SAND2013-9037P

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

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Sandia National Laboratories

Sept. 19, 2013

Codes and Standards Tech Team Meeting

Sandia is a multi-program laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy under contract DE-AC04-94AL85000

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Research, Engineering, and Applications Center for Hydrogen

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

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

Motivation: tubing and welds

- Tubing and piping are important components of hydrogen energy infrastructure
  - Relatively little work has been devoted to evaluation of tubing materials
- Orbital tube welding is an effective joining strategy for gas handling and dispensing manifolds
  - H-assisted fracture of welds has not been extensively characterized

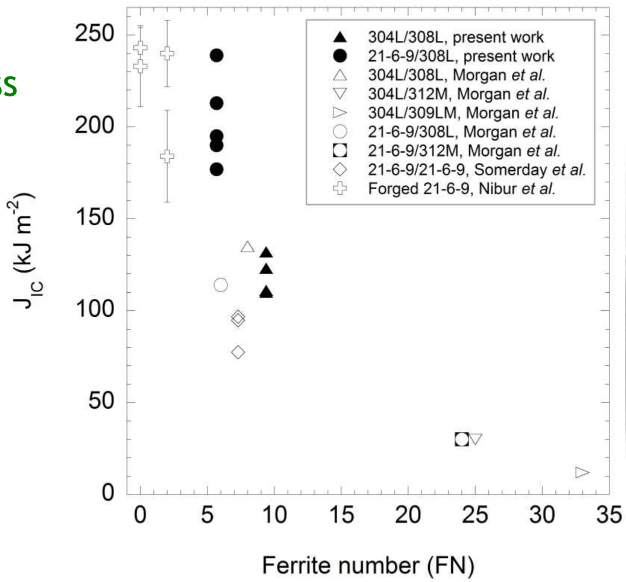
Conclusion from *Workshop on Hydrogen Compatible Materials* at SNL/CA (November 2010):

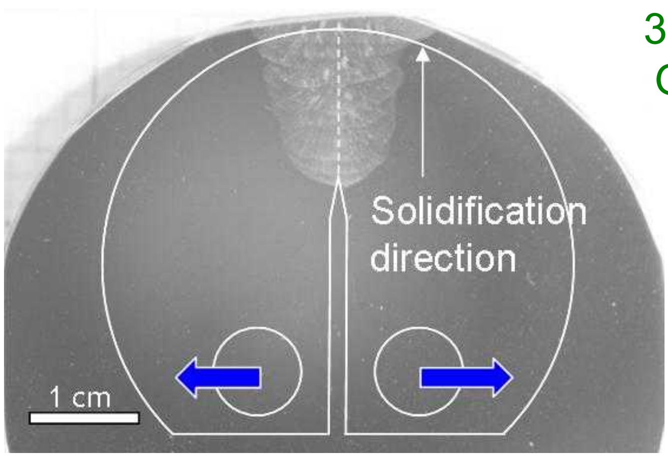
- Evaluation of welded structures is critical to deployment of hydrogen infrastructure and technology.

Previous work: austenitic stainless steel welds

- HE sensitivity of welds depends on vol% ferrite
- At low temperature, mechanisms of fracture are altered by hydrogen, although fracture toughness in the presence of hydrogen remains about the same as at room temperature
- However, geometry of tested welds not relevant to gas handling and dispensing manifolds

H-affected fracture toughness vs. vol% ferrite





304L/308L GTA weld

Recent Refs. from SNL/CA activities:
(1) Jackson et al., Corrosion Science **60** (2012) p. 136-144.
(2) Jackson et al., Corrosion Science (2013) online.

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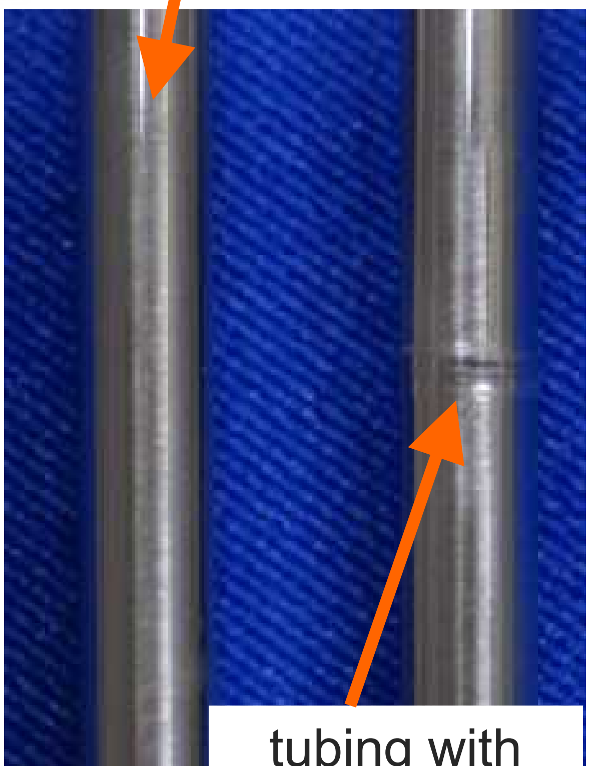
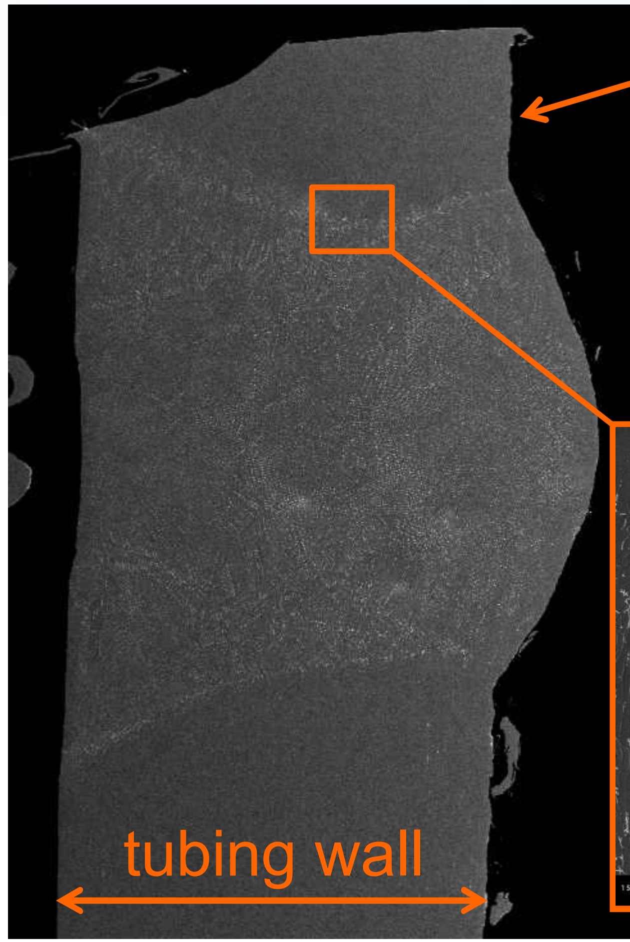
Orbital tube welds are relevant to gas handling and dispensing manifolds

As-received tubing

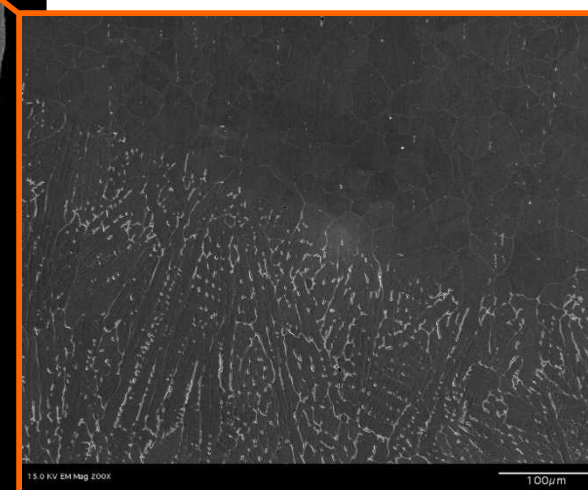


tubing with orbital weld



~1.3 mm (0.051")

inside surface

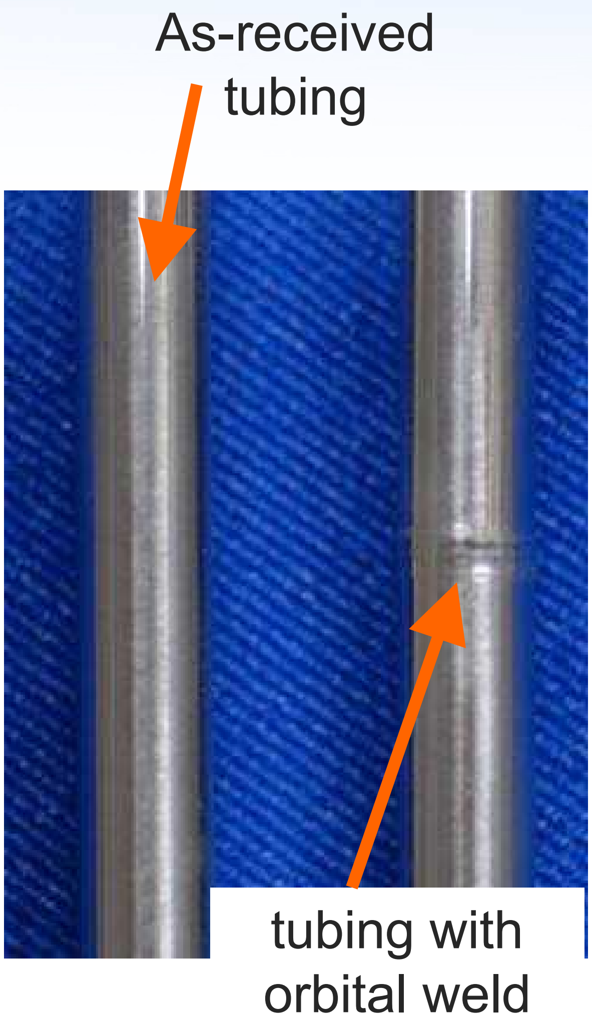


¼" OD 316L tubing

Tensile properties of tubing and orbital tube welds were evaluated

Testing scope: Uniaxial tension of 304/304L and 316L tubing

- As-received tubing
- *Internal hydrogen* (~140 wtppm)
  - Produced by thermal precharging (573K in 140 MPa H2)
  - Simulates hydrogen at stress concentrations
- *Orbital tube welds*
  - Different equipment, different welding personnel
- Effect of subambient *temperature*
  - 293 K (room temperature)
  - 223 K (-50˚C)



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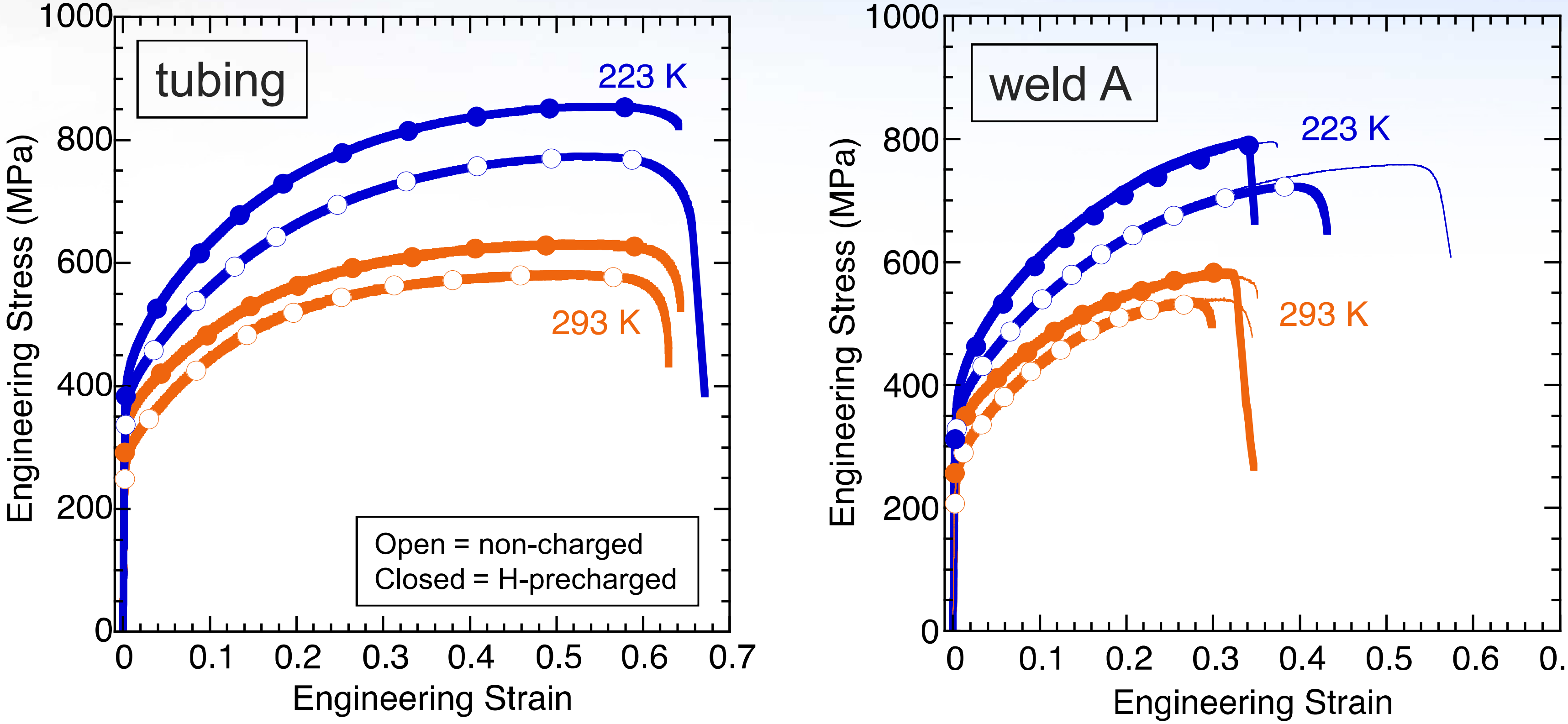
Testing matrix for welded tubing

Material/ Condition	Yield strength prior to welding (MPa)	Yield strength after welding (MPa)	Welder
Strain-hardened 304L (2A)	707	248	1
Recovered 304L (2C)	576	256	1
Annealed 304L (2F)	179	178	1
Annealed 316L (weld A)	286	243	2
Annealed 316L (weld B)	286	258	3
Sensitized 304/304L	varies	varies	n/a

- Sensitization provides “worst-case” impact of welding (or other thermal exposure) on microstructure

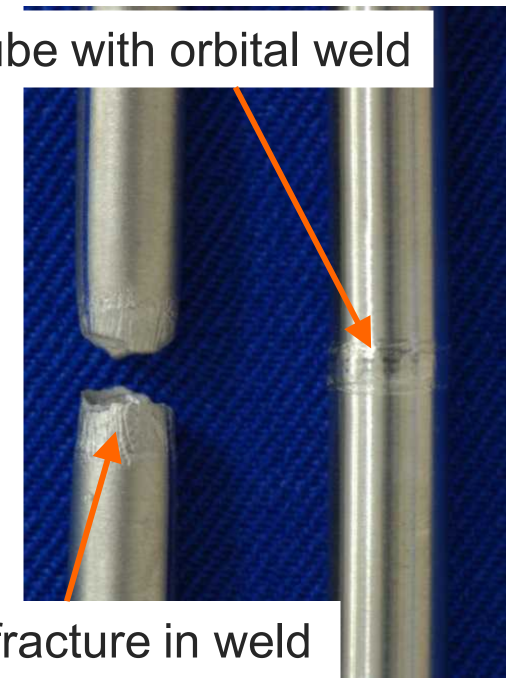
Type 316L tubing and welds show similar tensile properties with and without hydrogen

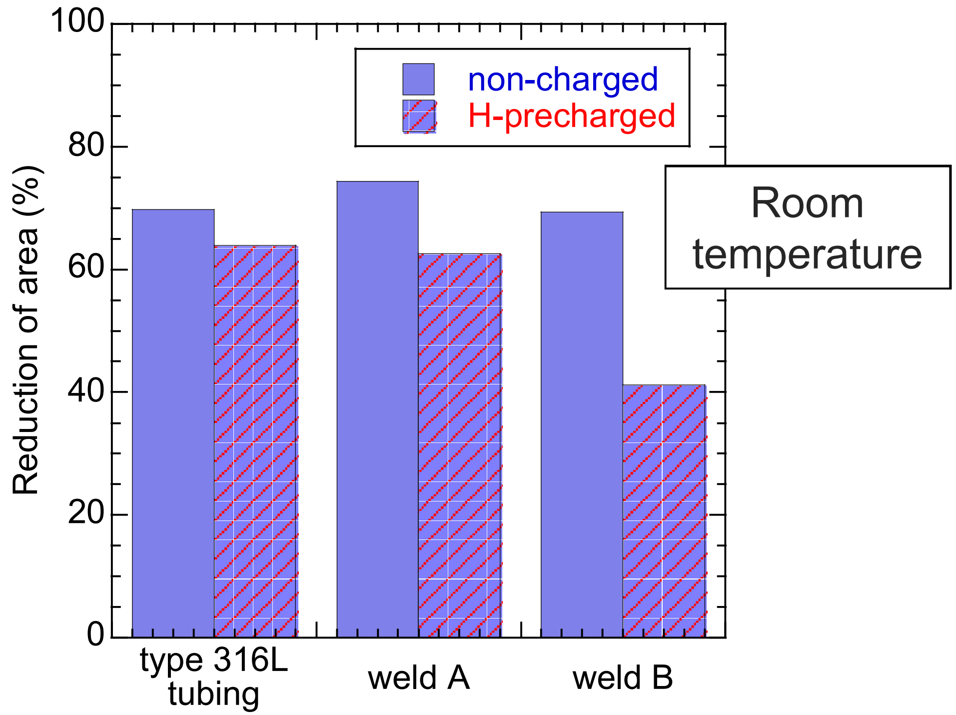


- Welded specimens show less elongation because deformation is restricted to the weld zone (reduction of area is similar for tubing and welds)
- Welded specimens show more variability in elongation than tubing without welds

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

- Tensile ductility of both weld specimens similar to as-received tubing (non-charged)
- Hydrogen-affected ductility similar in weld A and as-received tubing (H-precharged)





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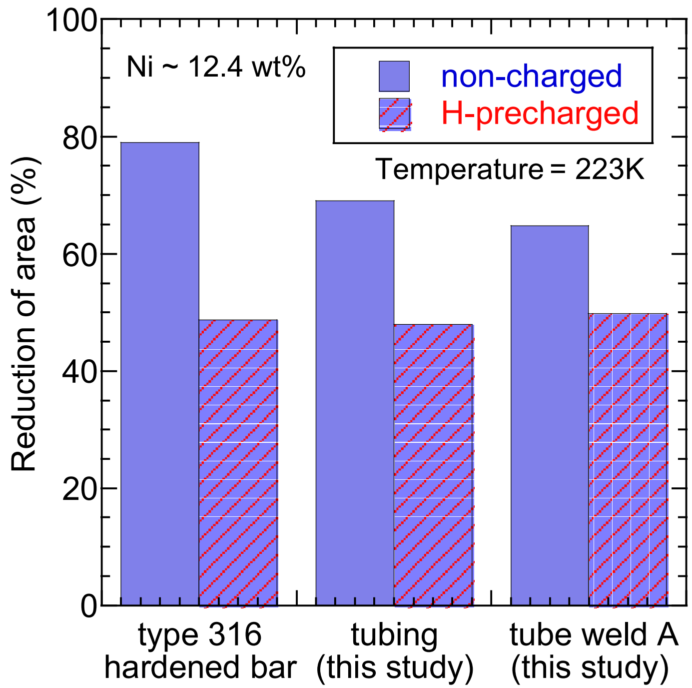
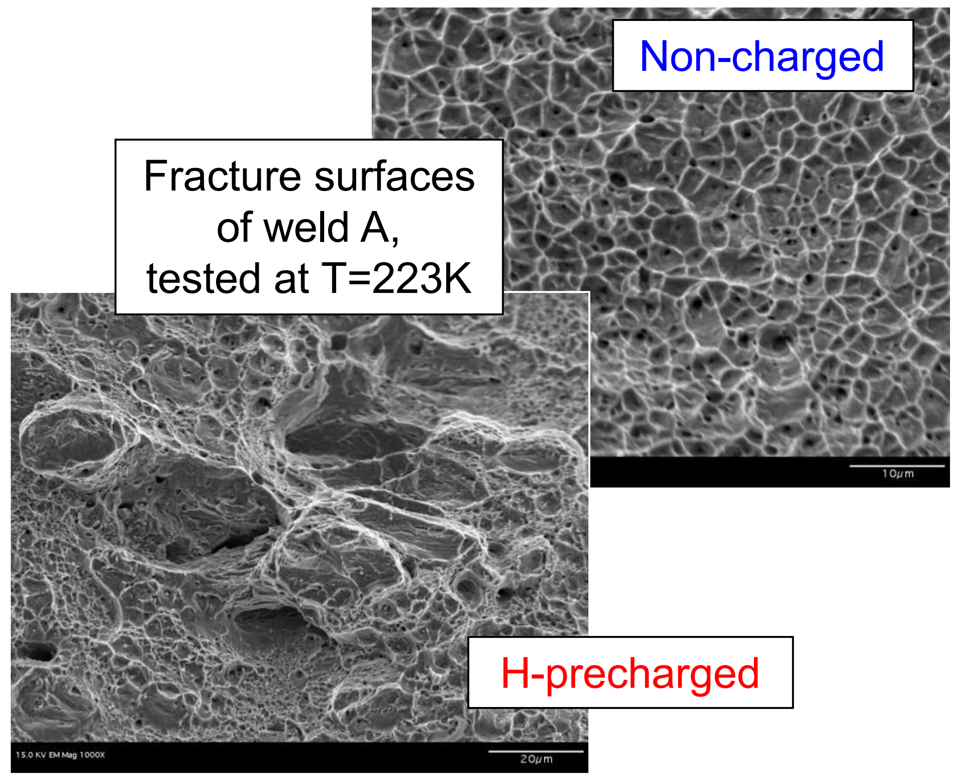
Effect of low temperature evaluated for orbital tube welds

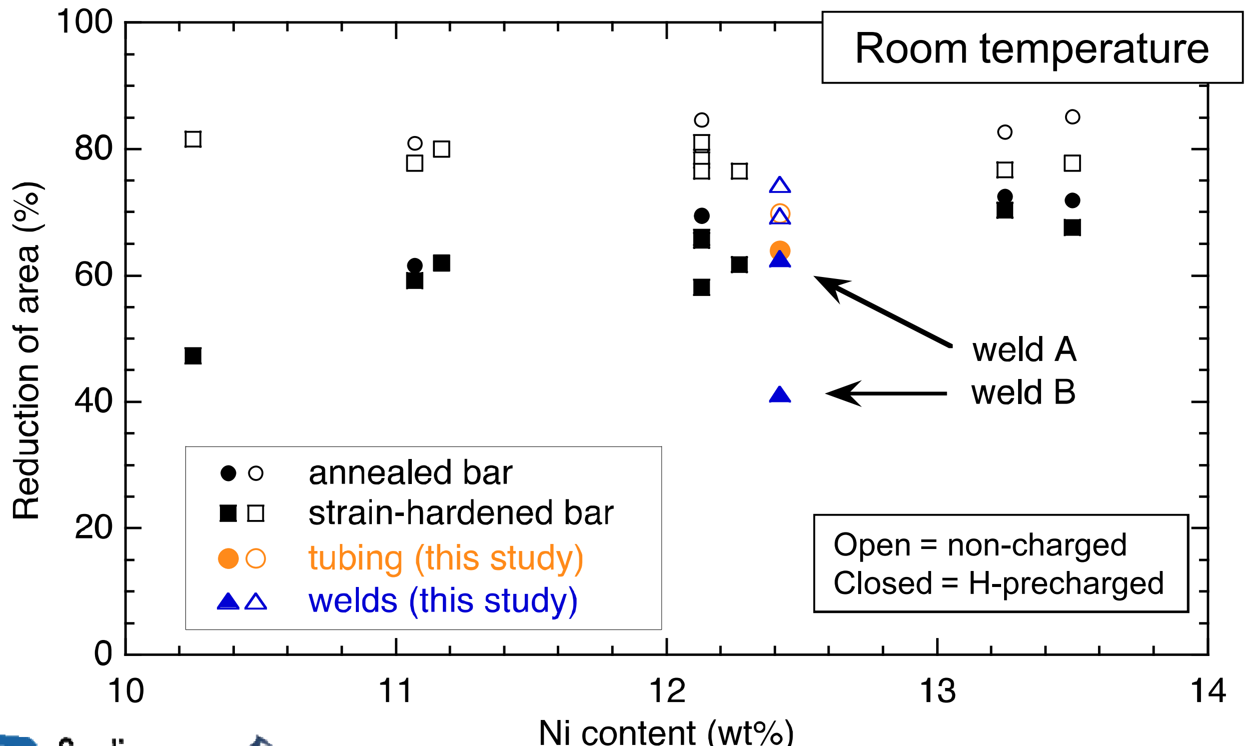
- Hydrogen-assisted fracture is enhanced at low temperature
- At temperature of 223K, welds and tubing show similar tensile ductility
- Fracture surfaces of welds show ductile features with the involvement of boundaries when H-precharged





Tubing and welds show similar ductility as bar materials

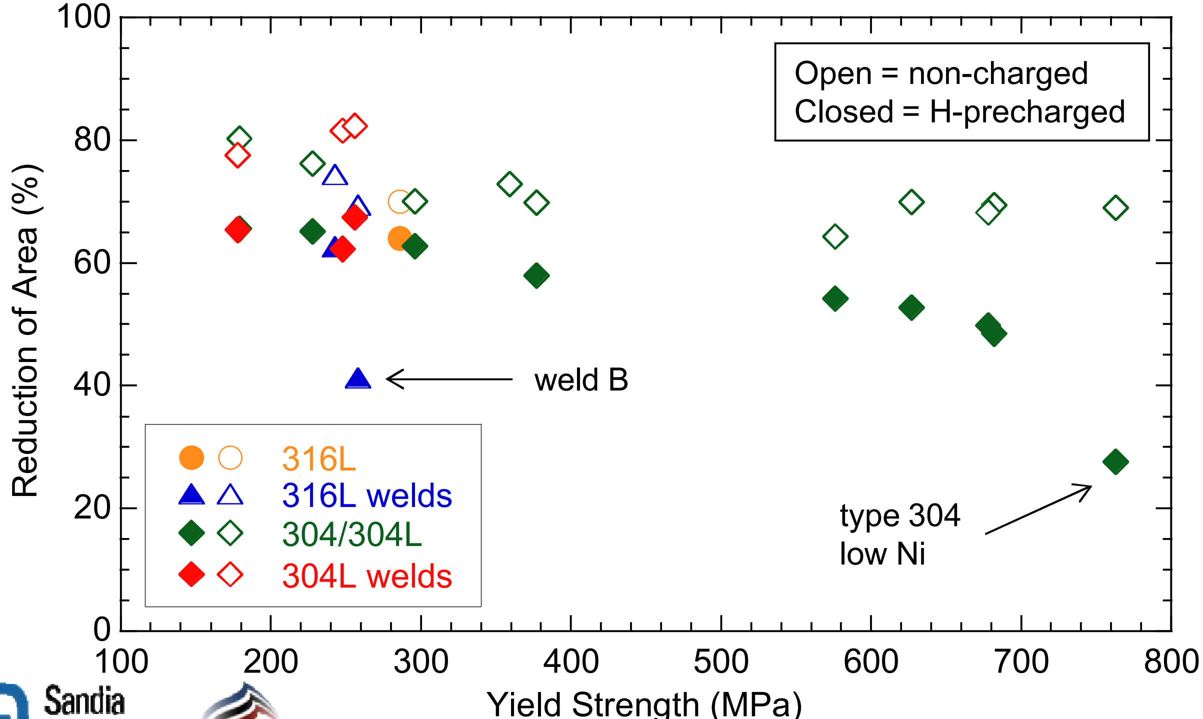
- Tensile ductility of type 316/316L austenitic stainless steels shows greater resistance to hydrogen with higher nickel content
- Tensile ductility of tubing specimens and welded specimens appear to follow same basic trend with nickel content
- Weld B displays lower ductility, but is still *very* ductile



Tensile ductility of type 316 & 316L austenitic stainless steels with different compositions

Type 304/304L tubing and welds show good ductility with internal hydrogen

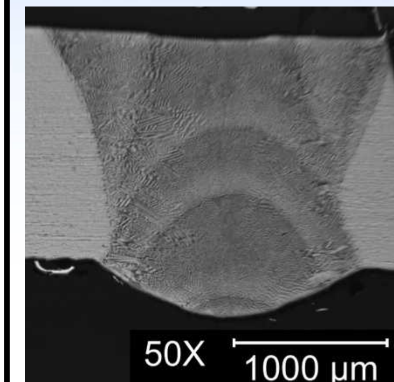
- Tensile ductility of type 304L austenitic stainless steel tubing can be similar to type 316L
- Tensile ductility depends on slightly on yield strength, but also on nickel content (low nickel alloys are more susceptible to hydrogen)
- Specimens with orbital tube welds show similar ductility to the tubing (however, strength of weld can be significantly lower)



- Five type 304/304L alloys
- One strain-hardened type 304L annealed to different strengths
  - Orbital tube welding of 3 different strength conditions
- Nickel content >10 wt%, except low Ni alloy (8.2 wt%)
- Type 304/304L tubing: 3.2 mm OD x 2 mm ID (nominal)

Relationship between welded microstructure and hydrogen-assisted fracture of welds is still emerging

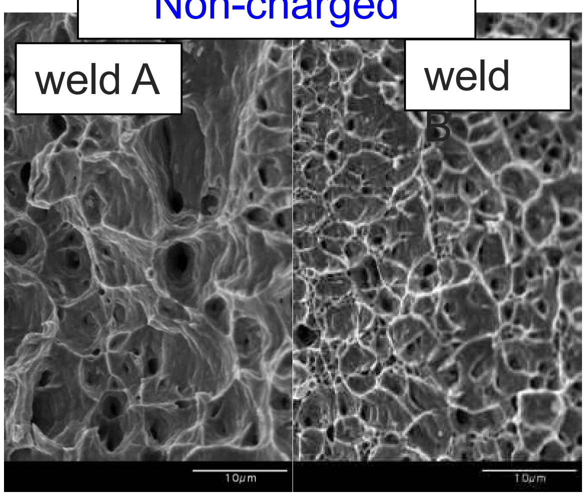
- Evaluation of microstructure and fractography shows hydrogen effects are consistent with experience from base materials (type 316L bar)
- Fracture features from weld A show no clear relationship to weld microstructure
- Fracture features from weld B suggest local regions with intrinsic relationship to weld microstructure



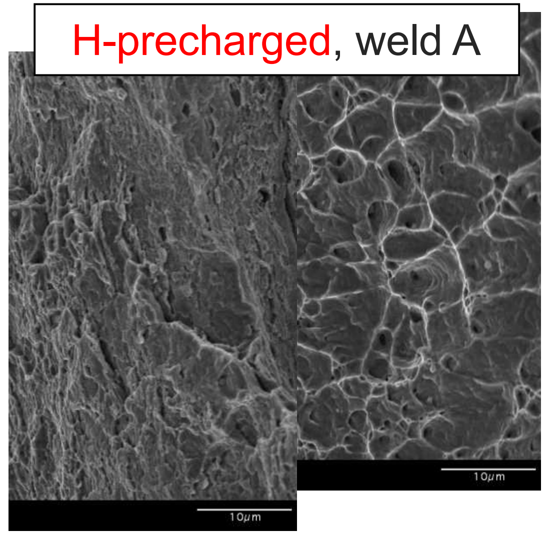
Non-charged

weld A

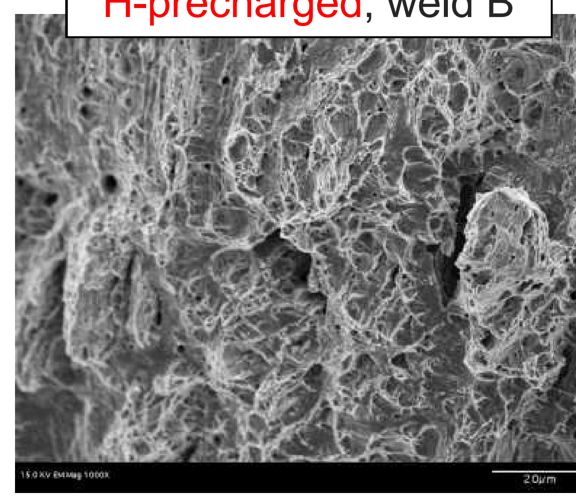
weld B



H-precharged, weld A



H-precharged, weld B



Room temperature fracture surfaces

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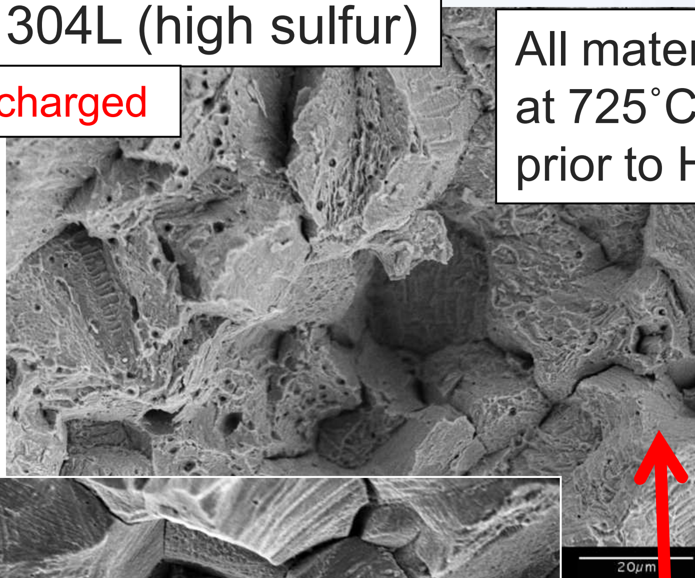
Compositional segregation (S & C) appear to enhance the effects of H

Type 304L (high sulfur)

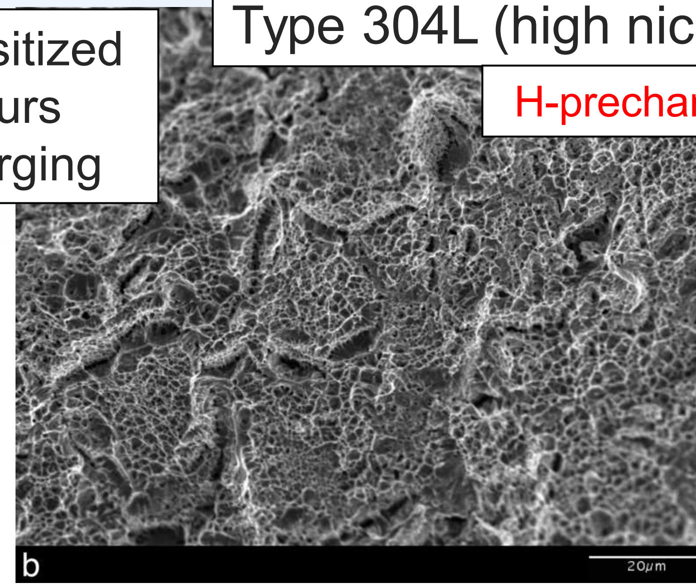
H-precharged

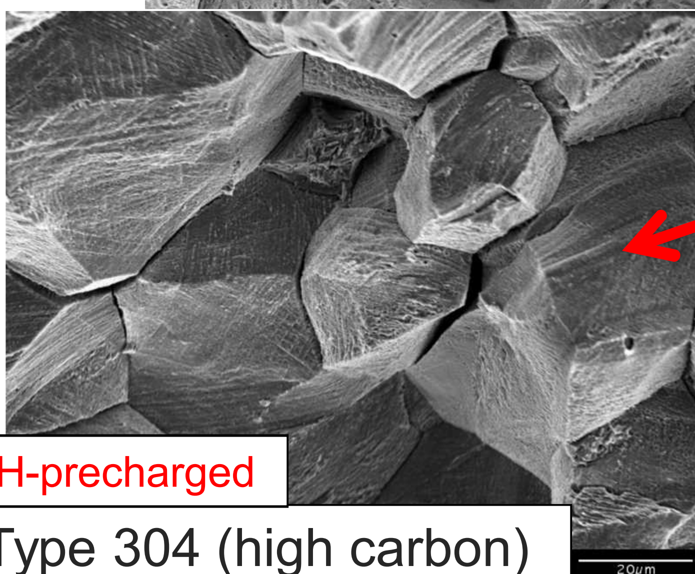


All materials sensitized at 725˚C for 4 hours prior to H-precharging

Type 304L (high nickel)

H-precharged





H-precharged

Type 304 (high carbon)

Undesirable “embrittled” fracture features

- Alloys with high carbon and sulfur show large reductions of ductility
  - Presumably due to the combined effects of segregation (sensitization) and hydrogen

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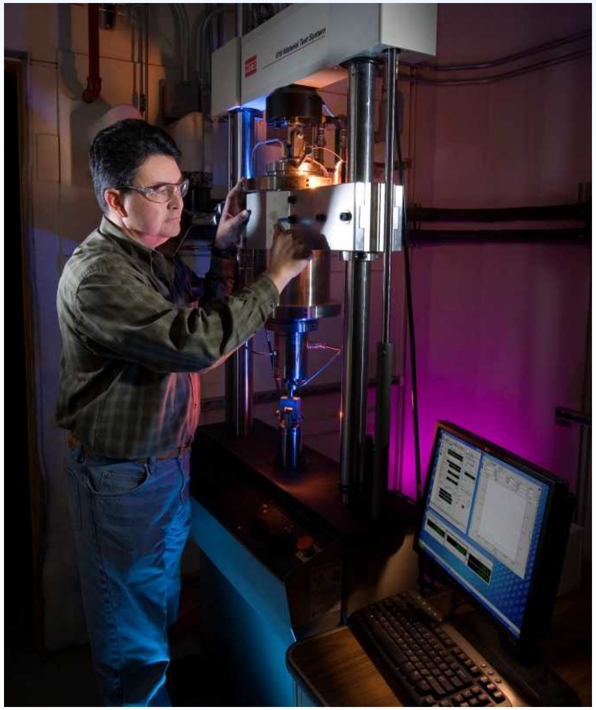
Summary: orbital tubing welds

- Hydrogen effects evaluated in austenitic stainless steel tubing and orbital tube welds
  - Tubing performs similarly to bar materials
  - Welded specimens display similar tensile ductility as tubing
  - Welded specimens remain very ductile after hydrogen precharging
    - Welding parameters can affect tensile ductility

- Conclusion: Orbital tube welds in austenitic stainless steels can display similar resistance to hydrogen embrittlement as the tubing from which the welds are manufactured

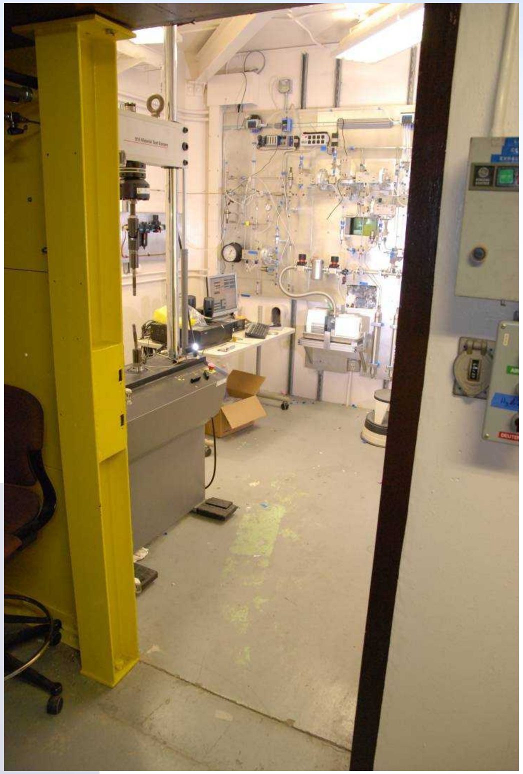
reacH2 Hosted meeting on Advancing Materials Testing in Hydrogen Gas at SNL/CA

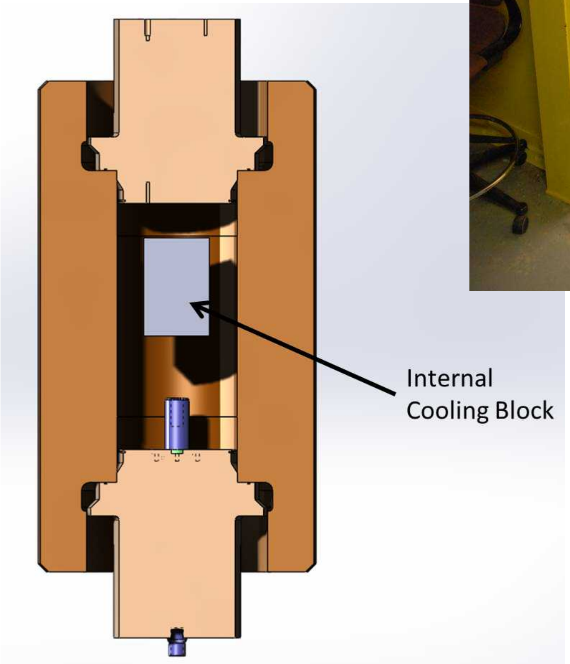


- Goal: exchange test system design details and initiate international collaboration on next-generation testing capabilities
- Attendees: ~25 people from universities, national labs, and industry world-wide
- Output:
  - Catalogue design concepts, best practices, and safety features
  - Determine test 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

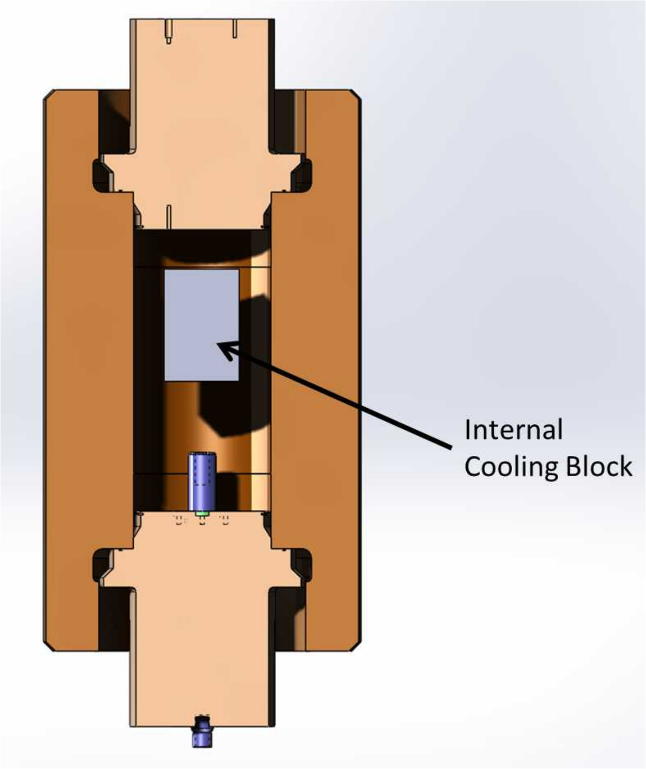
Development of variable-temperature testing in H_2 system progressing

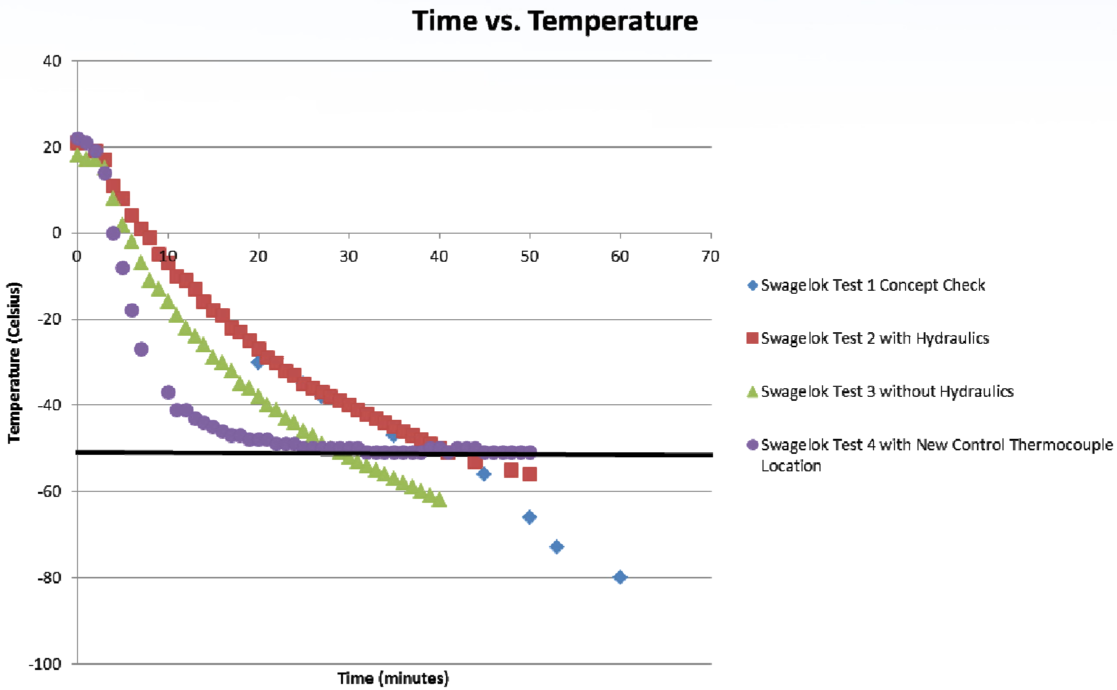
- Operational status
  - Dedicated test cell
  - Functioning test frame
  - Assembled gas manifold
- Current activity
  - Completing software for automated gas manifold
  - Refining prototype internal cooling mechanism for concept pressure vessel
  - Conducting thermal analysis of concept pressure vessel with internal cooling (Z. Harris, Boise State)





Prototype internal cooling mechanism yielded excellent temperature control





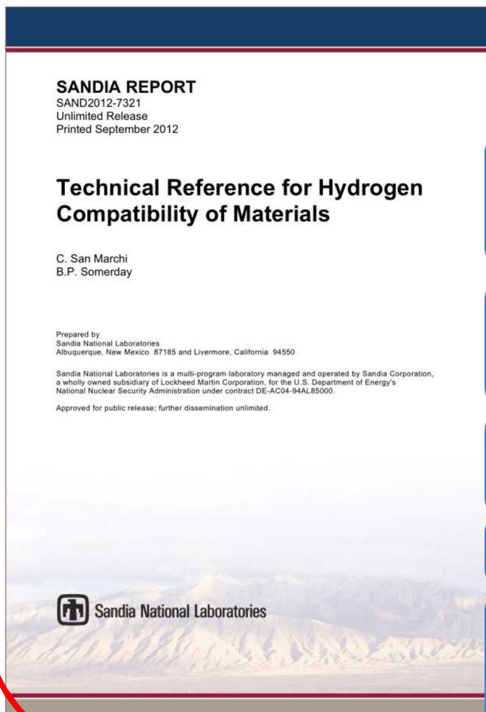
reacH2 Variable-temperature testing in H_2 system: next steps

- Demonstrate functionality of automated gas-handling manifold
- Refine design details of internal cooling mechanism for concept pressure vessel
  - Dimensions of cooling tube
  - Pressure-boundary feedthrough for cooling tube
- Conduct thermal analysis of concept pressure vessel with internal cooling mechanism (Z. Harris, Boise State)
- Submit detailed pressure vessel design with internal cooling mechanism for cost estimate from potential manufacturer
  - Still identifying source of funds for pressure vessel

Technical Reference and Technical Database for Hydrogen Compatibility of Materials

- OpenEI website: http://en.openei.org/wiki/Gateway:Hydrogen
- Sandia website: current release of information
- Preliminary datasets for fatigue crack growth of materials in gaseous hydrogen

Existing formulation


SANDIA REPORT
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Technical Reference for Hydrogen Compatibility of Materials

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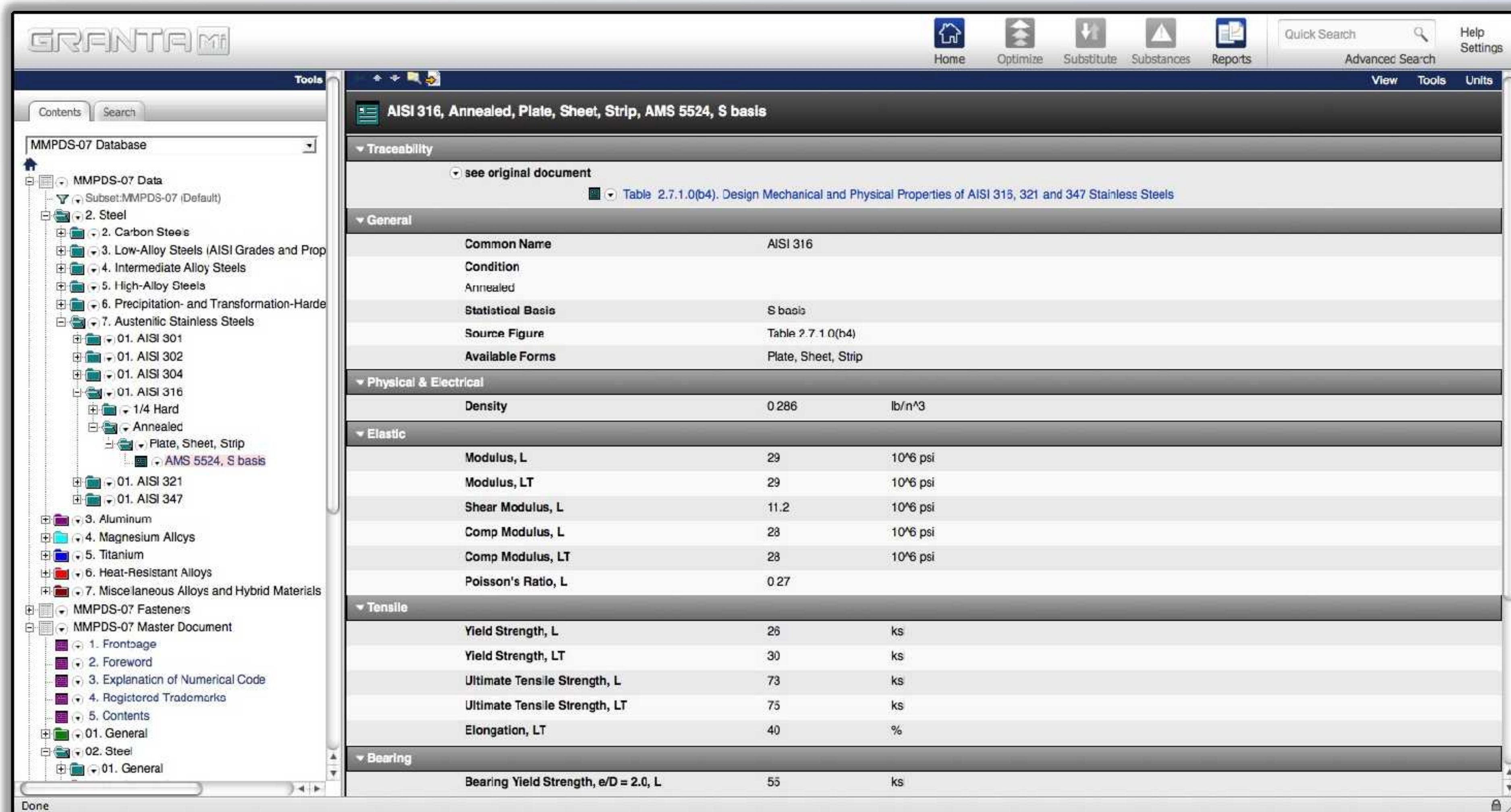
Future includes

- Comprehensive database structure

Reference information	Database information
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

Example of robust database tool for design: Granta MI



Materials databases are evolving into sophisticated data management tools

- Many institutions and industries are adopting sophisticated tools for data management
  - Warehouse and disseminate data from numerous sources
  - Analyze data sets and improve quality control
  - Harmonize the structural properties and materials used in design of engineering systems
  - Automatically populate engineering tools with design data
  - Minimize redundant testing activities
  - Aid materials innovation
- Sandia National Laboratories is a member of the Material Data Management Consortium (MDMC)
  - Other members include ASM, Boeing, NASA, Raytheon, Oak Ridge National Laboratory, Los Alamos National Laboratory and several others
  - Potential leverage for building tools to facilitate qualification of materials for hydrogen service

Summary: Technical Reference

- The *Technical Reference for Hydrogen Compatibility of Materials* is a handbook of structural materials data
- The *TR* is also an instrumental tool for managing hydrogen compatibility of materials, and aids identification of :
  - Important trends in the response of materials
  - Testing parameters that are sensitive to hydrogen
  - Gaps in our fundamental understanding of hydrogen embrittlement and gaps in the available data
- A database component of the *TR* will enable qualification of materials for hydrogen service
  - Necessary for managing large collections of data
  - Requires collaboration of stakeholders and sharing of information

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)

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Back-Up Slides

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

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SNL and I^2CNER 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

I^2CNER

- **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**

Materials Processing


S. Takaki (PI)


A. Macadre

Optimize cost, performance, and safety of H_2 components

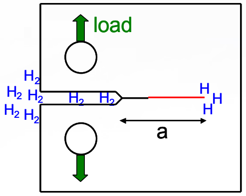
Friction and Wear

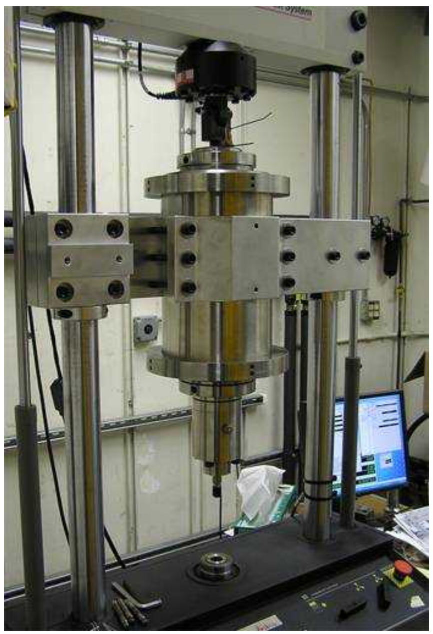

J. Sugimura (PI)


B. Somerday (Lead PI)


R. Kirchheim (PI)

Maintaining AIST-SNL collaboration to harmonize test methods and standards

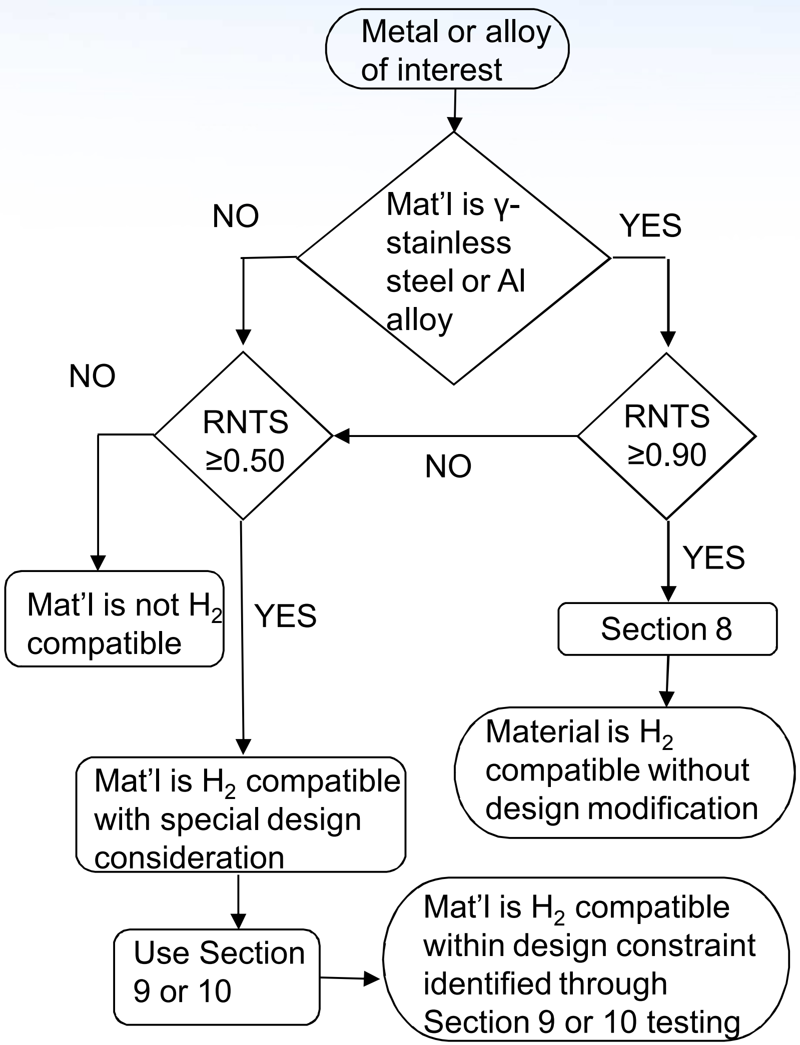




- Two joint activities documented in project plan (Jan. 2012)
  - Validate and promote method for measuring "initiation" threshold of ferritic steels in H_2
  - Explore basic mechanisms of H_2-assisted fracture in stainless steels
- Round-robin test matrix on two ferritic pressure vessel steels (one 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)

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



Materials selection for hydrogen service includes diverse range of product

Hydrogen delivery
- e.g., hydrogen pipelines: carbon steels
- Challenge: cyclic pressure

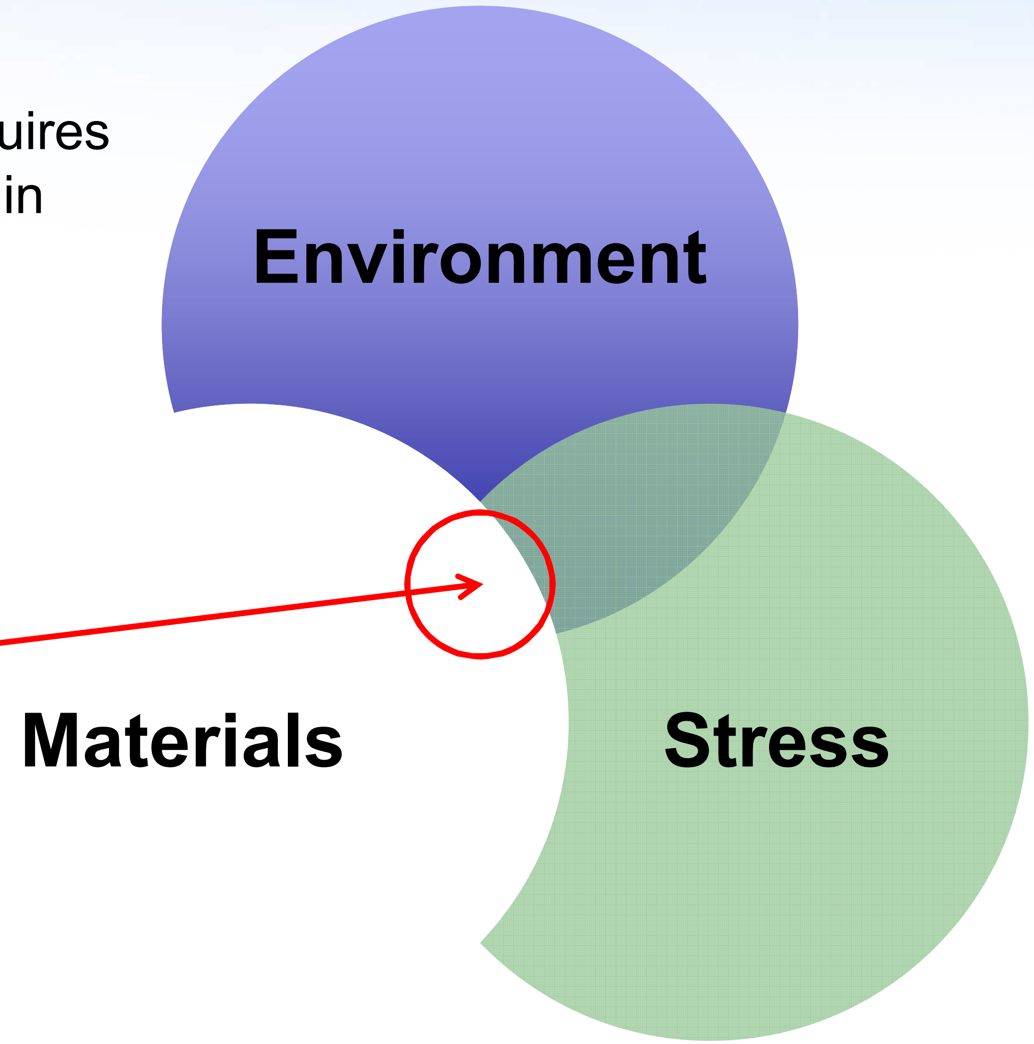
Mobile storage (fuel tanks)
- e.g., hydrogen forklifts: Cr-Mo ferritic steels
- Challenge: cycling ~6/day

Pressure manifold components
- ***Austenitic stainless steels***
- Challenges: low temperature, lower-cost alternatives (e.g., aluminum), alloy content, ***welding***

Structural properties must be measured in gaseous hydrogen

- *Compatibility* of materials for hydrogen service generally requires structural properties measured in gaseous hydrogen
- These measured structural properties are used directly in design to establish *suitability*

- Design space: Structural properties are needed to inform design
- Technical Reference: handbook of materials properties



Materials qualification requires a significant investment in evaluating materials

- Existing materials standards are largely insufficient for specifying materials for hydrogen service
  - Type 316/316L austenitic stainless steel is ***one known*** example of material that is very sensitive to hydrogen within the allowable compositional range
  - Implicit bounds on the strength of a material may need to be made explicit (hydrogen embrittlement is sensitive to strength)
- Standards that attempt to qualify a material require *multiple tests* on *multiple specimens* from *multiple batches* of material certified to the same designation
  - Quantification of a specific parameter (e.g., tensile ductility) usually requires a minimum of 9 tests (and could require more than 30)
  - In comparison, reports in the literature often represent single tests
  - Standards often require multiple parameters (eg fracture and fatigue)
  - Welds must be additionally qualified (testing x3 per ASME KD-10)
  - This is a lot of data!!

reacH2

A mechanism is needed to manage and disseminate materials qualification information

- Testing in gaseous hydrogen is expensive and time-consuming; few facilities exist
  - Access to materials properties measured in gaseous hydrogen should not be allowed to become a roadblock to commercialization of hydrogen technologies
- Databases aid qualification activities, materials selection and engineering analysis; however,
  - Text-based data presentation does not enable efficient communication of information (e.g. paper reports)
  - Paper reports limit comparison and integration of multiple data sets
- Robust software tools exist for managing databases of materials properties, as well as the pedigrees of the materials and the testing methods

Publications and Presentations

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- T. Michler, C. San Marchi, J. Naumann, S. Weber, M. Martin: "Hydrogen environment embrittlement of stable austenitic steels". *Intern J Hydrogen Energy*, vol. 37, 2012, pp.16231-16246.
- C. San Marchi, B. Somerday. "Technical Reference for Hydrogen Compatibility of Materials", Sandia National Laboratories Report SAND2012-7321, 2012.
- (invited presentation) C. San Marchi, "Overview of Gaseous Hydrogen Embrittlement Testing at Sandia", Korean Society of Mechanical Engineering, Annual Fall Meeting, Special International Session on Hydrogen Effects in Materials, Chagwon, Korea, Nov. 2012.
- (invited presentation) C. San Marchi, A. Harris, D. Dedrick, "Investigation of the Hydrogen Release Incident at the AC Transit Emeryville Facility", Korea Research Institute of Standards and Science (KRISS), Daejeon, Korea, Nov. 2012.
- (invited presentation) C. San Marchi, "Standards for Qualifying Materials for Hydrogen Service", Korea Research Institute of Standards and Science (KRISS), Daejeon, Korea, Nov. 2012.
- B. Somerday, K. Nibur, and C. San Marchi, "Measurement of Fatigue Crack Growth Rates for SA-372 Gr. J Steel in 100 MPa Hydrogen Gas Following Article KD-10", ASME PVP2013, Paper No. 97455, 2013, submitted.
- B. An, H. Itouga, T. Iijima, C. San Marchi, and B. Somerday, "Hydrogen-assisted twin boundary fracture of type 304 austenitic stainless steel at low temperature investigated by scanning probe microscopy", ASME PVP2013, Paper No. 97355, 2013, submitted.
- C. San Marchi, L. Hughes, B. Somerday, and X. Tang, "Hydrogen-Assisted Fracture of Type 316L Tubing and Orbital Welds, ASME PVP2013, Paper No. 97538, submitted.

Additional References available in other current and previous projects: SCS005, SCS011, and SCS012