

# Sandia's Capabilities and Relevant Research

**H2@Scale Kickoff**  
**Colorado School of Mines**  
**April 14, 2020**

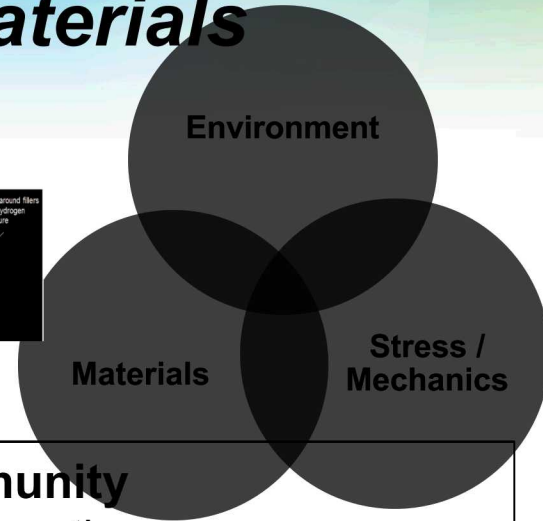
**Joe Ronevich and Chris San Marchi**  
**Sandia National Laboratories**

# Sandia maintains unique capabilities to support research on *Hydrogen Effects on Materials*



## Hydrogen Effects on Materials Laboratory

- In situ mechanical testing (P >100 MPa and 230K < T < 400K)
- Long-term, high-pressure H<sub>2</sub> exposure
- Pressure cycling at controlled temperature

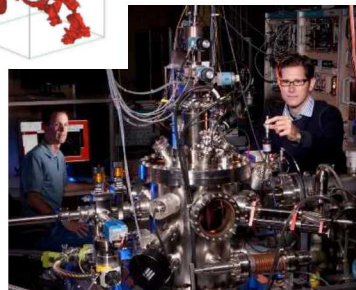
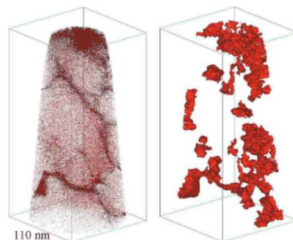


## Active materials science community

- Computational materials science expertise
- Full-suite of state-of-the-art materials characterization tools
- Joining laboratory (austenitic steels, non-ferrous materials)

## Hydrogen Transport and Trapping Laboratory

- Diffusion and permeation
- Thermal desorption spectroscopy
- Local-electrode atom probe tomography



## Hydrogen-Surface Interactions Laboratory

- Low-energy ion spectroscopy
- Ambient pressure x-ray photoelectron spectroscopy
- Kelvin probe atomic force microscopy

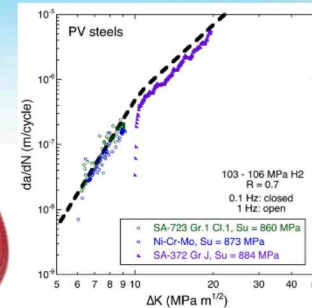
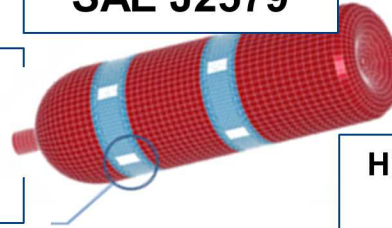
# Evaluation of *Materials Compatibility with H<sub>2</sub>* enables materials innovation

ASME article KD-10  
input on test  
methodology



First qualification data  
for high-pressure  
ASME vessels

Full-scale  
tank testing  
CSA HPIT1  
SAE J2579

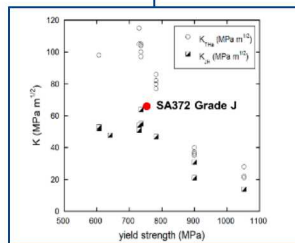


High-hardenability pressure  
vessel steel results  
presented to ASME KD-10  
for acceptance of curve fits  
based on data

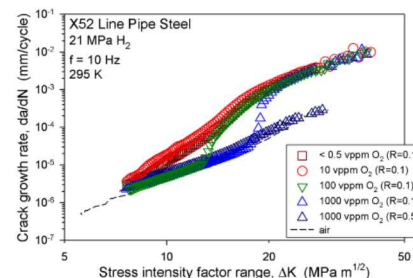
Platform for  
pressure cycling of polymers in  
GH2  
(up to 400 C)

2006                      2008                      2010                      2012                      2014                      2016                      2018

Platform for mats  
testing in GH2 at  
high pressure

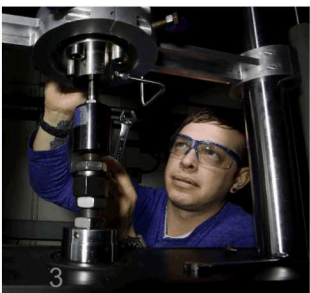


Modeled H<sub>2</sub> embrittlement  
mitigation through O<sub>2</sub> gas  
impurities



Critical assessment of  
statically loaded cracks

Platform for high-  
pressure GH2 over  
temperature range  
(-40 °C to +85 °C)



# General trends in hydrogen transport

- **Ferritic steels**

- Diffusivity:  $10^{-8}$  m<sup>2</sup>/s
- Solubility:  $10^{-8}$  H/M MPa<sup>-1/2</sup>
  - Lattice concentration (P=100 MPa): 0.3 ppm H/M
  - Trapping concentration: 100x greater than lattice concentration
- *Not amenable to gas-phase precharging*

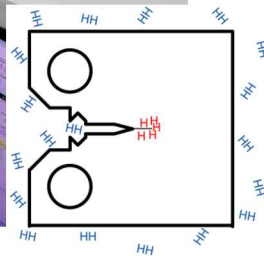
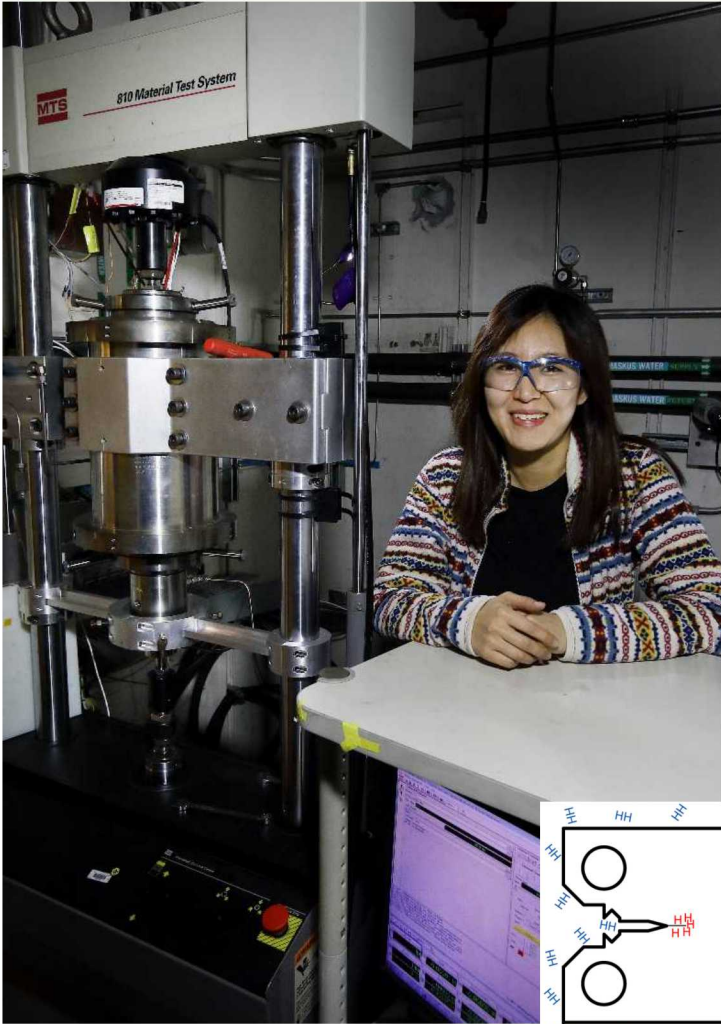
- **Austenitic stainless steels**

- Diffusivity:  $10^{-15}$  m<sup>2</sup>/s
- Solubility:  $10^{-4}$  H/M MPa<sup>-1/2</sup>
  - Lattice concentration (P=100 MPa): 0.2% H/M
  - Trapping concentration: <<1x lattice concentration
- *Gas-phase precharging is a well-developed technology*
  - Specimens can be shipped anywhere on dry ice and stored for extended time (months) at low temperature (-50°C)
  - Precharging time weeks to months depending on specimen size

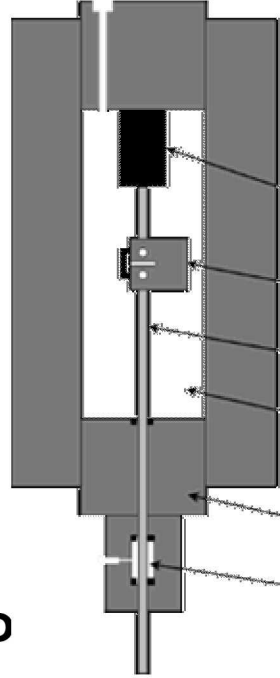
Solubility  
(ideal gas)

$$K = \frac{c_L}{\sqrt{P}}$$

# Fatigue and fracture measurements in high-pressure gaseous hydrogen



- Instrumentation
  - Internal load cell (w/ feedback)
  - Displacement measured on sample using LVDT or clip gauge
  - DCPD measurements possible
- Mechanical loading
  - Cyclic / monotonic
  - Load-ratio = 0.1 to 0.8
  - Frequency = 0.002 to 10 Hz
  - Loads: 1–15 kN
  - ASTM E1820, E647, etc
- Environment
  - Gaseous environment
  - Pressure  $\leq 120$  MPa
  - Room temperature (some lo temperature capability)



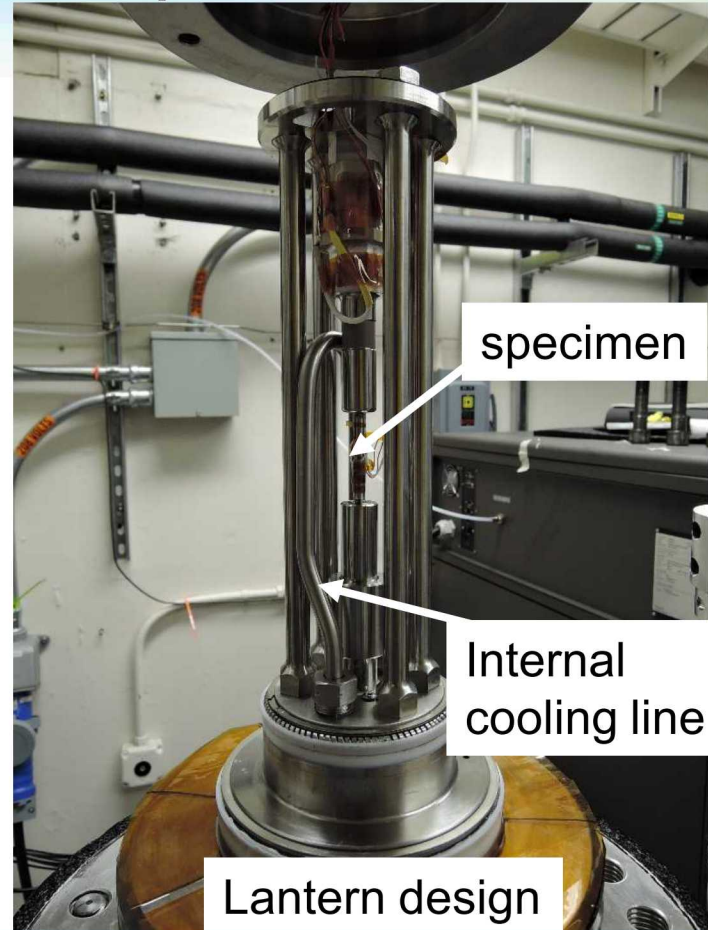
# Dynamic testing in high pressure hydrogen at sub-ambient temperatures (Cell 2)



Temperature  
-50C to 150C

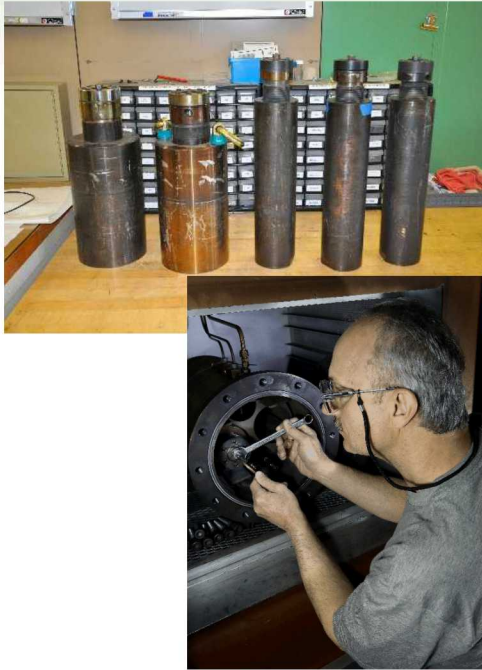
Pressure  
Up to 106 MPa

Mechanical Test  
Quasi-static  
or  
Cyclic loading



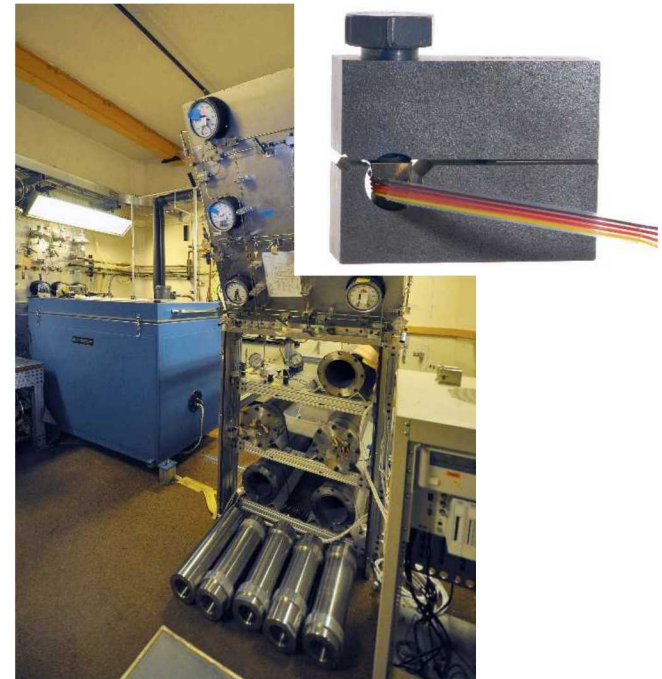
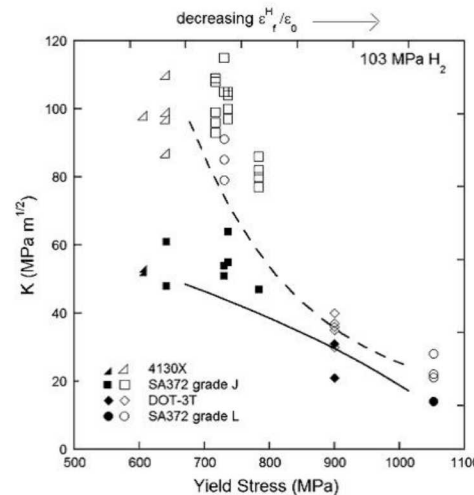
**Hydrogen refueling stations pre-chill gas and components experience temperatures from -40C to +85C**

# High temperature hydrogen pre-charging or statically loaded tests in hydrogen environment (Cell 5)



- Thermally pre-charge test specimens up to 138 MPa H<sub>2</sub> at 300C
- For austenitic stainless steel, results in approximately 1 at% hydrogen
  - 140 wppm in 300 series

- Statically loaded specimens (ASTM E1681) exposed to high pressure hydrogen to evaluate fracture thresholds

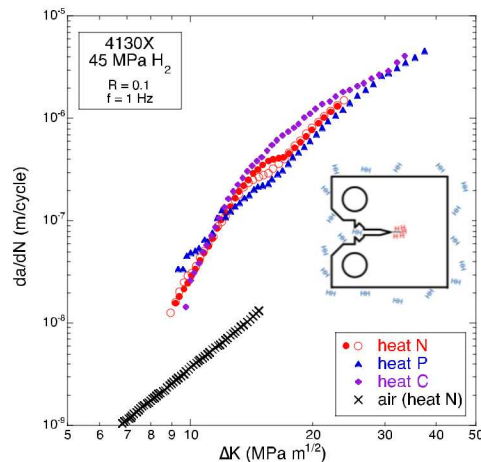


# Hydrogen-materials exemplar projects

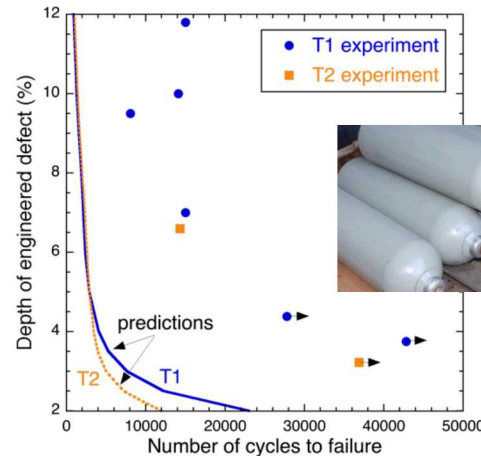
- 1) Evaluated pressure vessels for H<sub>2</sub> fuel cell fork lift trucks**
- 2) Residual stress effects on high strength pipeline steel welds**
- 3) Microscopy characterization at different length scales – 304L austenitic stainless steel**

# 1) Full-scale testing of pressure vessels enabled deployment of safe, low-cost fuel cell forklift fuel systems

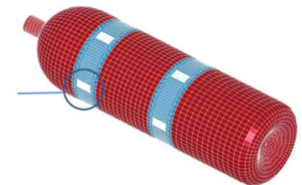
Materials testing and evaluation



Component testing



Lifetime prediction and SCS development

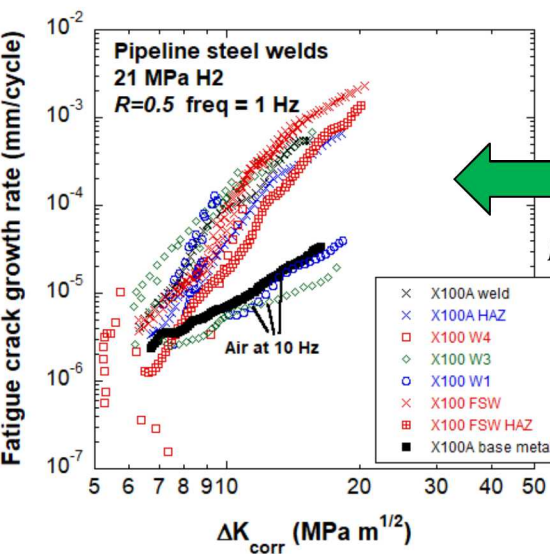
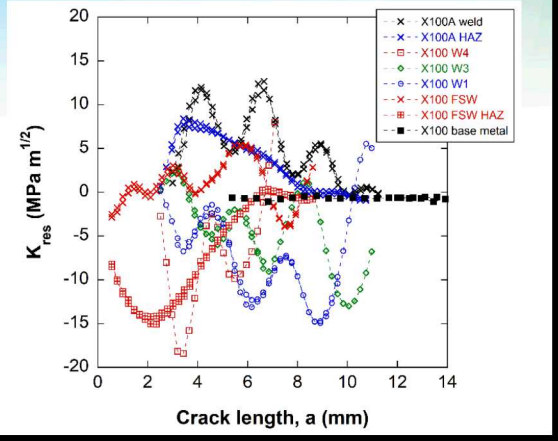
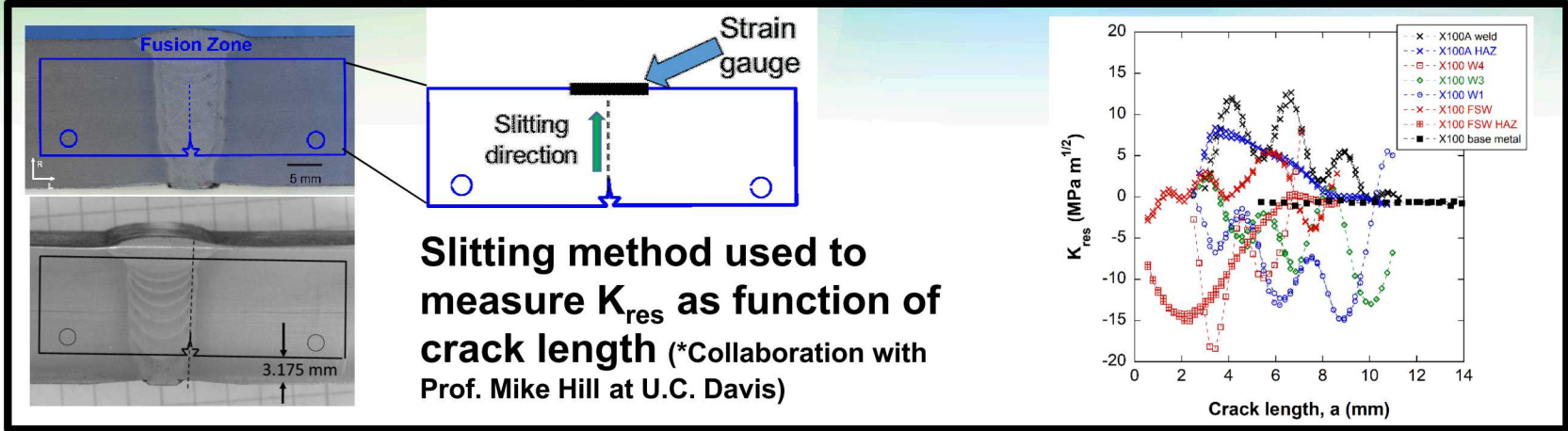


- Quantified uncertainties in the cycle life of hydrogen storage tanks for the lift-truck application
- Enhanced safety and market growth enabled through standards development (CSA HPIT1)
- Today, there are >23,000 clean and efficient fuel cell forklifts in service (and growing!)



Enabled H<sub>2</sub> fueled forklifts through combined full-scale & laboratory materials testing

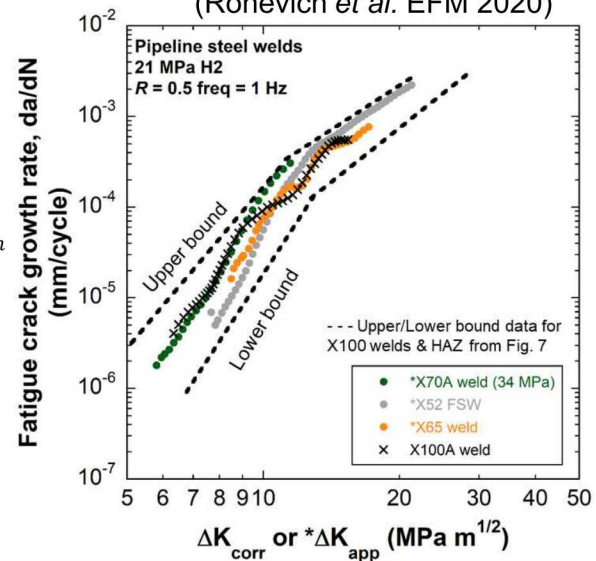
## 2) Fundamental understanding of hydrogen-assisted fatigue behavior of pipeline welds through measurements & correction for residual stress



**Corrected data normalizes FCGR curves to single R-ratio = 0.5 providing better utility and fidelity**

$$K_{norm}(a) = (\Delta K(a))^{1-n} * (K_{max-app}(a) + K_{res}(a))^n$$

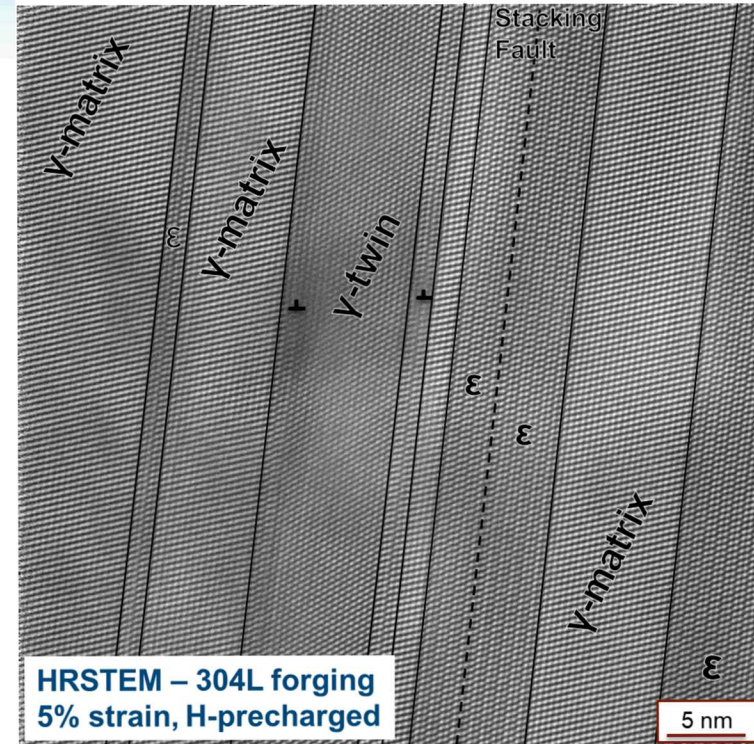
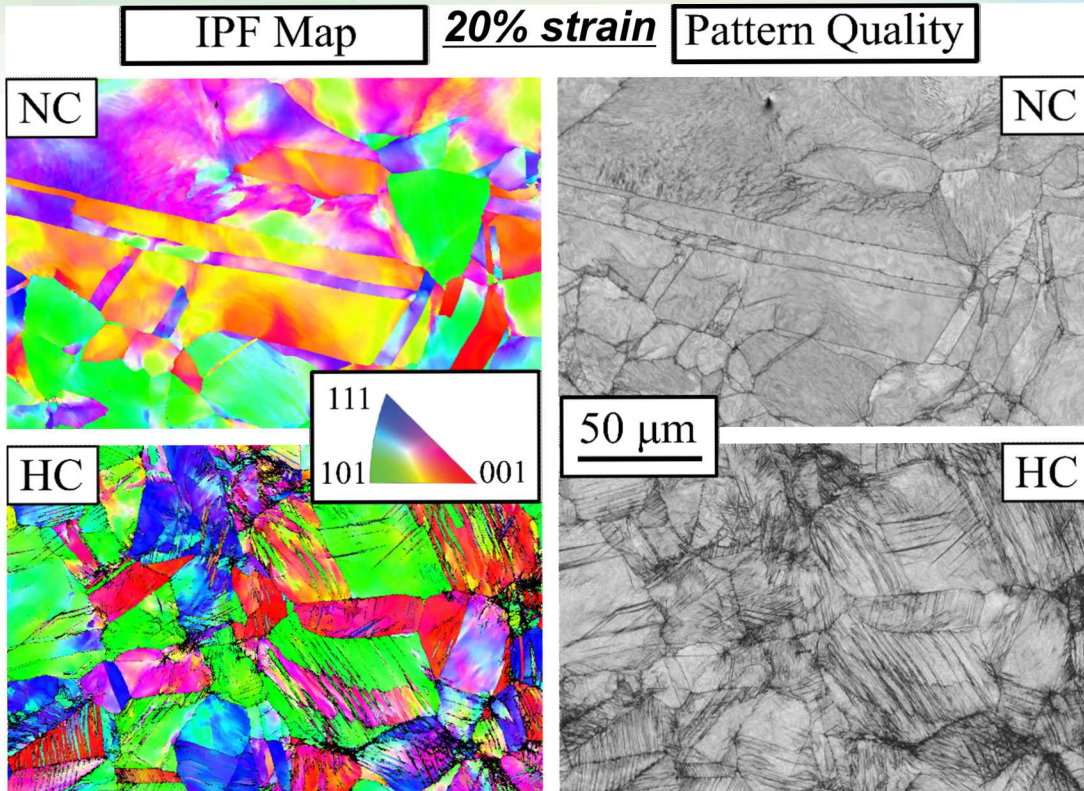
$$\Delta K_{corr} = K_{norm} * (1 - \bar{R})^n$$



**FCGR of high strength welds were comparable to lower strength welds, when residual stress is considered**

### 3) Characterization at different length scales – 304L

Julian Sabisch, Doug Medlin, Josh Sugar (SNL/CA)



- 304L deformed 5% and 20% without (NC) and with H-precharging (HC)
- Martensite is more common than twinning in H-precharged samples
- Twins and  $\epsilon$ -martensite are generally very thin (less than  $\sim 20$  {111} planes) while spanning through most of the grain. With twins appearing as faulted  $\epsilon$ -martensite

# Summary

- **Sandia National Laboratories has numerous H-related capabilities**
  - Thermal (gas-phase) H-precharging
  - Fatigue and fracture in high-pressure gaseous hydrogen
  - Hydrogen transport (TDS, diffusion/permeation)
  - Static loading/displacement testing
- **Should not overlook other capabilities and expertise, such as**
  - Expert materials science characterization
  - Computational materials science

# Acknowledgements

FCTO for funding much of this research

Hydrogen Effects on Materials Laboratory Team

- Chris San Marchi, Joe Ronevich, Jeff Campbell, Brendan Davis, James McNair, Brian Kagay

Microscopy

- Josh Sugar, Doug Medlin, Julian Sabisch

Modeling

- Jay Foulk, Coleman Alleman, Xiaowang Zhou, Norm Bartelt, Chris Nowak

Surface Science

- Farid El Gabaly, Rob Kolasinski, Konrad Thurmer

Trapping and Transport Lab

- Rick Karnesky

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