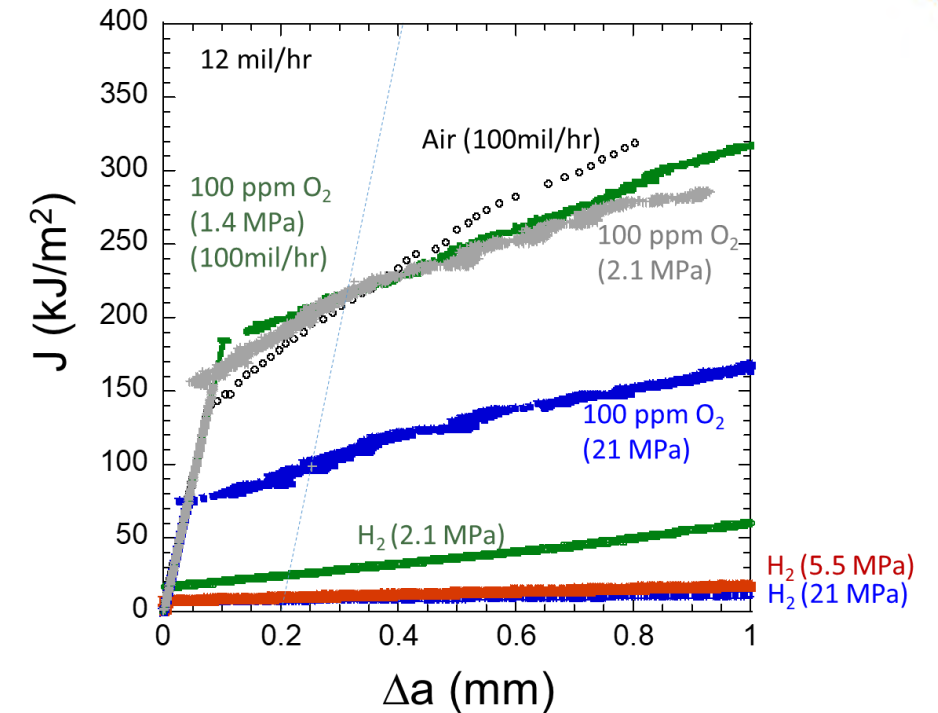
- Numerous examples of trace gases mitigating fatigue crack growth rate (FCGR) in laboratory conditions
- Example:
 - (1) Oxygen reduces FCGR to air
 - (2) Oxygen has no effect on FCGR in H₂

Oxygen and other impurities are known to affect measurements of fatigue and fracture



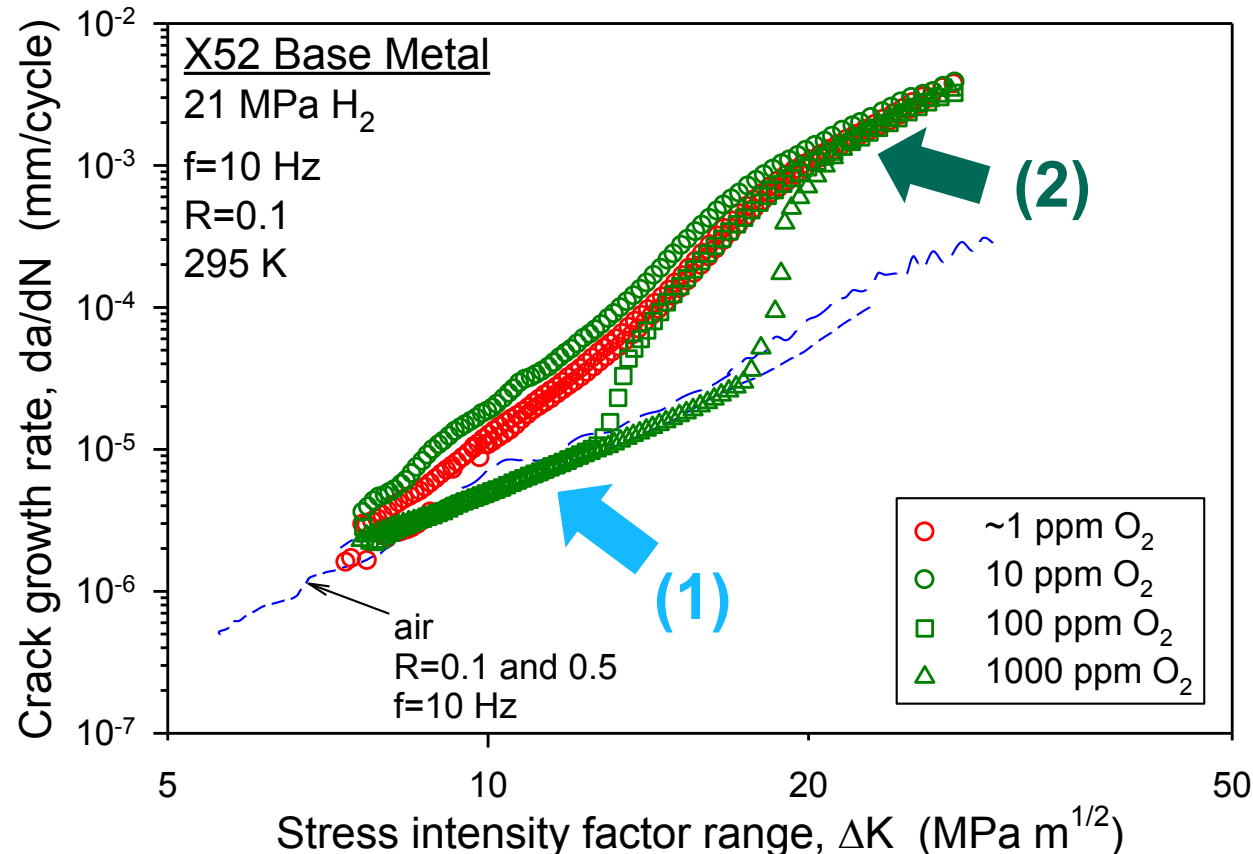
Oxygen moderated hydrogen-assisted fracture (J-R curves ASTM E1820)

- Fracture toughness K_{JH} values decreased by 80% in 21 MPa pure H_2
- 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



Sample ID	Environment	Test Pressure (MPa)	Actuator rate (mm/hr)	da/dt (m/s)	K_{JH} (MPa m ^{1/2})
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
X100-51	Air	-	2.5	5.0E-7	217
X100-52	Air	-	2.5	1.4E-7	202
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

Oxygen and other impurities are known to affect measurements of fatigue and fracture



- Fatigue crack growth tests are typically performed at 1 Hz ($\pm$ decade)
 - $da/dN = 10^{-5}$ mm/cycle
 - Time for $\Delta a = 1\text{mm}$: ~ 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?

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

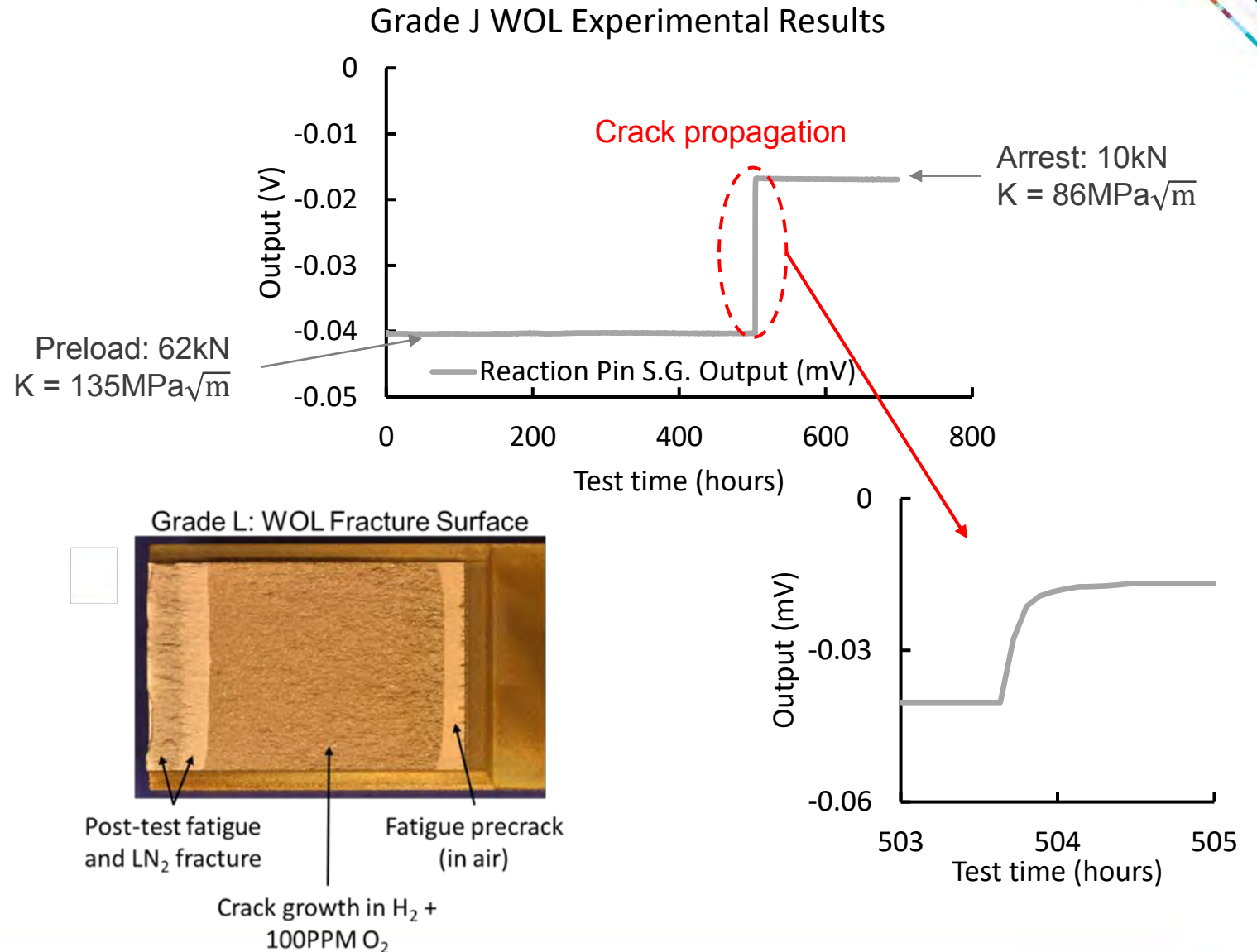
Wedge-opened loaded (WOL)



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



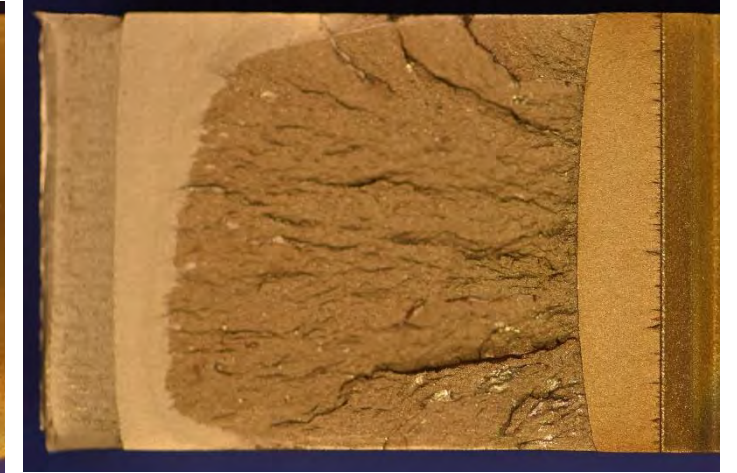
Materials tested and fracture surfaces

- $H_2 + 100\text{PPM}$ or $1000\text{PPM } O_2$:
 - SA372 Grade J steel (YS = 700 MPa)
 - SA372 Grade L steel (YS = 730 MPa)
 - X100 pipeline steel (YS = 760 MPa)
- Additional data:
 - Precipitation Hardened 13-8 stainless steel (YS = 1480 MPa)
 - ASTM A723 (YS = 1034 or 1206 MPa)
 - 4335 V (YS = 1275 MPa)
 - IN718 (YS = 1102 MPa)

Grade L



Grade J



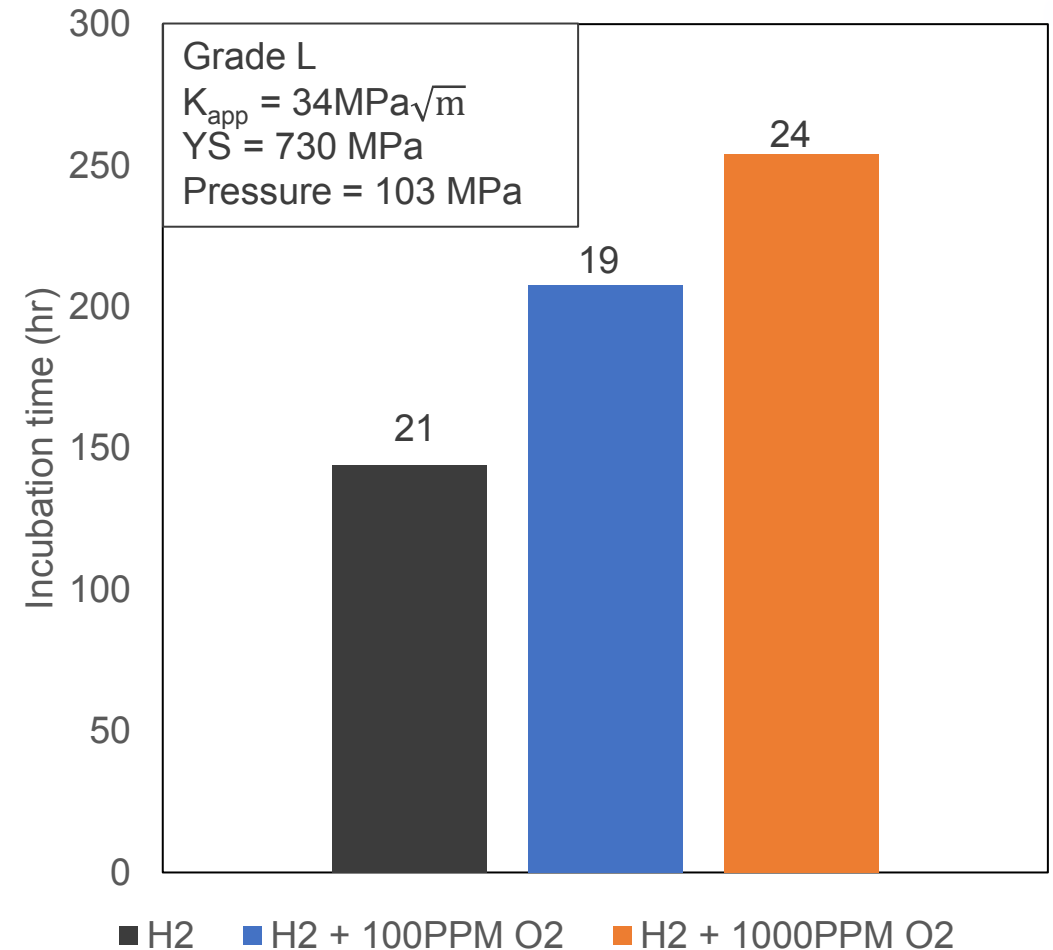
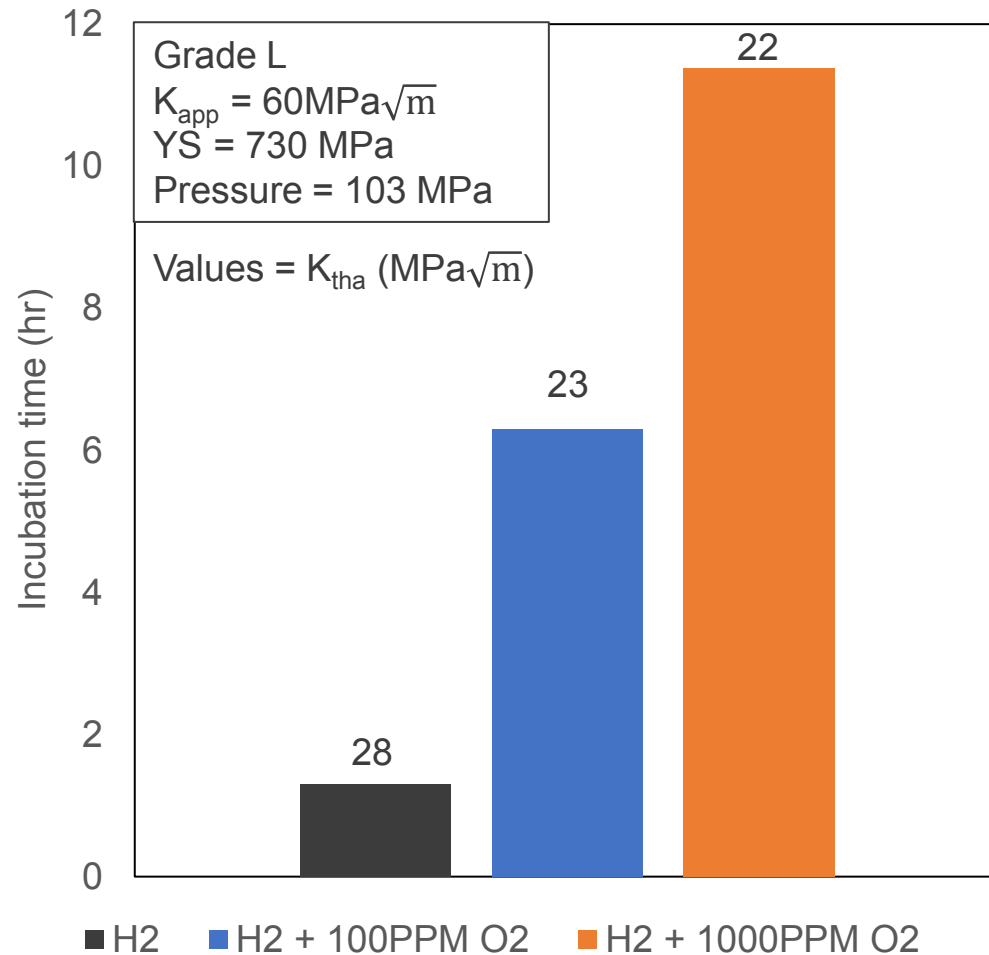
X100



13-8

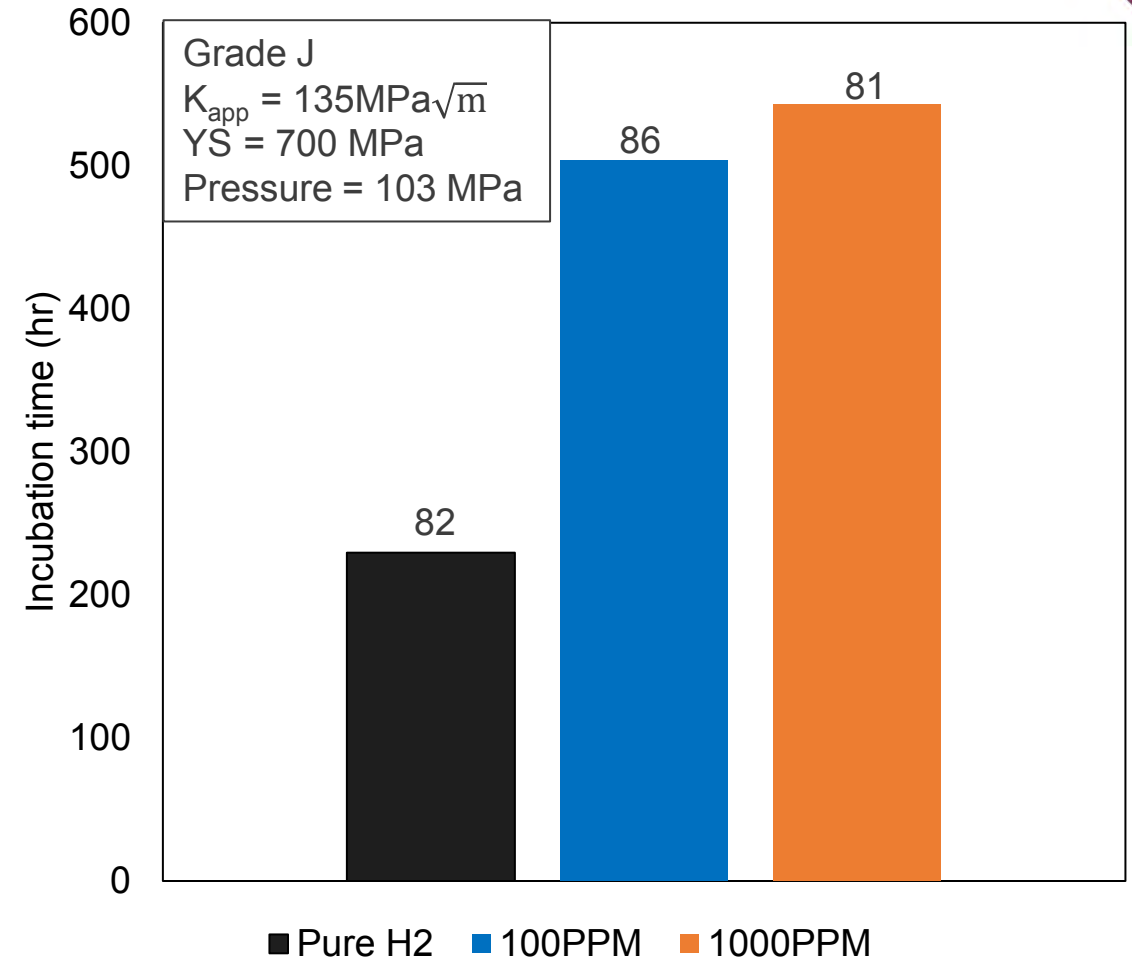
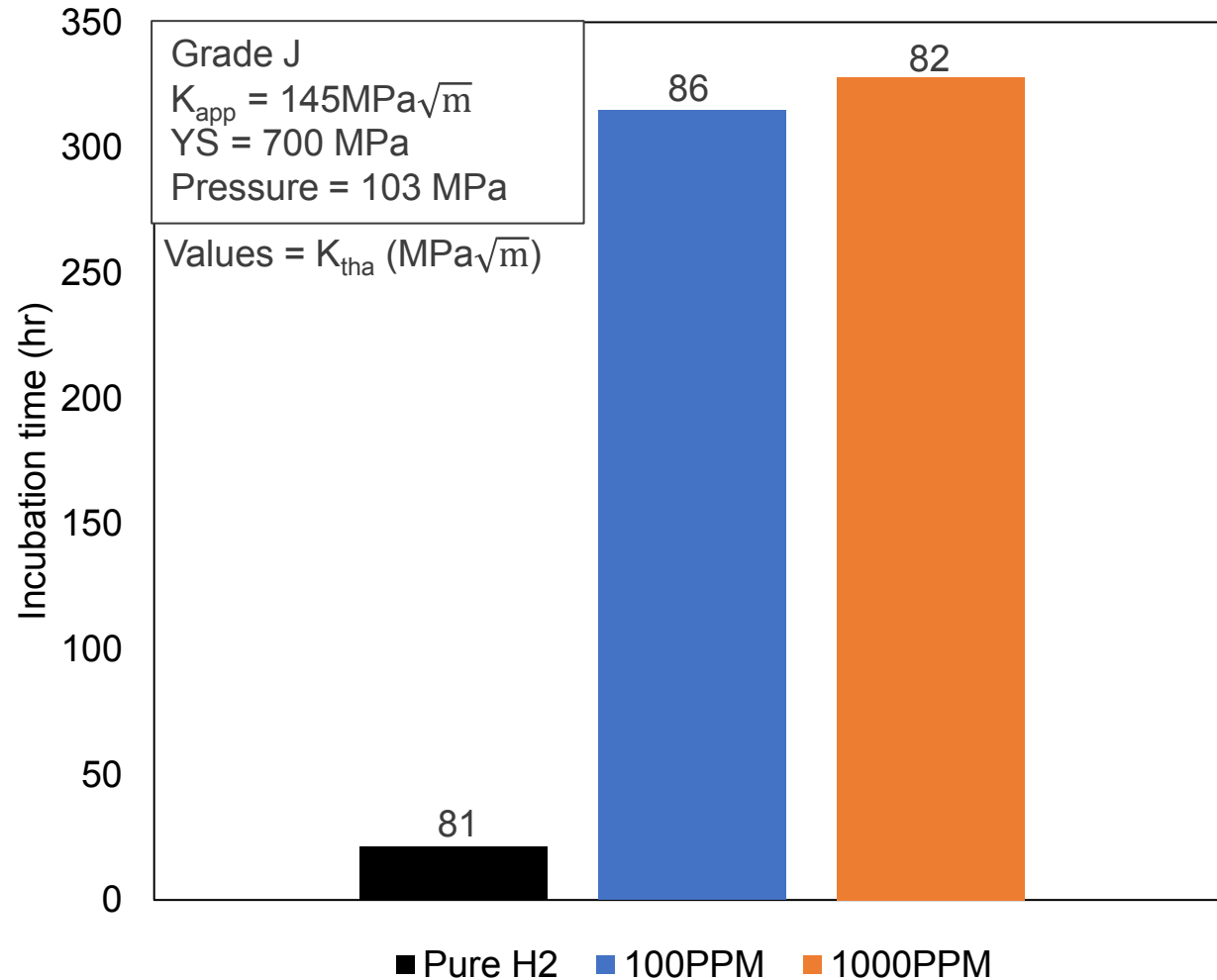


100PPM & 1000PPM O₂ delay incubation time



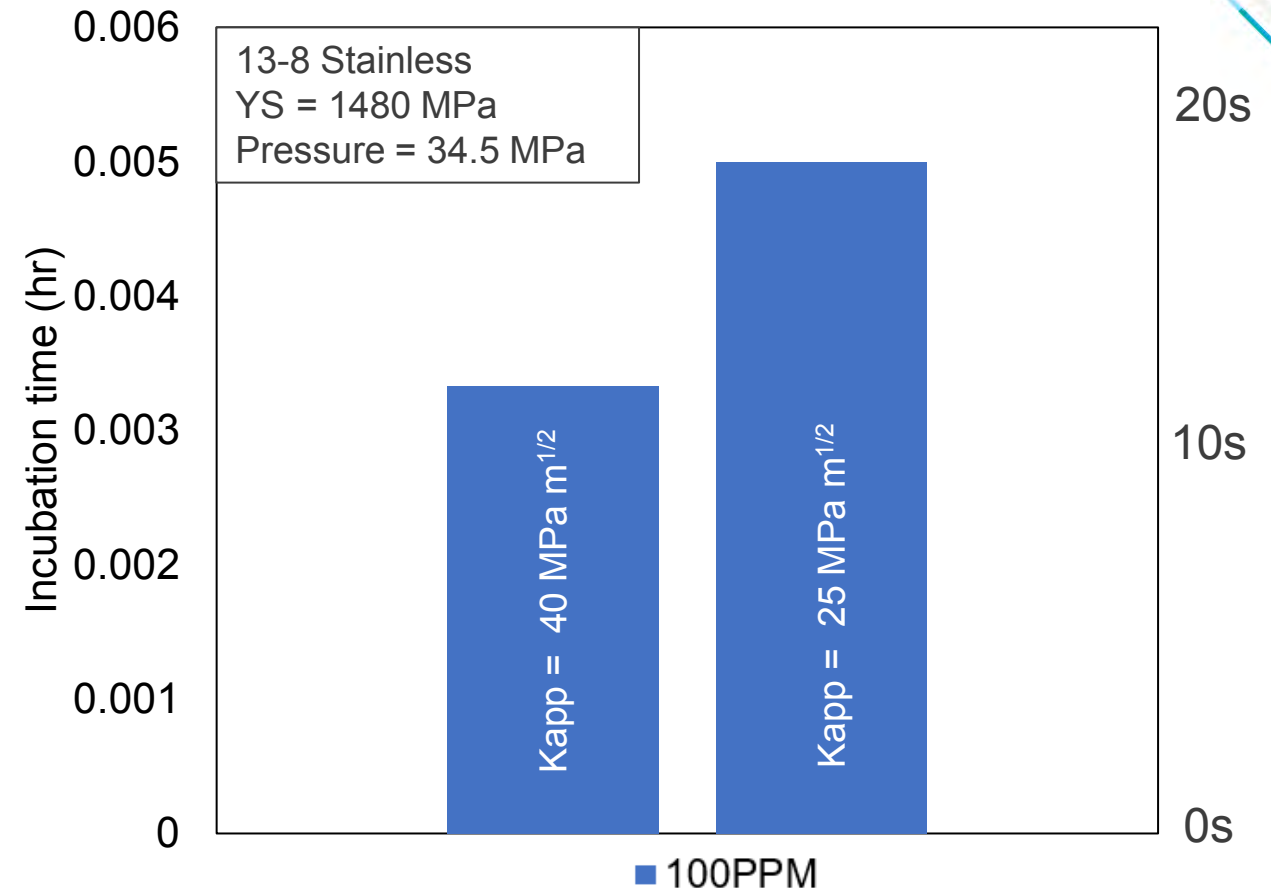
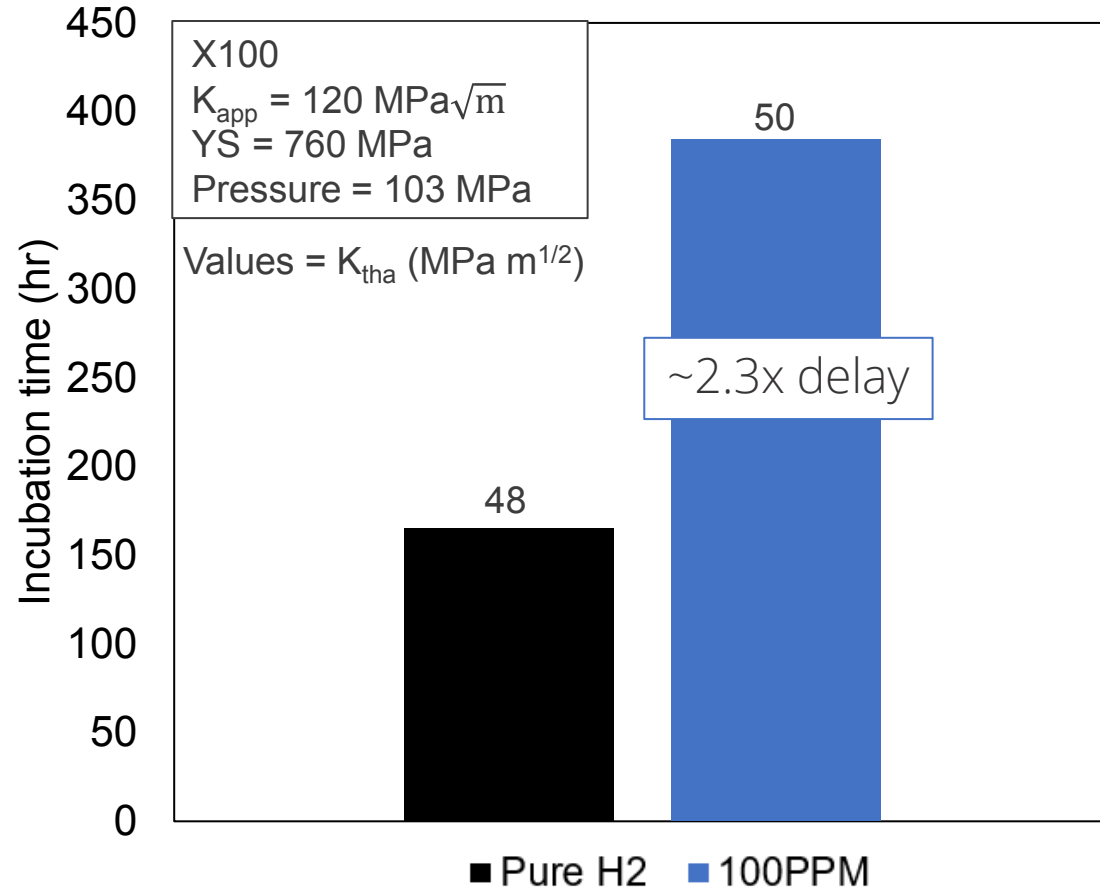
- Grade L shows a 5x delay at higher preload ($K_{app} = 60 \text{ MPa}\sqrt{\text{m}}$) and a 1.5x delay at lower preload ($K_{app} = 34 \text{ MPa}\sqrt{\text{m}}$)
- Similar crack arrest thresholds for all test conditions

100PPM & 1000PPM O₂ delay incubation time



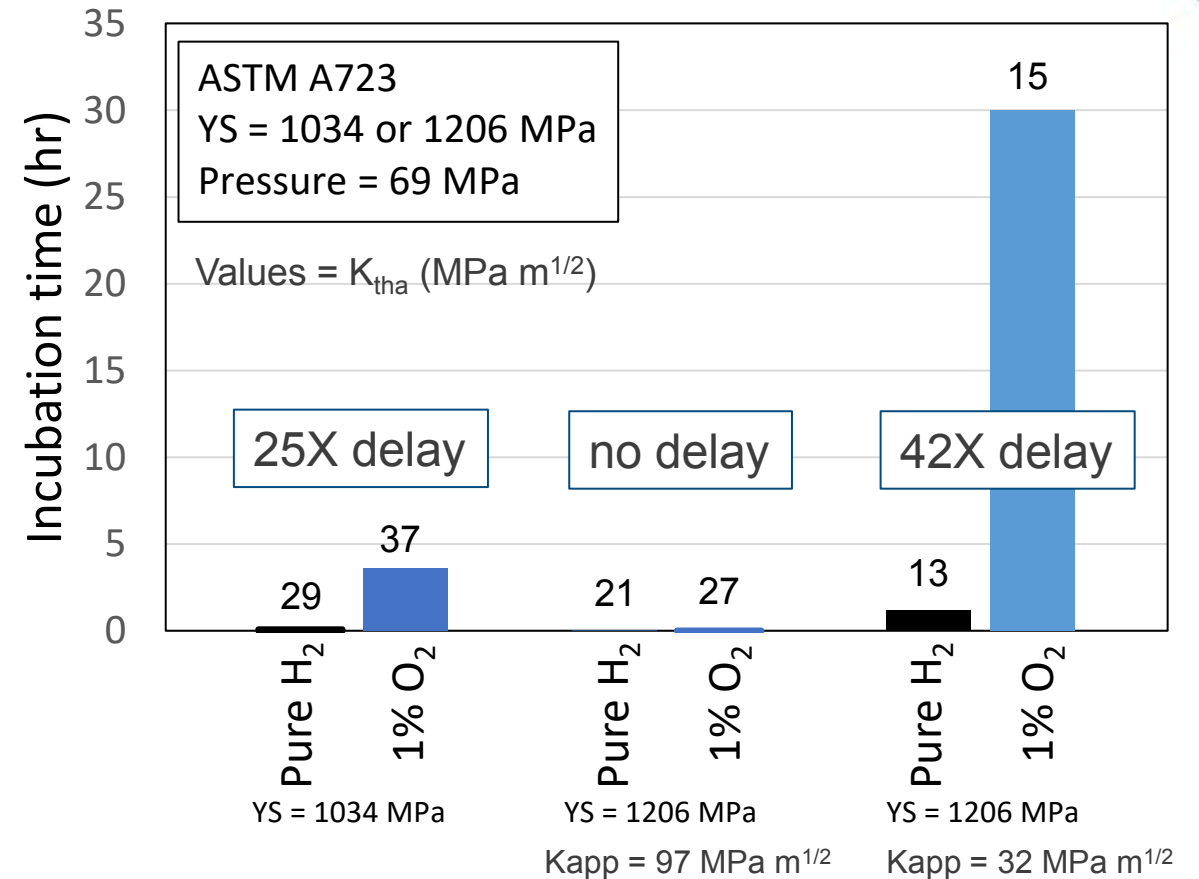
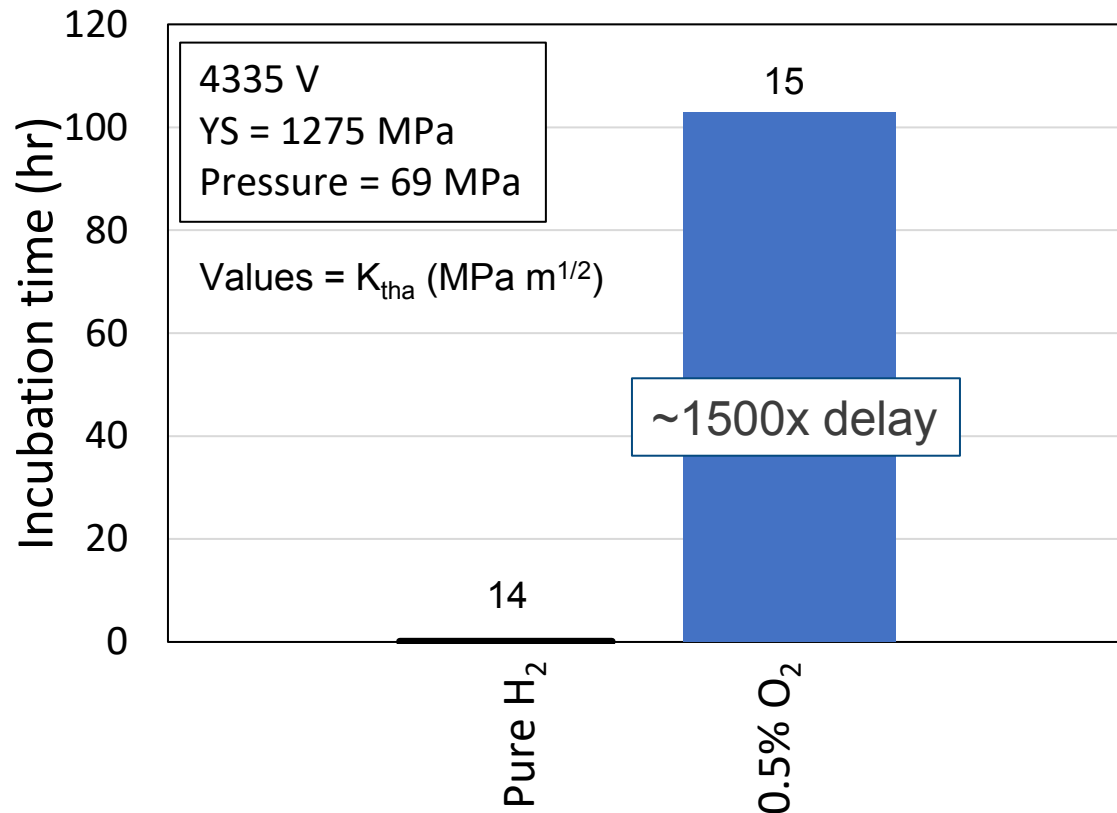
- Similarly, the Grade J material showed delays of 15x at a higher preload ($K_{app} = 145 \text{ MPa}\sqrt{\text{m}}$) and a 2.2x delay increase at a lower preload ($K_{app} = 135 \text{ MPa}\sqrt{\text{m}}$)
- K thresholds were within $\pm 5 \text{ MPa}\sqrt{\text{m}}$ of average for both the pure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

100PPM O₂ delays incubation time for X100, but 13-8 fractured immediately



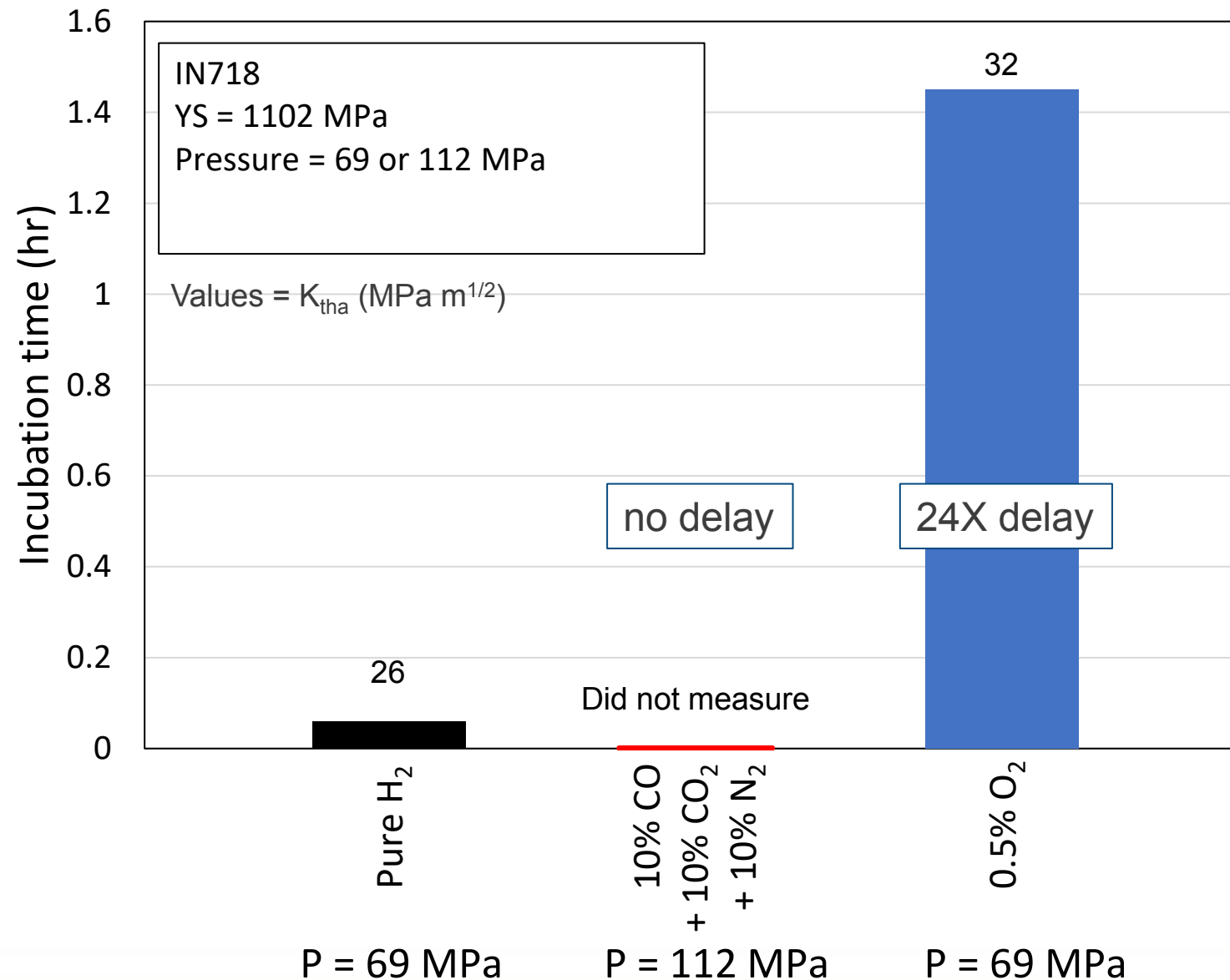
- X100 also saw a delay with the addition of 100PPM O₂
- Both 13-8 samples fractured ($a/W > 97\%$) within seconds of exposure to H₂ + 100PPM O₂

Oxygen delays incubation time in some cases, but does not prevent cracking



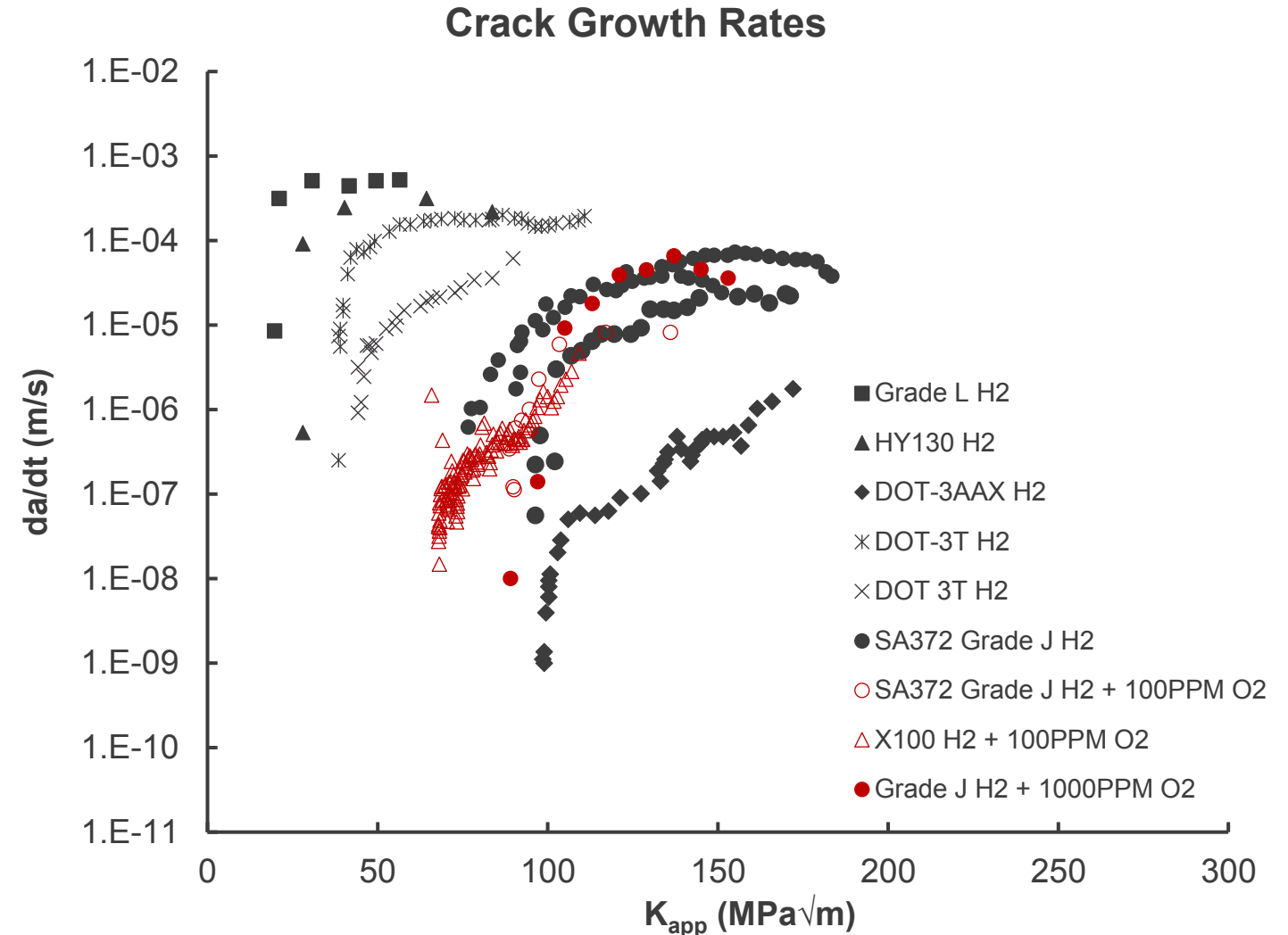
- At same K_{app} = 97 MPa m^{1/2}, the higher strength grade showed no delay of incubation time
- When K_{app} was lowered to 32 MPa m^{1/2}, then a measurable delay was recorded.

CO and CO₂ had no measurable delays in incubation time



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

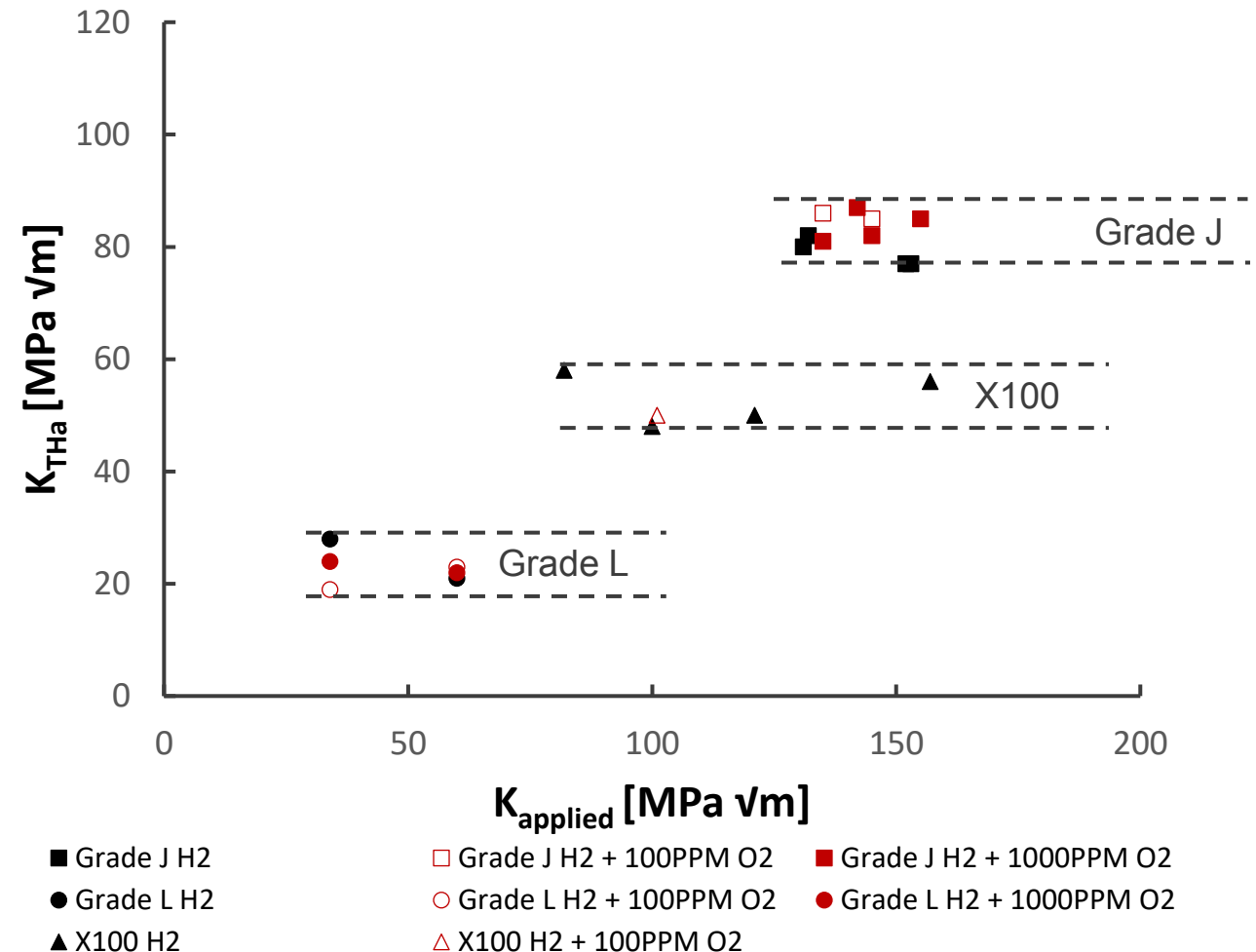
- Crack growth rates (da/dt) fall within the expected ranges from tests in pure hydrogen at similar pressures
- Adding oxygen to a high pressure hydrogen environment (103MPa) often increases the incubation time
 - The addition of oxygen impurities does not prevent subcritical crack growth or seem to effect crack arrest thresholds



K_{THa} unaffected by oxygen content

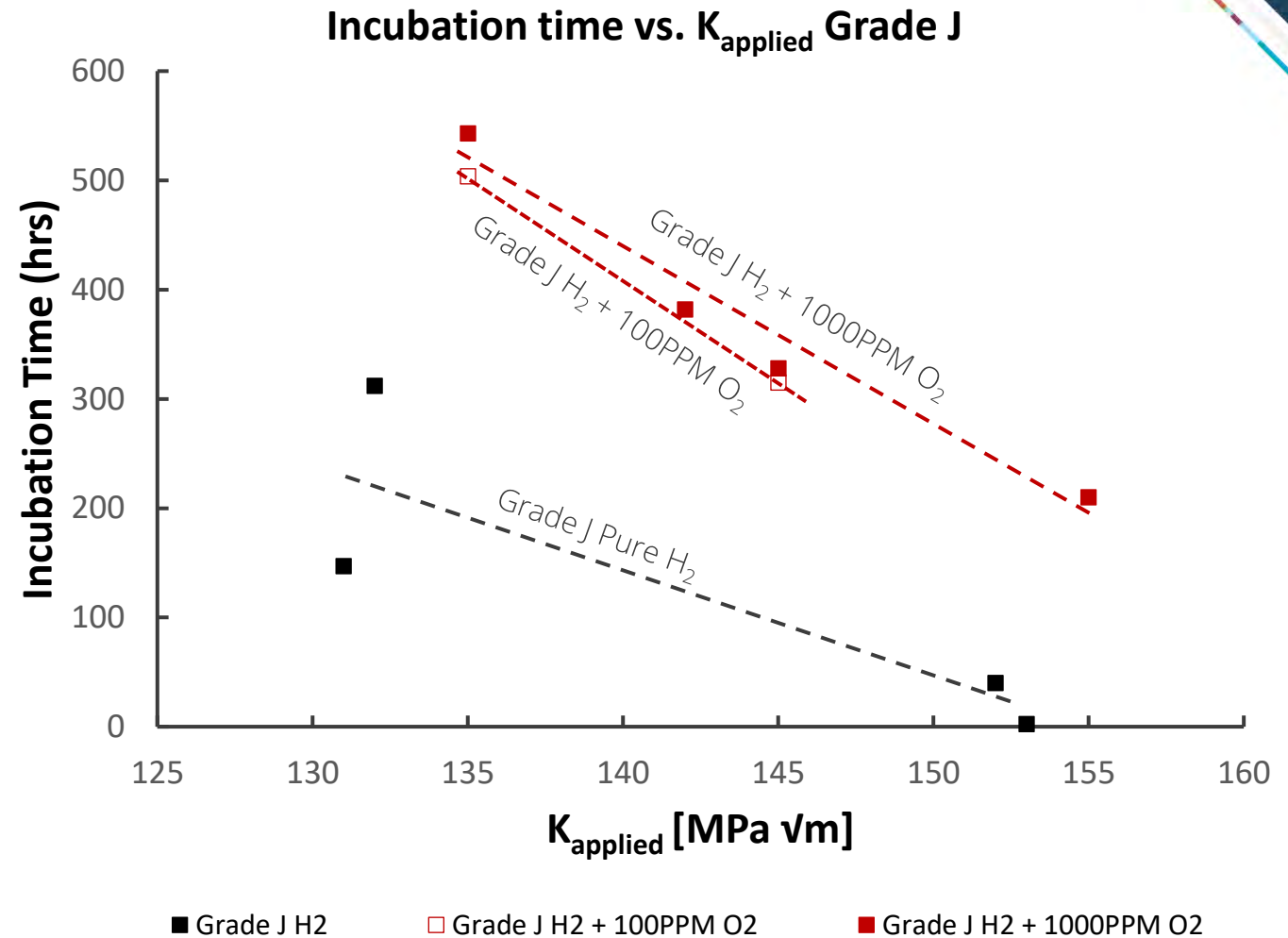
- Constant displacement fracture tests were carried out in pure hydrogen and mixed gas (100 and 1000PPM oxygen) environments at 103MPa (15ksi)
- K_{THa} independent from oxygen content
 - All tests with pure hydrogen and oxygen impurities fall within an apx. $10\text{MPa}\sqrt{\text{m}}$ range
- The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

Crack arrest threshold vs. applied preload



Summary

- Introducing 100PPM oxygen increased the incubation time by factors between 1.5x and 50x, 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
- Thank you for your attention!
 - rwheel@sandia.gov



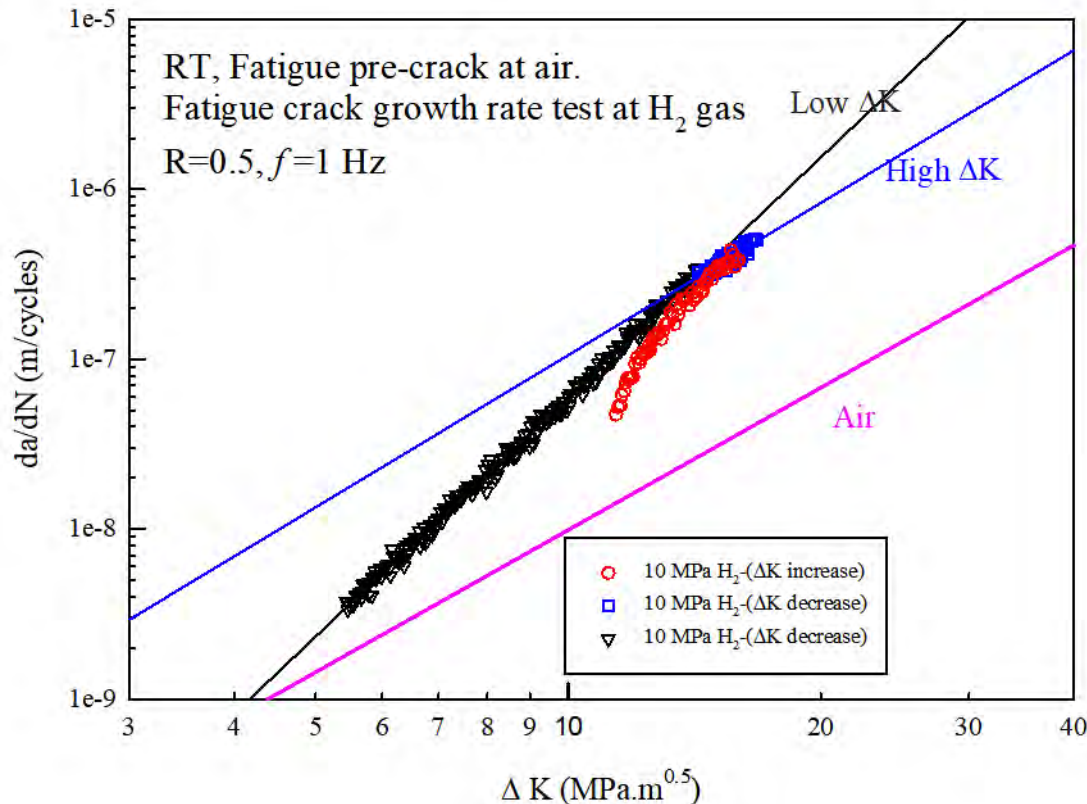
Effect of fatigue pre-crack condition for fatigue crack growth rate test

2023. 07. 21.

Un Bong BAEK*

Korea Research Institute of Standards and Science
Division of Industrial Metrology
Yuseong, DaeJeon, Korea

KRISS 한국표준과학연구원



Cr-Mo steel.

Δ K control

Crack size measurement : DCPD method

● Fatigue Pre-crack in Air

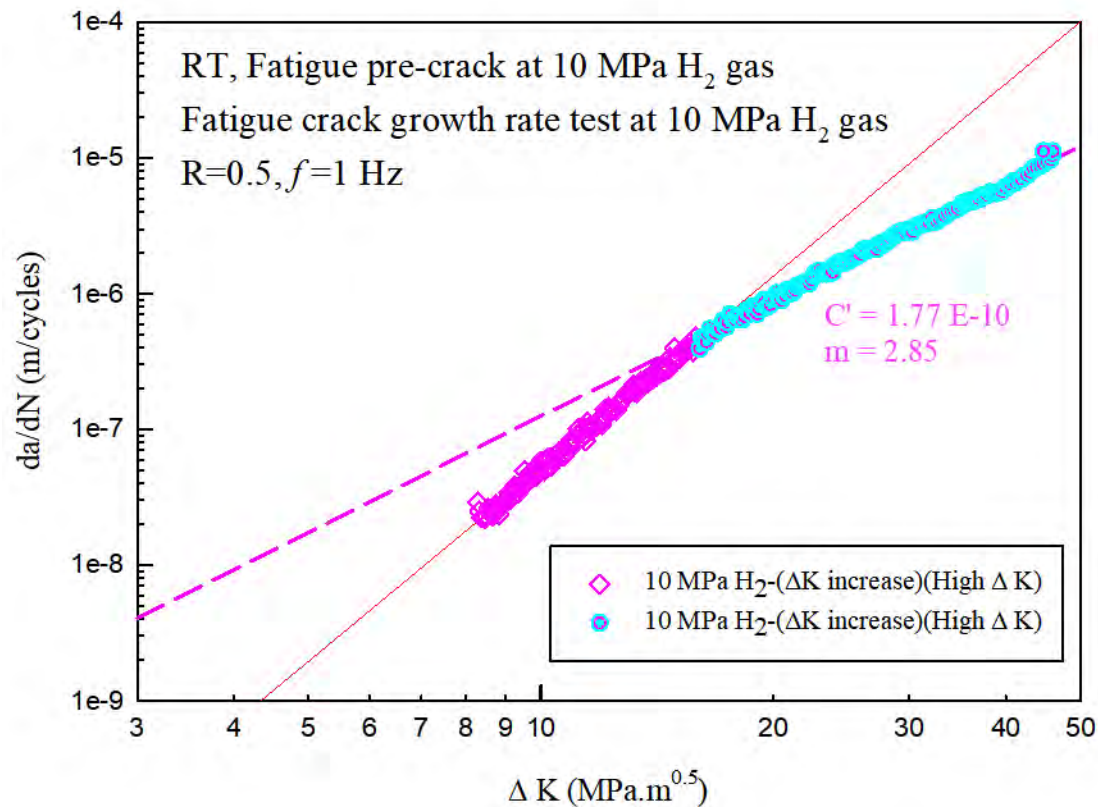
- Loading ratio: **0.1** / Frequency: 10 Hz
- Target K max : **15 MPa.m^{1/2}**
- Initial Notch size : 13 mm
- Target Crack size : 17 mm

● FCGR Test (ΔK Increase) in 10 MPa H₂

- Loading ratio: **0.5** / Frequency: 1 Hz
- Initial K max : **22.353 MPa.m^{1/2}**
- Initial Crack size : 17 mm
- Target Crack size : 21 mm

● FCGR Test (ΔK Decrease) in 10 MPa H₂

- Loading ratio: **0.5** / Frequency: 1 Hz
- Initial K max : **33.335 MPa.m^{1/2}**
- Initial Crack size : 21 mm
- Target Crack size : 35 mm



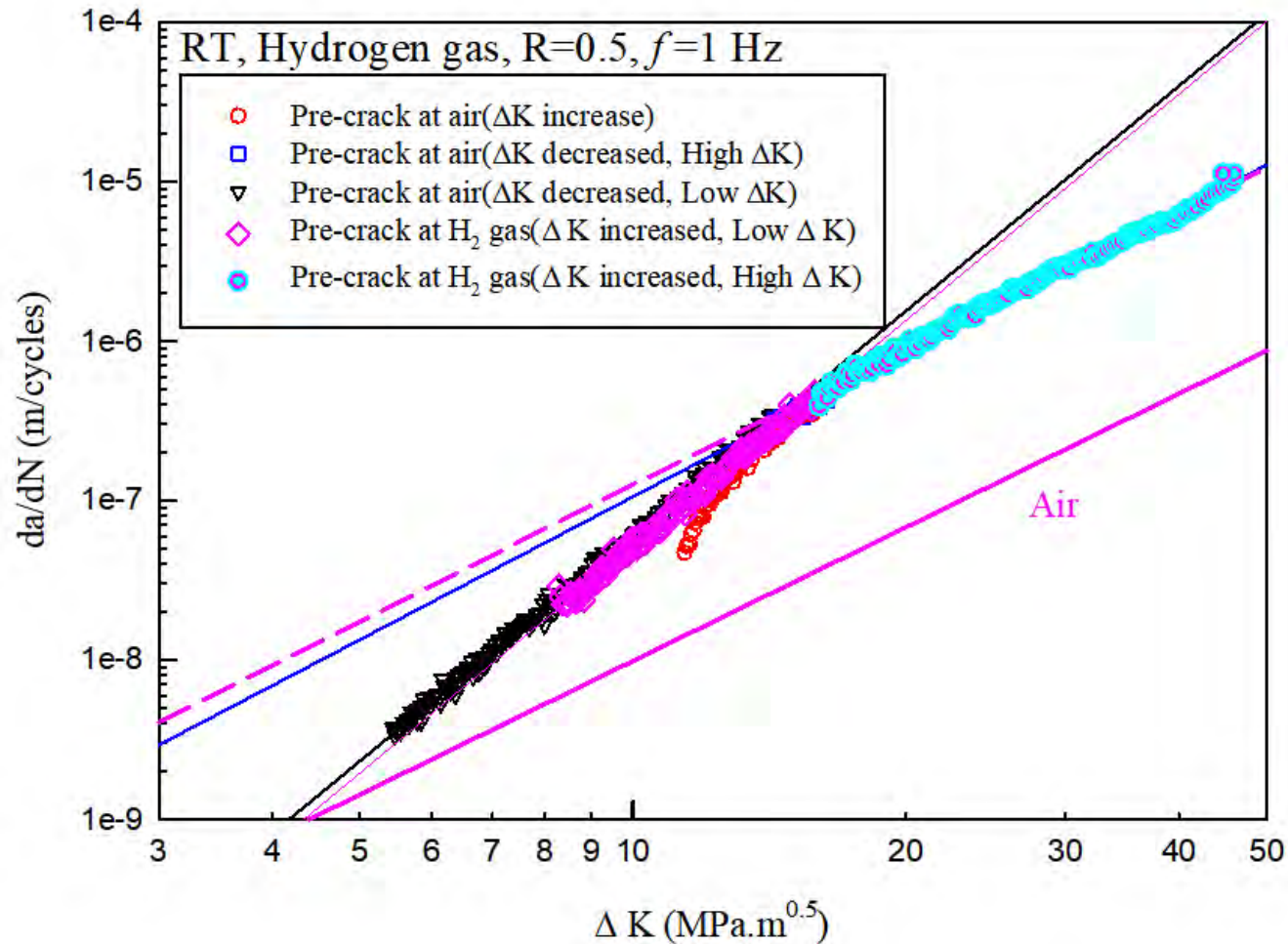
● Fatigue Pre-Crack in 10 MPa H₂

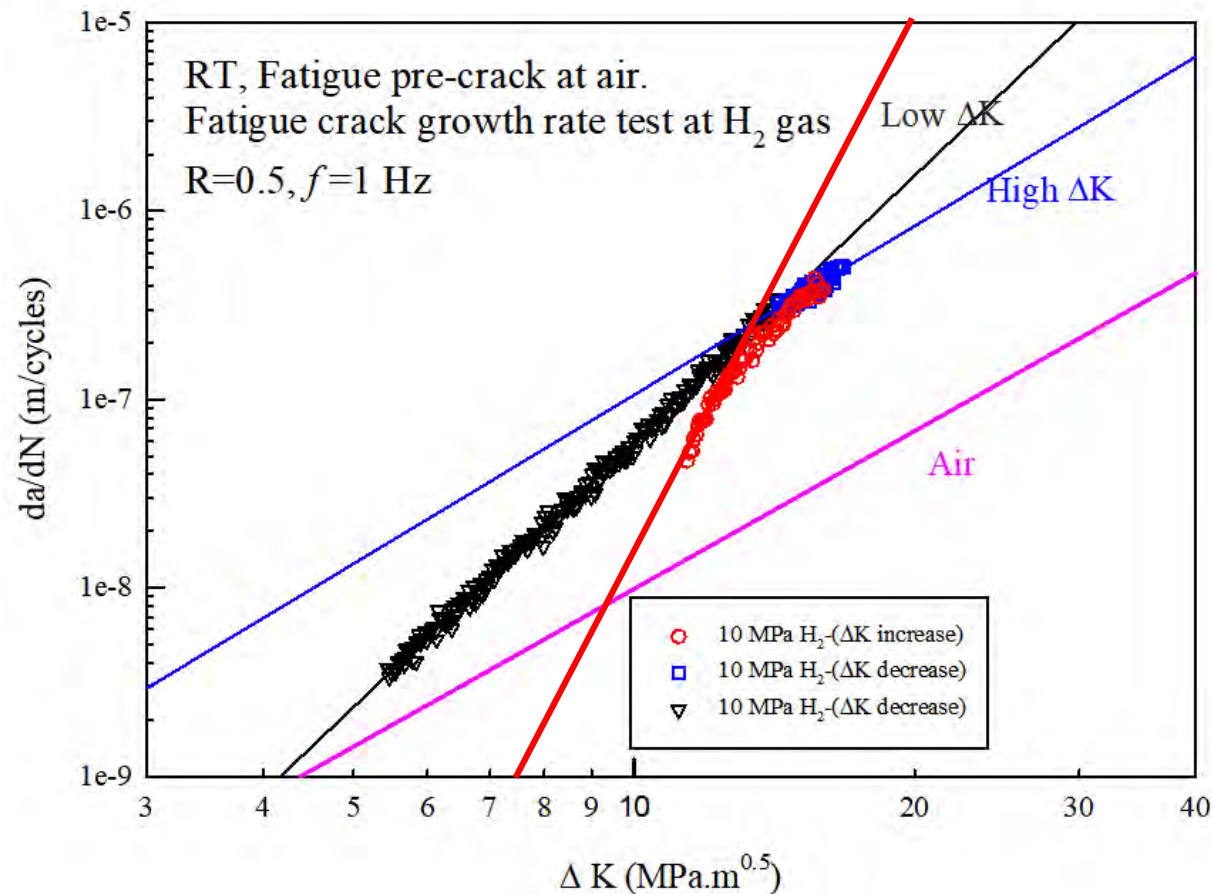
- Loading ratio: **0.1** / Frequency: **2 Hz**
- Target K max : **16 MPa.m^{1/2}**
- Initial Notch size : 13 mm
- Target Crack size : 16 mm

● FCGR Test (ΔK Increase) in 10 MPa H₂

- Loading ratio: **0.5** / Frequency: **1 Hz**
- Initial K max : **16 MPa.m^{1/2}**
- Initial Crack size : 16 mm
- Target Crack size : 35 mm

Repeatability





It is recommend to make pre-cracks in a hydrogen gas environment.



Thank you for your attention !!



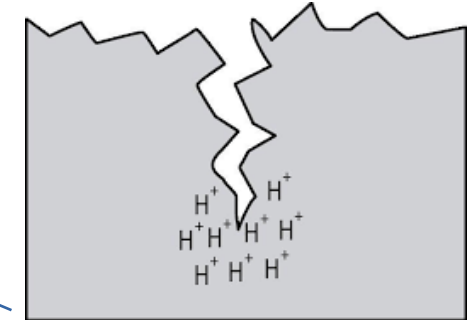
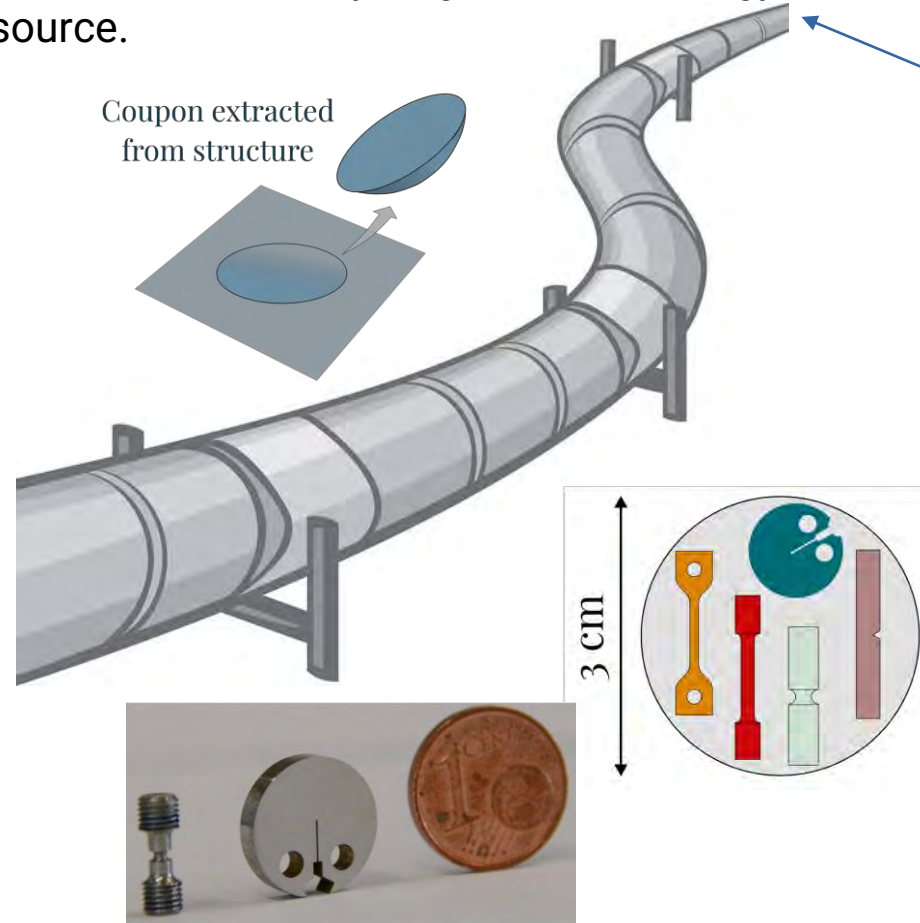
Characterization of Hydrogen Embrittlement of Line Pipe Steels Using Sub-Size Specimens

C. Soret, M. Polo, S. Belkacemi, L. Meirelles Santana,
D. Lopes, P-J. Marchais, F. Bourguignon, J. Besson,
Y. Madi



Background and challenges

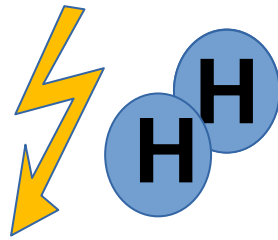
Hydrogen transport using existing dedicated natural gas pipelines (40,000 km in France, values to €50 billion in assets, **80 years**) is a proposal that could allow the use of hydrogen as an energy source.



Effect of HE on existing pipeline steels?



Measure **characteristics of material** in structures from small **coupons**



Hydrogen can be produced with excess energy from other renewable sources, such as wind and solar.

MESSIAH ANR project



Mini-Éprouvettes pour le Suivi en Service des structures avec Application au transport d'Hydrogène

Mini-samples for in-service monitoring of structure with application to hydrogen transportation



Objectifs:

- ✓ Develop a procedure for the mechanical characterization of plastic and fracture behaviour from in-situ millimetric coupons
- ✓ Extend this procedure to study hydrogen embrittlement of structures
- ✓ Propose a methodology for analyzing the results and transferring them to standard specimens and structures

Who is involved?

➡ Partners



Meso-seggregation in large forged parts.



End-user — gas transportation ($\text{CH}_4 + \text{H}_2$ in existing pipes).



H_2 production, storage and transportation.



Tube producer



Code developper and distributor



➡ PhD students

Numerical
tools and
POC

- A. El Tuhami Ouazani : Non-local GTN model with remeshing (finished 2022)
- C. Belhadj : Testing protocol for mini-specimens — test database (finished 2022)

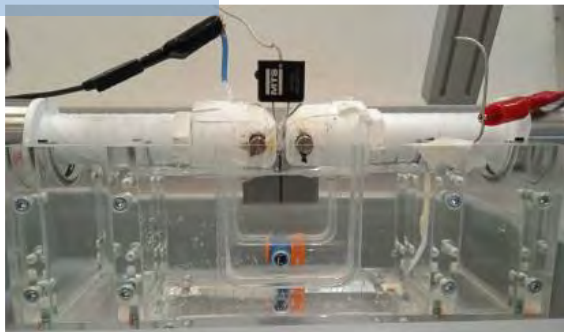
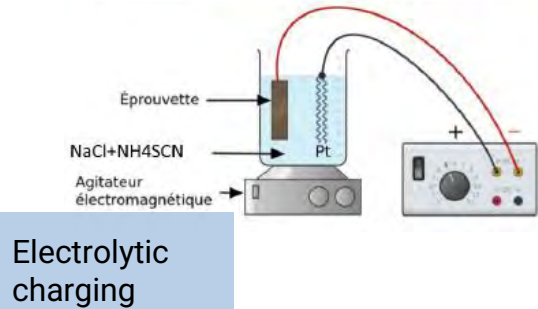
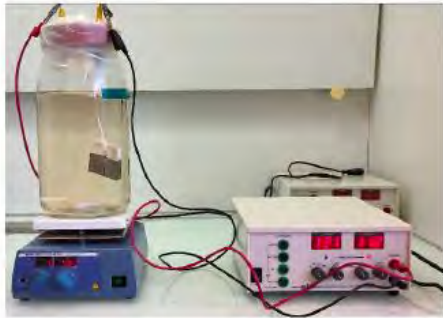
A. Christal : Master Curve in segregated C–Mn steels (started 2020)

S. Belkacemi : Study of welds in a X52 “vintage” line pipe steel (started 2022)

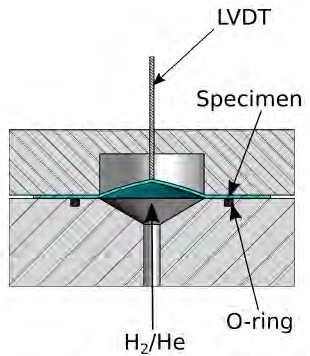
L. Meirelles Santana : Comparison of a “vintage” and a “modern” X52 steel (started 2022).

D. Lopes Pinto : FE simulation of the effect of H on plasticity and fracture (started 2022).

Hydrogen experiments: MESSIAH Project



Gaseous charging



Different methods of charging

1. Mechanical testing under gas pressure (in-situ) : **Mines (Subsize)**, GRTgaz (Standard), Air liquide (Disc)
2. Electrolytic charging then mechanical testing (ex-situ) : **Mines** (standard)
3. Hydrogen electrolytic concurrent charging technique (in-situ) : **Mines** (standard)

Focus on H₂ gas charging methodology

Mini-samples and H₂ Gaseous experiments (CMAT)



Vertical gas
pressurized tensile
test machine for sub-
size specimens

- Small pressurized chamber (1.8 liters, up to 250 bar) with 4 Sapphire windows
- Backlighting (shadow images)
- ATEX environment not needed
- Load cell of 5kN inside the chamber
- High resolution displacement Gauge
- Small rate (0,3µm/min)
- Gas analysis: O₂ and H₂O measurement impurities

Gas analysis equipments



O₂



H₂O

Analysis of Oxygen:

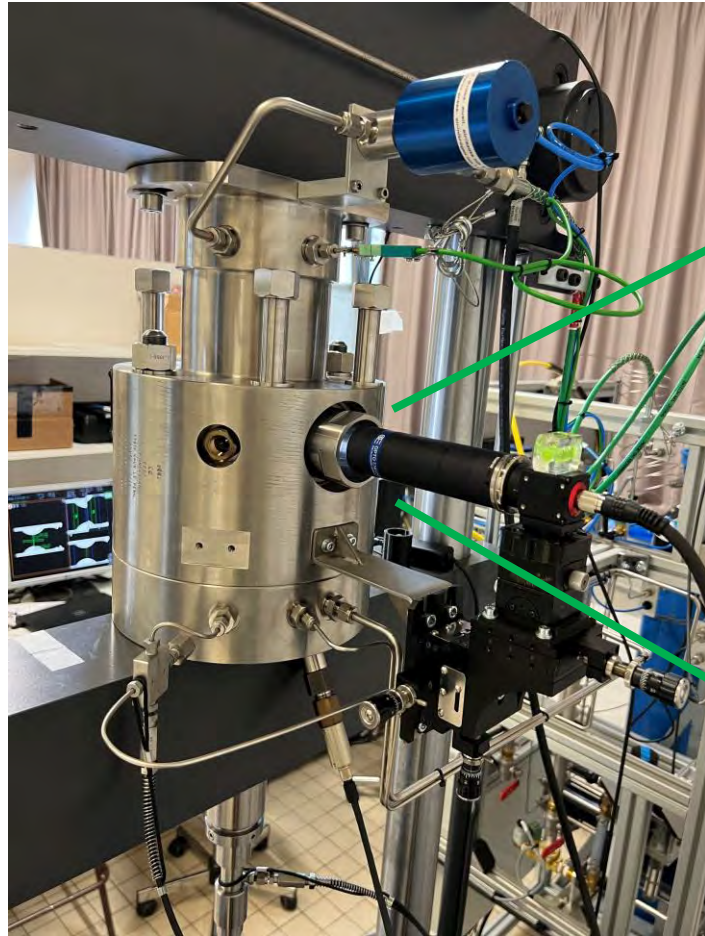
- ✓ Analysis of oxygen concentration with a resolution of up to 0.1 ppm
- ✓ Two electrochemical analysis cells (bypass system):
 - Percent cell: analyzes for high O₂ % content
 - Accurate RACE cell: analyzes below 200 ppm

Moisture

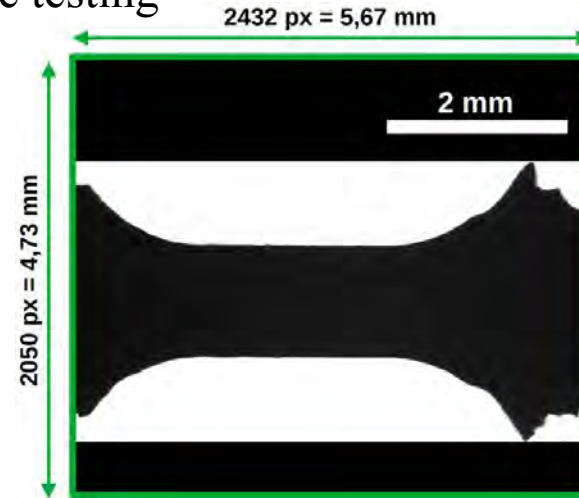
- ✓ Stainless steel drying head with reduced volume
- ✓ Rapid measurements by transitioning from a dry environment to a more humid environment
- ✓ Accuracy of $\pm 2^{\circ}\text{C}$ dew point ($\sim \pm 1,6$ ppm)

Measurements by imposing a small leak flow
Real time data for both equipments

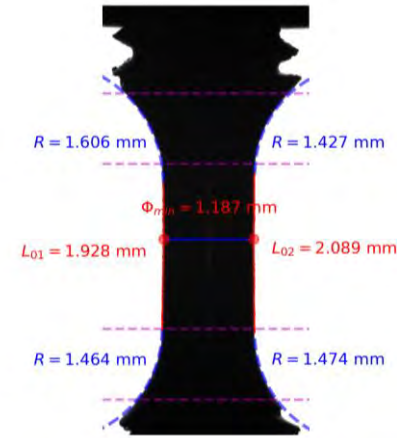
Optical extensometer methodology (Edge Tracing technique ET)



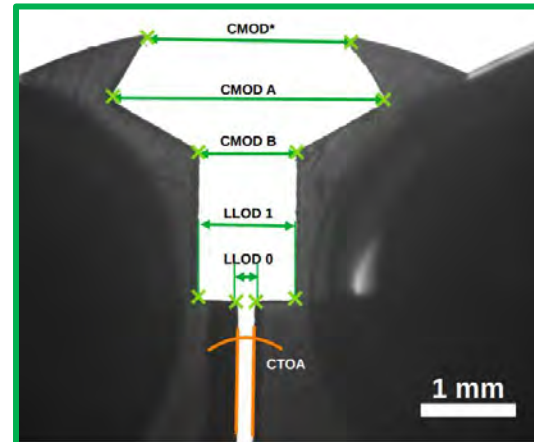
Tensile testing



Metrology

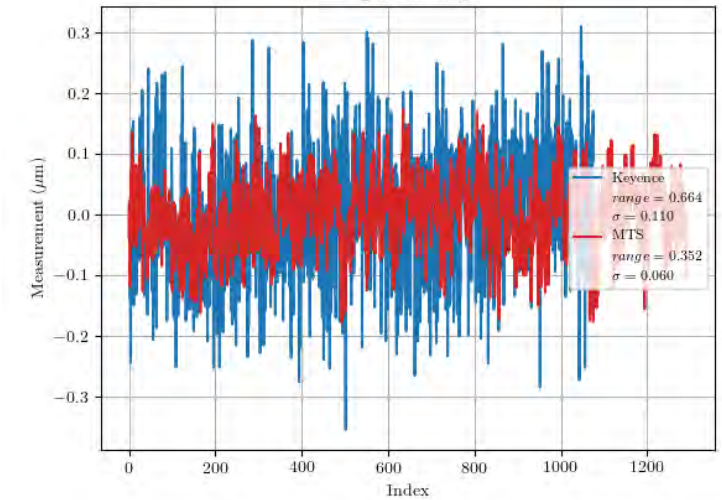


ET
[Shokeir et al., FFEMS, 2022]



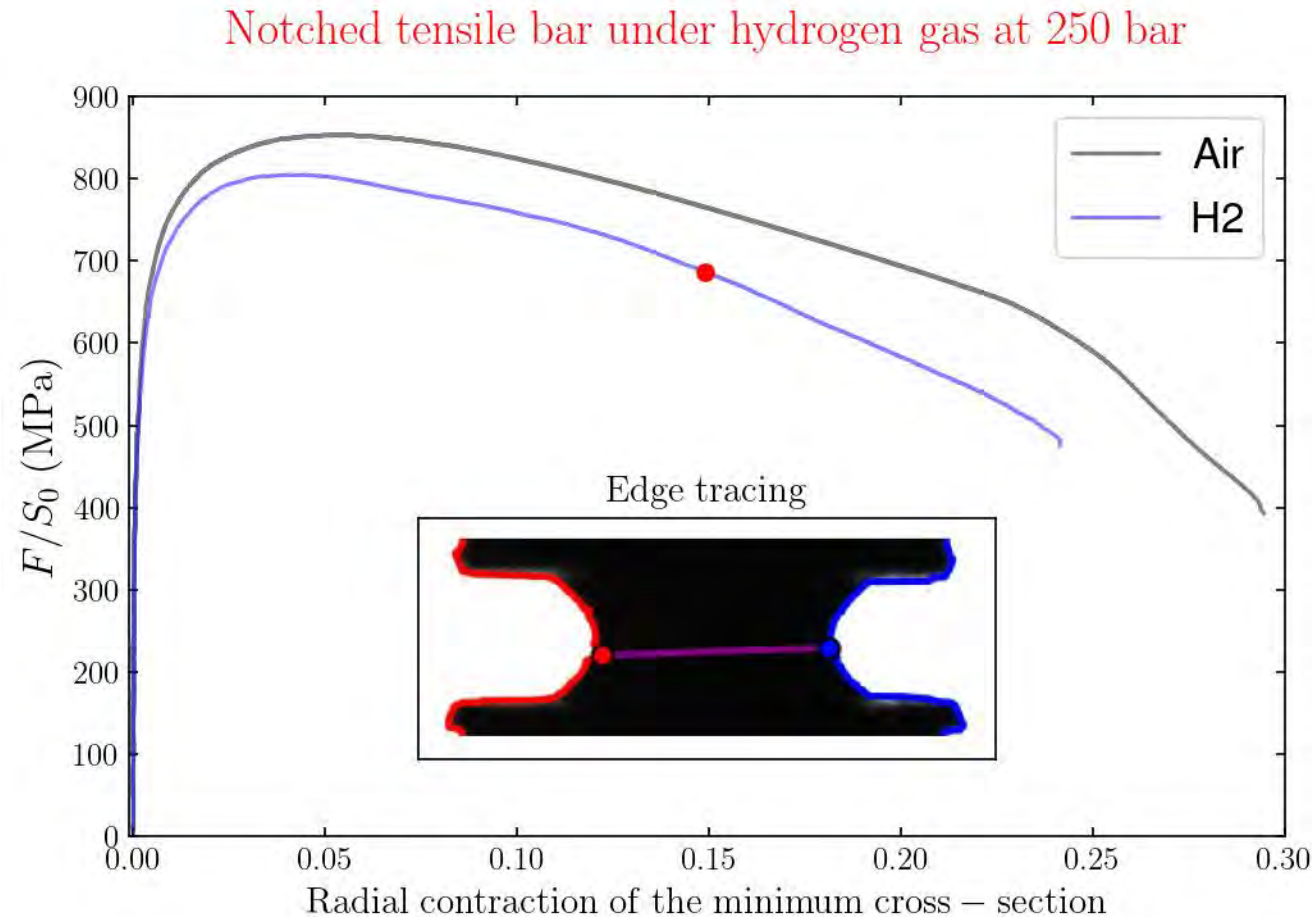
Toughness testing

Signal stability



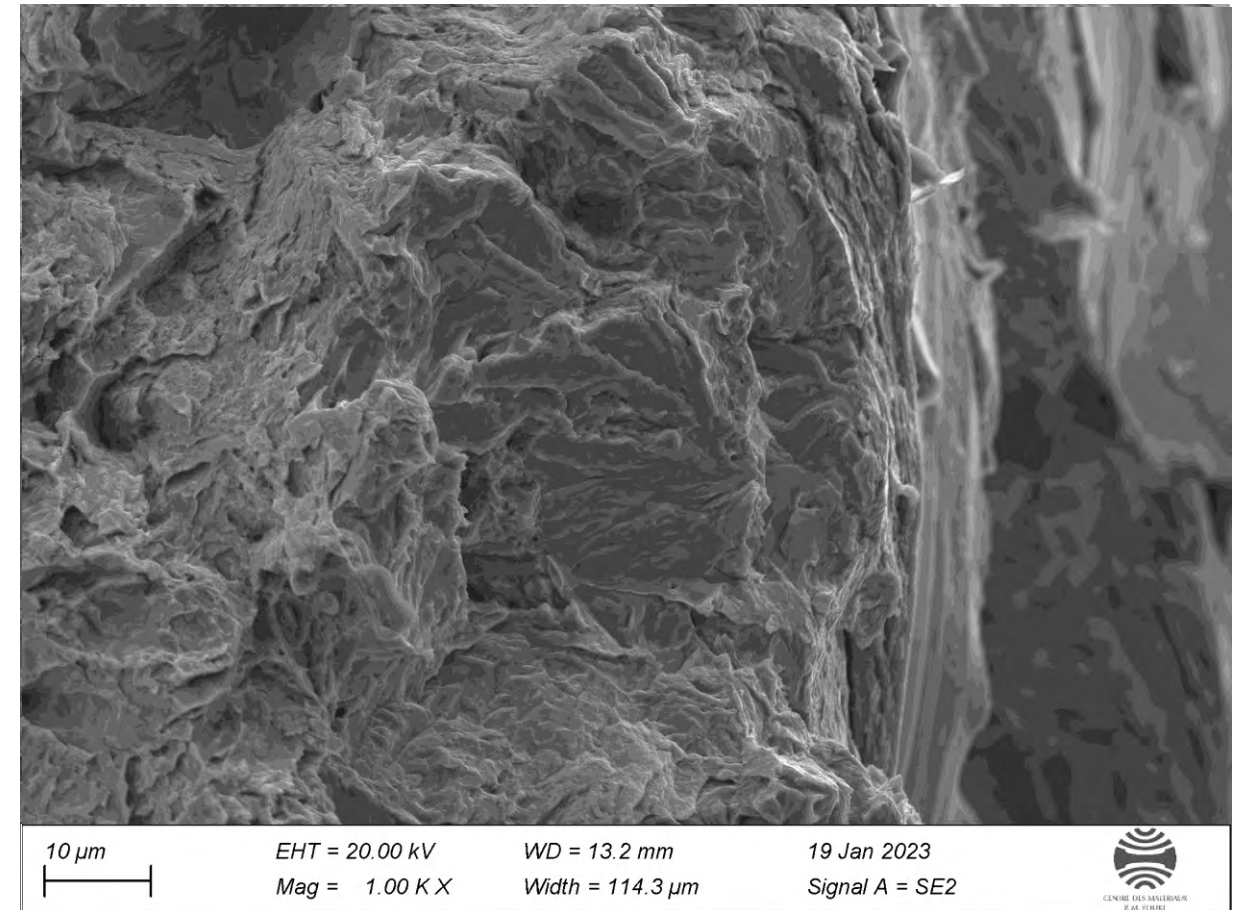
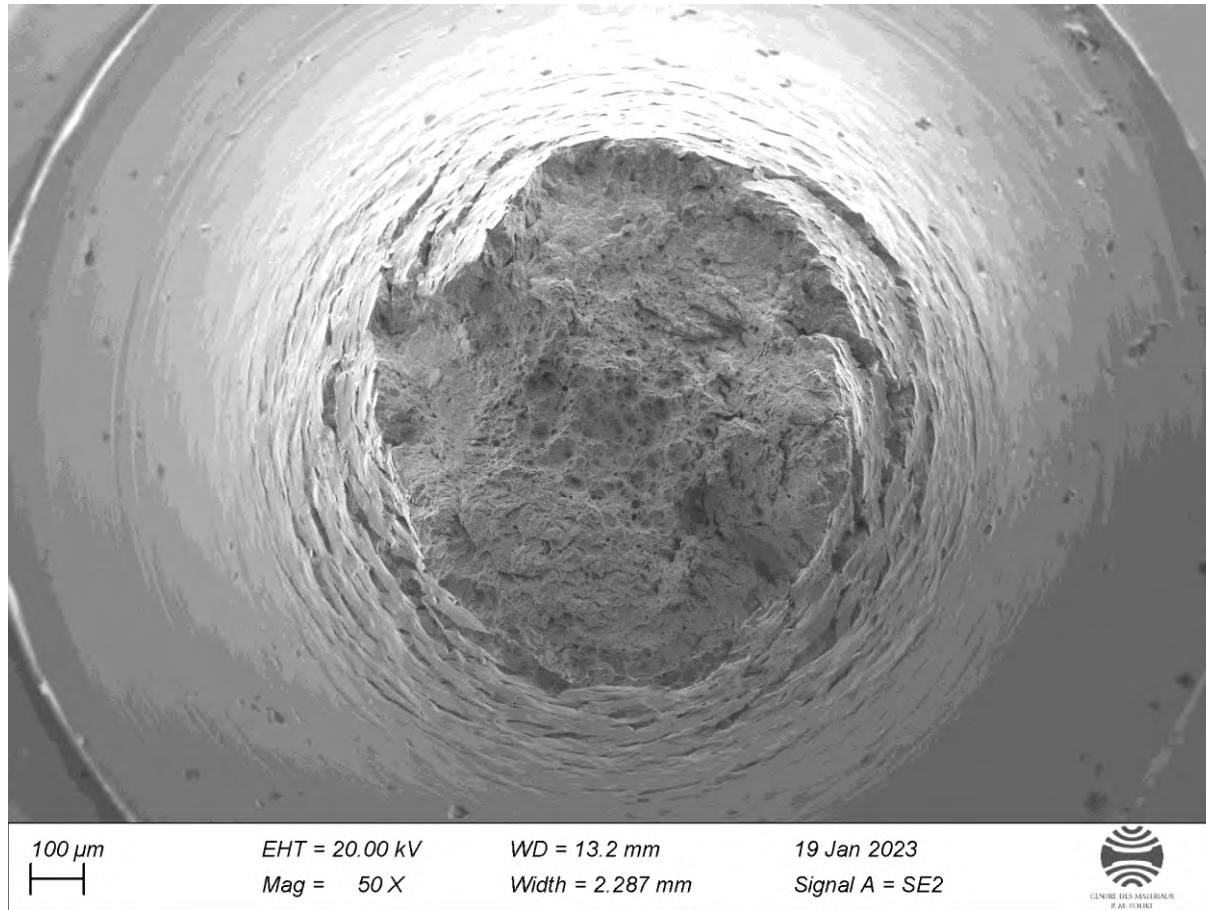
Signal stability
(resolution: $0.1 \mu\text{m}$)

First H₂ gaseous test at CMAT (E355 modern steel)



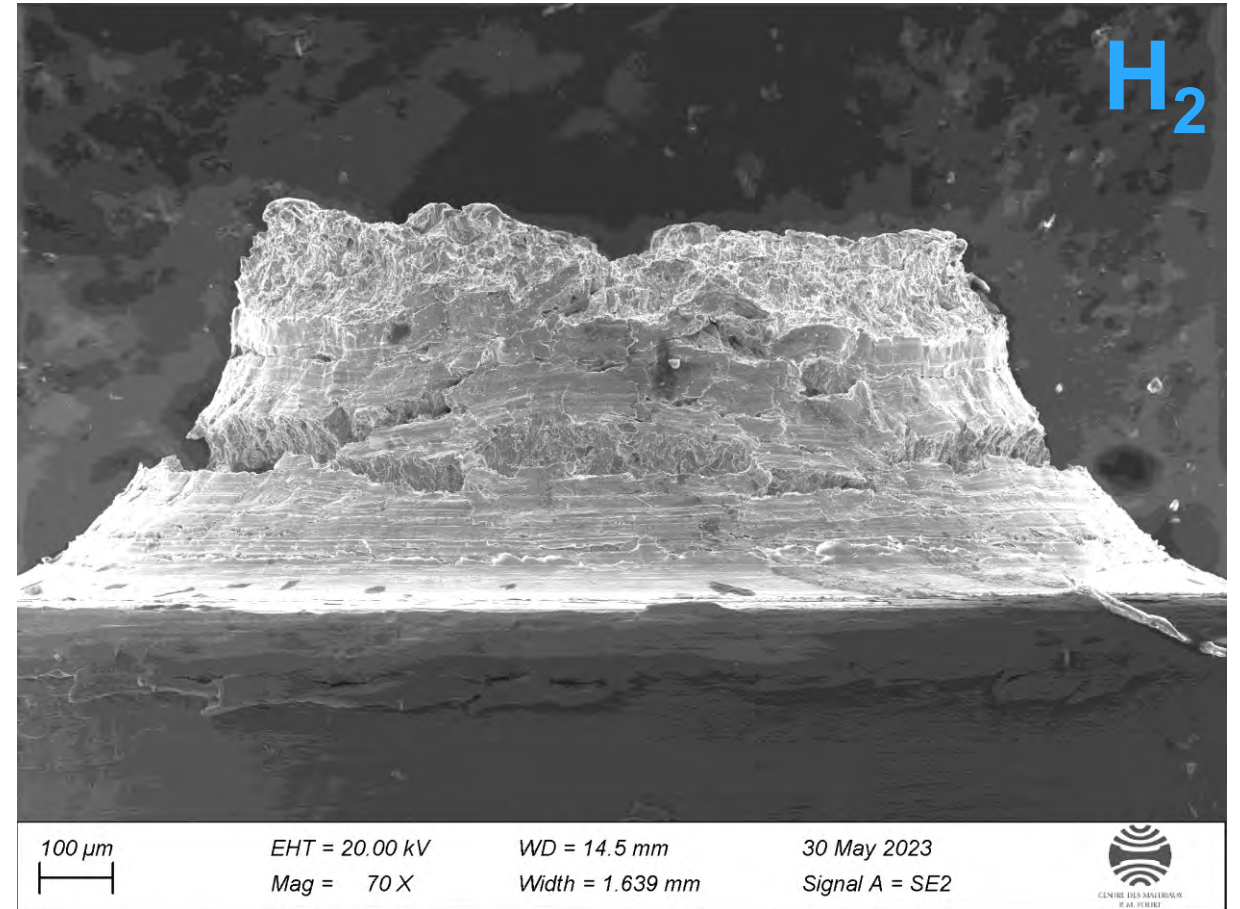
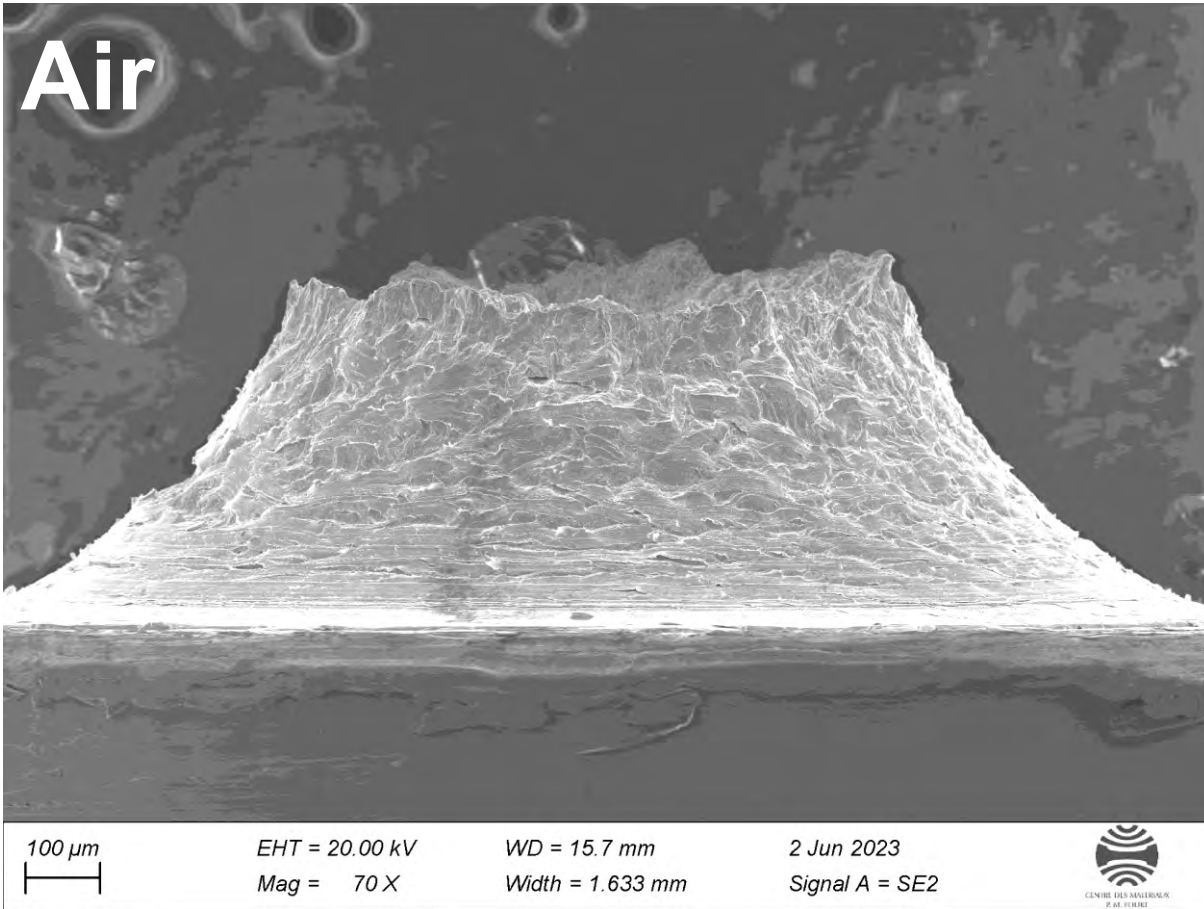
Focus on the left notch

SEM fracture surface



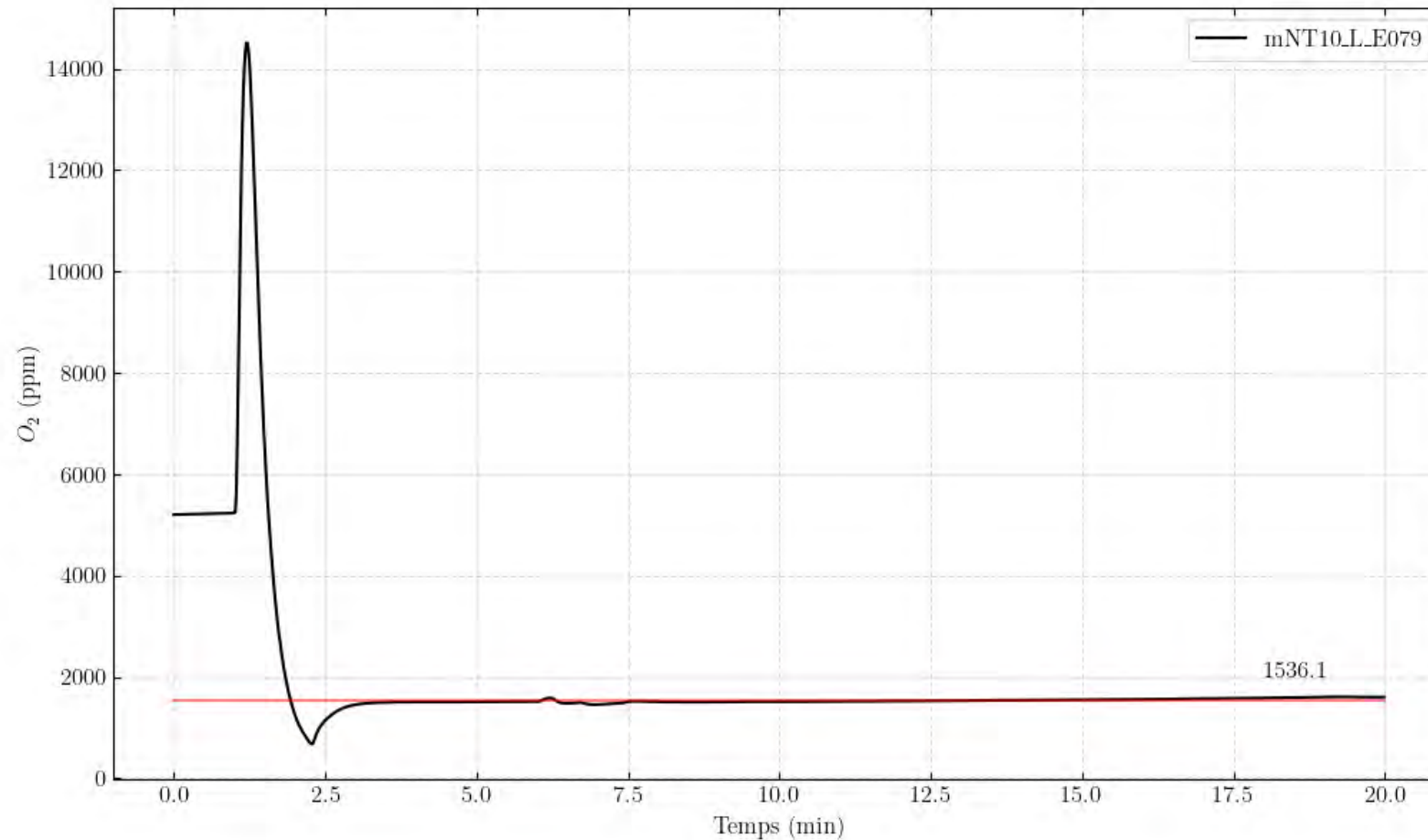
Ductile fracture in the center, quasi-brittle fracture near the external surface

SEM Side views



Formation of multiple cracks from different
free surfaces

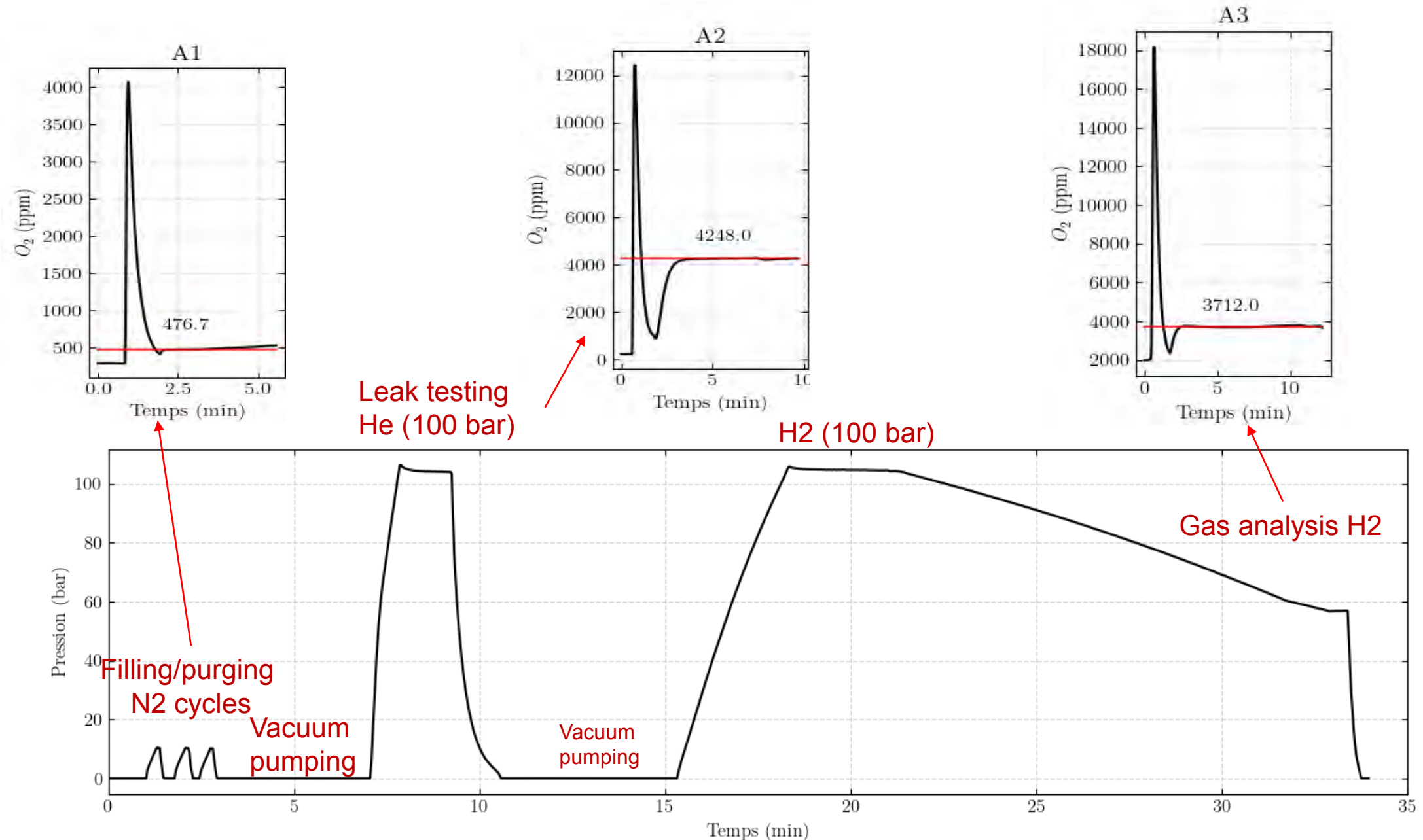
Gas analysis: O₂



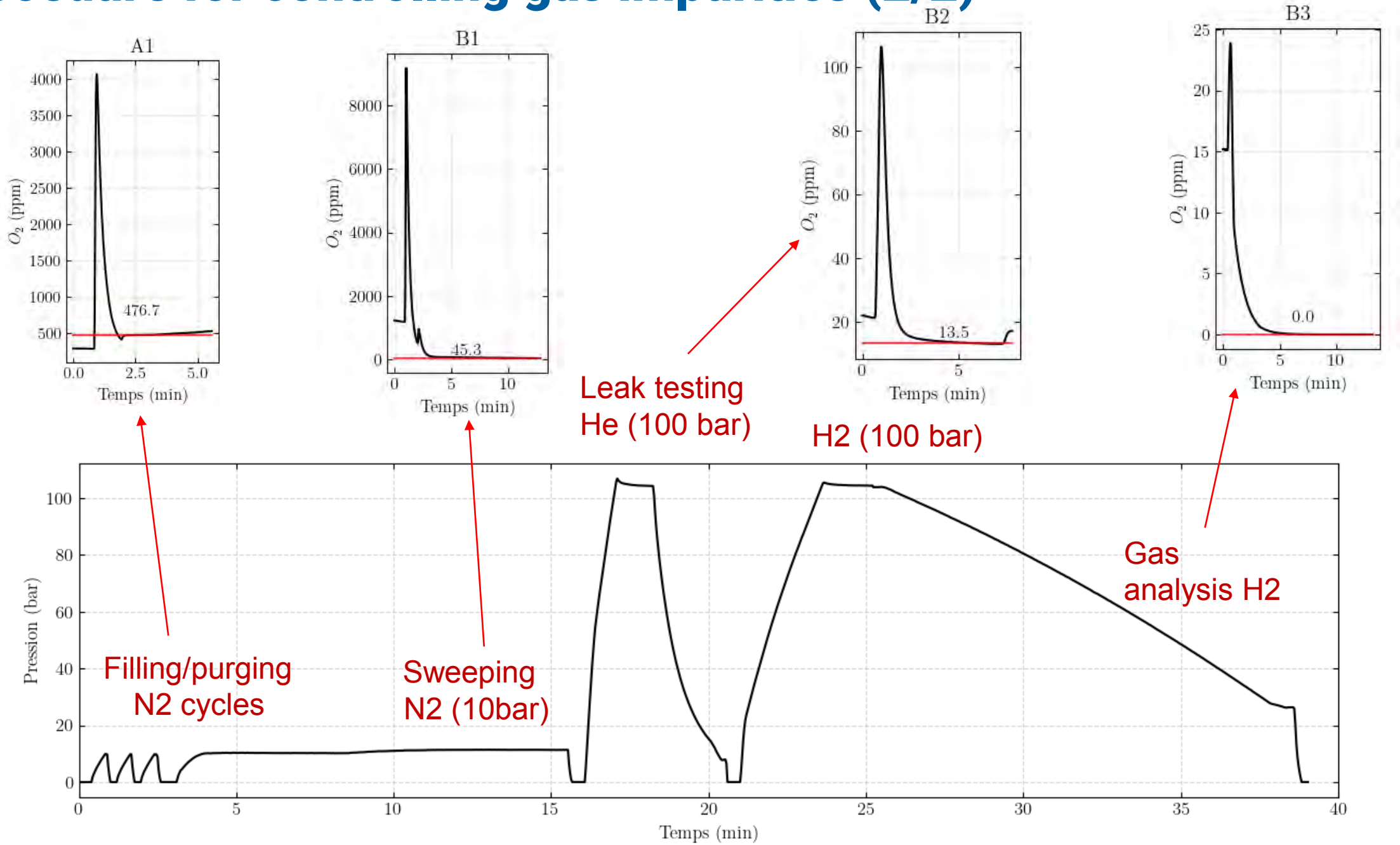
The gas analysis after the mechanical test showed:

An oxygen concentration of 1500 ppm!

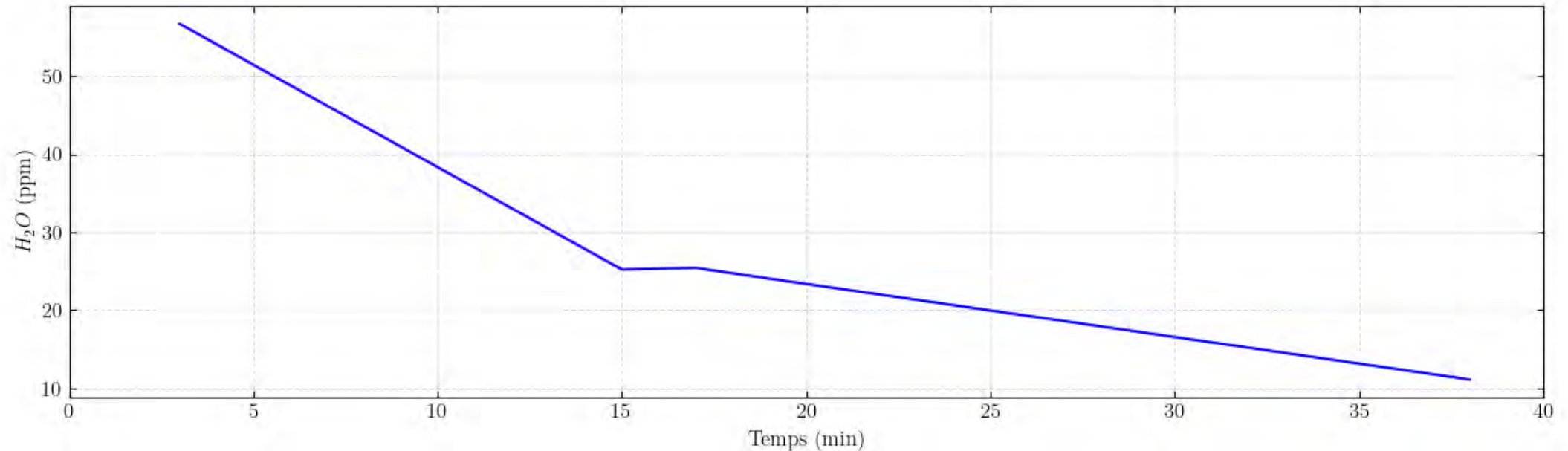
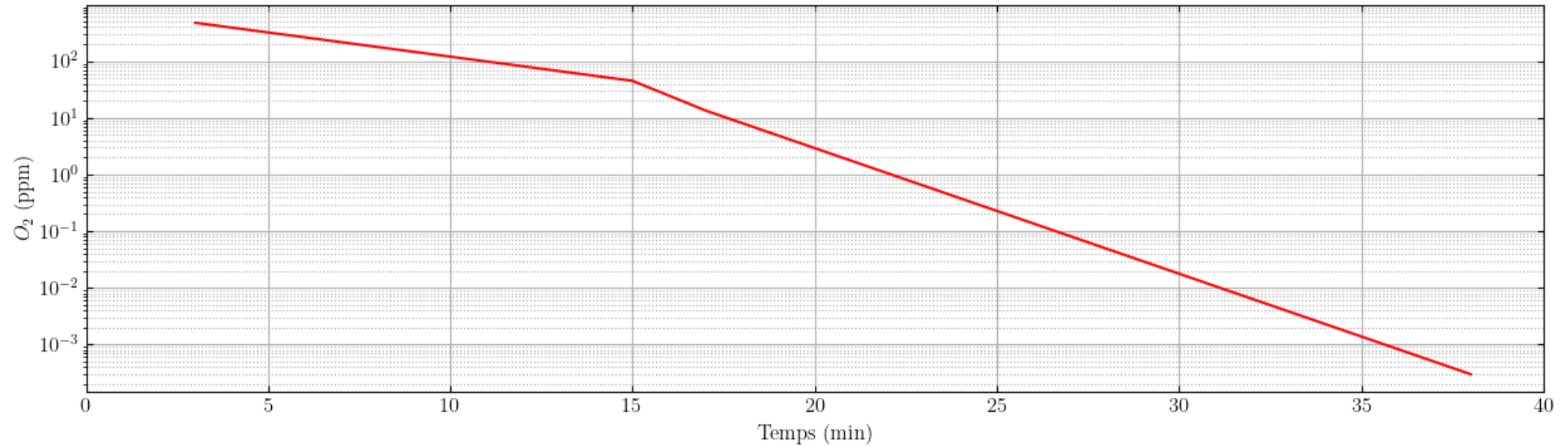
Procedure for controlling gas impurities (1/2)



Procedure for controlling gas impurities (2/2)



Moisture is more difficult to control

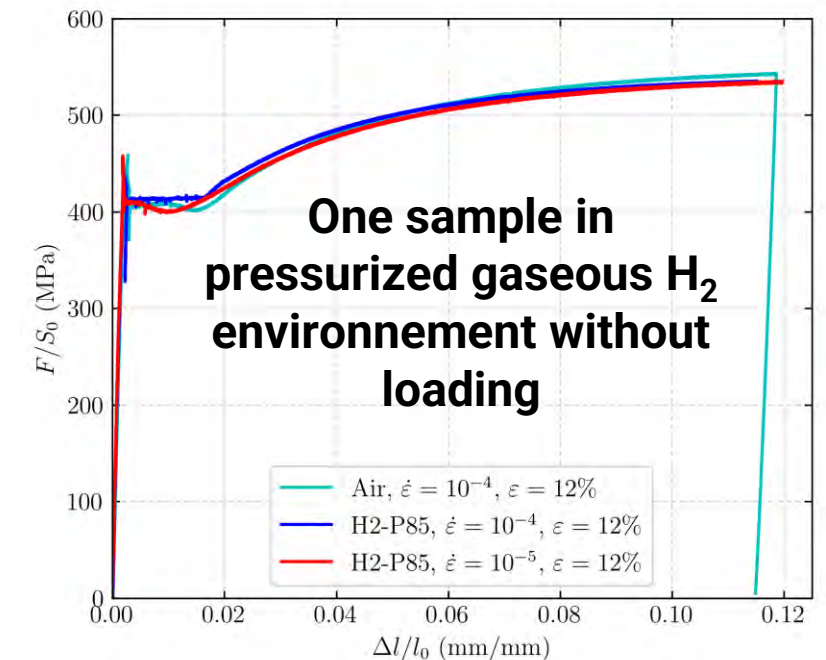
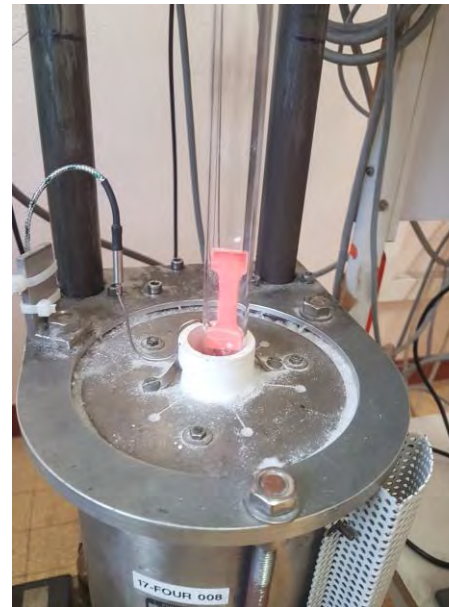
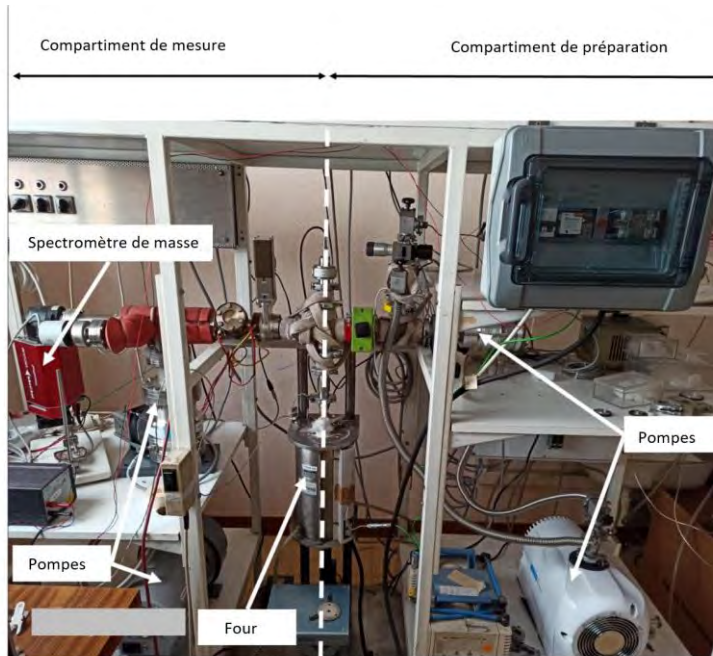


Pre-charging in gaseous H₂ before testing in gas?

- ❑ TDS analysis
- ❑ Material: X52 Vintage linepipe steel
- ❑ 4 samples analysis: Air (1), gaseous pressurized H₂ with and without plastic loading (3)
- ❑ Effect of plasticity? Effect of surface phenomena for gaseous charging method?



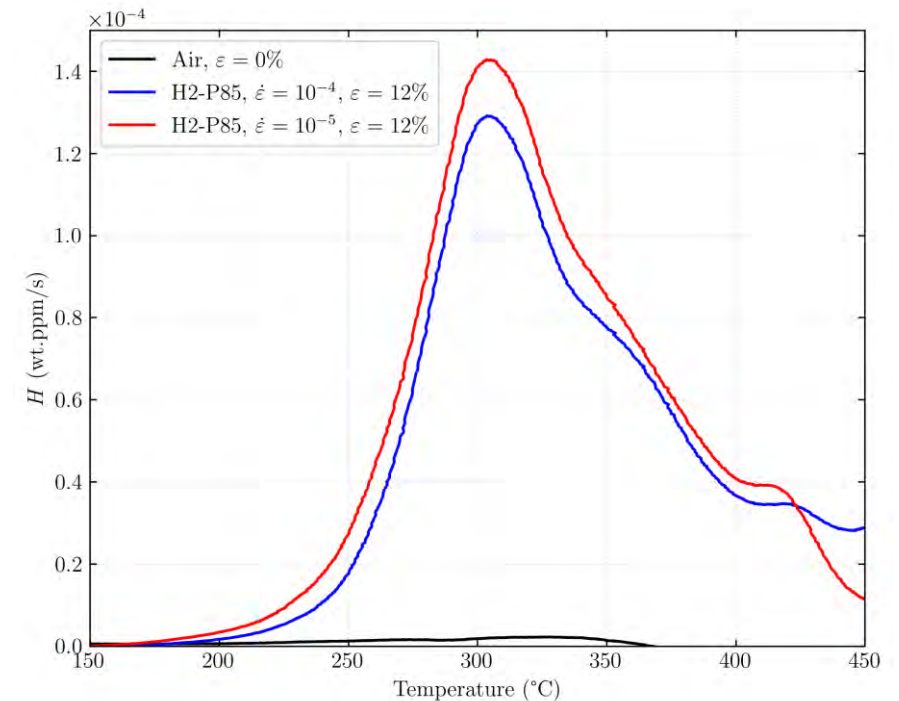
Collaboration
F. Christien



Pre-charging in gaseous H₂ before testing in gas?

- ❑ Sample mass: $L_0=20\text{mm}$, $ep=4\text{mm}$, $m=6,24\text{g}$
- ❑ Surface roughness R_a : less than $1\text{ }\mu\text{m}$
- ❑ Measurement by quadrupole mass (Q-Mass) spectrometry
- ❑ Desorption time before TDS starting: 75 min
- ❑ Heating rate: 10°C/min
- ❑ Diffusible hydrogen: desorbed below 450°C
- ❑ Background subtraction with air reference curve

Charging method	Strain Rate (s^{-1})	Strain	Cumulative H ₂ (wt. ppm)
AIR	10^{-4}	12%	Reference
P85 bar H2	-	-	4×10^{-6}
P85 bar H2	10^{-4}	12%	0.085
P85 bar H2	10^{-5}	12%	0.090



- ❑ **Significantly more hydrogen is observed for a mechanically loaded sample**
- ❑ No effect of strain rate (confirmed on ST samples tested until rupture)

Conclusions – Future works



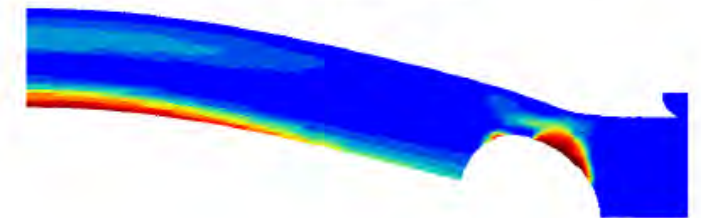
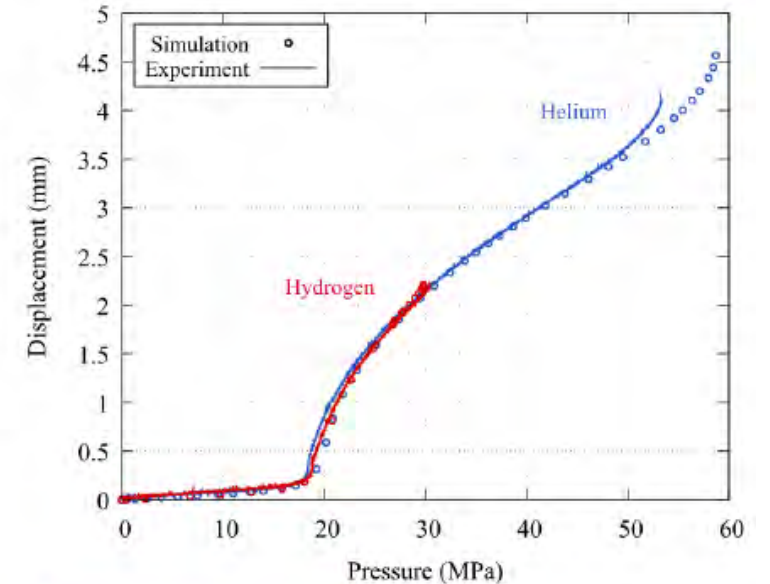
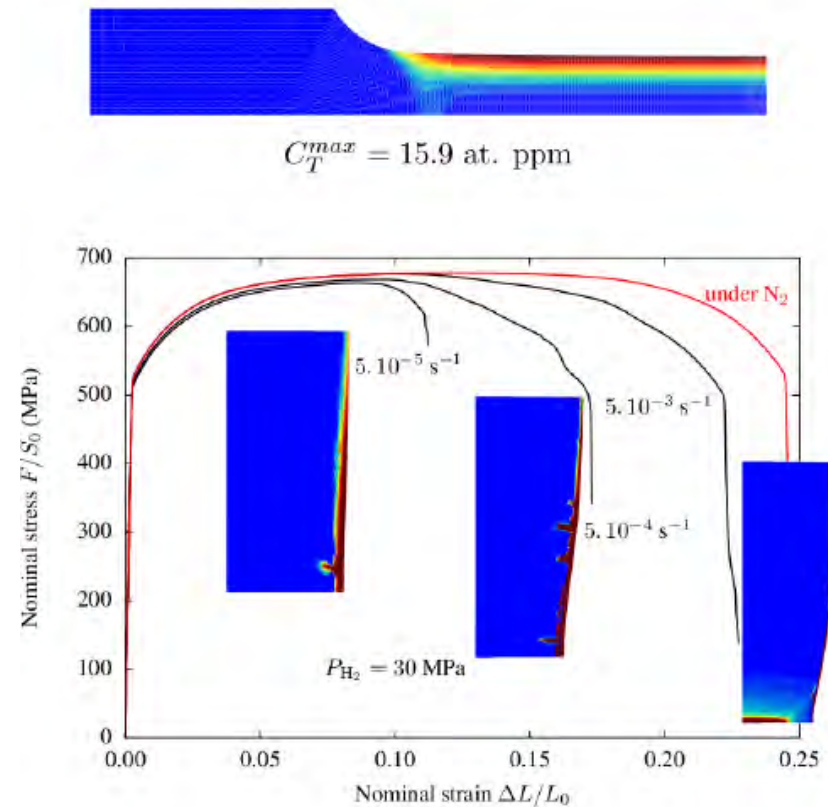
- ✓ **Presentation of H₂ Gas Pressurized Tensile Test Machine for Sub-size Specimens**
 - ✓ Successful application of optical control and measurements in mST/mNT and mDCT experiments
 - ✓ Development of a procedure for controlling the H₂ environment tests using O₂ and H₂O gas analysis measurements
 - ✓ No pre-charging condition is needed before the test. A strong interaction between H₂ diffusion and plasticity is observed → more analyses are needed
- ✓ **Ongoing large-scale experimental campaign**
 - ✓ A comprehensive experimental campaign is currently underway, including (miniature) tensile, (miniature) notch tensile, toughness samples, SPT and disc tests
 - ✓ Most of tests are conducted both with and without hydrogen
- ✓ **Messiah H₂ Modelling developments
(Daniella Lopes-Pinto PhD thesis)**

Modelling of HE: plasticity, damage, and coupling with hydrogen

- ✓ Crack initiation at the surface for hydrogen testing
- ✓ Loss of ductility due to HE



Daniella Lopes





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Chair program MESSIAH
(ANR-20-CHIN-0003)

Thank you, questions?

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