Reducing the Cost of Fatigue Crack Growth Testing for Storage Vessel Steels in Hydrogen Gas

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DOE project award # EE-0008829

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DOE Hydrogen Program

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AMR Project ID
#IN029

Project Goal

Goal: Reduce the cost of fatigue crack growth rate (FCGR) measurement in H_2 gas through efficient test design

Service life of H_2 storage vessels at fueling stations is dictated by fatigue crack growth

- ASME code provides methodology for calculating fatigue-limited design life for H_2 storage vessels
- Requires FCGR relationship measured for vessel steel in service environment, i.e. H_2 gas

Measurement of FCGR in H_2 gas is *time consuming* and *expensive*:

This project aims to :

- **Reduce test system run-time costs: Accelerate fatigue testing by optimizing progression through defined ΔK range**
- **Reduce labor costs by making multiple measurements from each test specimen (min. 2X cost reduction)**
- **Enable cost-effective measurement of fatigue crack growth rate at lower ΔK, which will facilitate market adoption of H_2 storage vessels at fueling stations**

Overview

Timeline

- Project Start Date: 3/24/20
- Project End Date: 5/31/24

Barriers

- Barriers addressed
  - K. Safety, Codes and Standards, Permitting

Budget

- Total Project Budget: $770,420
  - Total DOE Share: $616,270
  - Total Cost Share: $154,150
  - Total DOE Funds Spent*: $521,686.66
  - Total Cost Share Funds Spent*: $130,421.65

    * As of 03/8/2024

Partners

- Project lead: Kevin Nibur, Hy-Performance Materials Testing, LLC
- Team member: Brian Somerday, Somerday Consulting, LLC
- H-Mat Partner: Sandia Natl. Lab.

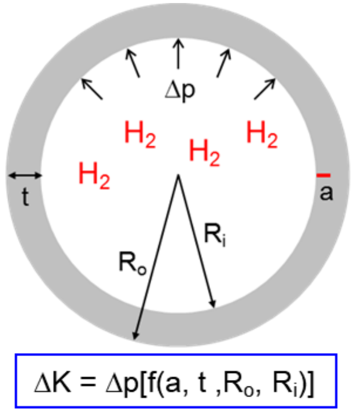
Relevance/Potential Impact

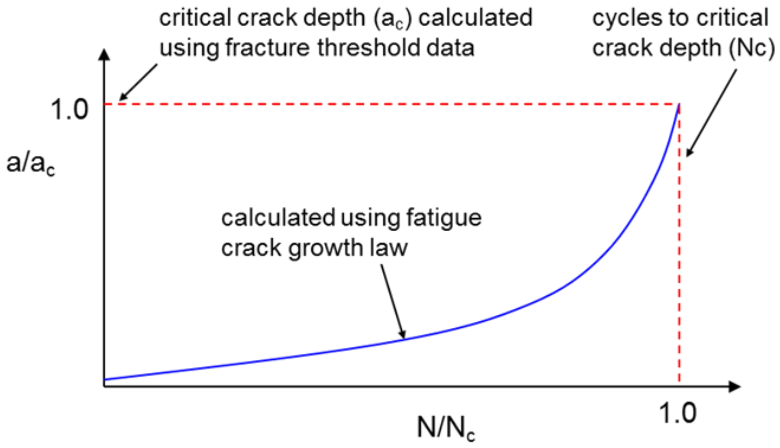
- This project complements existing design standards which ensure safe operation of stationary hydrogen storage vessels
  - Reducing the burden associated with fatigue crack growth testing enables increased accuracy of hydrogen storage vessel life prediction
  - Market adoption of hydrogen storage vessels at fueling stations will reduce GHG emissions
- Impact: Fully informed design life calculations
  - Current design standards (ASME pressure vessel code) require extensive materials testing in hydrogen gas
    - These testing requirements are expensive and time consuming
    - Reducing the cost and time associated with fatigue crack growth testing significantly reduces this testing burden
    - Reduced testing costs will reduce overall fueling station costs
  - Maximize design life of stationary hydrogen storage vessels
    - Reducing the cost and time associated with fatigue crack growth testing enables measurements at low ΔK which are necessary to demonstrate increased vessel design life
    - Increased storage vessel life will reduce overall fueling station costs
  - Prescribed properties (design curves) in ASME code have replaced need for testing in certain cases
    - Reducing the cost and time associated with fatigue crack growth testing enables the expansion of applicability and improves accuracy of prescribed design curves

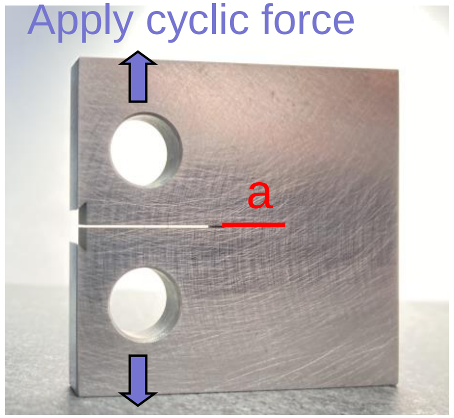
Approach: Introduction

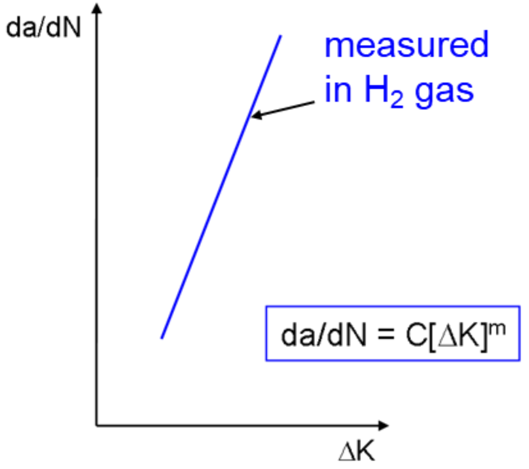
Hydrogen pressure vessel design utilizes fatigue crack growth relationships to determine safe design life

- A crack in the vessel wall with length ***a*** experiences stress due to pressurization
- ***K*** is a fracture mechanics parameter used to characterize stresses at the crack tip
- Crack extension (***da***) occurs with each pressurization cycle (***N***)









Laboratory specimens are used to characterize the increment of fatigue crack growth per loading cycle (***da/dN***) as a function of the cyclic stress at the crack tip ***ΔK***

- ***Fatigue crack growth rate relationships are expressed using log/log plots of da/dN vs ΔK***

Approach: The Problem

Fatigue crack growth rate (FCGR) measurement in hydrogen gas is expensive

- Cost drivers for FCGR testing in H_2:
  - Protracted test durations due to required low cyclic loading frequency (e.g. f = 0.1 Hz)
    - This barrier is unique to testing in environments like H_2 gas since hydrogen transport into the metal relies on various kinetic processes
  - Time to assemble and disassemble complex apparatus containing test specimen
    - This barrier is unique to testing in gaseous environments since robust pressure containment must be integral to the test system

- This project aims to reduce cost by optimizing test parameters while retaining data accuracy:
  - Shorten duration of da/dN vs. ΔK measurement by limiting crack extension (complete for intermediate ΔK)
  - Perform multiple FCGR tests on single specimen (complete for intermediate ΔK)

Approach: Proposed Solution

- This project aims to reduce cost by optimizing test parameters while retaining data accuracy:
  - Shorten duration of *da/dN* vs. *ΔK* measurement by limiting crack extension
  - Perform multiple FCGR tests on single specimen

- *K*-decreasing test procedures allow multiple measurements from each specimen
- Steep *K*-gradients limit the crack extension needed to measure *da/dN* vs *ΔK* over large range of ΔK

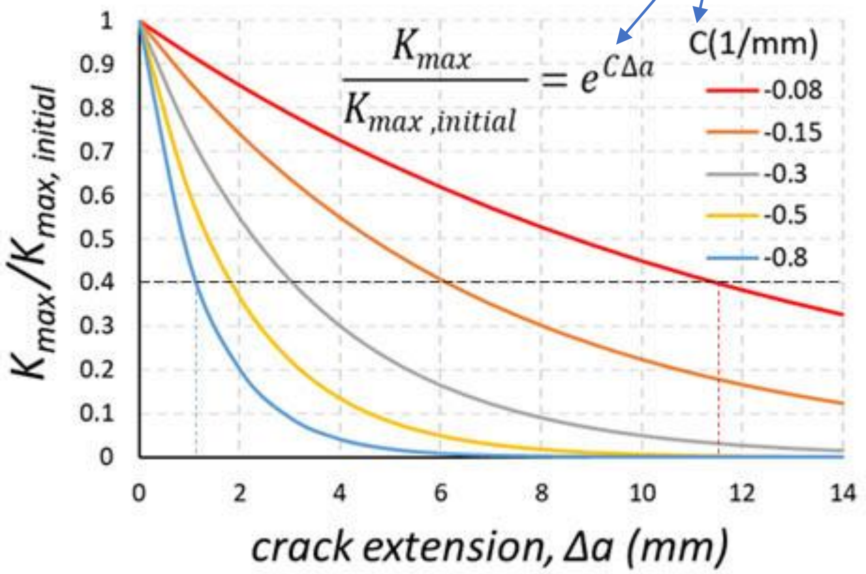
Load history effects compromise measurement accuracy and limit steep, decreasing *K* gradients

- "Load history effects" is a term used to capture various mechanical phenomena including crack closure and residual stress that result when crack tip stresses are changed rapidly during crack extension

Measurement inaccuracy due to load history effects constitutes the primary technical barrier to this approach

- **Goal**: Identification of the optimal ***K*** vs Δ***a*** profile (*K* gradient) that minimizes test duration (reduces crack extension) without promoting load history effects

C defines gradient



Steeper *K* vs Δ*a* profile results in less crack extension and shorter test time

Approach: Milestone table for funding period 3

Milestone	4.1	Measure baseline fatigue crack growth threshold in H2 gas at 30 Hz and C=-0.08 mm^{-1}
Milestone	4.2	Identify the steepest K-gradient that may be used for measurement of fatigue crack growth threshold in H2 gas without introducing load history effects. Demonstrate that measurements of fatigue crack growth rate made with this optimized K-gradient are consistent with the baseline measurements from Subtask 4.1.
Milestone	4.3	Demonstrate that multiple measurements of near threshold (i.e. low ΔK) da/dN vs ΔK relationships at different load ratios (R) can be made from a single specimen and that these measurements do not deviate from the baseline results in Subtask 4.2.
Milestone	5.1	Complete final report providing basis for standardization of optimized fatigue crack growth testing procedures for storage vessel steels in H2

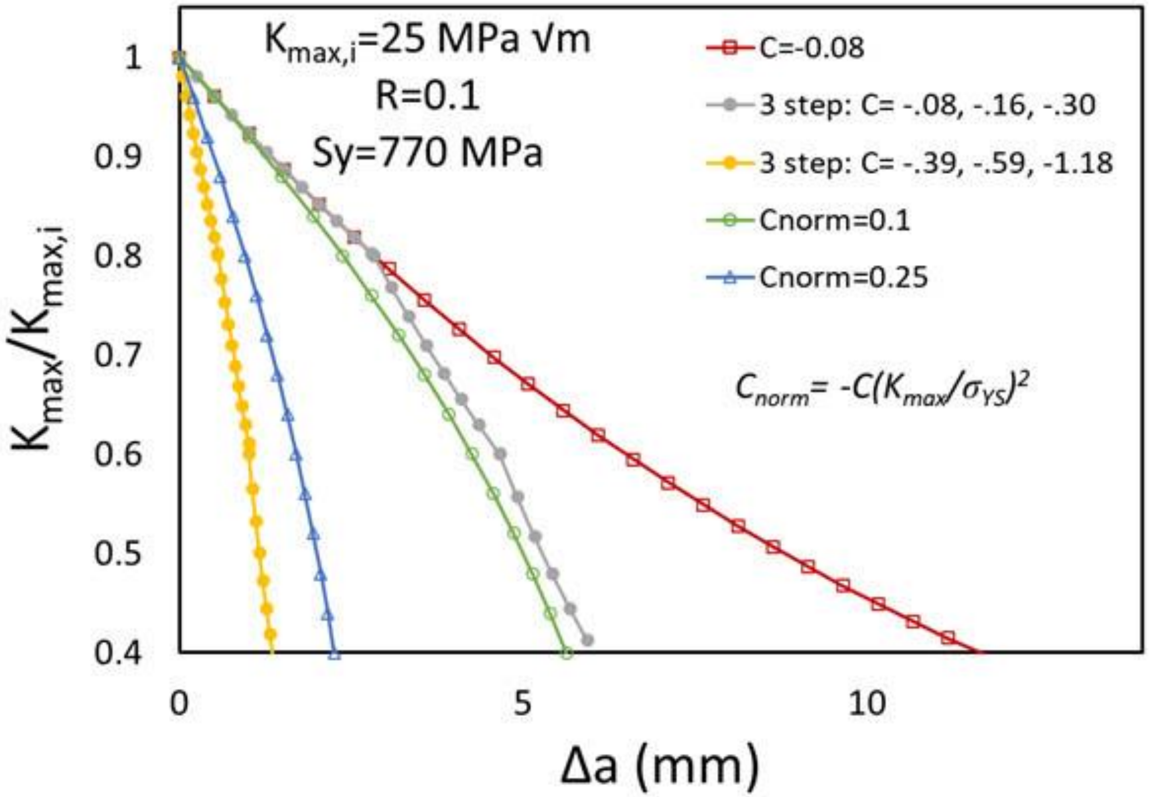
Approach: Safety Planning and Culture

- This project has submitted a safety plan to the Hydrogen Safety Panel (HSP)
- Compressed hydrogen gas presents potential hazards that must be carefully mitigated
  - Likely leak points are captured and vented
  - Secondary containment of test chamber with nitrogen blanket during testing captures unexpected leaks
  - Laboratory ventilation system is triggered by hydrogen sensors
  - Near-misses are considered as safety violations. Any near-miss triggers a re-evaluation of the process and new procedures or equipment are implemented to prevent repeat occurrence.

Prior Accomplishments: Variable *C* shed profiles optimized test efficiency

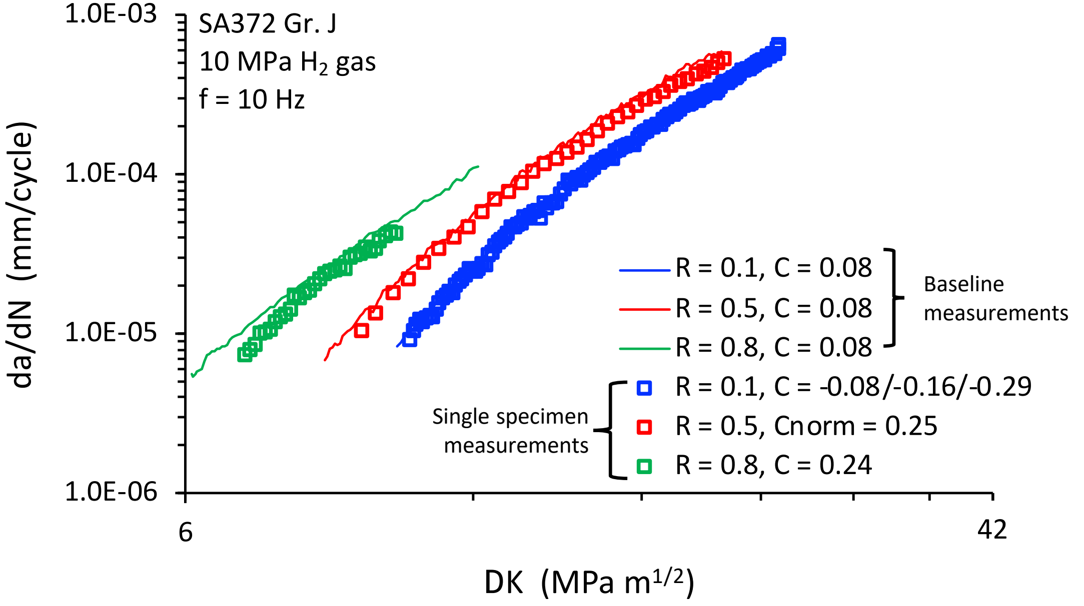
- Three-step scheme for *K*-shedding developed in year 1 was improved by continuously varying *C* following the expression for C_{norm}

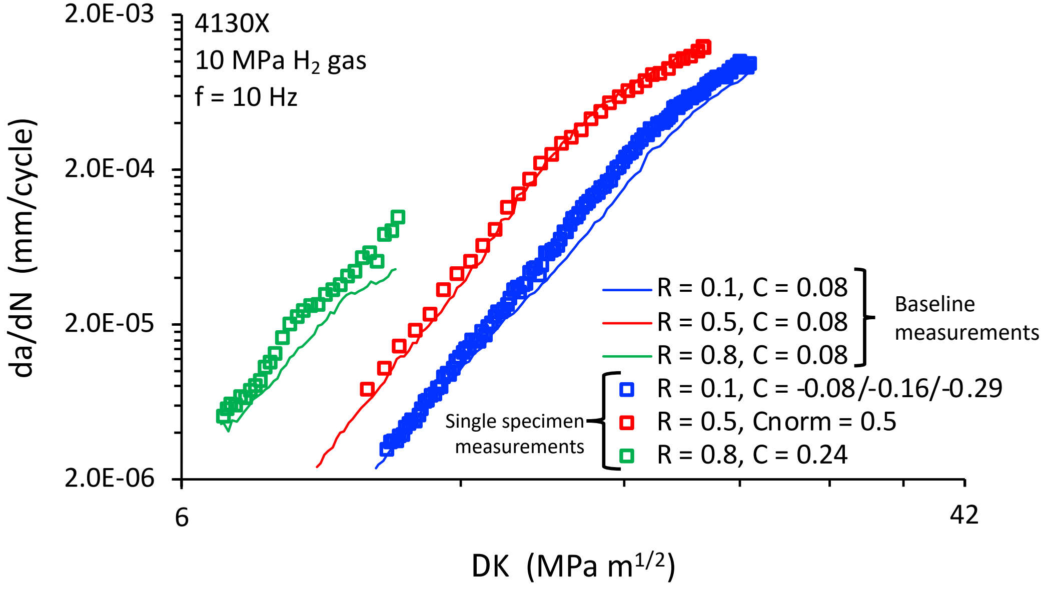
- *K*-gradient without load history effects depends on initial K_{max} and material strength
- C_{norm} parameter accounts for both factors
- $C = {}^{-C_{norm}}/_{(K_{max}/\sigma_{YS})^2}$
  - During *K*-shed, *C* decreases as K_{max} decreases



Steeper *K* vs Δ*a* profile results in less crack extension

Prior Accomplishments: Improved test efficiency has been demonstrated over intermediate ΔK range





- da/dN vs ΔK relationship measured at R= 0.1, 0.5, and 0.8 sequentially using single specimen of each material
  - Optimize K vs Δa profiles defined using either 3-step shed or C_{norm}
- <u>Each relationship is consistent with baseline measurements</u>
- Test time reduced compared to C=+0.08 1/mm:
  - 4130X: 13% of run time and 1/3 setup time
  - SA372 grade J: 36% of run time and 1/3 setup time
  - Cost: specimen preparation, test setup reduced to 1/3; run time cost reduce to 1/3 or less

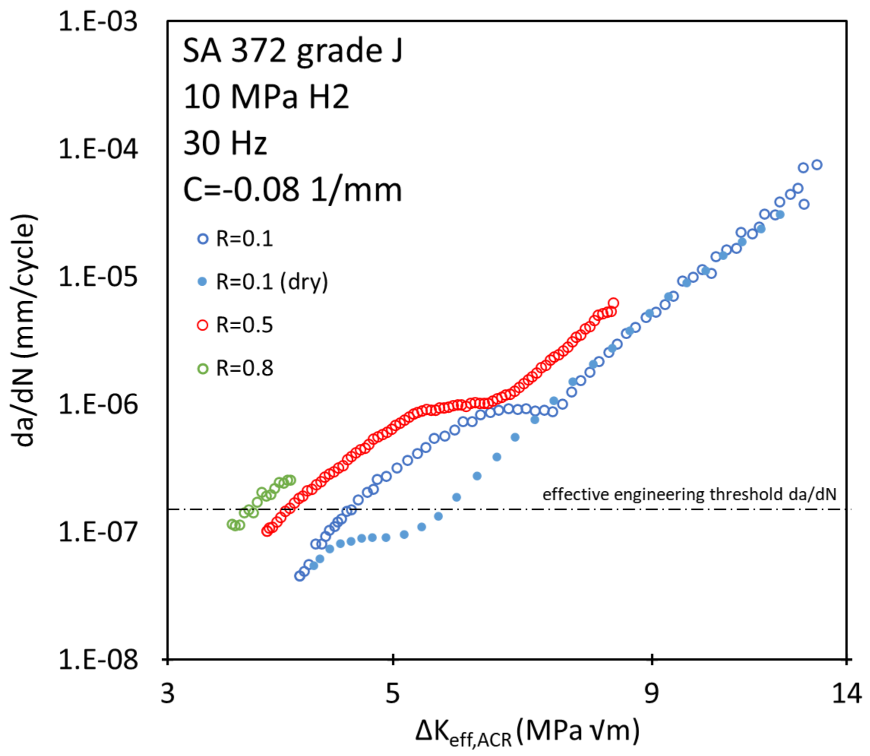
Demonstrates Milestone 3.2 and Go/No-Go #2

Prior Accomplishments: Baseline fatigue crack growth rate threshold measurements at R=0.1, 0.5 and 0.8

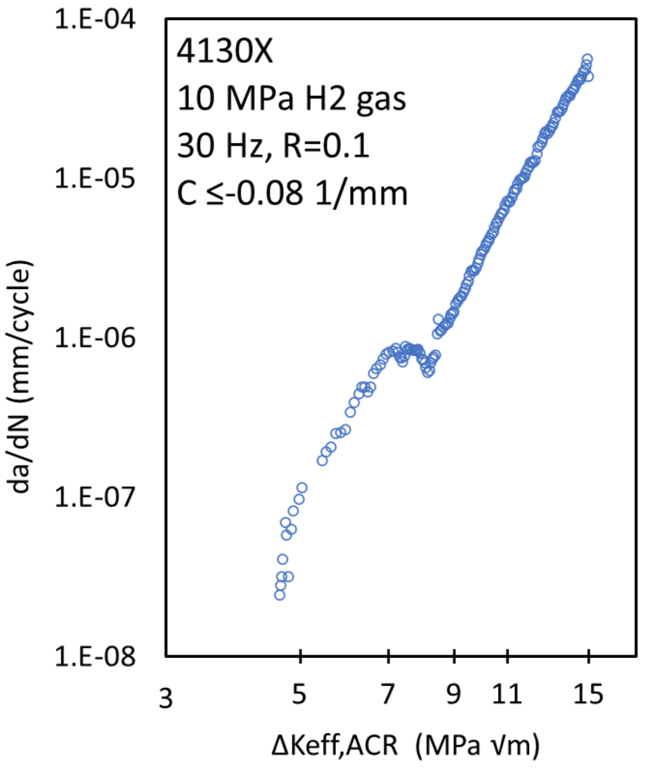
- Previous test method optimization focused on intermediate ΔK range
- Similar test method optimization applied to lower (near-threshold) ΔK range
- Baseline FCGR thresholds have been measured for SA372 grade J
  - Baseline measured at R=0.1, 0.5 and 0.8 at C=-0.08 1/mm
- Baseline FCGR threshold has been measured for 4130X at R=0.1

Demonstrates Milestone 4.1

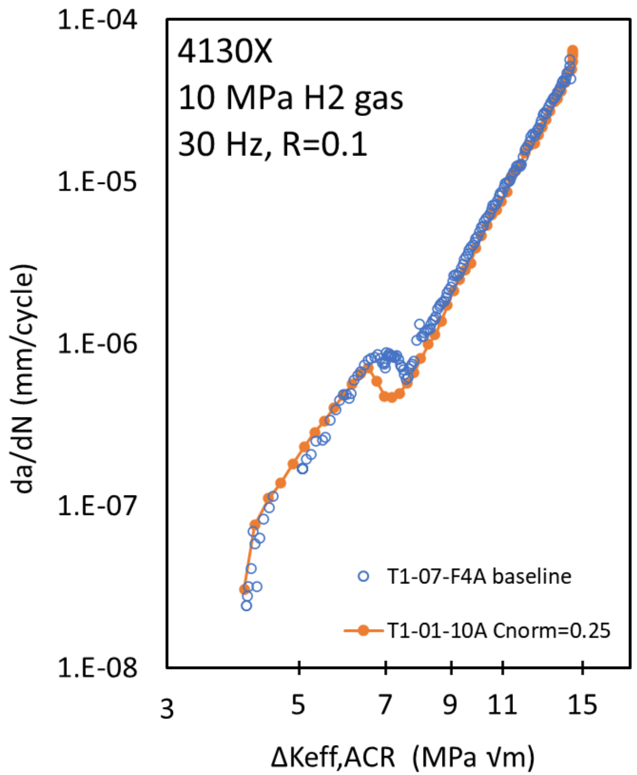
SA372 gr.J baseline FCGR



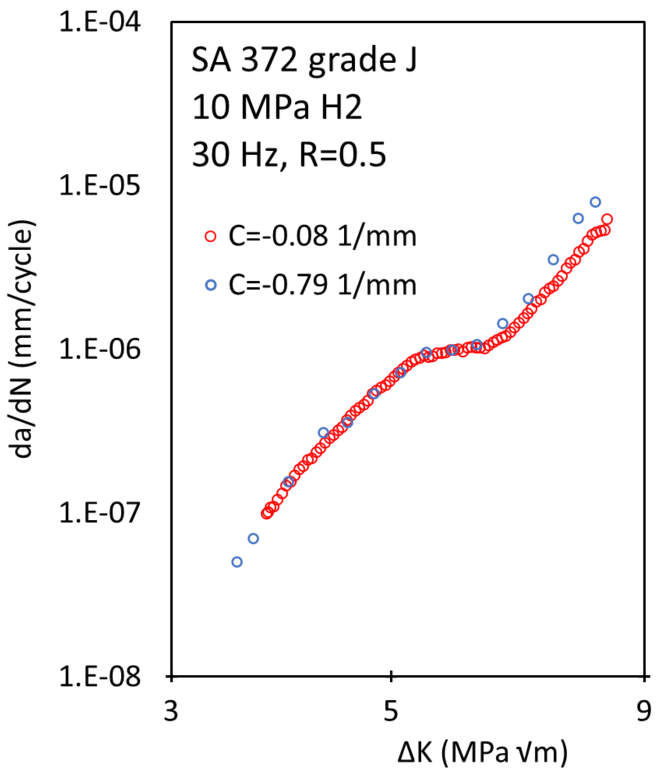
4130X baseline FCGR



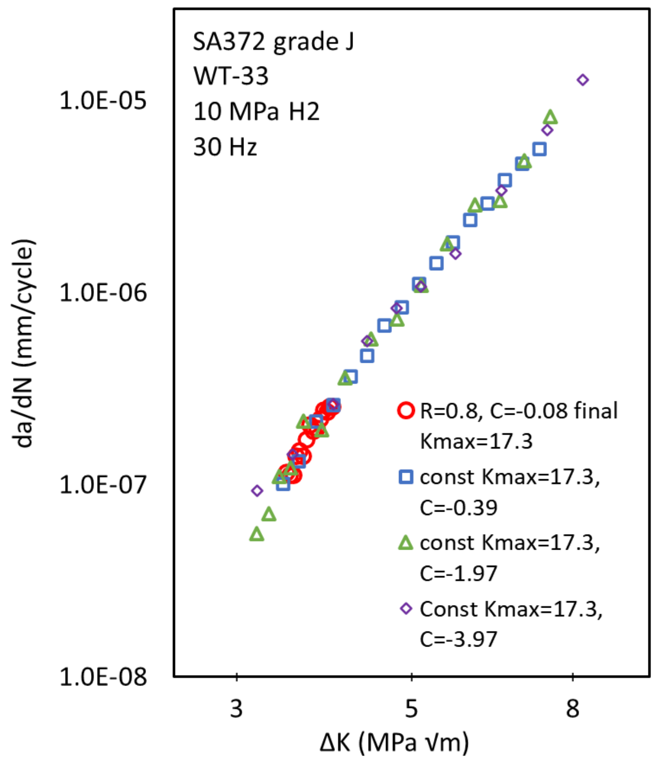
Prior Accomplishments: Optimized K vs Δa shed profiles in near-threshold region reduce test duration by up to 90%



K vs *a* profile defined by C_{norm} =0.25 provides crack growth rates consistent with baseline measurements at *C*=-0.08 1/mm
Impact: <u>Up to 90% reduction in number of cycles required to measure ΔK_{th}</u>



K vs *a* profile defined by *C* =-0.79 provides crack growth rates consistent with baseline measurements at *C*=-0.08 1/mm while <u>reducing number of cycles required by over 80%</u>



K vs *a* profile defined by *C* =-1.97 to -3.94 at const K_{max} provides crack growth rates consistent with baseline measurements at R=0.8 and *C*=-0.08 1/mm while <u>reducing number of cycles required by 90% or more</u>

Demonstrates Milestone 4.2

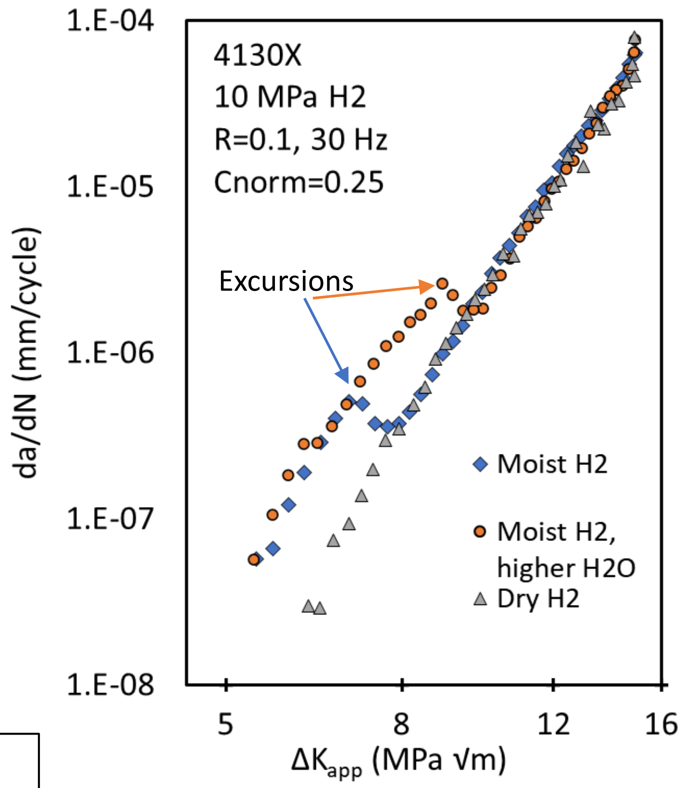
Prior Accomplishments: Role of water vapor has been elucidated which enhances understanding of near-threshold fatigue crack growth behavior in hydrogen gas

- Excursion in da/dN vs ΔK curve occurs when trace levels of water vapor are present
- Below excursion, da/dN may vary by up to an order of magnitude depending on water vapor content in hydrogen gas

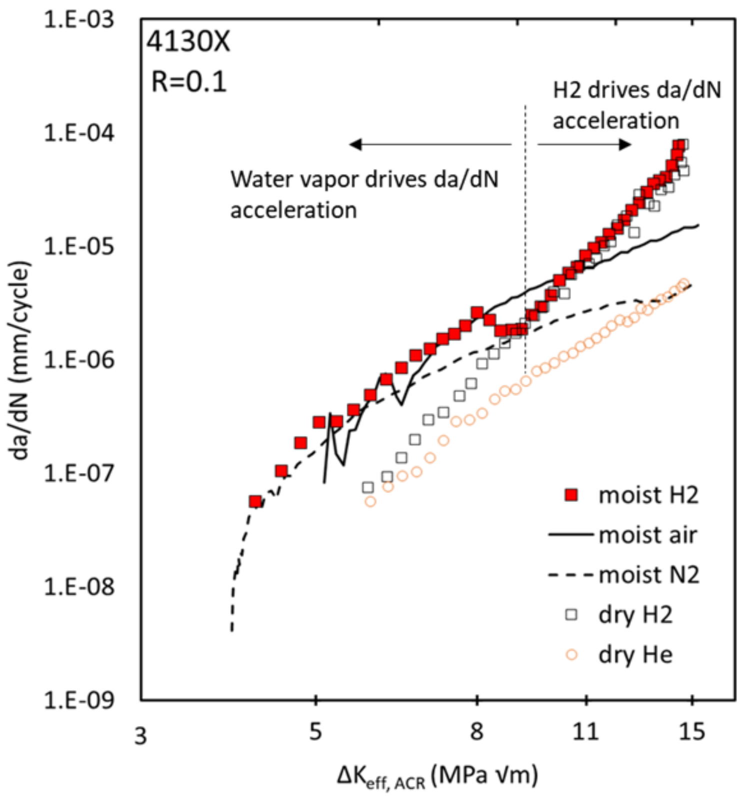
<u>Above excursion</u> – gaseous hydrogen is dominant driver of *da/dN* acceleration

<u>Below excursion</u> – water vapor is dominant driver of *da/dN* acceleration

Impact: Discrepancies in prior data may be better understood when considering variations in water vapor content. Also, da/dN vs ΔK measurements used for storage vessel design must consider this variable to ensure data is relevant to the application

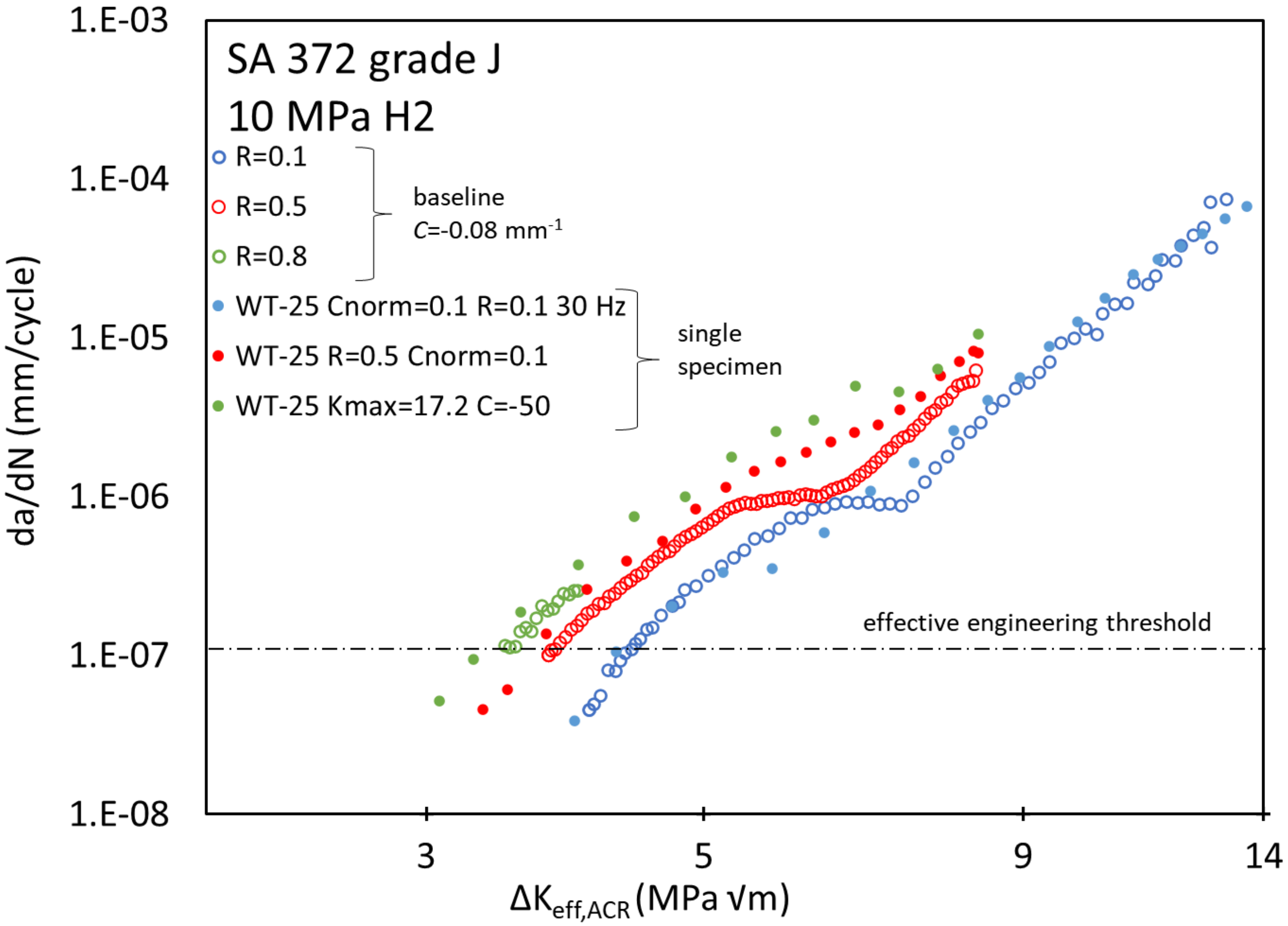


Trace levels of water vapor (>~2-5ppm) was identified as the cause of the observed excursions in da/dN vs ΔK plots.



Accomplishments: Multiple near threshold da/dN vs ΔK relationships at different load ratios (R) have been made from a single specimen using C_{norm} shed profile

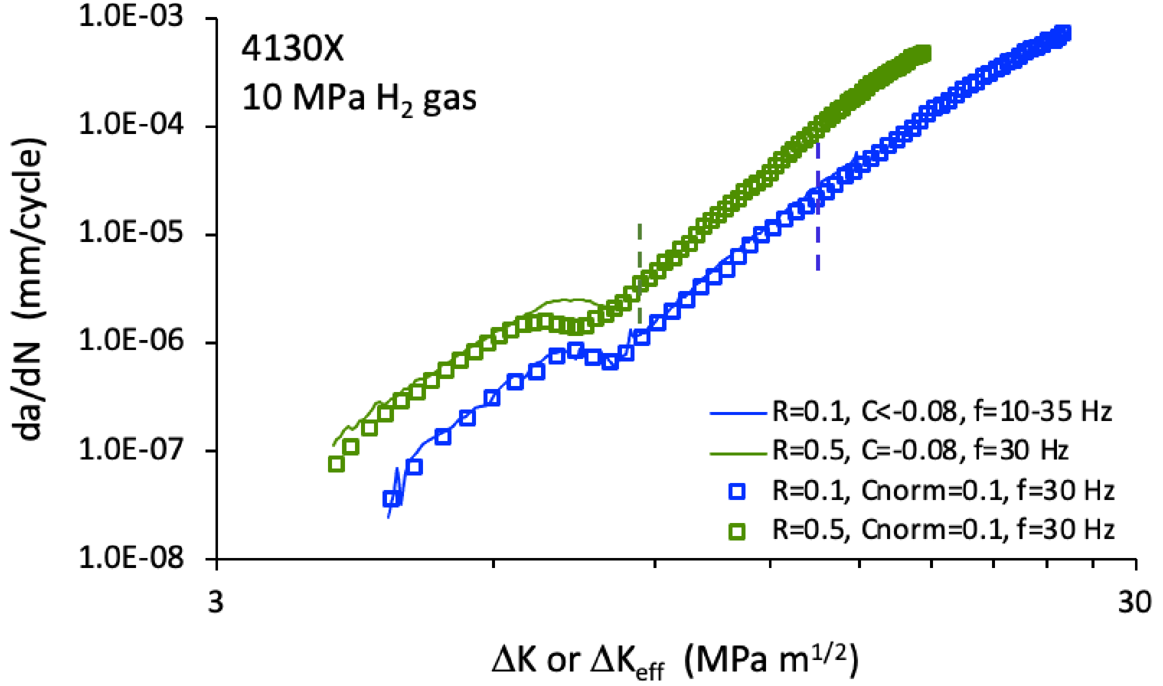
- ΔK_{th} and near threshold da/dN vs ΔK relationships at different R have been measured sequentially on a single specimens using optimized K vs Δa shed profiles based on C_{norm}
- Use of accelerated shed profiles reduced combined measurement time from to 55 million cycles to 8 million cycles



Demonstrates Milestone 4.3

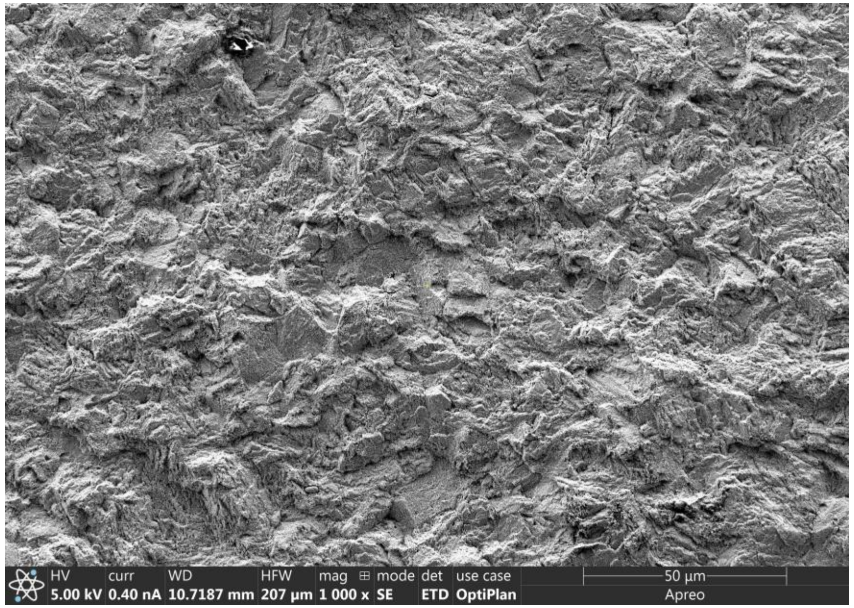
Accomplishments: C_{norm} shed parameter has been demonstrated over full range of ΔK at R=0.1 and 0.5

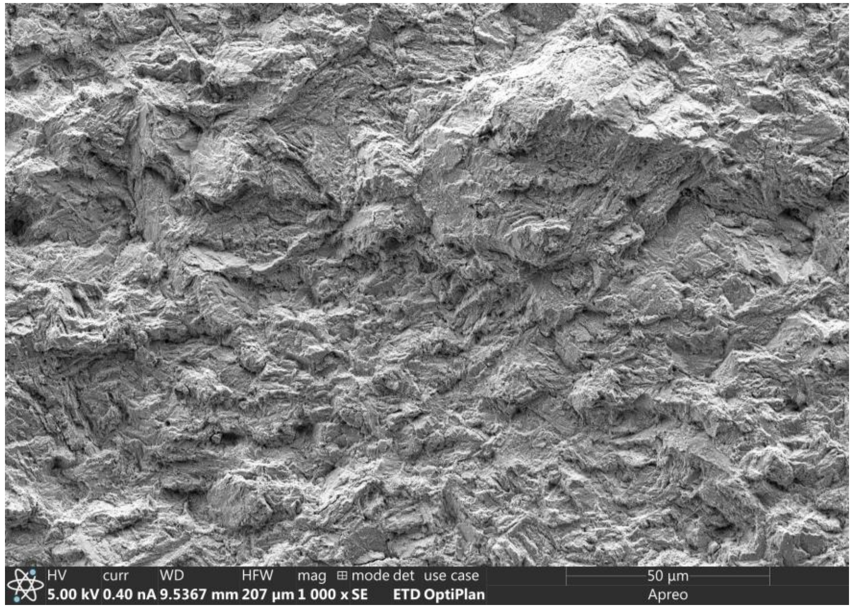
- Prior work demonstrated ability to use C_{norm} parameter at either intermediate ΔK or near-threshold ΔK separately
- Testing this quarter: Use of C_{norm}=0.1 allows K-decreasing measurements to be conducted from:
  - Initial K_{max}>36 MPa $m^{1/2}$ (R=0.5)
  - Initial K_{max}>30 MPa $m^{1/2}$ (R=0.1)
- Ensure C_{norm} parameter unlikely to be mis-applied for intended testing applications

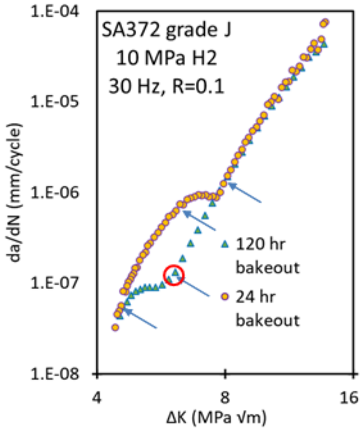


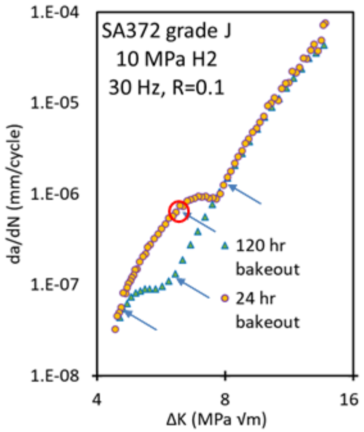
Accomplishments: SEM examination did not reveal evident difference if fatigue mechanism between moist and dry environments

- Scanning electron Microscopy was performed on six test specimens to compare fracture surface features
- Comparisons made at similar ΔK
- Fractur surfaces appear similar despite different measured da/dN



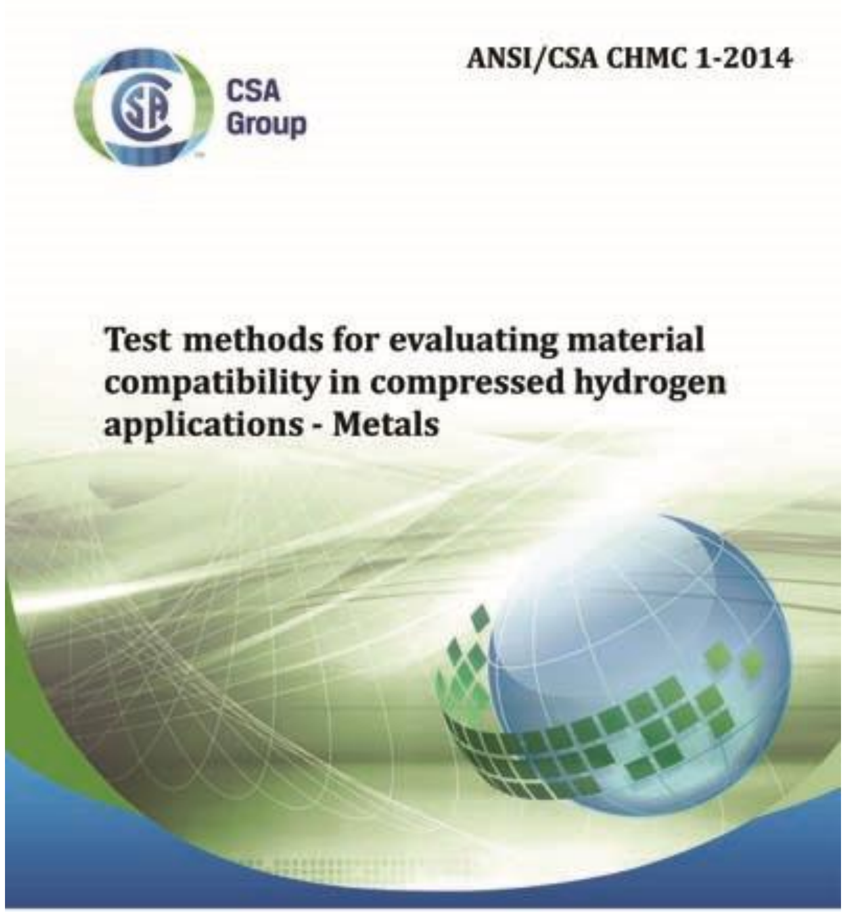






CSA CHMC1 Technical Sub-committee has begun updating standard

- CSA CHMC1 sub-committee has reconvened to update the CHMC1 test standard.
  - CHMC1 is currently the only standard that is specifically and solely intended to provide guidance for conducting mechanical testing in hydrogen gas
- Fatigue crack growth testing procedures developed in this project will be proposed as updates to the relevant sections of this document
- Project findings with regard to water vapor content have drafted into proposed revisions
- Publication of updated CHMC1 is targeted for 2024/2025 time frame



Accomplishments and Progress: Responses to Previous Year Reviewers' Comments

- Comment: "The project has good accomplishments and progress to date. The data generation is the easiest part, with ultimate ASME and ASTM buy-in being the most difficult. Initial feedback from the various committees on whether this could be accepted would have been appreciated."
  - response: We agree with this sentiment. Realistically, the approach needs to be identified and demonstrated before it can be considered for standardization. Results of this project have been shared throughout the project period with relevant ASTM committees where the reception has been encouraging. We are also in the process of proposing these procedures for the updated CHMC1 testing standard.

- Comment: "It seems that some testing capacity is being underutilized. Other approaches to increasing testing efficiency, such as the National Institute of Standards and Technology "daisy chain" method, could be compared to the approach in this project."
  - response: The "daisy chain" method at NIST addresses different issue than those addressed with this project. One of the primary goals of this project (see slide 2) was to enable cost effective measurement of fatigue crack growth rate at threshold and in the near threshold region of low ΔK. The "daisy chain" approach at NIST necessitates that fatigue crack growth testing must be performed at constant load amplitude. Testing in the near threshold region of the da/dN vs ΔK relationship is not conducive to testing at constant load amplitude. Moreover, the benefit of the daisy chain, as we understand, is the ability to perform duplicate tests simultaneously, but the constant load method is less efficient. Our approach is to optimize efficiency of each test and to facilitate multiple test objectives (e.g. different R values) from each test specimen. The two different approaches could not be combined simultaneously. Any direct comparison between the two is difficult; it would depend on the specific needs and whether duplicate measurements at intermediate ΔK are needed or if measurements at different parameters or near threshold are needed.

- Comment: "The project is tremendously impactful. Reducing FCGR times and costs by 66% (for the systems that have been tested in this project) is a significant advancement of the current state of the art as described by the ASTM protocol. The project serves the DOE goals and objectives in the space of safety assessment and codes and standards development for the hydrogen infrastructure."
  - response: Thank you, we appreciate this comment

Collaboration and Coordination

- Sandia National Laboratories (H-Mat partner)
  - Perform validation experiments at higher hydrogen gas pressure
  - Fractography and other analytical tools
- ASTM International
  - Project progress is shared with the Task Group on Load History Effects, ASTM E08.06.03
  - Mutually beneficial relationship provides this project with critical oversight and provides task group with new observations on load history effects
  - Procedures developed from this project may lead to new ASTM standard practice for FCGR measurement in hydrogen gas
- CSA
  - The CSA CHMC1 task group has reconvened to update CHMC1. The findings of this project are being considered for this revision

Remaining Challenges and Barriers

- This project is near its completion. All technical challenges and barriers have been overcome
- Final reporting, including journal submissions are in process
- Realizing the full impact of this work will require that standardization methods incorporate the results of this project
  - A primary goal for the remainder of this project is to initiate standardization changes that will allow the findings of this project to be used to reduce the testing barriers currently present for stationary hydrogen storage vessels such as ASME KD-10

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

Proposed Future Work

- FY2024:
  - Prepare final report and companion journal articles to complete Task 5

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

Summary

- Procedures for measuring fatigue crack growth rate in hydrogen gas for storage vessel steels have been optimized to reduce cost by reducing the amount of total crack extension
  - Reduces duration of measurement to 11-36% of baseline measurement (Go/No-go #1)
- These procedures have been used to enable measurement of three different fatigue crack growth rate relationships, each at a different R-ratio, using a single test specimen
  - Reduces test setup time and cost to 1/3 that required for a single measurement per specimen (Go/No-go #2)
- Optimized K vs Δa profiles reduce amount of crack extension required to measure ΔK_{th} by 80-90% or more
  - Measuring ΔK_{th} at frequency as high as 30 Hz further reduces test duration without compromising data accuracy