# R&D for Safety, Codes and Standards: Materials and Components Compatibility

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**DOE Hydrogen Program**
**2023 Annual Merit Review and Peer Evaluation Meeting**
**June 5-8th, 2023**

**AMR Project ID# SCS005**
**WBS 6.2.0.801**

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# Project Goal

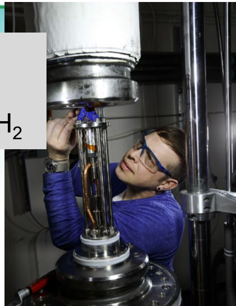
**The hydrogen compatible materials and components project has several objectives:**

1) **Optimize the reliability and efficiency of test methods for structural materials and components in hydrogen gas.**

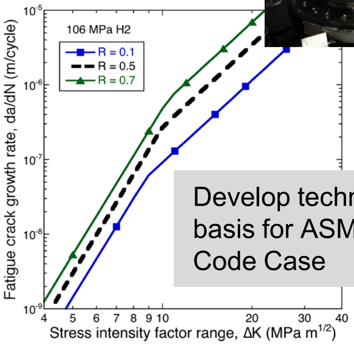
2) **Generate critical hydrogen compatibility data for structural materials to enable technology deployment.**

3) **Create and maintain information resources such as the "Technical Reference for Hydrogen Compatibility of Materials”.**

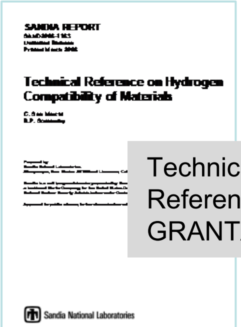
4) **Demonstrate leadership in the international harmonization of standards for qualifying materials and components for service with high-pressure gaseous hydrogen.**



Unique testing capabilities in high pressure $H_2$



Develop technical basis for ASME Code Case



SANDIA REPORT

Technical Reference on Hydrogen Compatibility of Materials

Sandia National Laboratories

Technical Reference and GRANTA database

# Overview

## Timeline

- Project start date: Oct 2003
- Project end date: Sept 2023*

* Project continuation and direction determined by DOE annually

## Budget

- FY22 DOE Funding: $441K
- FY23 DOE Funding: $676K
    - Tank life extension tasks operating with carryover funds

## Barriers

A. Safety Data and Information: Limited Access and Availability

F. Enabling national and international markets requires consistent RCS

G. Insufficient technical data to revise standards

## Partners

- **SDO/CDO participation:** CSA, ASME, SAE, ISO
- **Industry:** FIBA Technologies, Tenaris-Dalmine, JSW, Swagelok, NASA-WSTF, Hexagon Digital Wave, Luna Innovations Inc.
- **International engagement:** AIST-Tsukuba (Japan), I2CNER (Kyushu University, Japan), MPA Stuttgart (Germany), KRISS (Korea)

# Relevance and Impact

*Objective*: Enable technology deployment by **performing and applying foundational research toward the development of science-based codes and standards** that enable the deployment of hydrogen technologies

| Goals from SCS 2021 report | Impacts |
|---|---|
| Facilitating the creation, adoption, and harmonization of regulations, codes, and standards (RCS) for hydrogen and fuel cell technologies | • **Provided technical basis for Code Case: Hydrogen Crack Growth Rate Constants for B31.12 Pipeline Materials** – Functional design curves accommodate pressure and load ratio<br>• Leadership in international codes related to hydrogen compatibility of materials<br>  • UN GTR and R134<br>  • ISO 197/WG 29 for Appendix C of ISO 15916/JWG 30<br>  • ISO TC164/SC 1/WG9 technical reviews on new test method for testing in H2<br>  • SAE 2579 and companion TIR (in development) |
| Conducting research to generate the valid scientific bases needed to define requirements in developing RCS | • **Accepted ASME PVP Conference Papers** *July 2023*<br>  • Demonstrated that fatigue and fracture measurements are independent of orientation and location in high-quality thick-walled pressure vessel steels in hydrogen gas<br>  • Demonstrated that pressure corrections to ASME design curves fit fatigue data for pressure vessel steels tested at lower pressure (550 bar) providing technical basis for code changes that would remove over-conservatism of ASME code.<br>  • Extended use of cross-hole tube geometry testing to allow evaluation of material in final product form (material: XM-19, a high strength alternative to 316L).<br>    • Measured negligible influence of internal hydrogen on crack initiation and cycles to failure over range of frequencies, establishing basis for expedited testing. |

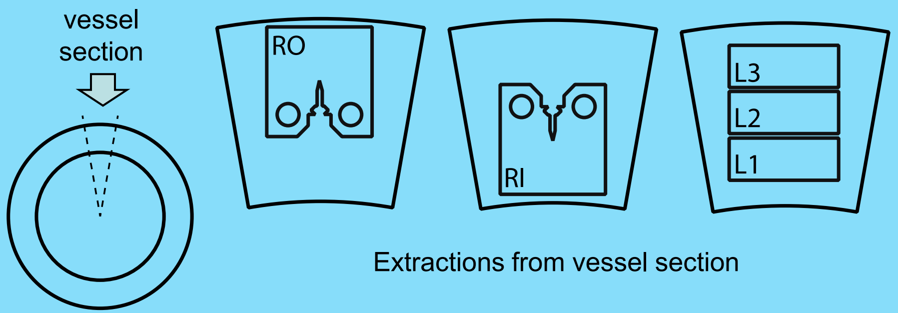
# Project Approach and Milestones

| FY23 Milestone | Status |
|---|---|
| Develop the technical basis for a pressure compensated fatigue crack growth relationship for ASME B31.12 code case for hydrogen pipeline | • **Developed technical basis for Code Case: Hydrogen Crack Growth Rate Constants for B31.12 Pipeline Materials**<br>    • Establishes functional design curves that accommodate pressure and load ratio<br>    • ***Presented to ASME B31.12 code committee***<br>• *Related outcome*<br>    - Published ASME PVP Conference paper showing ASME Code Case 2938 design curves with pressure correction fit pressure vessel data at 1000 bar and 550 bar<br>        o Provides technical basis for modification of code to allow pressure correction of fatigue design curves<br>        o Current code provides pressure-independent design curves for pressure of 1,000 bar<br>        o Pressure correction formulation for high pressure (400-1,000 bar - PVs) is necessarily different than at low pressure (< 200 bar – pipelines). |

# Approach

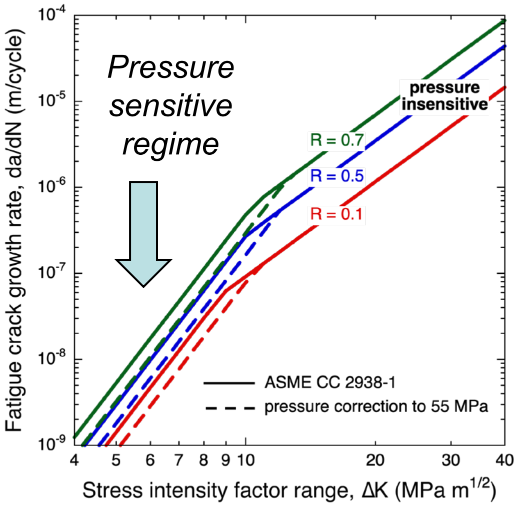
## Advancing technical basis for fatigue design rules and test methods through critical assessment & comprehensive testing in gaseous hydrogen environments

### 1) Are fatigue/fracture behavior dependent on orientation and location in quench & tempered pressure vessel steels?



***Approach:***
**Perform fatigue/fracture tests at different locations/orientations within thick-walled pressure vessel in gaseous hydrogen**

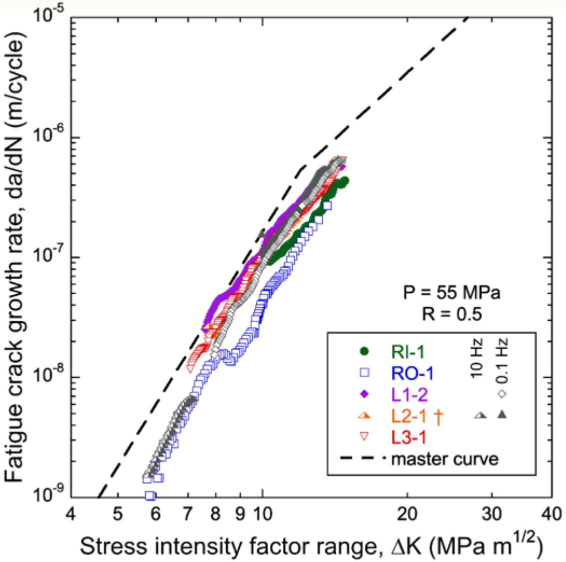
### 2) Can pressure corrections to ASME design curve be justified for lower pressures?

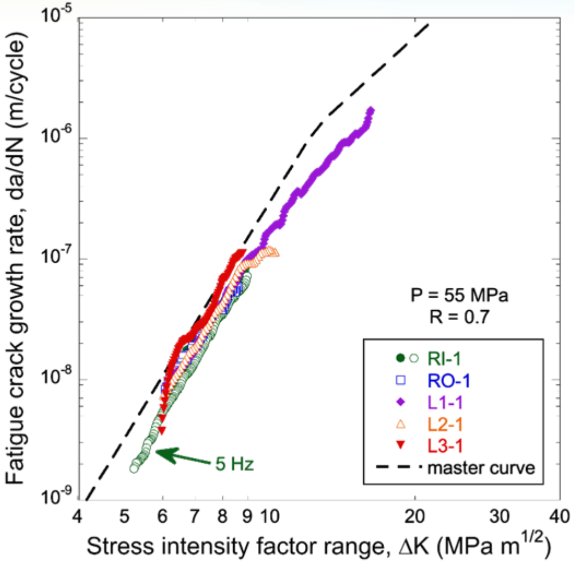


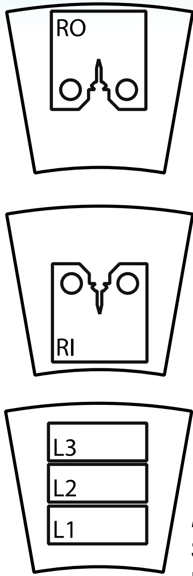
***Approach:***
**Measure fatigue crack growth rate at lower pressures for pressure vessel steels**

# Accomplishment

## Demonstrated fatigue and fracture in thick-walled pressure vessels can be independent of orientation and location







*Ref*: Bortot, Ortolani, San Marchi, Ronevich PVP2023-106417

**<u>Impact</u>: Addresses uncertainty about the uniformity of fatigue and fracture properties in thick-walled pressure vessels**

# Accomplishment

## Developed pressure-dependent fatigue design curves for hydrogen pressure vessel (Q&T) steels

At high ΔK:

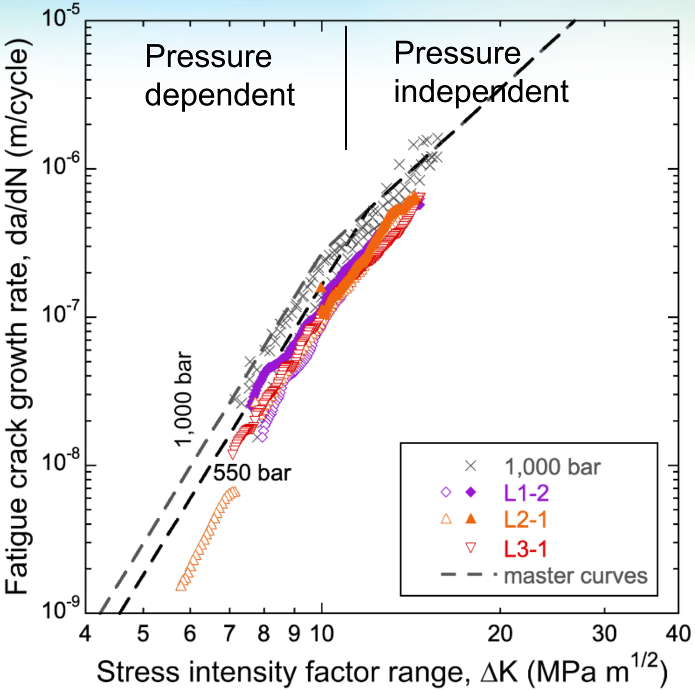
$$\frac{da}{dN} = 1.5x10^{-11}\left[\frac{1+2R}{1-R}\right]\Delta K^{3.66}$$

At low ΔK:

$$\frac{da}{dN} = 3.5x10^{-14}\left[\frac{1+0.4286R}{1-R}\right]\Delta K^{6.5} f(P)$$

$$f(P[bar]) = 0.19 + 0.000763\ P$$

(for $400 < P\ (\text{bar}) < 1{,}100$)

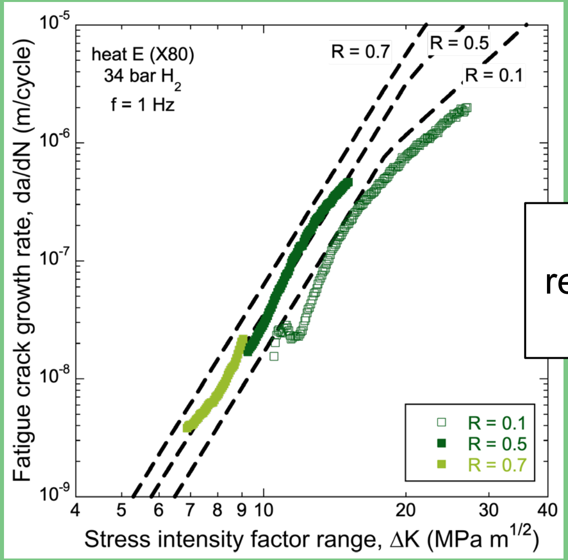


**Impact:** Provides technical basis for modification of code to allow pressure correction of fatigue design curves

# Accomplishment

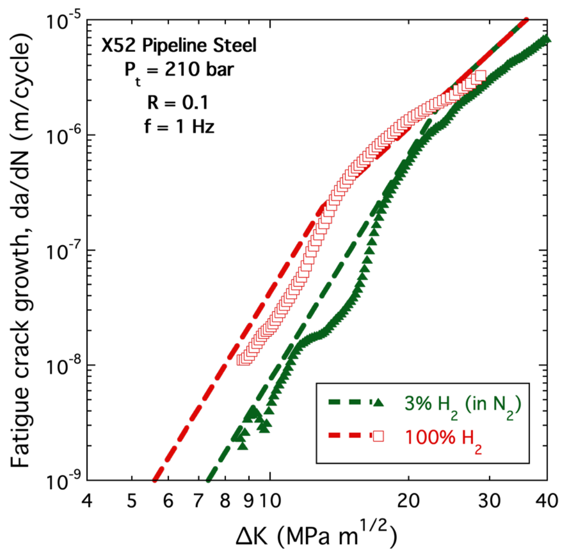
## Developed technical basis for fatigue design curves dependent on load-ratio and pressure for pipeline steels and presented to ASME B31.12 committee

### Load-ratio dependence



Dashed lines represent fatigue design curves

### Pressure dependence



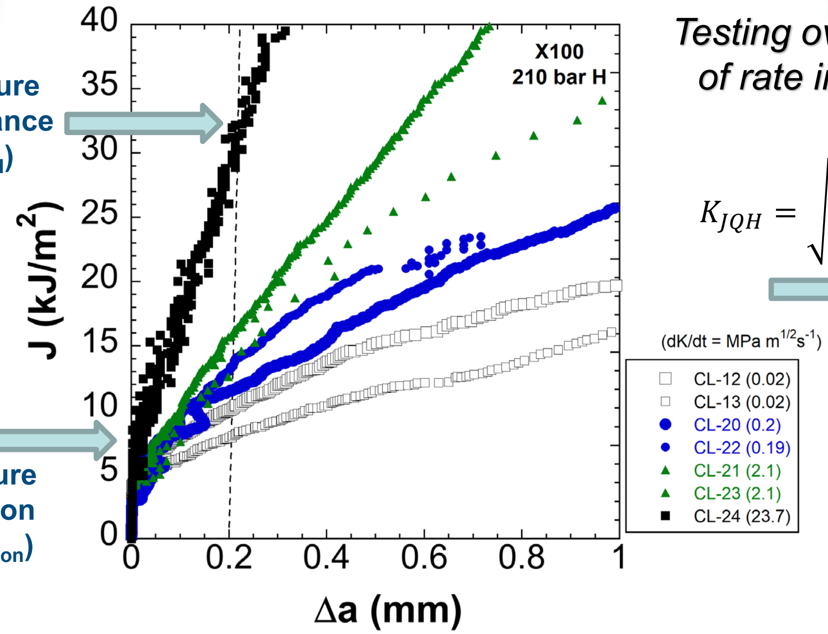
**Impact**: Provides simple relationships that reduce testing burden for structural integrity assessment of hydrogen pipelines

# Accomplishment

## Fracture resistance ($K_{JQH}$) is a function of testing rate; however fracture initiation ($K_{initiation}$) is less rate sensitive

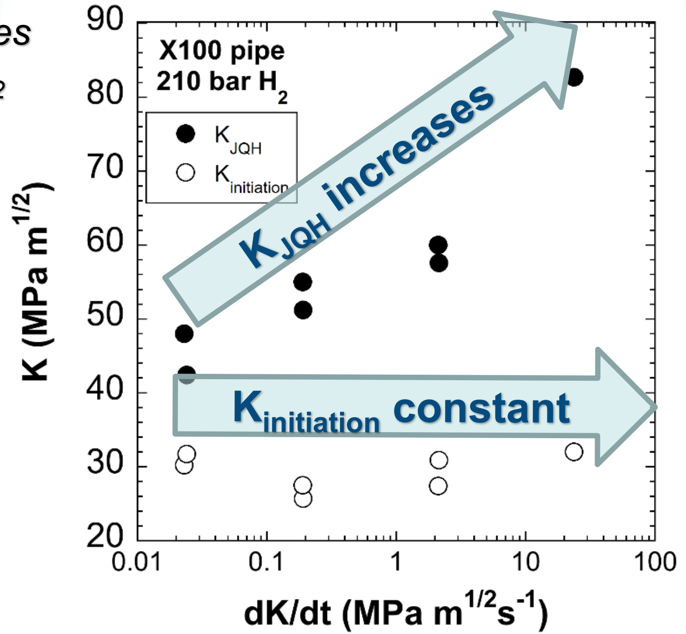
**Fracture resistance ($J_{QH}$)**

**Fracture initiation ($J_{initiaion}$)**



*Testing over 3 decades of rate in 210 bar $H_2$*

$$K_{JQH} = \sqrt{\frac{EJ_{QH}}{(1-\nu^2)}}$$

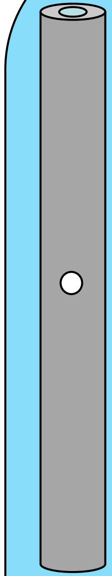


**Impact: Demonstrates importance of testing rates when assessing fracture resistance in gaseous hydrogen**

# Approach

## Develop test methods to characterize hydrogen effects on fatigue of components and component-like structures

1) Is there a consistent relationship between crack initiation and failure in manufactured tubing?
2) Will test frequency influence results?


Cross-hole tube specimen

**Innovative test method**

- Instrumented fatigue testing of tubing
- Cross-hole simulates stress concentration
- Internal H obtained via thermal hydrogen-precharging
  - temperature of 300˚C
  - pressure of 138 MPa

**Material**: XM-19

- high-strength austenitic stainless steel
  - Alternative to 316L

# Accomplishment

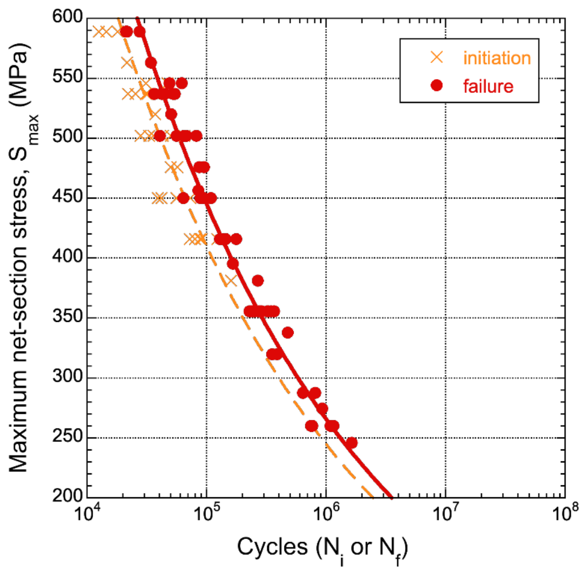
## Internal hydrogen had no apparent effect on fatigue life of XM-19

- crack initiation and test frequency were also assessed

### *Impact*

**Crack initiation occurred consistently at 2/3 cycles to failure**

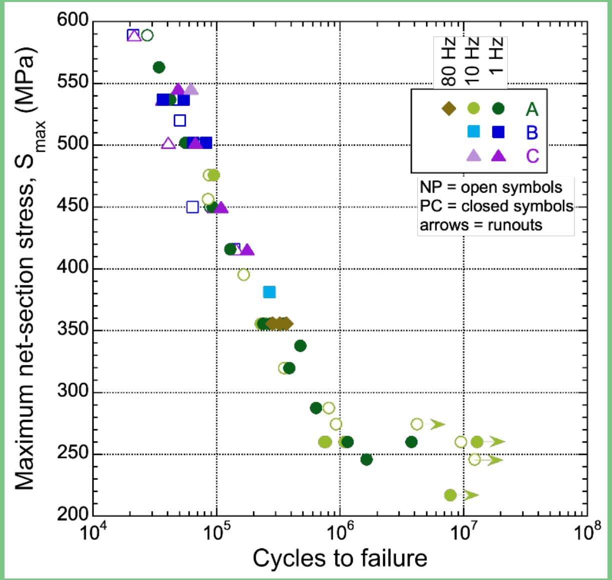
- ➢ **Simple relationship: initiation to failure**



### *Impact*

**Frequency did not influence cycles to failure (in range of 1 to 80 Hz)**

- ➢ **Opportunity for accelerated testing**



# Response to Previous Year Reviewers' Comments

## (Note: This project was not reviewed in 2022, therefore addressing FY21 comments)

- *FY21 Reviewer Comment:* It is unclear why SNL is looking at the effects of hydrogen in an atmosphere containing oxygen, a condition that SAE J2719 does not allow.
  - **As natural gas operators consider blending hydrogen, use of oxygen as a hydrogen embrittlement mitigator is being discussed. Our goal in looking at oxygen effects was to better understand the long-term effects of oxygen more broadly than use of hydrogen in mobile applications.**
- *FY21 Reviewer Comment:* It would be good to see a specific focus on the tank life extension work regarding the impact of cycles at less than full pressure and the impact of different types of usage on tank life. There is talk in industry about cycle life testing being too conservative since cylinders are often not fully emptied before being refilled.
  - **Agreed. We are performing sensitivity studies to help provide guidance to industry on how they can achieve maximum life out of their vessels while still ensuring safe (and conservative) operation. Results have been presented at several conferences, PVP2022 and ICF15(2023).**
- *FY21 Reviewer Comment:* Generating test methods on hydrogen compatibility for metal and non-metal materials should ultimately be published with ASTM International (ASTM) and the American Society of Mechanical Engineers (ASME) for stationary applications and SAE International (SAE) for transportation.
  - **We have developed the technical content for a Code Case including fatigue design curves which incorporate pressure and load ratio. This work has been presented to the ASME B31.12 Hydrogen Piping and Pipelines Code committee. This would extend the design curves beyond the ASME Code Case 2938 and greatly reduce the need for testing of pipeline materials. ASTM committees have not shown interest in hydrogen test methods.**

# Collaborations

- ***Standards Development Organizations (SDOs)***
  - ISO: involvement with several working groups to provide expertise to harmonize test methods
  - ASME BPVC: Code case adds design guidance to Article KD-10; ASME community and stakeholders are engaged in tank life extension discussion as well as ***requesting assistance on fatigue life versus fatigue crack growth methodologies***
- **Industry partners**
  - Partners communicate materials testing gaps/needs and provide technology-relevant materials (FIBA Technologies, Tenaris-Dalmine, Swagelok, Endress-Hauser and others)
  - International MOU: evaluation of Ni-Cr-Mo PV steels, motivation of Code Case for ASME BPVC and future testing plans (threshold fatigue crack growth and $R < 0$)
  - NASA-WSTF, Luna Innovations, and Digital Wave: ***non-destructive evaluation*** of metal liner in tanks
  - Becht and Air Products: comparison of actual service environments and design criteria, evaluation of margin in design and opportunity for life extension of large-volume, high-pressure storage vessels
- ***International research institutions***
  - ***Performance-based fatigue evaluation*** in the context of SAE is focus of R&D collaboration with international community, including collaborative research activity in Japan (Kyushu Univ), Germany (MPA Stuttgart), Korea (KRISS) and China (Zhejiang Univ)

# Remaining Challenges and Barriers

- Long-time scales (kinetics) associated with hydrogen-materials interactions challenges our ability to interrogate materials
  - Acceleration of testing is challenging and generally requires equal parts creativity and patience
  - Surface effects are difficult to characterize and even more difficult to quantify – thus establishing bounding behavior can be challenging
    - Particularly challenging at low pressures where effects of impurities can dominate.
- International consensus on codes and standards
  - **Consensus has always been a significant challenge** and requires patience and sustained interaction
  - Technical basis for code changes have been presented but acceptance can be prolonged.
- Next generation materials/microstructures cannot be identified without fundamental understanding of the physical processes
  - **Advanced scientific computing** and innovative experimentation are needed to integrate new materials into design

→ H-Mat℠ Hydrogen Materials Compatibility Consortium

# Proposed Future Work

Any proposed future work is subject to change based on funding levels

**Remainder of FY23**

- ***Test method development for targeted data***
  - Evaluate adaptation of cross-hole tube geometry for testing in gaseous hydrogen to permit evaluation of material classes other than austenitic stainless steels (to-date only testing w/ internal H has been achieved)
- ***Harmonization of standards***
  - Draft technical basis to incorporate pressure-dependent relationships into ASME Code Case 2938 to reduce over-conservatism for hydrogen pressure vessels operated at pressure <1,000 bar
  - Promote international dialogue on materials/components with UN GTR/ECE and ISO and SAE
- **Informational resources**
  - Revise Technical Reference (pressure vessel steels & stainless steels)
- ***Tank life extension***
  - ***Assess pressure variations on design life of tanks***
    - Present results of fatigue design sensitivities of Type 2 hydrogen vessels which address tank life extension at International Conference on Fracture (June 2023).
  - ***Evaluation of NDE techniques for tanks***
    - Demonstrate eddy current technique is feasible means **to detect flaws on Type 2 tanks** (Digital Wave)
    - In collaboration with Luna Innovations, evaluate NDE techniques ability to characterize various crack depths using C-ring (SBIR project)

# Proposed Future Work

Any proposed future work is subject to change based on funding levels

**FY24** (project continuation and direction determined by DOE annually)

- ***Stress-based fatigue design methodology to complement fracture mechanics***
  - **Evaluate other test configurations** in gaseous hydrogen to demonstrate potential for component-like testing in manufactured hardware (such as tubing for SAE J2579 & ISO)
- ***Quantification and guidance on role of environmental variables***
  - Leveraging outcomes from other projects, develop guidance on **role of environmental variables** for applicable standards, such as service temperature
  - Develop formalism for **pressure dependence on** fracture resistance
- ***Evaluate role of kinetics on hydrogen fatigue and fracture behavior***
  - Evaluate relationship between hydrogen-assisted **fracture and testing rate** on different geometries, and materials classes (potential change to CSA CHMC1 and other standards)
  - Explore emerging **role of moisture on fatigue crack growth**
- ***Engage API 1104 and ASME B31.12 on questions about welds***
  - Requirements for **hardness of welds** are viewed by some stakeholders as overly constraining (testing leveraged through HyBlend; code engagement in this project)

# Summary

- ***Target knowledge and data gaps with respect to hydrogen compatibility of materials***
  - Fatigue and fracture measurements of high-quality thick-walled vessels are not dependent on orientation or location
  - Design curves in ASME CC2938 can be pressure corrected for lower pressure conditions, reducing unnecessary conservatism; partners plan to present new formulation to code committee
  - A series of testing rates were examined for pipeline steels in hydrogen gas and it was shown that fracture initiation is less dependent on testing rate than hydrogen-assisted fracture
- ***Test method development and harmonization of standards***
  - Developed technical basis fatigue design curves of line-pipe steels that incorporates pressure-scaling and load-ratio dependence; presented to ASME B31.12 code committee
  - Developing TIR for SAE to capture current state of knowledge on materials testing and acceptance to SAE J2579
  - Invited as subject-matter expert for UN regulation R134 and ISO documents (independent 3rd party)
- ***International partnerships***
  - *Research institutions*: AIST (Japan) , Kyushu Univ. (Japan), KRISS (Korea), MPA Stuttgart (Germany)
  - *Industry*: Tenaris-Dalmine (Italy), FIBA Technologies (US), Hexagon Digital Wave (US), NASA-WSTF (US), Becht (US), Luna Innovations (US)