SAND2019-14323PE

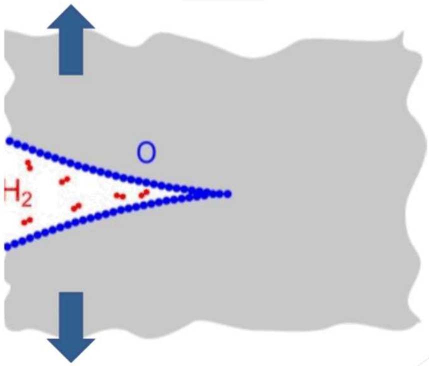
SAND2019-XXXX

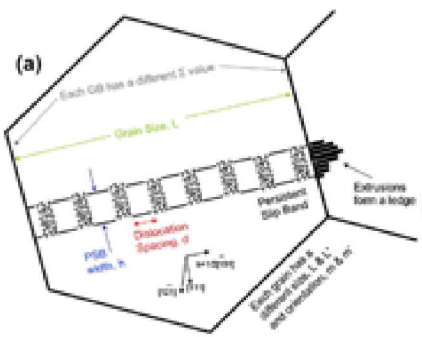
Austenitic Stainless Steel Research at Sandia National Laboratories and H-Mat Consortium

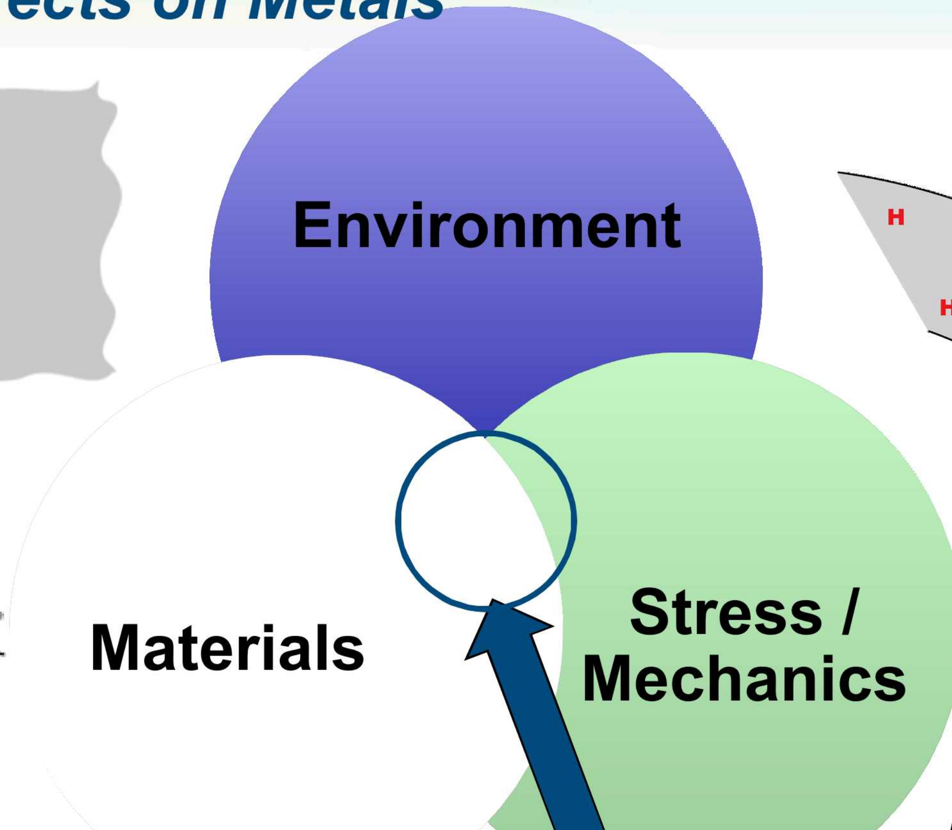
Chris San Marchi (H-Mat co-lead) and Joe Ronevich
H-Mat co-lead: Kevin Simmons (PNNL)
H-Mat Lab Partners: SRNL, ORNL, ANL

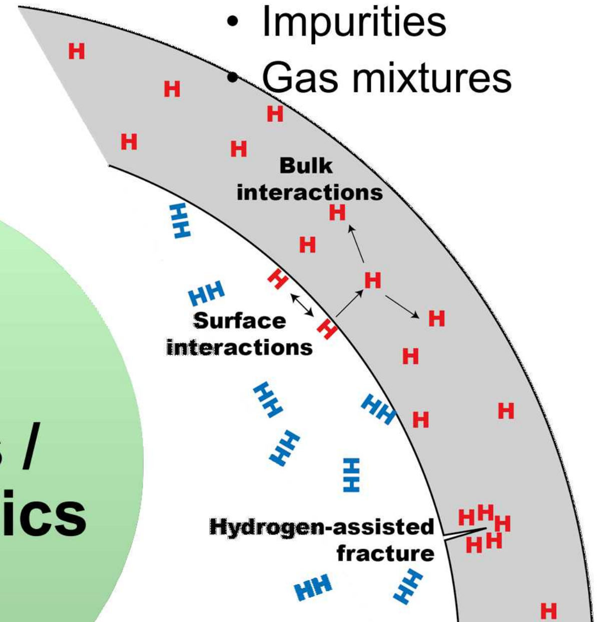
DOE EERE H2@Scale, UIUC Project Kick-off Meeting
November 21, 2019

Consider the intersection of *environmental*, *mechanics* and *materials* variables to understand *Hydrogen Effects on Metals*









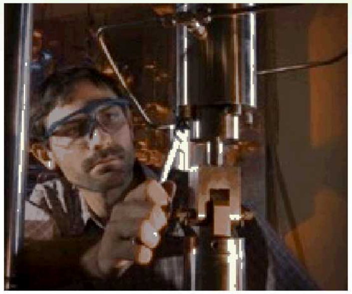
Environment
- Low temperature
- High pressure
- Impurities
- Gas mixtures

Materials
- High-strength
- Hydrogen-enhanced plasticity
- Boundary cracking
- Surface passivation

Hydrogen embrittlement occurs in ***materials*** *under the influence of* ***stress*** *in hydrogen* ***environments***

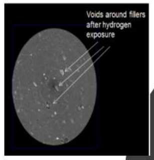
Mechanics
- Autofrettage
- Short crack behavior
- Fatigue crack initiation
- Fracture resistance

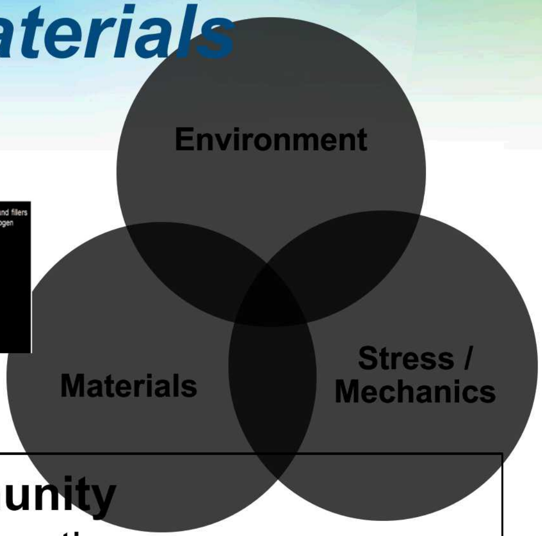
Sandia maintains unique capabilities to support research on *Hydrogen Effects on Materials*



Hydrogen Effects on Materials Laboratory

- In situ mechanical testing
  (P >100 MPa and 230K < T < 400K)
- Long-term, high-pressure H2 exposure
- Pressure cycling at controlled temperature



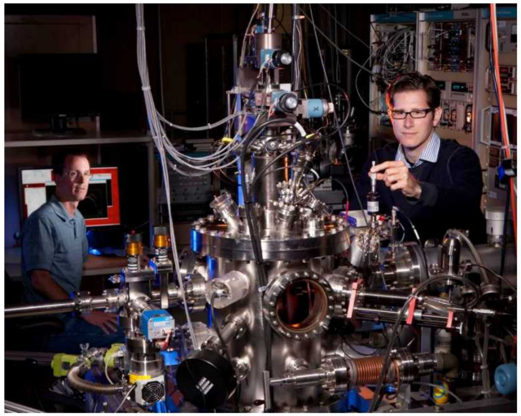


Active materials science community

- 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



Hydrogen-Surface Interactions Laboratory

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

Sandia has rich tradition of research at intersection of materials science and hydrogen technologies

Globally-recognized science leadership in materials compatibility

Safety, Code and Standards

- Develop **science-based test methods** to qualify for metals and polymers for H2
- **Harmonize** methods and materials in partnership with **international community**
- Disseminate information and dispel myths

Delivery

- Advanced materials and welding technologies for hydrogen transmission
- **Microstructure-based assessment** of materials and mechanisms

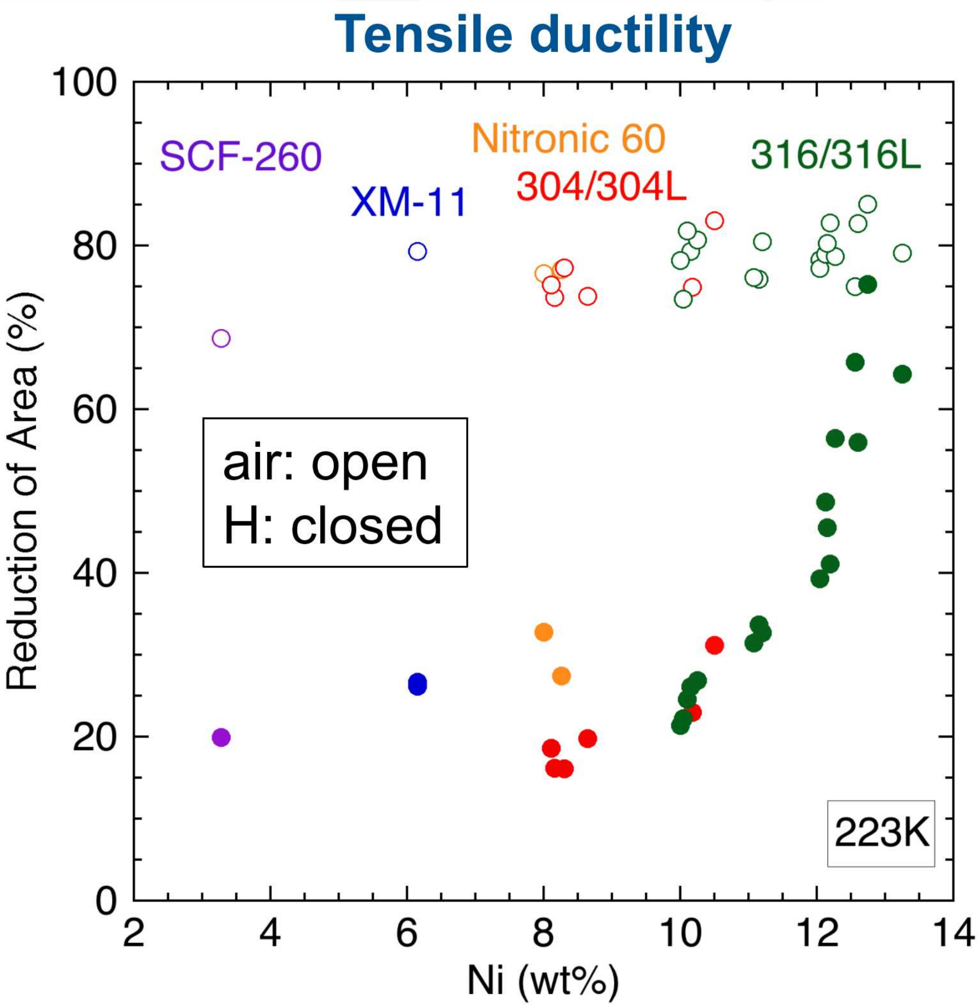
Storage

- **Reduce cost and weight** of BOP
- Develop methods for screening materials using computational materials science

Internally-funded activities

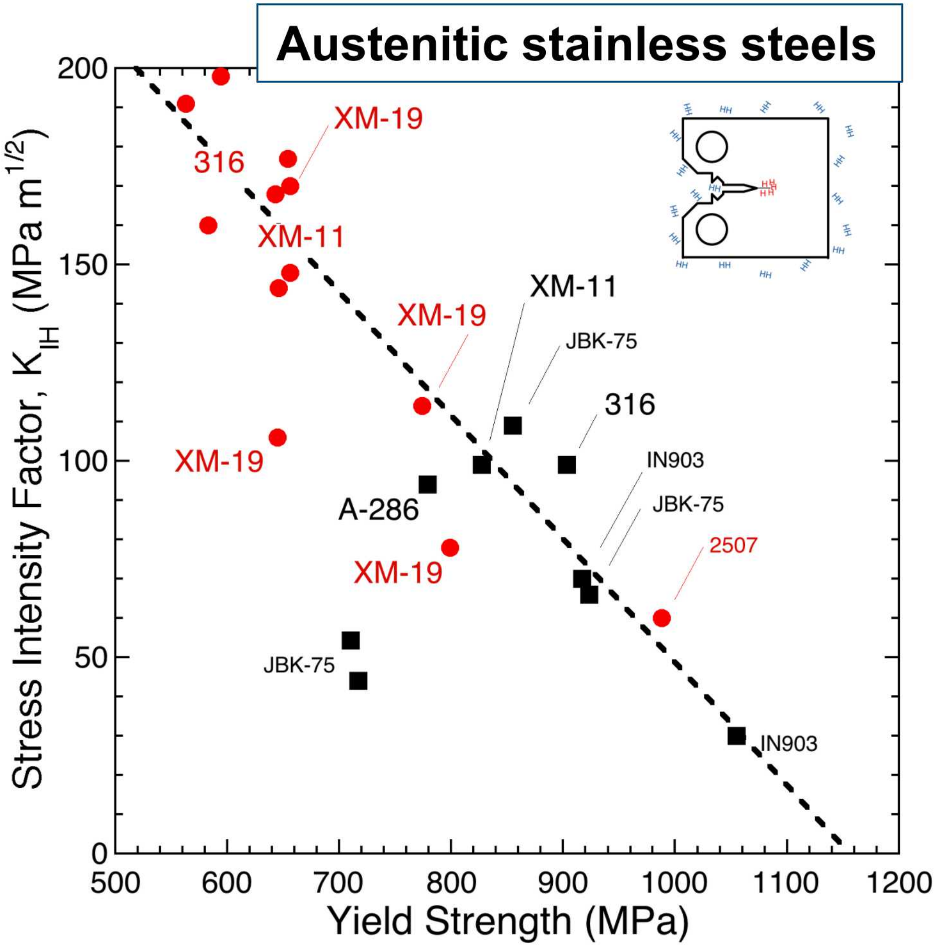
- Passivation of metal surfaces to mitigate hydrogen embrittlement
- Hydrogen-assisted fracture of additively manufactured austenitic stainless steels
- Fundamental microstructure-hydrogen interactions in **single crystals** (and **oligocrystals** in H-Mat)
- **Computational materials science** of hydrogen-defect interactions in engineering alloys (also in H-Mat)
- **Advanced characterization** of deformation and fracture mechanisms due to long-term exposure of hydrogen isotopes
- Fundamentals of grain boundary fracture in model engineering metals

Extensive database exists for tensile behavior in hydrogen environments



- Strength properties are generally not affected by hydrogen
- Nickel correlates with ductility in hydrogen as well as any other indicator
- Austenite stability is not as good an indicator when nitrogen-strengthened alloys are considered

Typical austenitic stainless steel maintain high fracture resistance in hydrogen environments



- Fracture resistance in gaseous hydrogen (K_{IH}) is reduced by >50% for all steels on this plot
- General trend of lower fracture resistance with higher strength
- More highly alloyed and/or complex alloys can have lower fracture resistance

Diverse range of austenitic stainless steels have been evaluated in fatigue, including high-strength alloys

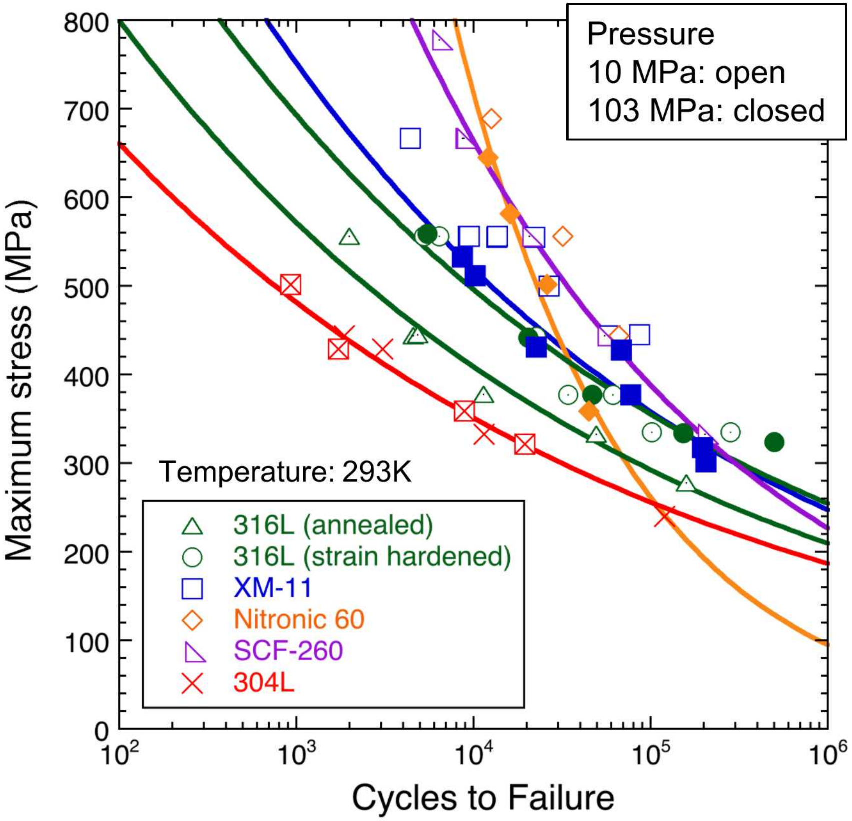
material	Yield (MPa)	Tensile (MPa)	Cr	Ni	Mn	N	Typical allowable stress (MPa)
316L	280	562	17.5	12	1.2	0.04	115
CW 316L	573	731	17.5	12	1.2	0.04	218
304L	497	721	18.3	8.2	1.8	0.56	195
XM-11	539	881	20.4	6.2	9.6	0.26	207
Nitronic 60	880	1018	16.6	8.3	8.0	0.16	218
SCF-260	1083	1175	19.1	3.3	17.4	0.64	333

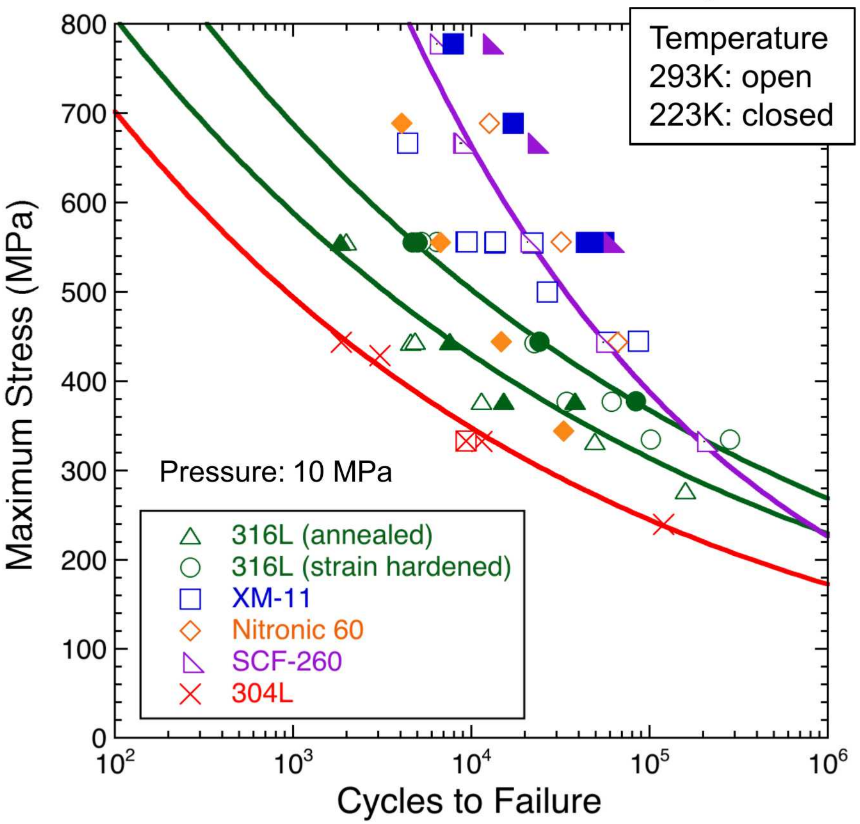
Wide range of strength

Wide range of Ni/Mn content

Fatigue life data shows less dramatic effects from hydrogen than tensile data

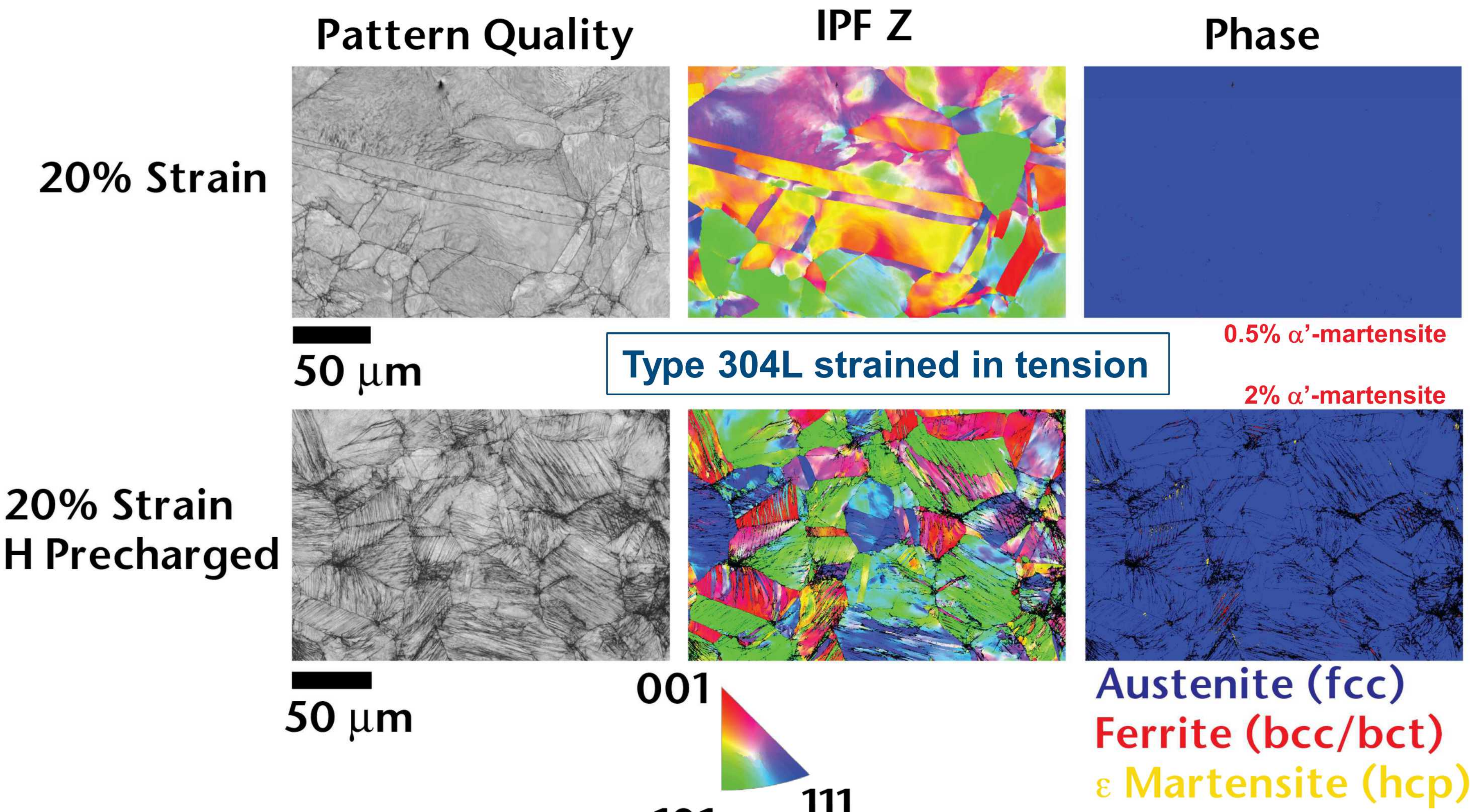
CNT specimens
$R = 0.1$, $f = 1\text{Hz}$





- Pressure has modest effect on fatigue life
- Low temperature increases fatigue life (or has no effect)
- Very high-strength alloys might be an exception in some cases

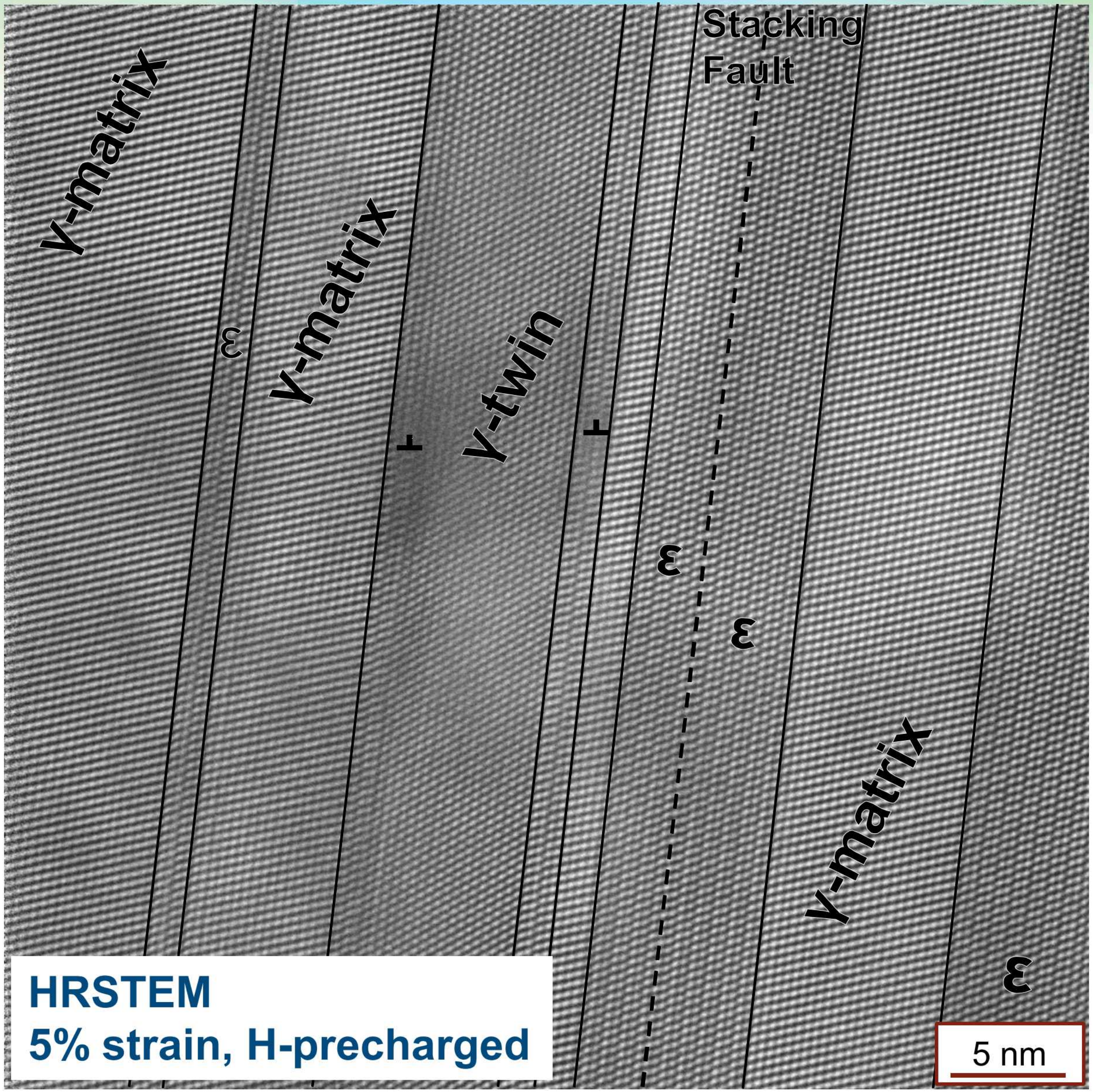
Hydrogen has significant effect on deformation character of austenitic stainless steels



Josh Sugar (SNL/CA)

Characterization at the nanometer length scale reveals unique structure

- HR-STEM shows some interface dislocations ($\frac{1}{6}\langle 112\rangle$ and $\frac{1}{3}\langle 111\rangle$) with no dislocations observable within twins, matrix, or ε-martensite
- Martensite is more common here than twinning (typical for H-precharged samples)
- Twins and ε-martensite are generally very thin (less than ~20 $\{111\}$ planes) while spanning through most of the grain. With twins appearing as faulted ε-martensite



HRSTEM
5% strain, H-precharged

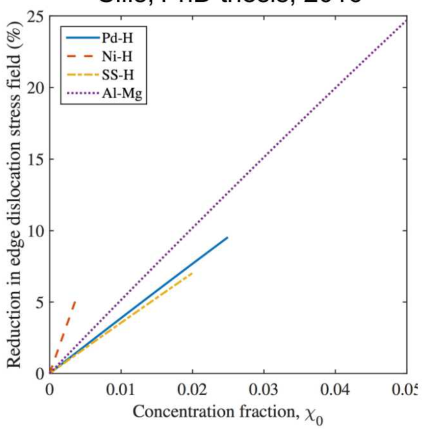
Julian Sabisch &
Doug Medlin (SNL/CA)

Re-examination of the HELP mechanism

Recently, the mechanisms underlying HELP have been examined by several research groups:

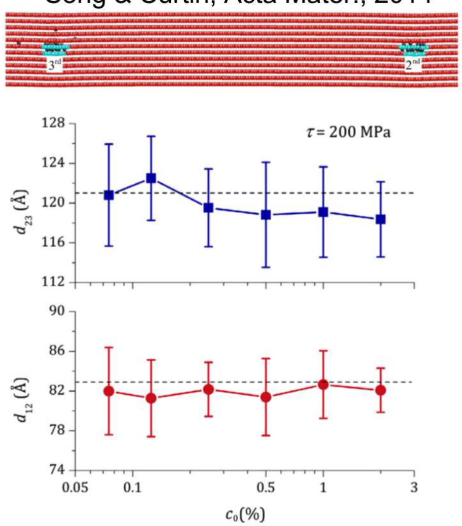
Stress field screening

Sills, PhD thesis, 2016



Small stress field reduction is small

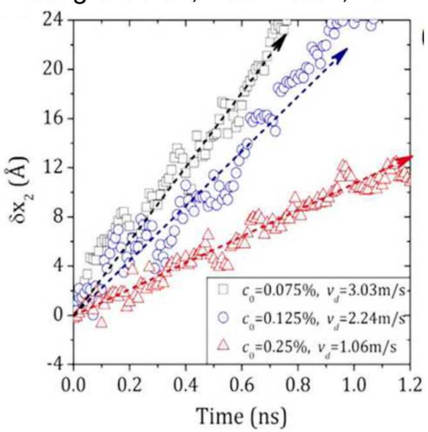
Song & Curtin, Acta Mater., 2014



Pile up separation distance not affected by H

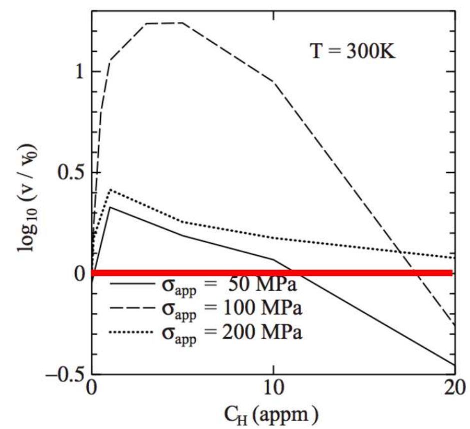
Increased mobility

Song & Curtin, Acta Mater., 2014



H *reduces* dislocation mobility with extended dislocations (FCC, HCP, BCC non-screw)

Katzarov et al., Phys. Rev. Mater., 2017



H *sometimes* increases mobility of BCC screws

- Similar findings by Gu et al (JMPS, 2018) and Yu et al (JMPS, 2019) with DDD

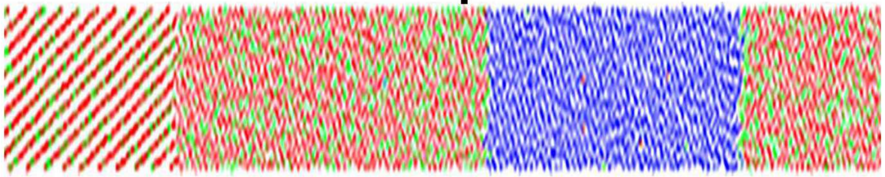
Neither *stress-field screening* nor *hydrogen-enhanced dislocation mobility* offer robust explanation of hydrogen effects in metals

Ryan Sills (Rutgers)

Nanoscale simulations are essential to illuminating hydrogen-deformation interactions

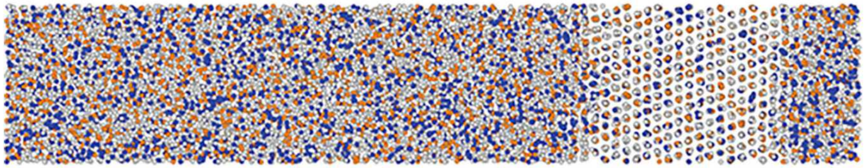
- Molecular dynamics, for example, provide opportunity to evaluate experimentally derived hypotheses
  - Atomic (MD) simulations require robust interatomic potentials
  - However, interatomic potentials for complex alloy systems are generally limited and inadequate

Literature potential



Newly developed Fe-Cr-Ni potential

fcc $Fe_{0.6}Ni_{0.2}Cr_{0.2}$, atom map, T_m = 2100 K



- Current activity focused on developing Fe-Cr-Ni-H interatomic potential to investigate fundamental processes:
  - Deformation structure interactions in the presence of hydrogen
  - Role of twinning and phase changes on deformation
  - Evolution of damage structures

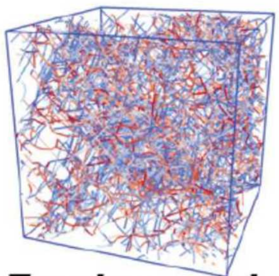
Xiaowang Zhou (SNL/CA)

Engineering performance depends on mechanisms manifest at nanometer length scales

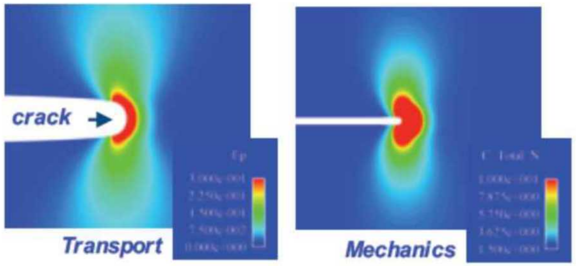
Approach: Integrate innovative computational & experimental activities across length scales

Advanced computational materials science

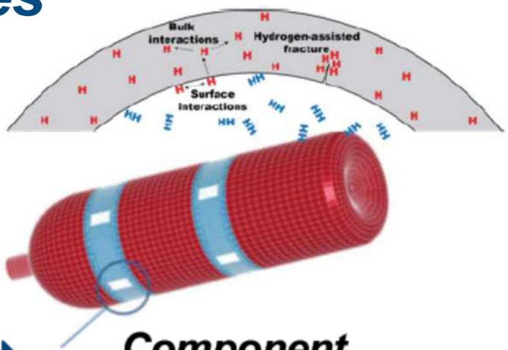
Innovative experimental capabilities



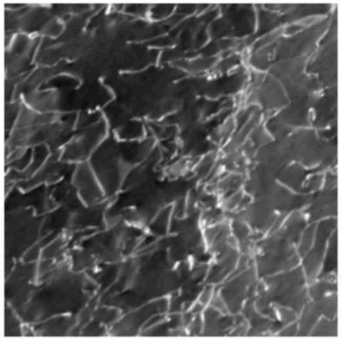
Fundamental hydrogen-materials interactions

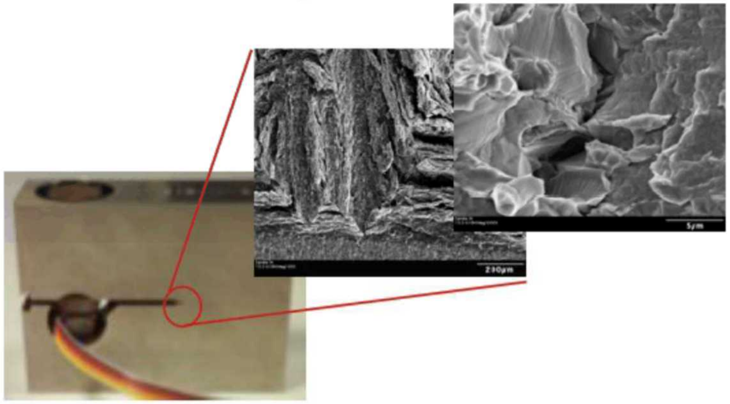


Materials response



Component design





Thermodynamics of H-interactions at nm-scale

Micromechanisms of materials behavior at μm-scale

Structural performance at the engineering scale

H-Mat addresses materials-compatibility science questions

Metals

Task M1
High-strength ferritic steel microstructures

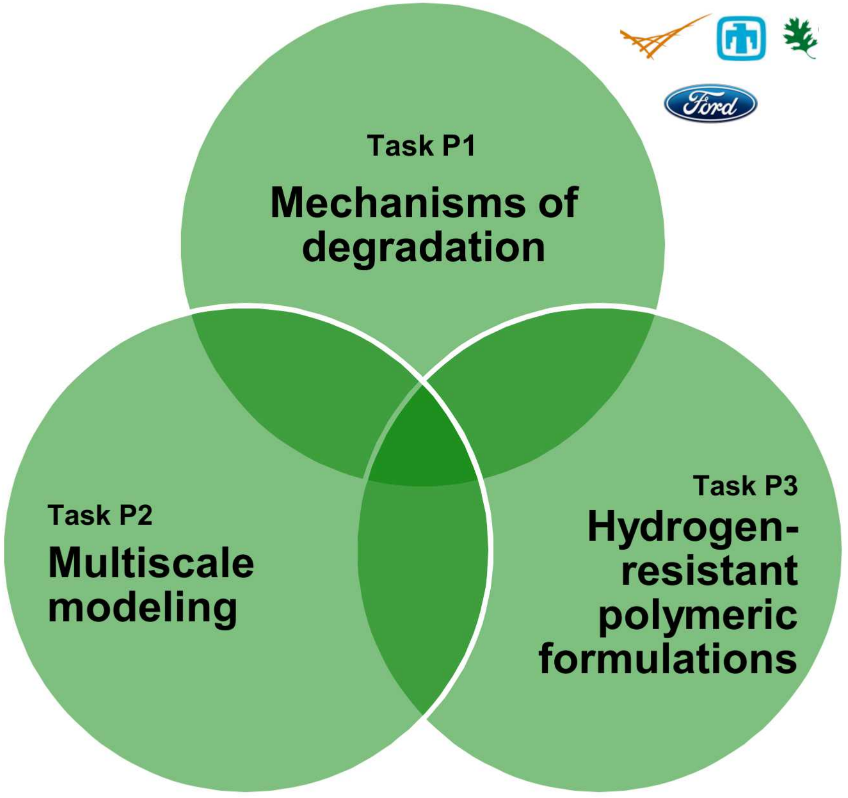
Task M2
High-strength aluminum alloys

Task M3
Transferability of damage and crack nucleation

Task M4
Microstructure of austenitic stainless steels

Task C1
Materials for cryogenic hydrogen service

Polymers



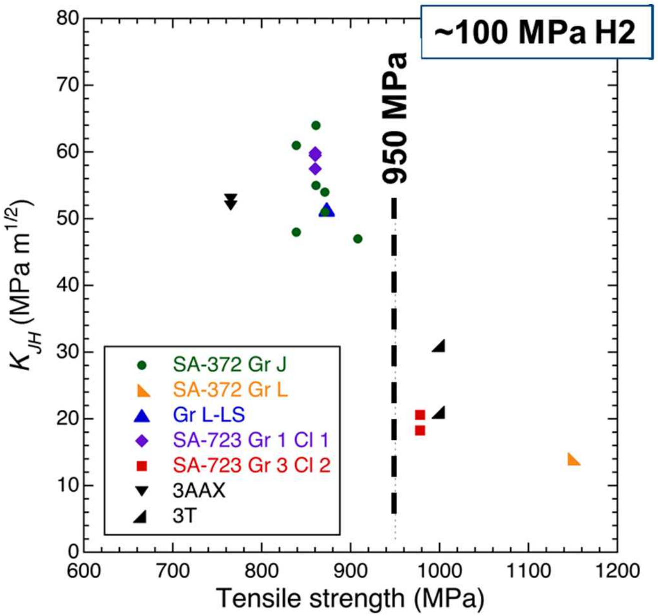
Relevance and Objectives

Motivation: elucidate the mechanisms of hydrogen-materials interactions to inform **science-based strategies to design the microstructure** of metals with improved resistance to hydrogen degradation

Task	Relevance and Objective
High-strength ferritic steel microstructures	Develop a mechanistic understanding of hydrogen-induced fracture processes in ferritic steel microstructures to improve fracture resistance of low-cost steels with tensile strength >950 MPa
High-strength aluminum alloys	Elucidate mechanisms of hydrogen embrittlement in high-strength aluminum alloys and the role of moisture in hydrogen surface interactions in this class of materials
Transferability of damage and crack nucleation	Understand the mechanics of hydrogen-induced deformation and damage in fatigue environments at multiple length scales toward a framework to implement crack nucleation in structural design
Microstructure of austenitic stainless steels	Identify governing physical processes of hydrogen embrittlement in austenitic stainless steels to design microstructures that mitigate the adverse effects of hydrogen environments
Materials for cryogenic hydrogen service	Identify materials for cryo-compressed hydrogen storage onboard vehicles, and develop key technical metrics for viable structural materials in this application

Hydrogen-resistant, high-strength ferritic steel microstructures (task M1)

Science question:
Are there high-strength steel microstructures that can be resistant to hydrogen effects?



- Mechanical testing of steels in high pressure H_2
- Development of unique microstructures (e.g., austempering)
- Microstructural and fracture characterization
- Kelvin Probe Force Microscopy to investigate hydrogen distribution in different microstructures
- Modeling of Fe-C-H (DFT and MD) to explore preferential locations for hydrogen in microstructure from physics standpoint

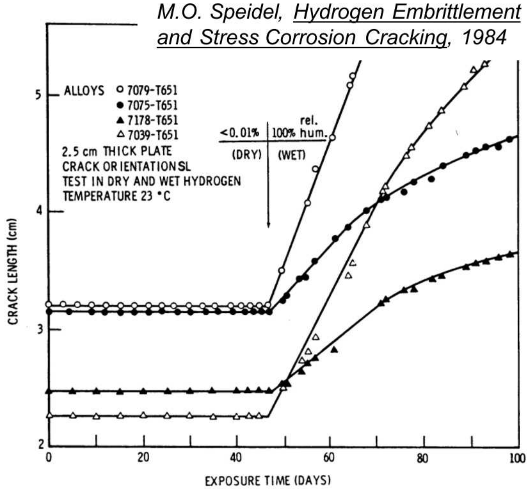
Engineering goals:

- **Achieve K_{JH} > 50 MPa m$^{1/2}$ for steels with UTS > 950 MPa**
- **Ferritic steel microstructures with tensile strength up to 1100 MPa and 50% increase of fracture resistance in high-pressure hydrogen**

High-strength aluminum alloys (task M2)

Science question:
What are the mechanisms of environmental embrittlement of high-strength aluminum alloys in high-pressure hydrogen?
(in particular, what is role of moisture?)

M.O. Speidel, Hydrogen Embrittlement and Stress Corrosion Cracking, 1984



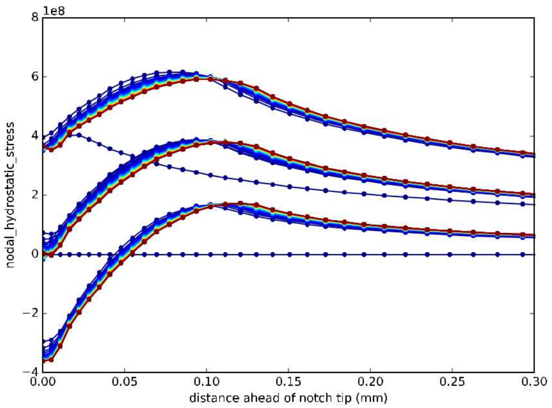
- Mechanical testing of aluminum in mixed gases (H_2 + H_2O) at high pressure
- Kelvin Probe Force Microscopy to investigate moisture on Al surfaces
- Modeling of moisture on Al surfaces to identify and quantify mechanisms of H uptake (DFT) and microstructural interactions of dissolved H (MD)

Engineering goals:
- **Hydrogen-compatible microstructures of aluminum alloys with yield strength >350 MPa that are insensitive to standardized moisture limits for fuel-grade hydrogen (5ppm H_2O)**
- **Specification of environmental conditions under which aluminum is not degraded in gaseous (and liquid) hydrogen environments**

Transferability of damage and crack nucleation in hydrogen environments (task M3)

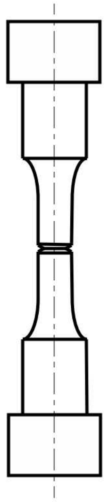
Science questions:
- **Can the mechanics of damage be generalized such that crack nucleation can be predicted in the context of design lifetimes?**
- **What are the mechanisms of hydrogen-defect interactions that lead to damage accumulation?**

- Atomistic modeling of defect structures to rank-order the effects of hydrogen on defect evolution
- Continuum modeling of test specimen geometry to develop normalization schemes correlating material evolution to fatigue crack nucleation
- Experimental evaluation and microstructural quantification of hydrogen-affected cyclic deformation and fatigue crack nucleation

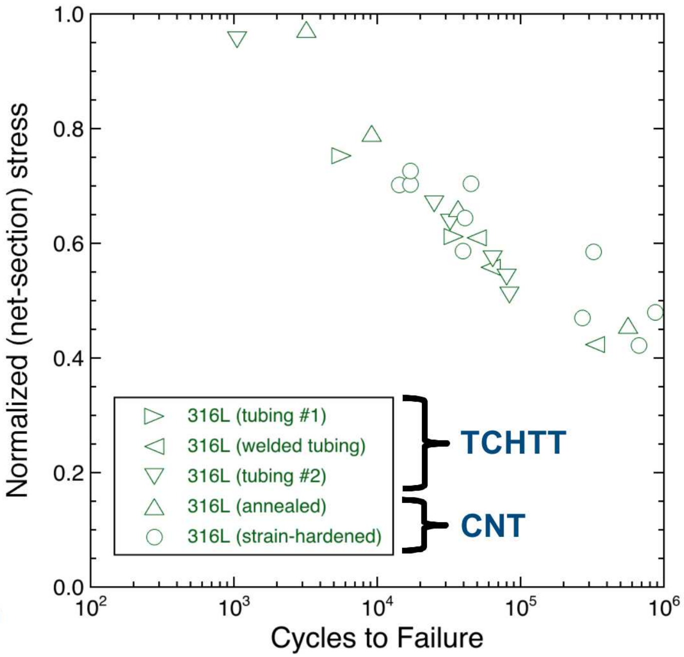


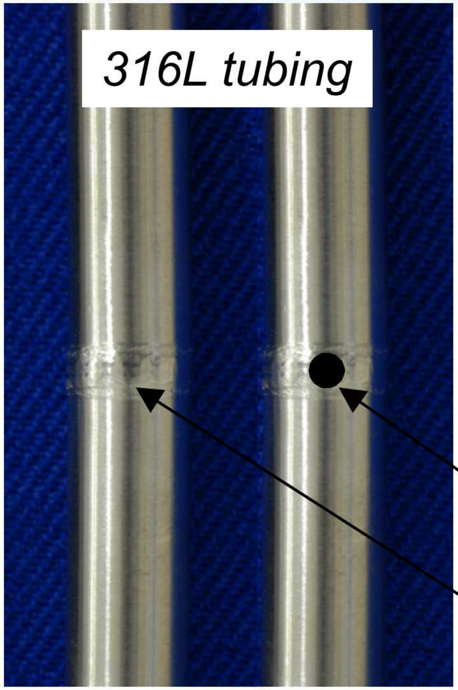
Engineering goals:
- **Framework for quantification of damage and crack nucleation that can be implemented in design to increase lifetime assessment by 50% compared to conventional fracture mechanics approach**
- **Microstructural requirements that minimize effects of hydrogen**

Experimental and computational studies explore mechanics associated with damage and cracking



CNT specimen





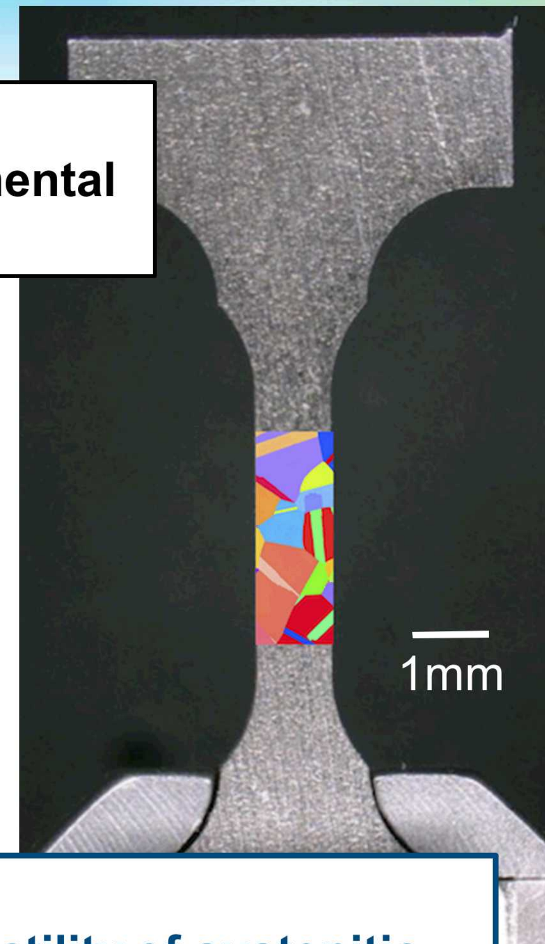
Transverse circular hole in tube tension specimen
Kt ~ 3

Behaves nominally the same as bulk specimen

- **Experimental evidence suggests that simple scaling parameters enable generalization of behavior**
- **Continuum simulations provide an assessment of similarity of mechanical fields to support generalizations**

Mechanisms of hydrogen-deformation interactions in austenitic stainless steels (task M4)

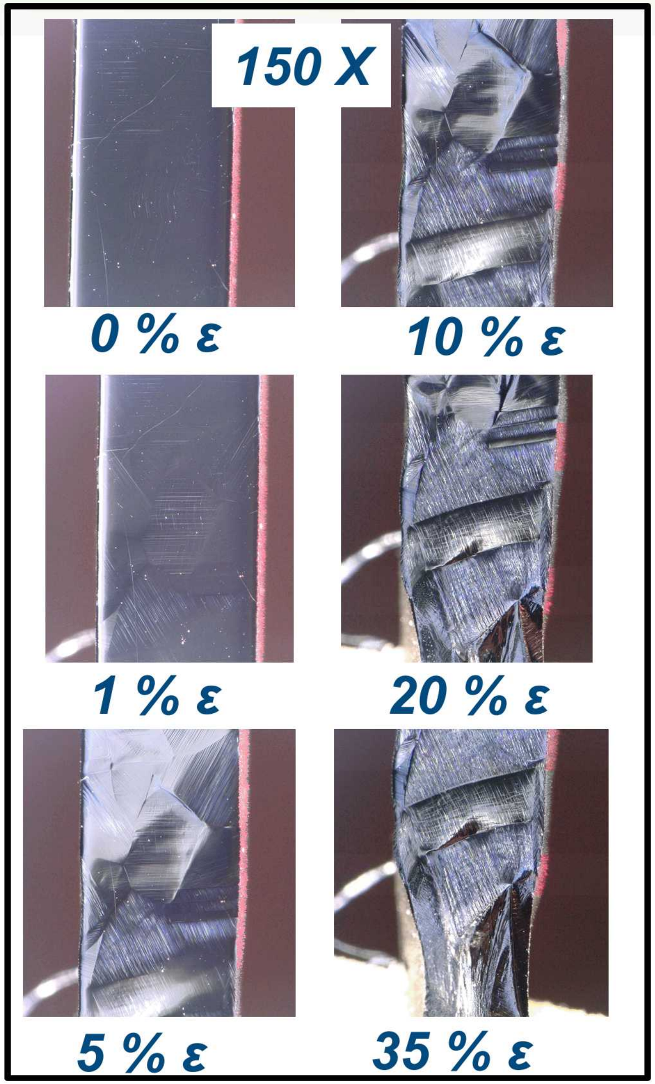
Science question:
How does hydrogen change deformation and fundamental boundary interactions in austenitic stainless steels?

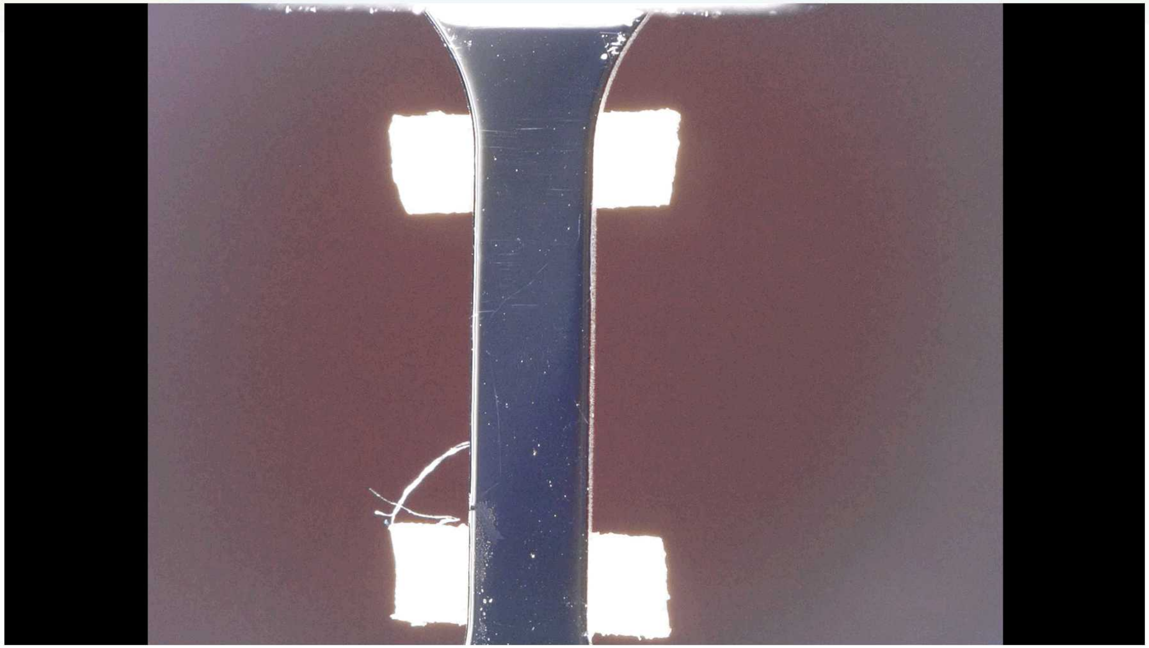


- Develop methods to test and evaluate single crystals (leveraged) and oligocrystals of austenitic stainless steels
- In situ testing and local characterization of strain and damage accumulation
- Micromechanical modeling of oligocrystals with internal hydrogen (CP) to illuminate mechanisms of hydrogen-microstructure interactions

Engineering goals:
- **Microstructural design concepts that improve ductility of austenitic stainless steels in high concentration of hydrogen**
- **Accessible micromechanical modeling tools (CP) sensitive to hydrogen transients, local microstructure, and phase transformations**

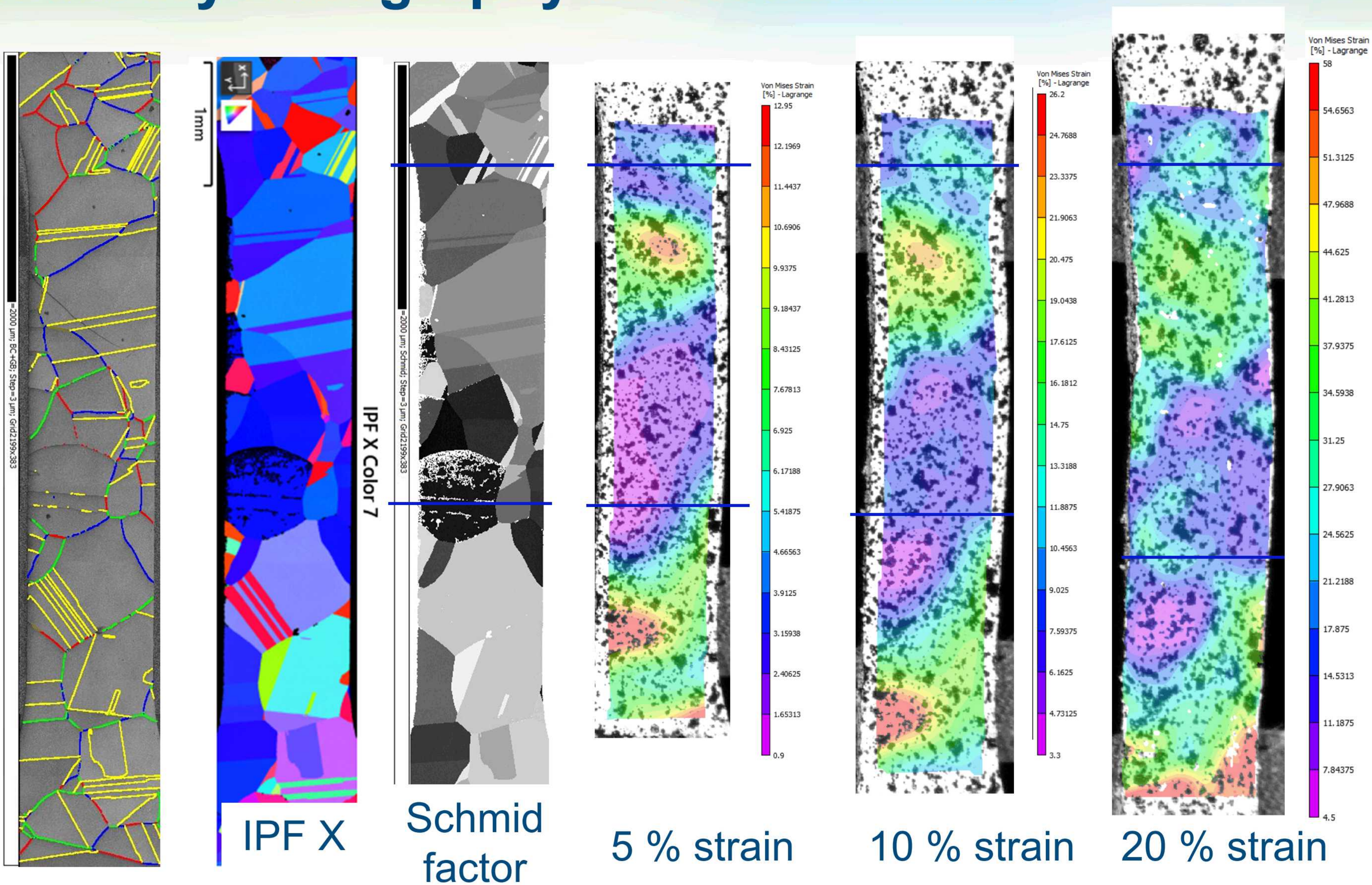
In situ mapping of oligocrystal specimens to inform evolution of deformation structures





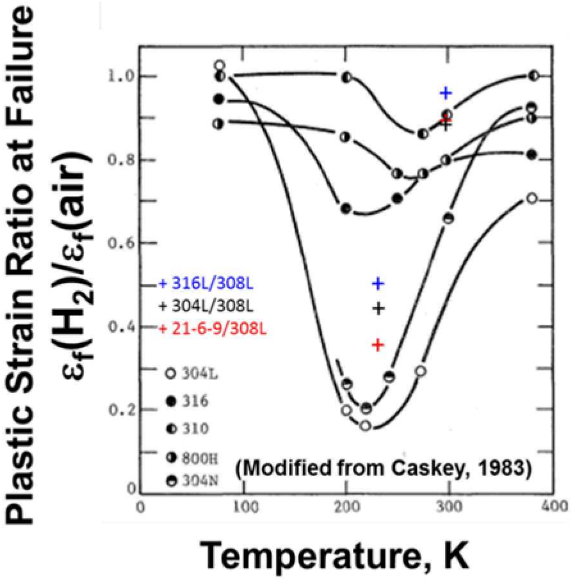
- Slip bands visible early in strain history
- Grain structure clearly visible after <10% strain
- Necking appears to be governed by local crystallography

EBSD and DIC provide relationship between local crystallography and local strain



Metals for cryogenic applications in hydrogen service (task C1)

Science question:
At cryogenic temperature, what are the kinetic versus mechanistic limitations to the manifestation of degradation in hydrogen environments?



- Identify effects of hydrogen-induced cracking at LH2 temperature
- Design of mechanical tests to elucidate kinetic factors on testing results (such as test rate)
- Evaluate weld microstructures at low-temperature with high hydrogen concentration

Engineering goals:
- **Guidance for long-term performance of materials in cryogenic environments**
- **Performance metrics (e.g., fatigue) for structural welds in cryogenic hydrogen applications**

Summary

- **Sandia has a rich history and active programs studying hydrogen effects in austenitic stainless steels at multiple length scales**
  - Fatigue and fracture in high-pressure gaseous hydrogen
  - Nanoscale characterization of deformation
  - Computational materials science and mechanics
- **H-Mat** is a growing consortium of partners to address the **materials science of hydrogen-induced degradation** of materials
- **H-Mat** integrates advanced **computational materials science** and **innovative experimental capabilities** from microstructural to engineering length scales

H-Mat seeks to develop **science-based strategies** to **design materials microstructures** for resistance to hydrogen-assisted fatigue and fracture

H-Mat Hydrogen Materials Compatibility Consortium

Acknowledgment of national labs H-Mat team

Task	Lead	Principal Contributors
High-strength ferritic steels	Joe Ronevich	• Xiaowang Zhou, Catalin Spataru (computational) • Zhili Feng, Yanli Wang (material/microstructure) • Joy McNamara, Andy Duncan (KPFM) • Chris San Marchi (experimental)
High-strength aluminum alloys	Chris San Marchi	• Norm Bartelt, Xiaowang Zhou (computational) • Joy McNamara, Andy Duncan (KPFM) • Joe Ronevich (experimental)
Damage and crack nucleation	Jay Foulk	• Ryan Sills (computational – nanoscale) • Vincente Pericoli, Guy Bergel (computational – continuum) • Joe Ronevich, Chris San Marchi (experimental)
Austenitic stainless steels	Coleman Alleman	• Jay Foulk (computational) • Brian Kagay (characterization/experimental) • Chris San Marchi, Joe Ronevich (experimental)
Cryogenic hydrogen service	Aashish Rohatgi	• Daniel Merkel (experimental) • Chris San Marchi (materials)