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Project ID # SCS005

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

Sandia National Laboratories

Brian Somerday
(Presenting)
Principle Investigator

Team Members: Chris San Marchi, Aaron Harris

DOE Hydrogen and Fuel Cells Program Annual Merit Review and Peer Evaluation Meeting
May 14, 2013

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Overview

Timeline

- Project start date: Oct. 2003
- Project end date: Sept. 2015
- Percent complete: 83%

MYRD&D 2012 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

Budget

- Project funding DOE share: $6.4M
- Funding received in FY12: $0.8M
- Planned funding for FY13: $0.4M

Partners

- **SDO/CDOs:** CSA, ASME, SAE, ISO
- **Industry:** FIBA Technologies, European cylinder manufacturer, Swagelok
- **Universities:** Boise State
- **Govt:** DOE Pipeline Working Group
- **International:** I2CNER (Kyushu University, Japan), AIST-Tsukuba

Project Approach

The Safety, Codes and Standards program coordinates critical stakeholders and research to remove technology deployment barriers

Partnerships with industry, labs, academia

Identify R&D needs → *Perform High-Priority R&D* → *Impact Codes and Standards*

Harmonize Internationally

Regulations, Codes and Standards (SAE, GTR, IEC)
International Standards (ISO)
International Agreements (IEA, IPHE)

NFPA ICC INTERNATIONAL CODE COUNCIL ASME SETTING THE STANDARD

Objectives/Relevance

Objective: Enable technology deployment by providing science-based resources for standards and hydrogen component development and participate directly in formulating standards

Barrier from 2013 SCS MYRDD	Project Goal
A. Safety Data and Information: Limited Access and Availability	Develop and maintain material property database and identify material property data gaps
F. Enabling national and international markets requires consistent RCS	Develop more efficient and reliable materials test methods in standards Design and safety qualification standards for components (SAE J2579, ASME Article KD-10) and materials testing standards (CSA CHMC1)
G. Insufficient technical data to revise standards	Execute materials testing to address *targeted* data gaps in standards and critical technology development

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FY2013 Approach and Milestones

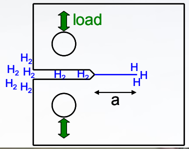
MYRD&D 2012 Barrier	FY13 Milestone	Status
A. Safety Data and Information: Limited Access and Availability	Investigate and propose concepts for material property database	Contributed materials compatibility data to OpenEI website
F. Enabling national and international markets requires consistent RCS	Optimize fatigue crack growth rate measurements for pressure vessel steels in H_2 and report results to ASME Enable completion of standards through committee leadership and data evaluation	Completed test matrix on two steels over range of H_2 pressure Publication of CHMC1 (Part 3) and SAE J2579 expected in 2013
G. Insufficient technical data to revise standards	Measure benchmark tensile fracture properties of H_2-exposed orbital tube welds in collaboration with industry partner Develop capability for variable-temperature testing in high-pressure H_2 gas	Completed testing of two welds at ambient and low temperature Two Boise State student teams designing pressure vessel concepts according to Sandia specifications

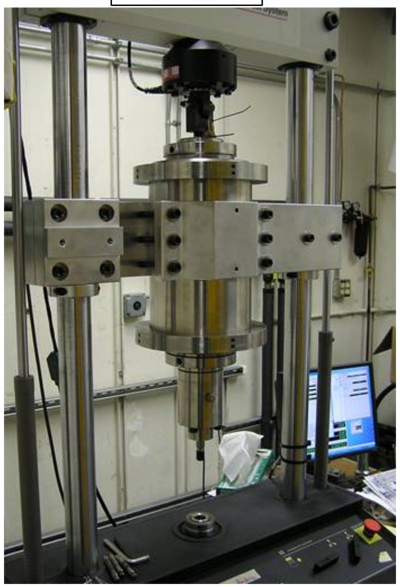
Materials Compatibility and Components project impacts multiple standards

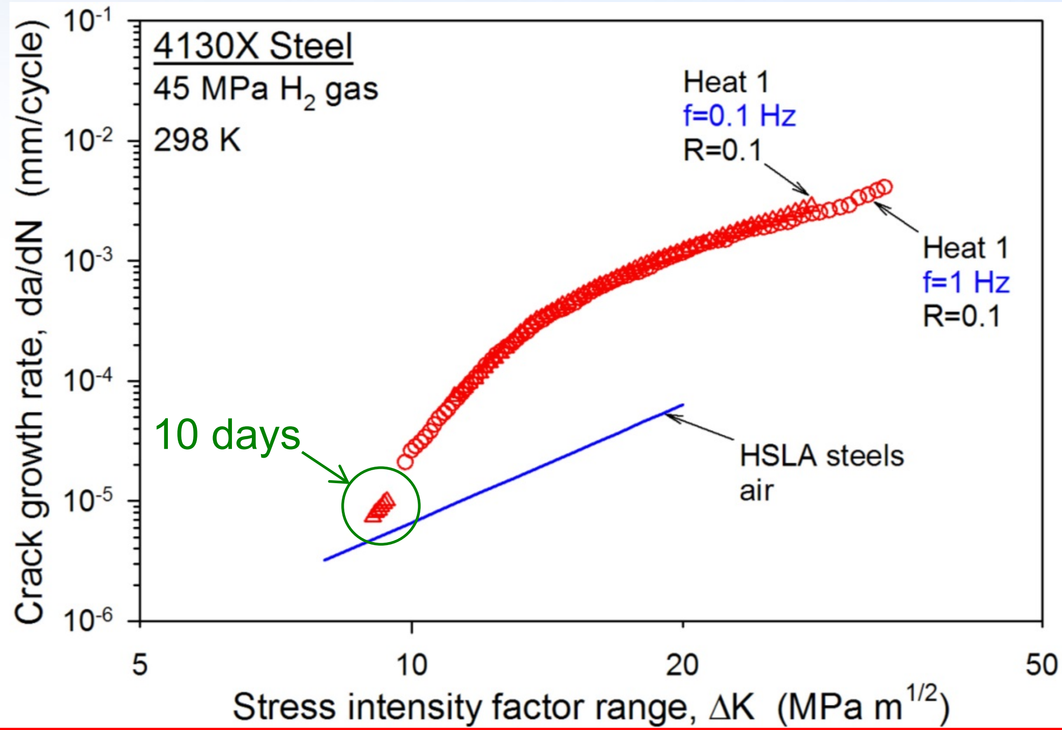
- CSA CHMC1
  - Materials testing and data application standard
  - Sandia provides leadership in technical committee and document preparation
  - Publication of Part 3 expected in 2013
- SAE J2579
  - Hydrogen vehicle fuel system standard
  - Sandia serves as U.S. technical lead on addressing hydrogen embrittlement
  - Publication expected in 2013
- ASME Article KD-10
  - Standard on high-pressure hydrogen tanks for transport and storage
  - Sandia provides data on exercising and improving materials test methods
  - Reporting progress on optimizing fatigue crack growth testing to former chair of ASME Project Team on Hydrogen Tanks

Previous Accomplishment:

Fatigue testing in H_2 must be optimized to balance efficiency and data reliability







- Load-cycle frequency, *f*, currently in ASME KD-10 (0.1 Hz) not practical
- *Goal: establish test procedure that shortens test duration without compromising data quality*
- Approach identified in FY11-12: measure *da/dN vs ΔK* at high frequency, apply correction based on limited low-frequency data

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Accomplishment:

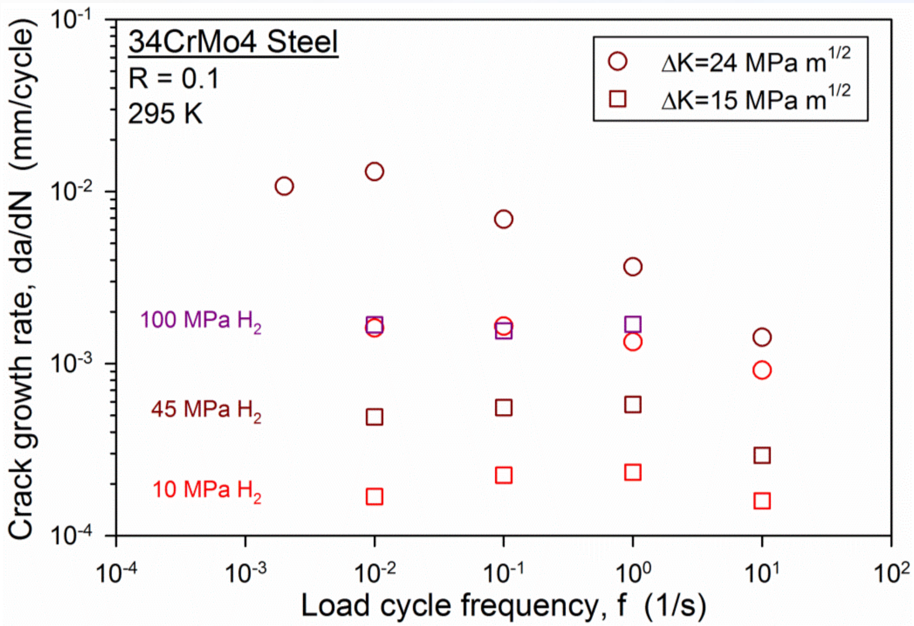
Completed test matrix to evaluate *da/dN* corrections: two steels at three pressures

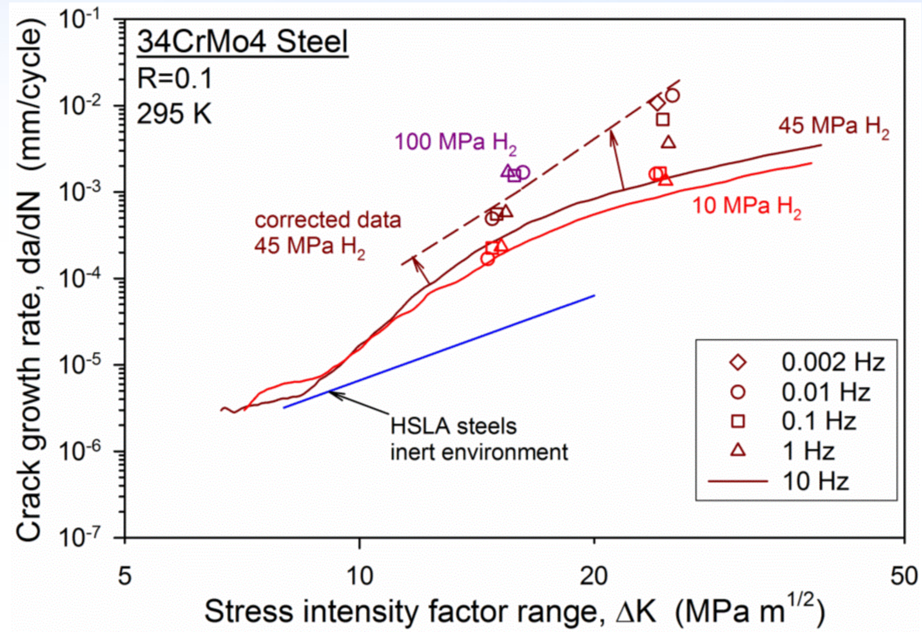
- Steels provided by two partners: FIBA Technologies and European steel cylinder manufacturer

Steel	S_u (MPa)	H_2 pressure (MPa)	Test frequency (Hz)	Load ratio	Status
SA372 Gr. J	890	10	10	0.1	Complete FY12-13
		10	variable	0.1	Complete FY12-13
		45	10	0.1	Complete FY11-12
		45	variable	0.1	Complete FY11-12
		100	10	0.1	In progress
		100	variable	0.1	Complete FY12-13
34CrMo4	1045	10	10	0.1	Complete FY12-13
		10	variable	0.1	Complete FY12-13
		45	10	0.1	Complete FY11-12
		45	variable	0.1	Complete FY12-13
		100	10	0.1	Complete FY12-13
		100	variable	0.1	Withdrawn

Accomplishment:

For higher-strength steel, *da/dN* correction depends on H_2 pressure and *ΔK* level

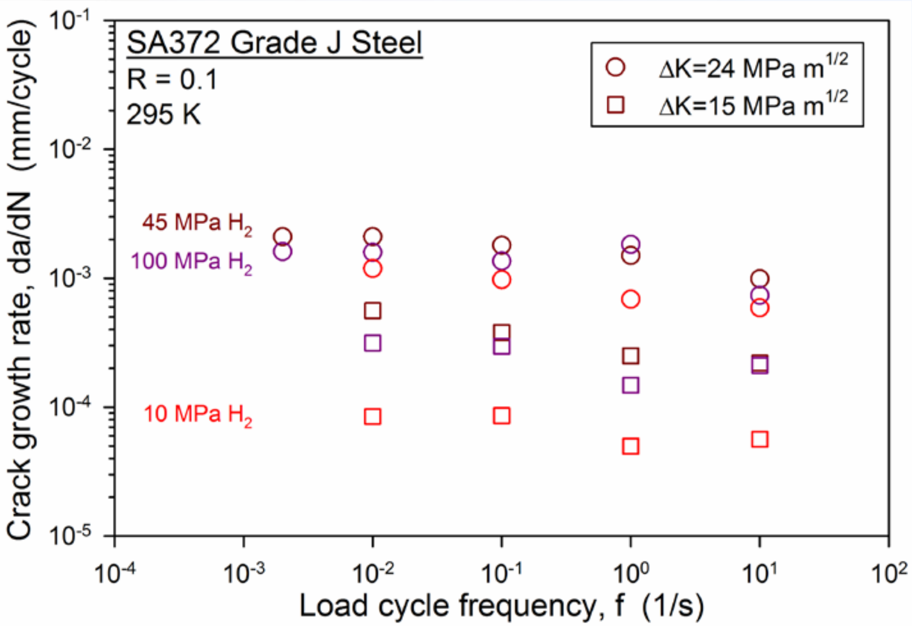


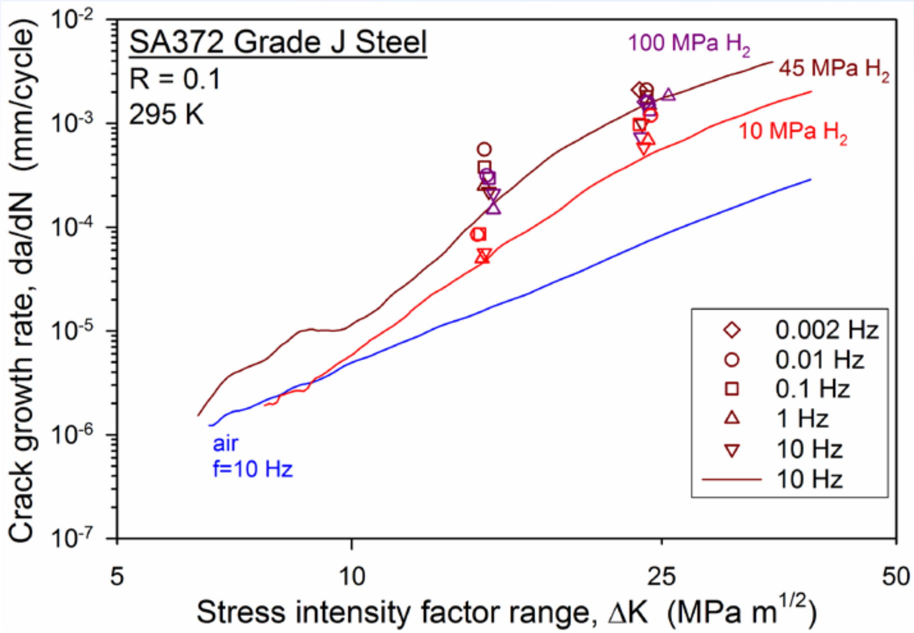


- Crack growth rate correction based on upper bounds in *da/dN vs f* data
- Upper-bound *da/dN* attained at limiting load-cycle frequency
  - Example: at 10 MPa H_2 pressure, *da/dN* reaches upper bound at *f* ~ 1 Hz for both *ΔK* levels
- Possible modified test method: measure *da/dN vs ΔK* at 10 Hz, determine correction based on upper-bound *da/dN* measured at limiting frequency
- ASME Project Team dissolved, but communicating data to former chair

Accomplishment:

For lower-strength steel, *da/dN* correction less sensitive to H_2 pressure





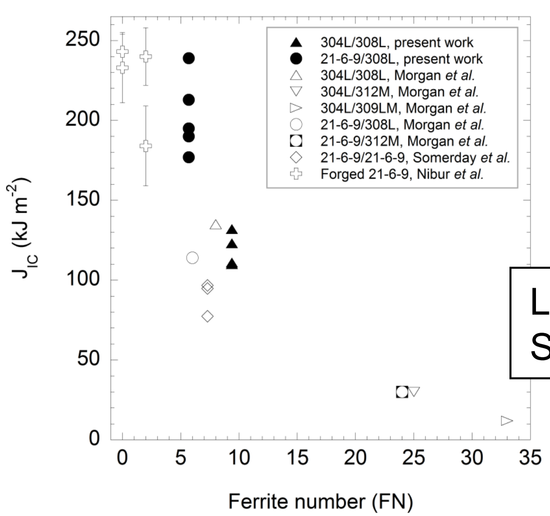
- Similar to 34CrMo4 steel, crack growth rate correction based on upper-bound *da/dN* defined at limiting load cycle frequency
- Unlike 34CrMo4 steel, upper-bound *da/dN* similar at 45 MPa H_2 and 100 MPa H_2 for both ΔK levels
- Modification to test method may include defining limiting test pressure for lower-strength steels
- ASME Project Team dissolved, but communicating data to former chair

Previous Accomplishment:

Hydrogen compatible materials workshop: weld evaluation critical to technology deployment

- Welds are effective for joining metal components; reduce leaks in pressure systems compared to fittings
- Weld microstructures can be more susceptible to hydrogen embrittlement (HE), limiting performance of pressure systems
- Data gap: mechanical properties of technologically relevant, H_2-exposesd welds

H-affected fracture toughness vs. vol% ferrite

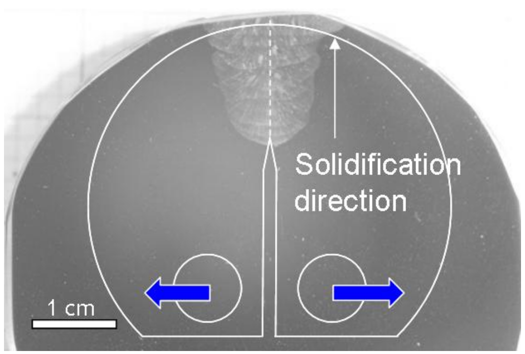


Jackson et al., Corrosion Science, 2012

Leveraged internal Sandia programs

Previous work

- HE sensitivity of welds depends on vol% ferrite
- geometry of tested welds not relevant to typical H_2 components



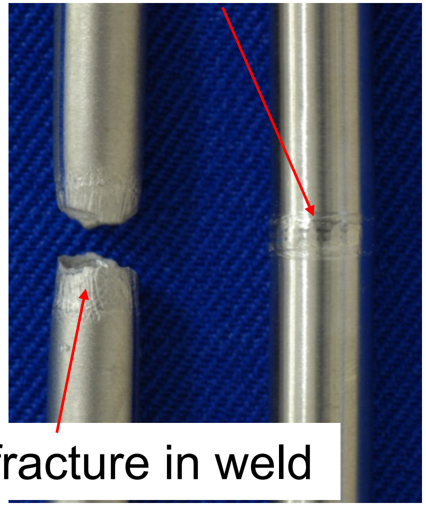
304L/308L GTA weld

Accomplishment:

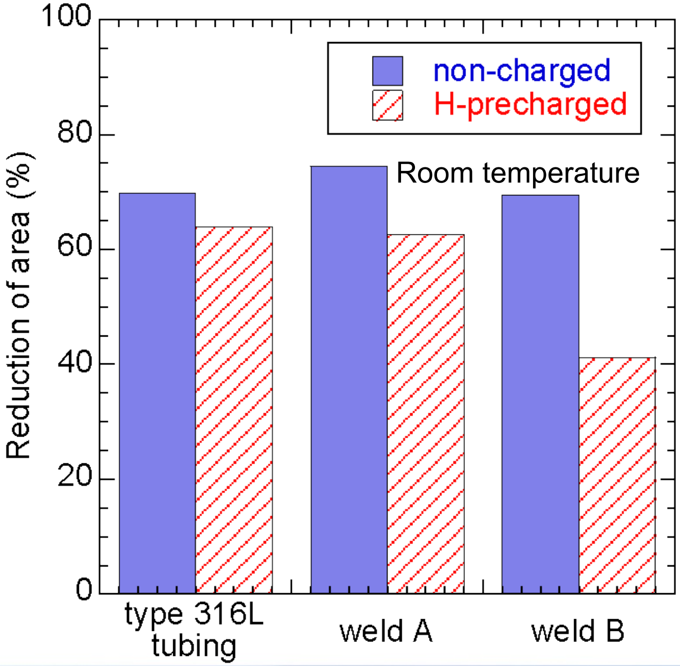
Effects of welding practice investigated for orbital welds in type 316L tubes

- Orbital tube welds are important technology for H_2 distribution systems
- Type 316L austenitic stainless steel currently represents state-of-the-art for H_2 distribution components, e.g., tubing and valves
- Orbital tube welds (type 316L) produced with industrial partner (weld A) and internally using commercial equipment (weld B)
- Lengths of tubing with or without an orbital weld were tested in uniaxial tension

tube with orbital weld



fracture in weld

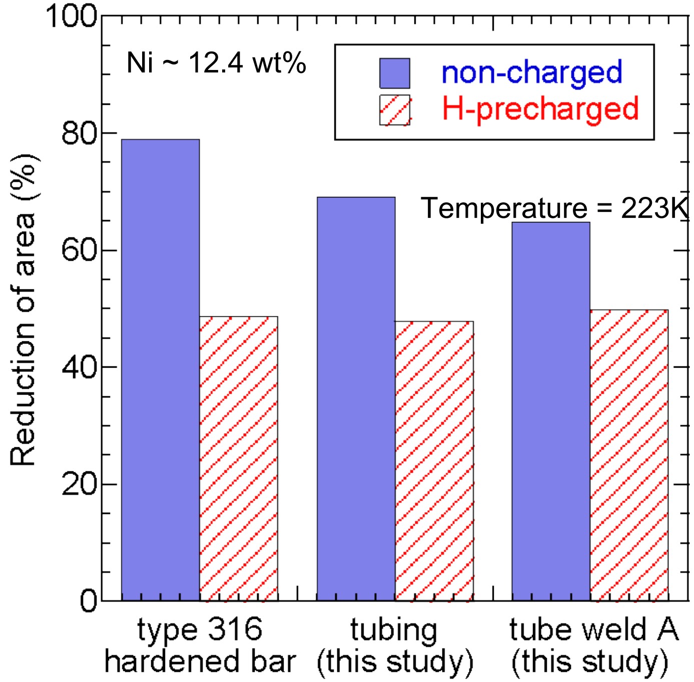


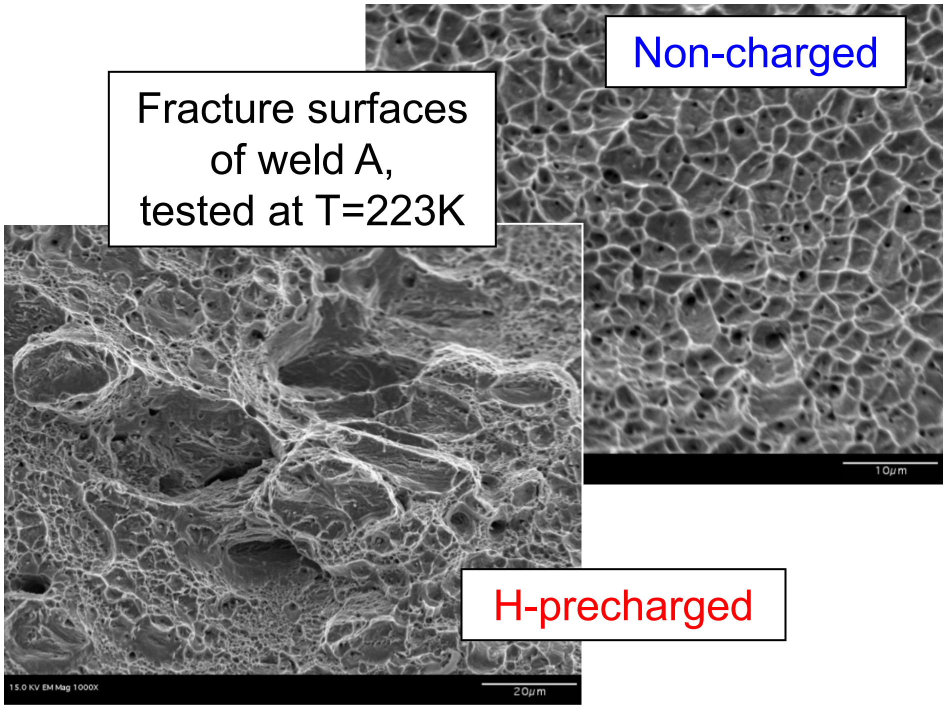
- Tensile ductility of both welds similar to non-welded tubing
- Hydrogen-affected ductility similar in weld A and non-welded tubing
- Weld B shows greater effects of hydrogen, but remains ductile

Accomplishment:

Effect of low temperature evaluated for orbital tube welds

- Hydrogen-assisted fracture in austenitic stainless steel generally enhanced at low temperature
- At temperature of 223K, like at room temperature, hydrogen effects on ductility similar for non-welded tubing and weld A
- Fracture surfaces show ductile features with the involvement of boundaries when H-precharged, similar to bar materials





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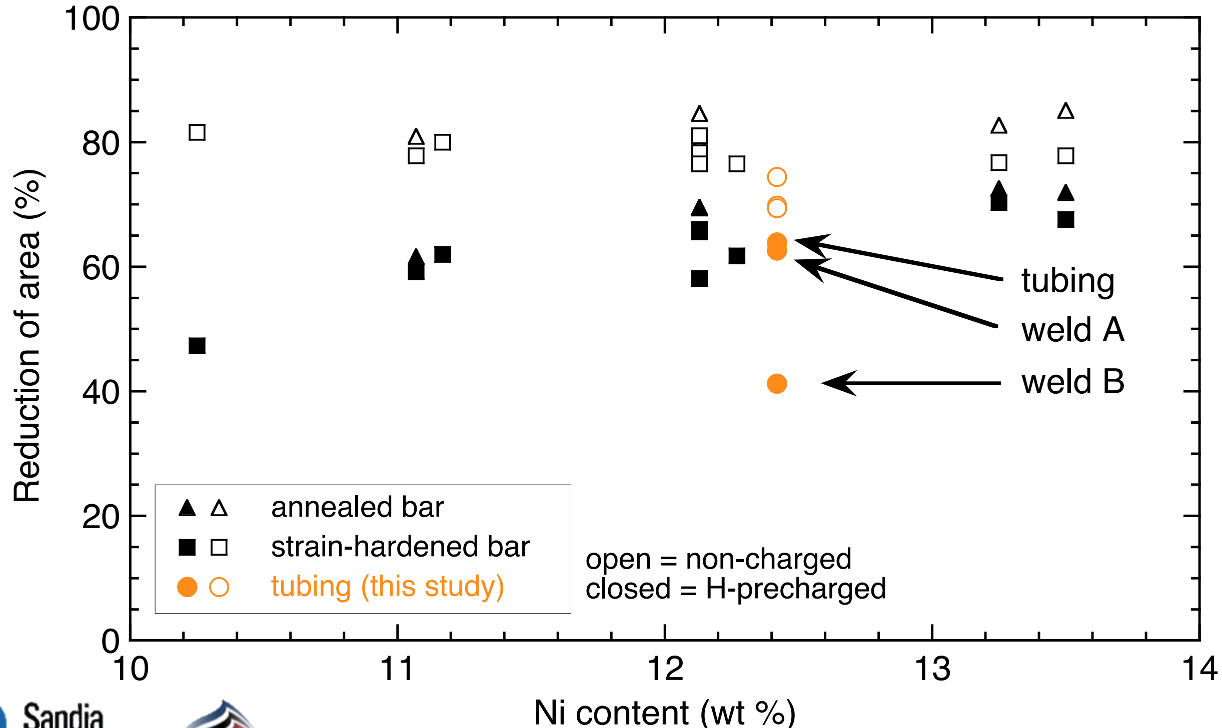
Accomplishment:

Tensile ductility of tubing and welds similar to data for bar materials

- Tensile ductility of type 316/316L austenitic stainless steels follows a trend of greater resistance to hydrogen with higher nickel content
- Tensile ductility data for non-welded tubes and orbital welds appear to follow same basic trend with nickel content
- Lower tensile ductility of weld B under investigation



Points for annealed and strain-hardened bar represent type 316 & 316L austenitic stainless steels with different composition (plotted as function of nickel content)

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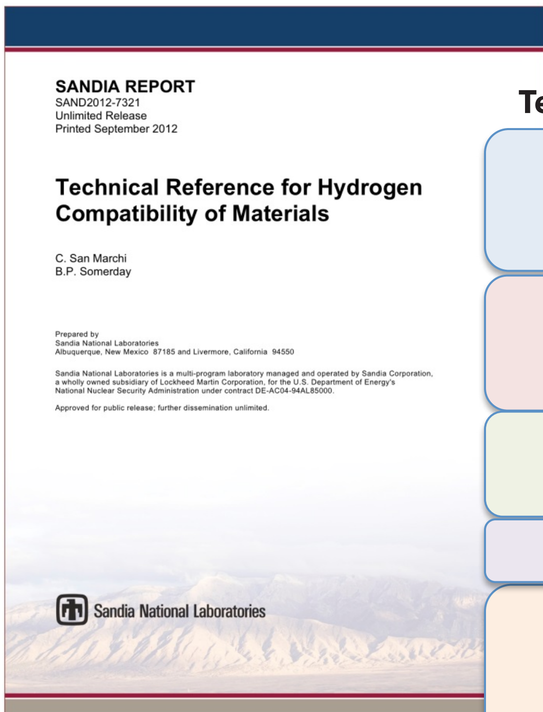
Accomplishment:

Technical Reference and Technical Database for Hydrogen Compatibility of Materials

- Information placed on OpenEI website: http://en.openei.org/wiki/Gateway:Hydrogen
  - Updated full public report on Technical Reference for Hydrogen Compatibility of Materials (SAND2012-7321), 292 pages
  - Datasets for fatigue crack growth of materials in gaseous hydrogen


SANDIA REPORT
SAND2012-7321
Unlimited Release
Printed September 2012

Technical Reference for Hydrogen Compatibility of Materials

C. San Marchi
B.P. Somerday

Prepared by
Sandia National Laboratories
Albuquerque, New Mexico 87185 and Livermore, California 94550

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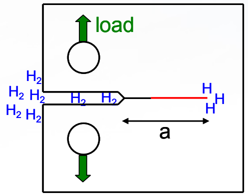
Technical Reference	Technical Database
1100 Carbon steels └ 1100: C-Mn alloys	1100 Carbon steels ├ CIA85: tension, fracture, fatigue ├ SAN10: fracture, fatigue └ SAN11: fracture fatigue
1200 Low-alloy steels ├ 1211: Cr-Mo alloys └ 1222: Ni-Cr-Mo alloys	1200 Low-alloy steels └ NIB10: fracture, fatigue
1400-1800 High-alloy steels └ 1401: 9Ni-4Co	1400-1800 High-alloy steels
2000 Austenitic steels	2000 Austenitic steels
3000 Aluminum alloys ├ 3101: Pure aluminum ├ 3210: 2xxx-series alloys └ 3230: 7xxx-series alloys	3000 Aluminum alloys └ SAN11: fracture, fatigue

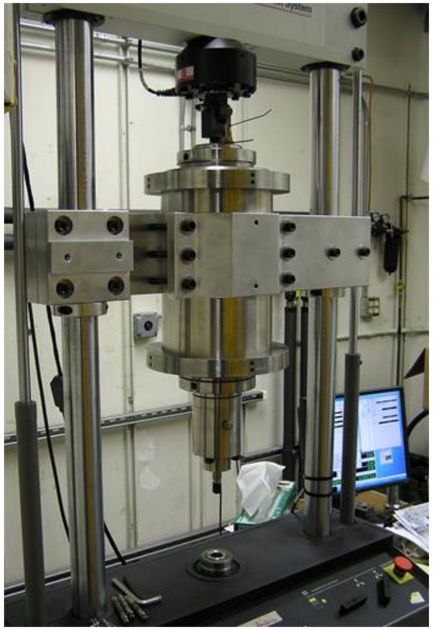
Collaborations

- Standards Development Organizations (SDOs)
  - Examples: CSA, SAE, ASME, ISO
  - Sandia technical staff lead and serve on committees
- Industry partners
  - Examples: FIBA Technologies, European cylinder manufacturer, Swagelok
  - Partners provide technology-relevant materials and input into materials testing conditions
- Universities
  - Example: Boise State University
  - Student design teams developing two pressure vessel concepts consistent with Sandia specifications for variable-temperature testing in H_2 system
- International research institutions
  - Example: International Institute for Carbon-Neutral Energy Research (I^2CNER), Dr. Brian Somerday (Sandia) serving as Lead PI for Hydrogen Structural Materials Division
  - Sandia influences and accesses basic research in I^2CNER (e.g., predictive H_2-assisted fatigue models) that complements applied research in Materials Compatibility project

Collaborations:

Maintaining AIST-SNL collaboration to harmonize test methods and standards





- Two joint activities documented in project plan (Jan. 2012)
  - Validate and promote method for measuring “initiation” threshold of ferritic steels in H_2
  - Explore basic mechanisms of H_2-assisted fracture in stainless steels
- Round-robin test matrix on two ferritic pressure vessel steels (one each from Japan and U.S.) started at AIST
- Joint publication on mechanisms of H_2-assisted fracture in stainless steels submitted to ASME
- Collaboration sustained through reciprocal visits to laboratory sites (~5/year)

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Proposed Future Work

Remainder of FY13

- Report results on fatigue crack growth measurements for SA372 Gr. J and 34CrMo4 steels in hydrogen gas to ASME and receive feedback
- Report and document results on tensile testing of H_2-exposed orbital tube welds at International Conference on Hydrogen Safety
- Evaluate commercial software for creating material property database
- Formalize format/structure of material property database: either (i) spreadsheet structure or (ii) commercial materials database structure
- Finalize pressure vessel design for variable-temperature testing in H_2 system

FY14

- Measure fatigue crack initiation resistance of H_2-exposed stainless steel tube welds
- Critically evaluate test method ("safety factor method") in CHMC1 Part 3 for qualifying materials for hydrogen service
- Develop validated methodology to account for fatigue crack initiation life in steel H_2 pressure vessels for consideration in ASME Article KD-10
- Develop R&D program with industry partner(s) to evaluate and improve resistance of high-strength structural metals to H_2-assisted fracture
- Leverage results on fatigue crack growth of pressure vessel steels in H_2 to enhance understanding of basic physics in collaboration with I^2CNER

Summary

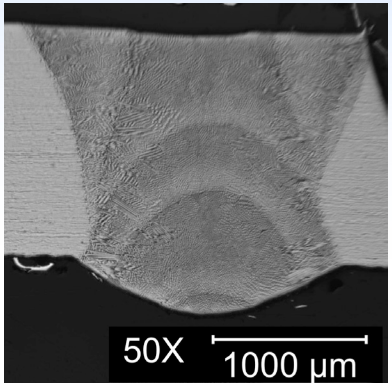
- Materials testing motivated by standards development and technology needs
  - Optimizing fatigue crack growth test method in ASME KD-10 to balance efficiency and data reliability
  - Measuring tensile properties of H_2-exposed tube welds in collaboration with industry partner
- Initiated potential pathway for creating public-access material property database
- Demonstrating leadership in materials testing by developing new variable-temperature system and hosting international meeting
- Concrete progress in developing standards that address hydrogen compatibility of components
  - Publication of Part 3 in CSA CHMC1 expected in 2013
  - Publication of SAE J2579 expected in 2012
- Maintaining active international collaborations
  - HYDROGENIUS/AIST (Tsukuba, Japan)
  - I^2CNER (Kyushu University, Japan)

Technical Back-Up Slides

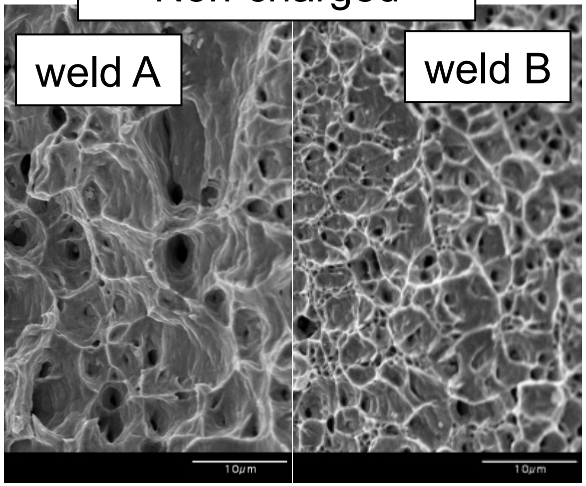
Accomplishment:

Relationship between microstructure and H_2-affected ductility of welds still unclear

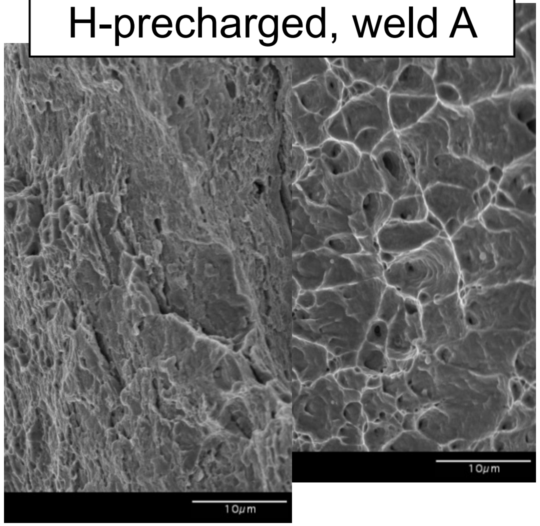
- Evaluation of microstructure and fractography shows hydrogen effects are consistent with experience from base materials (type 316L orbital tube welds)
- Fracture features from H-precharged weld A show no distinct relationship to weld microstructure
- Fracture features from H-precharged weld B suggest local regions with relationship to weld microstructure



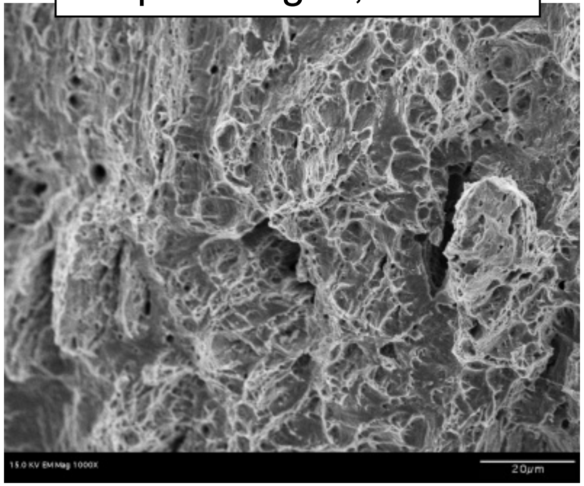
Non-charged



H-precharged, weld A



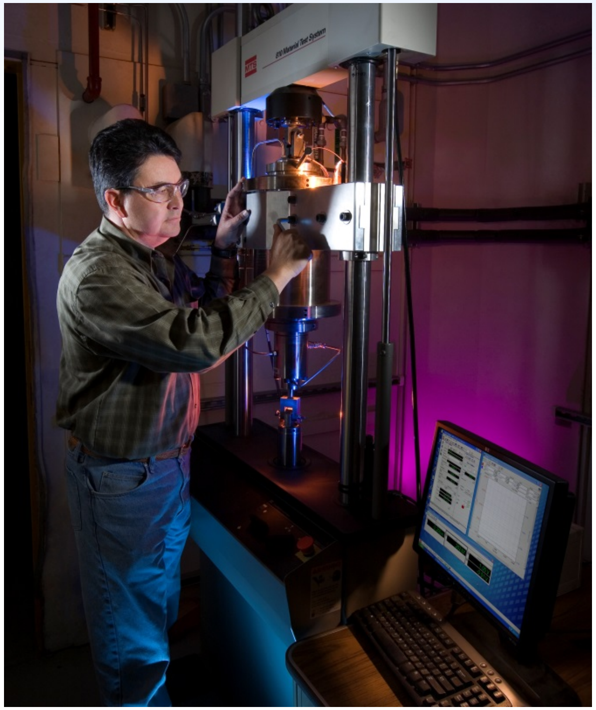
H-precharged, weld B



Room temperature fracture surfaces

Accomplishment:

Hosted meeting on Advancing Materials Testing in Hydrogen Gas at SNL/CA

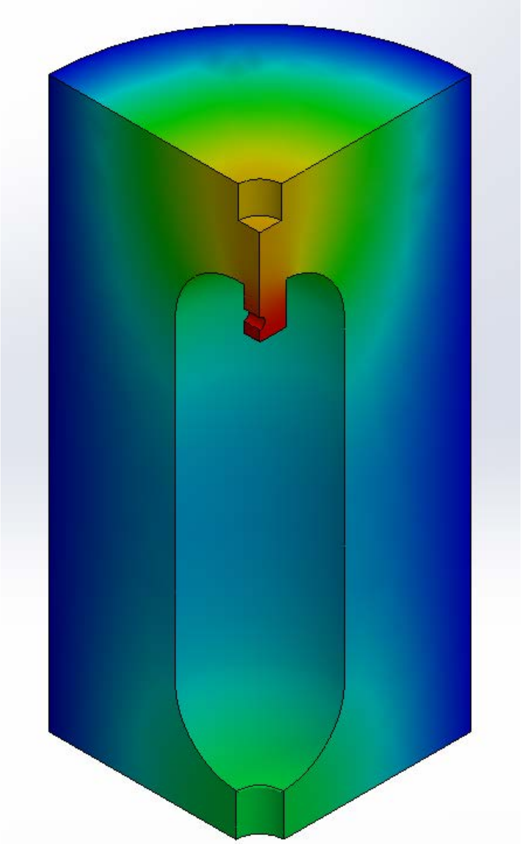


- Goal: exchange test system design details and initiate international collaboration on next-generation testing capabilities
- Attendees: ~25 people from universities, national labs, and industry world-wide
- Output:
  - Catalogue design concepts, best practices, and safety features
  - Determine test system limits
  - Identify gaps in existing testing capabilities
  - Make meeting presentations available to the public
  - Identify pathways and resources for development of capabilities
  - Identify collaboration opportunities

Accomplishment:

Development of variable-temperature testing in H_2 system progressing

- Operational status
  - Dedicated test cell
  - Functioning test frame
  - Assembled gas manifold
- Current activity
  - Designing software for automated gas manifold
  - Stress/thermal analysis of pressure vessel concepts
- Two Boise State University student teams designing vessel concepts
  - Team 1: large-bore vessel
  - Team 2: small-bore vessel
  - Both teams emphasizing thermal analysis



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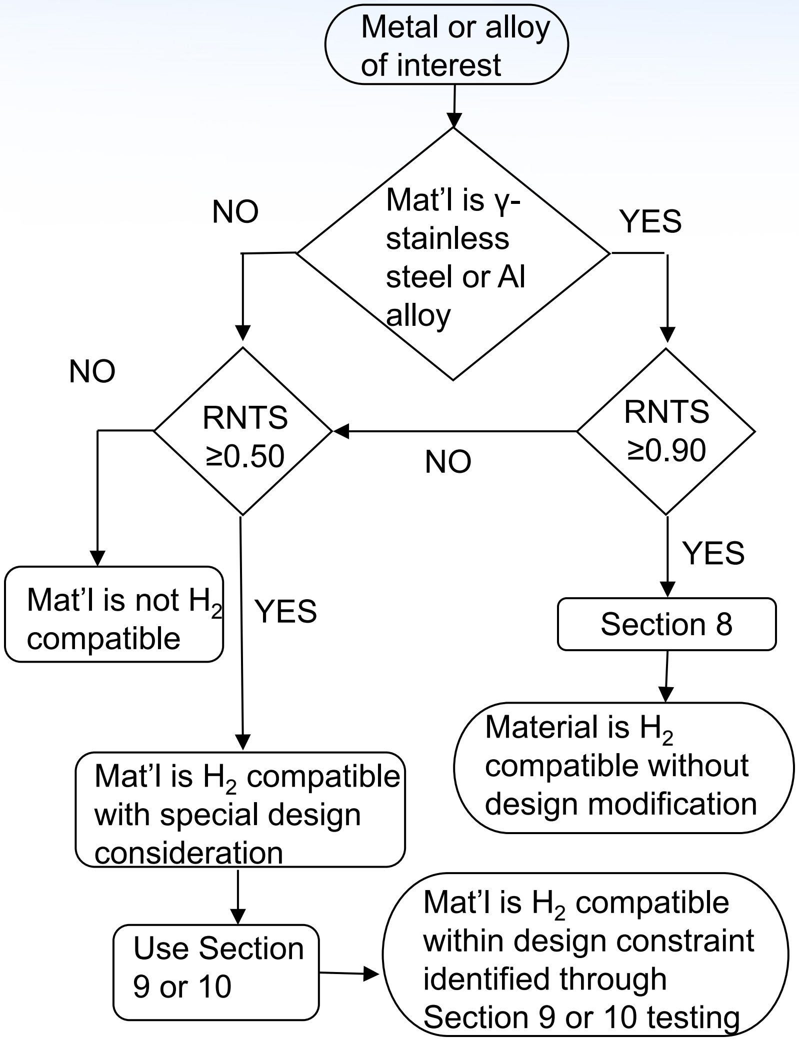
Accomplishment:

CSA CHMC1: standardized method to qualify materials for hydrogen service

- Parts 1, 2:
  - Previously published as Phase I
  - Specific methods for conducting tests in gaseous hydrogen
- Part 3: Material Qualification
  - Three different qualification procedures (Sections 8, 9, 10)
  - Section 8 : Stringent Pass/fail for SS and Al
  - Section 9: Determine safety factor to account for worse-case H_2 effect on mechanical properties
  - Section 10: Use measured mechanical properties to qualify material for a specific component design
  - Section 11: Procedures are provided to allow a materials specification to be qualified
    - Once specification is qualified, further testing is not required



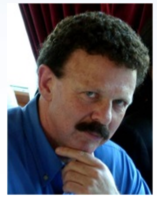
Collaborations:

SNL and I²CNER leverage applied and basic research for common goal

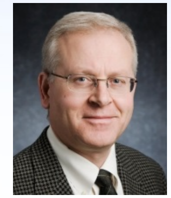
Fatigue and Fracture


S. Matsuoka (PI)


Y. Murakami (PI)


R. Ritchie (PI)


I. Robertson (UI PI)


P. Sofronis (PI)


N. Aravas

I²CNER

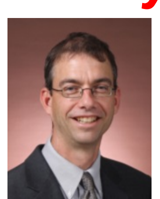
- **Predictive models based on physics of gas-surface interactions, H migration, and material degradation**
- **Advanced methods for measuring fatigue, fracture, and wear properties in H_2 environments**

- **Next-generation materials having improved resistance to H_2-induced degradation at higher strength levels**

Friction and Wear


J. Sugimura (PI)

Optimize cost, performance, and safety of H_2 components


B. Somerday (Lead PI)


R. Kirchheim (PI)

Materials Processing


S. Takaki (PI)


A. Macadre