Microstructural Engineering and Accelerated Test Method Development to Achieve Low Cost, High Performance Solutions for Hydrogen Storage and Delivery

2020 DOE Hydrogen and Fuel Cells Program Review Presentation

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Project ID IN021

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Overview

Timeline and Budget

- Project Start Date: February 1, 2020
- Project End Date: February 28, 2023
- Total Project Budget: $1,804,560
    - Total Recipient Share: $360,912
    - Total Federal Share: $1,443,648
    - Total DOE Funds Spent*: $5,315

* As of 3/31/2020

Barriers – Hydrogen Delivery

B. Reliability and Costs of Gaseous Hydrogen Compression

D. High As-Installed Cost of Pipelines

E. Gaseous Hydrogen Storage and Tube Trailer

Partners

Project Lead: Colorado School of Mines

Los Alamos National Laboratory

National Renewable Energy Laboratory

WireTough

U.S. Steel

General Motors

H-Mat Consortium (Sandia National Lab)

POSCO (cost share participant)

Relevance

Project Objectives:

a) Develop lower cost steel alloys with novel microstructural design for use in hydrogen refueling infrastructure such as storage, compressors, and dispensing components.
b) Develop and validate accelerated test methods to efficiently evaluate variations in alloy and microstructure design through electrochemical hydrogen charging (versus hydrogen gas charging)

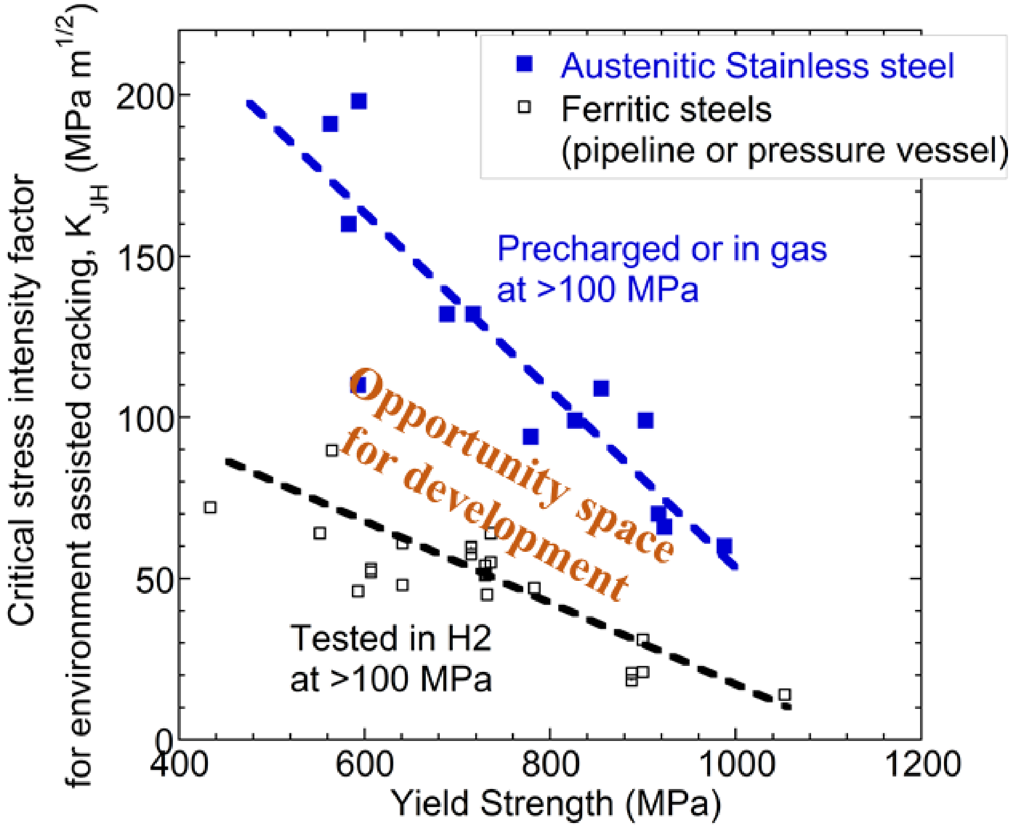
Current Year Objective:

Design and produce experimental alloys for the investigation.

Impact on Hydrogen Delivery Barriers:

- Identified initial lower cost austenitic steels in consultation with POSCO for hydrogen embrittlement susceptibility.
- Established initial project plans for each team member in kick-off meeting held April 14, 2020.

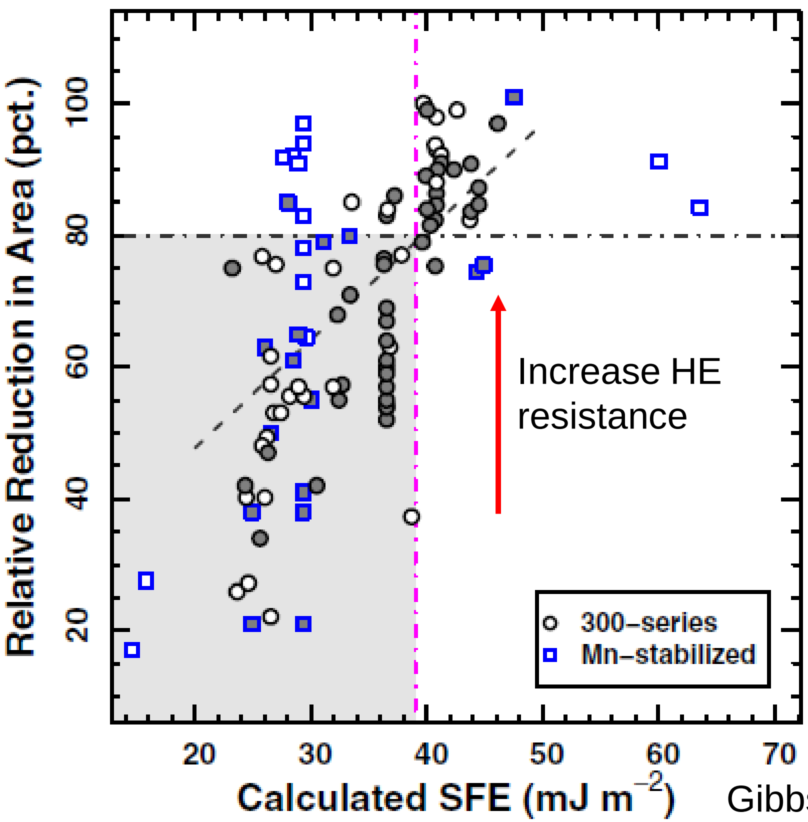
Overall Approach



Our approach involves developing lower cost austenitic alloys that meet or exceed the hydrogen embrittlement performance of austenitic steels and lower cost ferrite-austenite alloys that have intermediate hydrogen embrittlement performance between austenitic stainless steels and lower alloy ferritic steels.

Approach – Low Cost Austenitic Alloys

- Replace Ni with lower cost elements such as Mn to produce lower cost austenitic alloys (lower hydrogen diffusion)
- Utilize alloying approaches to achieve deformation mechanisms (through changes in stacking fault energy) known to be beneficial for hydrogen resistance.



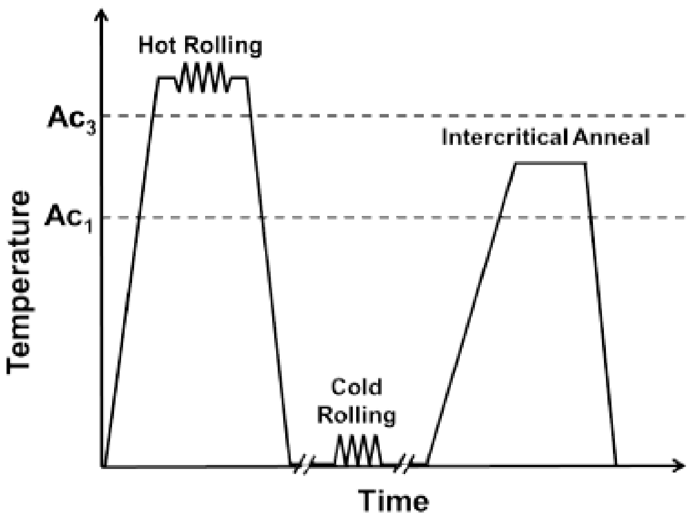
Gibbs et al., JOM, 2020

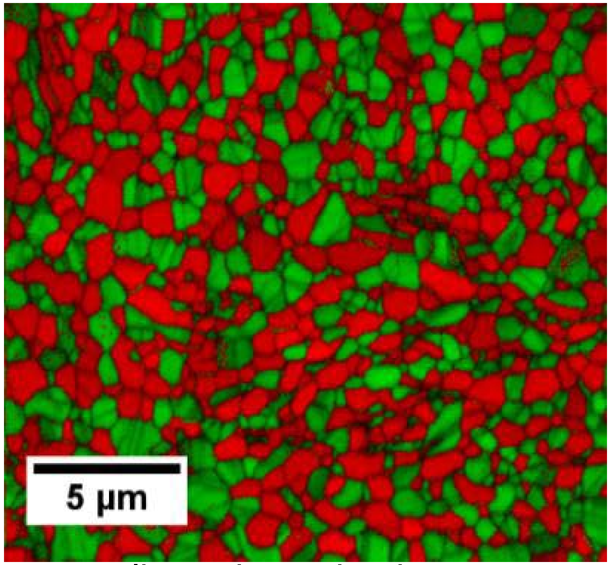
The plot indicates that hydrogen embrittlement susceptibility can be tuned through alloying that changes stacking fault energy

Approach – Ferrite-Austenite Microstructures

- Mixed ferrite-austenite microstructures may offer lower cost solutions for hydrogen gas transmission/storage with hydrogen embrittlement resistance sufficient for these intended applications
- Consider both medium Mn and duplex microstructure approaches
- Microstructure morphology can potentially be altered through thermomechanical processing to change HE resistance

0.1-0.2 wt.% C, 6-7 wt. % Mn



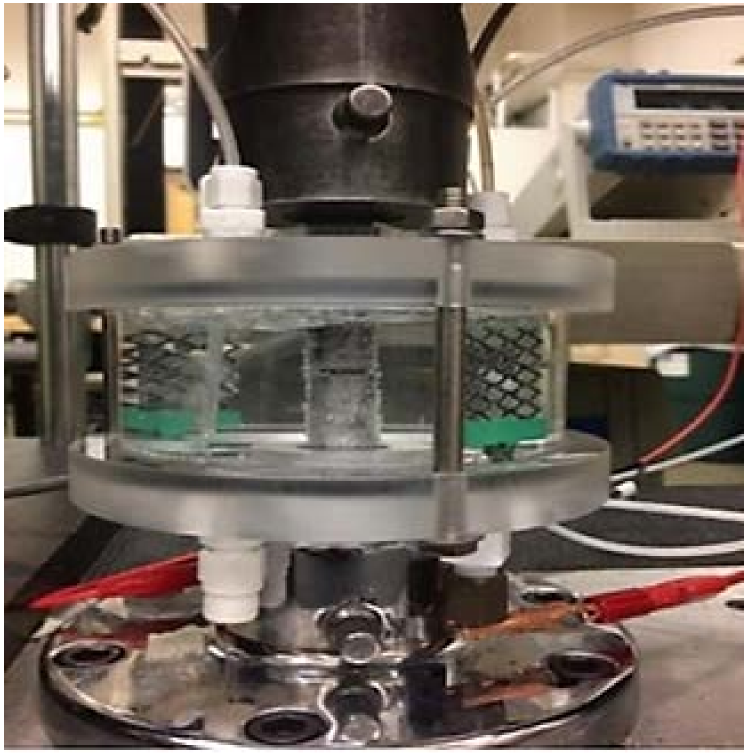


Poling, Ph.D. Thesis, 2016

Thermomechanical processing to achieve fine grain ferrite-austenite microstructures

Approach – Accelerated Testing

- Gaseous H-charging facilities are limited and expensive
- Objective: Identify test conditions that achieve comparable results between electrochemical and gaseous charging
  - Consideration of hydrogen transport characteristics associated with test conditions



Electrochemical hydrogen charging setup utilized to evaluate fracture toughness of steels in the presence of hydrogen-containing environments

Overall Project Outcomes

O1) Alloy and microstructure design strategies to achieve enhanced toughness and reduce cost for hydrogen infrastructure components used in compressors, dispensers, and storage.

O2) An economic analysis for the reduction in costs associated with the alloy strategies developed in this work compared to current alloys for these components, including 316 stainless steel for compressors and dispensers and ASTM 723 and 372 for pressure vessel storage.

O3) Development and demonstration of utilizing electrochemical charging methods as a surrogate accelerated testing method for gaseous hydrogen environments, which will enable cheaper and more broadly available testing to simulate gaseous hydrogen environments.

Tasks and Milestones

Task 1.0: Hire Staff (M1-M3). Hire a post-doctoral researcher, 2 graduate students, and undergraduate student to staff the project.

- **Milestone 1.1**: Hire and integrate all members of the project team. (M3)
  - Lawrence Cho (Research faculty), Pawan Kathayat (M.S. student), 1 grad and 1 undergraduate to be hired
- **Milestone 1.2:** Hold project kick-off meeting with all of the partners and H-Mat consortium. (M3)
  - April 14, 2020

Tasks and Milestones

Task 2.0: Alloy Design (M1-M9). Design the initial iteration of alloy composition and microstructures. This task will inform production of the initial set of alloys for continued testing and characterization.

- **Subtask 2.1:** Literature Review (M1-M6). We will review the literature to guide the alloy and microstructure design.
  - **Milestone 2.1.1** Complete initial literature review. (M6)
  - TWIP and ferrite-austenite steels, HE mechanisms, H-transport
- **Subtask 2.2:** Use computational tools and literature basis to inform alloy design (M2-M8).
  - **Milestone 2.2.1 (Go/No-Go Milestone)** Identification of approximately 6 alloying and microstructure strategies to hit the target strength levels of 400-800 MPa. (M9)
  - Mines to lead with input from U.S. Steel and project team

Tasks and Milestones

Task 3.0: Assessment of Alloys Provided by POSCO (M6-M12). Initial set of experiments on alloys made available by POSCO that have comparable microstructures to those sought in this work. The properties and deformation mechanisms will be analyzed and correlated to the microstructure and deformation mechanisms.

- Mines/LANL/Sandia – HE Testing and Characterization

POSCO Alloys

Designation: A1106/A1106M – 17

Standard Specification for Pressure Vessel Plate, Alloy Steel, Austenitic High Manganese for Cryogenic Application[1]

TABLE 1 Chemical Requirements

Element[A]	Composition, %
Carbon	0.35–0.55
Manganese	22.50–25.50
Phosphorus, max	0.030
Sulfur, max	0.010
Silicon[B]	0.10–0.50
Chromium	3.00–4.00
Copper	0.30–0.70
Boron, max	0.0050
Nitrogen, max	0.050

[A] Applies to both heat and product analyses.
[B] Silicon may be less than 0.10 %, provided total aluminum is 0.03 % or over, or provided acid soluble aluminum is 0.025 % or over.

TABLE 2 Tensile Requirements

Tensile strength, ksi [MPa]	116–141 [800–970]
Yield strength (0.2 % offset), min, ksi [MPa]	58 [400]
Elongation in 2 in. [50 mm], min, %[A]	22.0

- 2-3 lab chemistries with 0.25C and 25 or 30Mn
  - Cr, Ni, Al alloyed to increase SFE

Tasks and Milestones

Task 4.0: Test Bed Methodology (M6-M12). Develop a test bed methodology for fracture toughness testing in gaseous and electrochemical environments with associated analytical modeling to link the stress and strain states to performance in the hydrogen environments.

- **Milestone 4.1:** Determine test bed methodology for fracture toughness and fatigue crack growth testing, which includes specific guidance, based on analytical approaches, for the testing parameters that will be employed within the experimental capabilities of the available laboratories. (M12)
  - Mines/WireTough/Sandia

Tasks and Milestones

Task 5.0: Hydrogen Uptake Measurements (M6-M12). LANL and Sandia National Lab will conduct hydrogen permeability and diffusivity measurements in electrochemical and gaseous charging environments to guide fracture toughness testing. The results from this task will inform future mechanical testing parameters for hydrogen embrittlement testing.

- **Milestone 5.1:** Complete initial hydrogen permeability and diffusivity measurements to validate experimental approach and guide future measurements. (M12)
  - LANL/Mines/Sandia

Year 2 Tasks

- **Task 6.0: Produce experimental alloys (M12-M15). U.S. Steel**
- **Task 7.0: Alloy Screening (M11-M15). Mines**
- **Task 8.0: Hydrogen Embrittlement Testing (M13-M30). Mines/Sandia/WireTough**
- **Task 9.0: In-situ Mechanical Testing at LANSCE (M13-M24). LANL/Mines**
- **Task 10.0: Evaluation of Initial Alloy Designs (M13-M24). Project Team**

Year 2 Milestones

- **Milestone 6.1: Produce experimental alloys (M15). U.S. Steel**
- **Milestone 7.2.1: Alloy screening (M15). Mines**
- **Milestone 8.1: Initiate hydrogen embrittlement testing (M18). Mines/Sandia/WireTough**
- **Milestone 8.2.1: Gaseous and electrochemical hydrogen charging comparison (M21). Mines/Sandia**
- **Milestone 9.1. Establish methodology for in-situ mechanical testing at LANSCE (M18). Mines/LANL**
- **Milestone 10.1: Evaluation of Initial Alloy Designs (M24). Project Team**

Year 3

- **Task 11.0: Economic Assessment (M25-M36). NREL/WireTough/GM**
- **Task 12.0: Refine Electrochemical Testing Methodology (M25-M33). Mines/Sandia**
- **Task 13.0: Fatigue Crack Growth Testing (M25-M30). Mines/Sandia/WireTough**
- **Task 14.0: Phenomenological Model Development (M25-M31). WireTough/Mines**
- **Task 15.0: Complete Testing and Characterization (M25-M36). Project Team**

Year 3 Milestones

- **Milestone 11.1: Initiate market transformation plan (M27). NREL/WireTough**
- **Milestone 14.1: Phenomenological model for fatigue crack growth in H validated by experimental data (M30). WireTough/Mines**
- **Milestone 15.1: Complete testing and characterization to achieve refined alloy conditions (M33). Project Team**
- **Milestone 15.2: Hold project meeting with all team members to identify next steps and opportunities (M36). Project Team**

Accomplishments and Progress

Project not reviewed last year

- Hired graduate research assistant and Research Professor (Milestone 1.1)
- Begun literature review and determined critical parameters for alloy design (Milestone 2.1.1 and Milestone 2.2.1)
- Held project kick-off meeting April 14, 2020 (Milestone 1.2)
- Coordinated with POSCO to obtain initial set of alloys (Milestone 5.1)
- Begun acquiring equipment for hydrogen uptake measurements (Milestone 5.1)

Collaboration and Coordination

Organization	Relationship	Role
Colorado School of Mines	Prime	Project lead, management and coordination, hydrogen embrittlement testing, alloy design
Los Alamos National Lab	Sub-recipient	Hydrogen transport and in-situ experiments
National Renewable Energy Lab	Sub-recipient	Market transformation analysis
WireTough	Sub-recipient, cost share	Test bed methodology development, market transformation plan
U.S. Steel	Cost share	Produce designed alloys, input on alloy feasibility
POSCO	Cost share	Provide initial materials for assessment
General Motors	Non-funded collaborator	Provide input on hydrogen vehicle market
H-Mat (Sandia National Lab)	Funded partner	Testing in gaseous hydrogen, input on relevant metrics and previous work

Remaining Challenges and Barriers

- **Challenge**: Maintaining project schedule with lack of lab accessibility due to COVID-19
  - **Planned Resolution**: Focus early work on computational design of alloys; work within institutional guidelines to optimize lab usage; maintain communication with partners and DOE about action items, deadlines, and potential need to re-prioritize based on work opportunities

Proposed Future Work
Based on Project Year (3/1/20 – 2/28/21)

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

- **Milestone 1.1** – Finalize staffing
- **Milestone 2.1.1** – Complete literature review
- **Milestone 2.2.1** – Identify ~6 alloying and processing strategies to hit target strength levels
- **Milestone 4.1** - Determine test bed methodology for fracture toughness and fatigue crack growth testing
- **Milestone 5.1** – Complete initial H transport measurements

Technology Transfer Activities

- Project kick-off meeting on April 14, 2020
  - Participation from multiple groups/management level personnel from U.S. Steel and General Motors
- Report and presentation of the project to industrial sponsors the Advanced Steel Processing and Products Research Center
  - Representatives from 31 industrial sponsors who are users and producers of steel in addition to several guests attended workshops where project was presented

Summary

Objective: The goal of the proposed program is to develop lower cost steel alloys with novel microstructural design for use in hydrogen refueling infrastructure such as storage, compressors, and dispensing components. In support of this effort, we will also develop and validate accelerated test methods to efficiently evaluate variations in alloy and microstructure design through electrochemical hydrogen charging; these charging methods will be designed to produce comparable results to testing in hydrogen gas.

Accomplishments: Hired staffing, held project kick-off meeting, initiated procurement of initial alloys for testing, initiated literature review, began obtaining equipment for hydrogen transport measurements

Partners: Colorado School of Mines, Los Alamos National Laboratory, National Renewable Energy Laboratory, WireTough, U.S. Steel, General Motors, Sandia National Laboratory, POSCO

Technical Backup Slides

Ferrite-Austenite Approach Open Questions

- Alloying for austenite stability, stacking fault energy, cost
- Duplex versus Medium Mn approaches
- Achieving target strength and ductility levels

Austenitic Steel Open Questions

- Alloying for austenite stability, stacking fault energy, cost

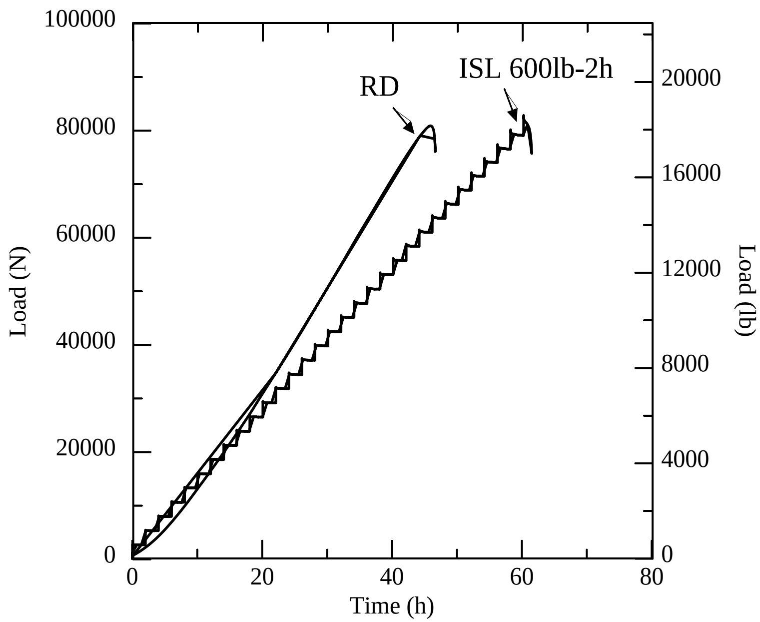
- Achieving target strength and ductility levels

Fracture Toughness in Hydrogen Approach (compared to gaseous H-charging)

Incremental Step Load (ISL):

- **Load is increased by set amount for a defined time and then specimen is held at constant displacement**

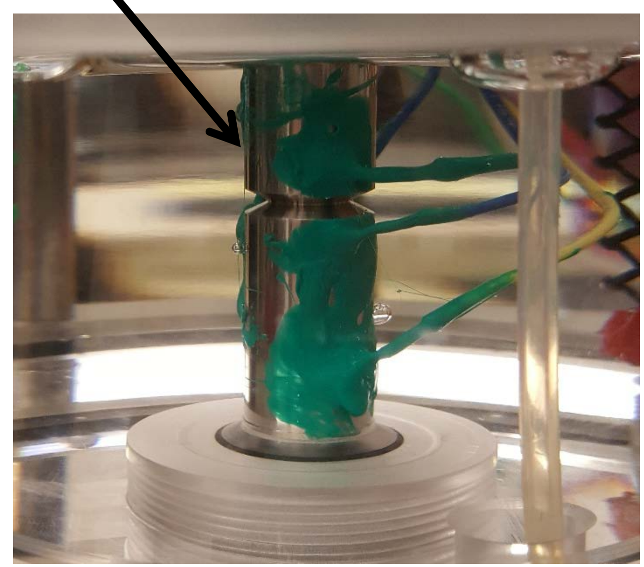
Rising Displacement (RD): continuously loaded throughout test



In situ **electrochemical charging:**

- **Solution: 0.5 M H_2SO_4**
- **5 mA/cm² current density**

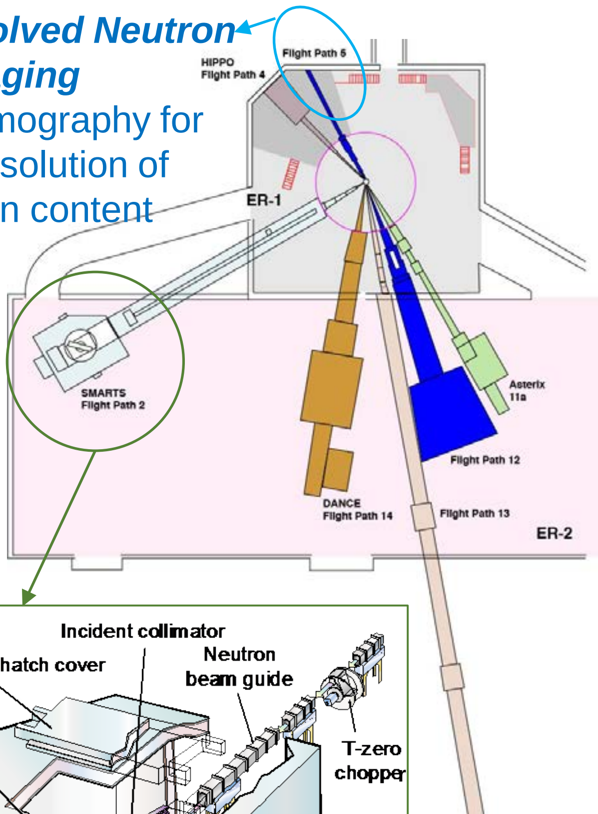
Circular notched tensile (CNT) specimens with sharp notch (K_t > 6)

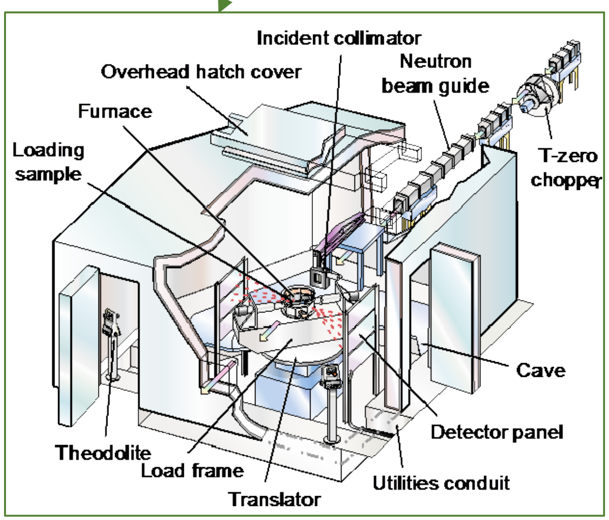


Kagay, Ph.D. thesis, Mines, 2019

In-situ loading paired with neutron diffraction used to interrogate hydrogen effects on deformation & phase transformations at Los Alamos Neutron Science Center

Energy Resolved Neutron Imaging
imaging/tomography for spatial resolution of hydrogen content

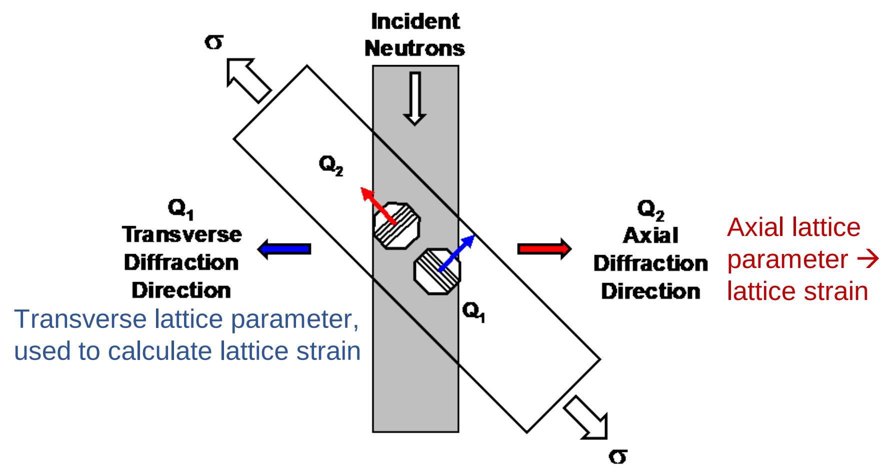




Spectrometer for Materials Research at Temperature and Stress
Neutron Diffraction w/ in-situ tensile/compression testing

We propose to:

1. Track lattice spacings as a function of applied strain during deformation of H pre-charged samples at *SMARTS*.
   i. Gain insight into effect of dissolved H on both phase stability and strain partitioning
   ii. Enhance understanding of microconstituents on strengthening mechanisms in alloys designed during this project



2. Potentially apply *ERNI* to compare light element (H or D) distributions in pre-charged alloys.
   i. Facilitate study of phase-specific H occupancy in multi-phase steels, further highlighting H-deformation interactions

LANSCE Images courtesy: https://lansce.lanl.gov/facilities/lujan/instruments/index.php
SMARTS schematic courtesy: P.J. Gibbs, et al. Mater. Sci. Eng. A, **609** (2014) 323–333