Materials for Cryogenic Hydrogen Storage Technologies

Project ID# ST200

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Overview

Timeline and Budget

- Project Start Date: 04/01/18
- FY18 DOE Funding (if applicable): $ 250K
- FY19 Planned DOE Funding (if applicable): PNNL: $649K
- SNL: $100K
- ANL: $100K
- ORNL: $150K
- Total DOE Funds Received to Date: $999K

Barriers

G: Materials of Construction

L: Lack of Tank Performance Data and Understanding of Failure Mechanisms

F: Codes and Standards

Partners

Pacific Northwest National Laboratory

Sandia National Laboratories

Oak Ridge National Laboratory

Argonne National Laboratory

Relevance

Objectives:

- Develop a material acceptance process that will provide detailed information to evaluate specialty resins, vessel liner options, and carbon fiber composite materials through thermomechanical testing.
- Investigate cryogenic material systems for use in 350+ bar cryo-compressed and sub-ambient (~20-30K) hydrogen pressure vessels.

Technical Barrier	Project Impact
Cryo-compressed hydrogen storage limits choice of materials	Improve resins, engineered fibers, and composites for cryogenic performance
Lack of material properties data and understanding of failure mechanisms at cryogenic temperatures	Investigate material properties throughout -253°C to +120°C
Lack of applicable codes and standards for materials acceptance	Develop acceptance testing criteria for base materials and engineered systems

Approach

- Experimentally test resin, carbon fiber composite, and welded aluminum and steel liner systems from -253°C to 120°C temperature range.
- Combined testing includes:
  - Cryogenic temperature tests
  - Thermal cycling from cryogenic to elevated temperatures (-253°C to +120°C)
  - Off-gassing under vacuum (10-6 torr)
  - Fatigue cycling at non-ambient conditions equivalent to the stress states in the composite at the maximum allowable working pressure and standard test cycles
- Numerical modeling will use the experimental results to predict the relative change in full tank burst properties at different temperatures

Project Tasks

Task 1:

Industrial Survey (PNNL)

- Collaborators from the pressure vessel and material supplier industry
- Focus on methods to qualify materials for use in cryo-compressed or cold gas hydrogen pressure vessels

Task 2:

Qualification Roadmap (PNNL)

- Illustrate path forward from coupon-level to full tank modeling and validation

Task 3:

Composite Test Methods and Geometries (PNNL/ORNL)

- Investigate performance of individual materials that make up a filament wound composite overwrapped pressure vessel
- Evaluate material properties at various temperatures

Task 4:

Liner Test Methods and Geometries (PNNL/SNL)

- Investigate polymeric and metallic liner material properties and weld performance
- Evaluate cryogenic mechanical fatigue performance
- Investigate TIG and friction stir weld performance

Task 5:

Temperature Effects Testing (PNNL)

- Test mechanical properties of Task 3 at temperatures from -253˚C to +120˚C
- Includes tensile, short beam shear, shear, and fatigue testing as a function of thermal cycling

Task 6:

FEA of Tank-Level Performance (PNNL/ANL)

- Estimate tank pressure retaining and burst performance as a function of operating temperature using FEA methods for filament wound composite tank cylinders

Task 7:

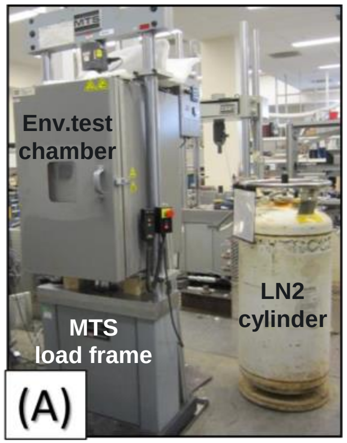
Acceptance Testing Criteria (PNNL)

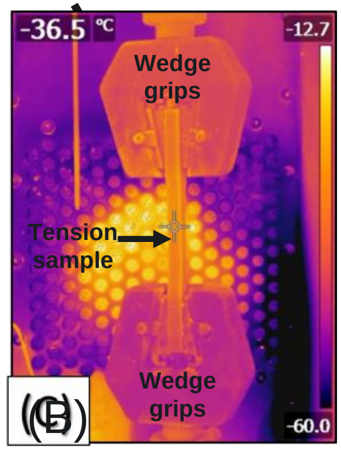
- Review allowable coefficient of variance and statistical requirements based of test data scatter

Approach

PNNL's Cold/Cryo-temperature Mechanical Testing Capabilities

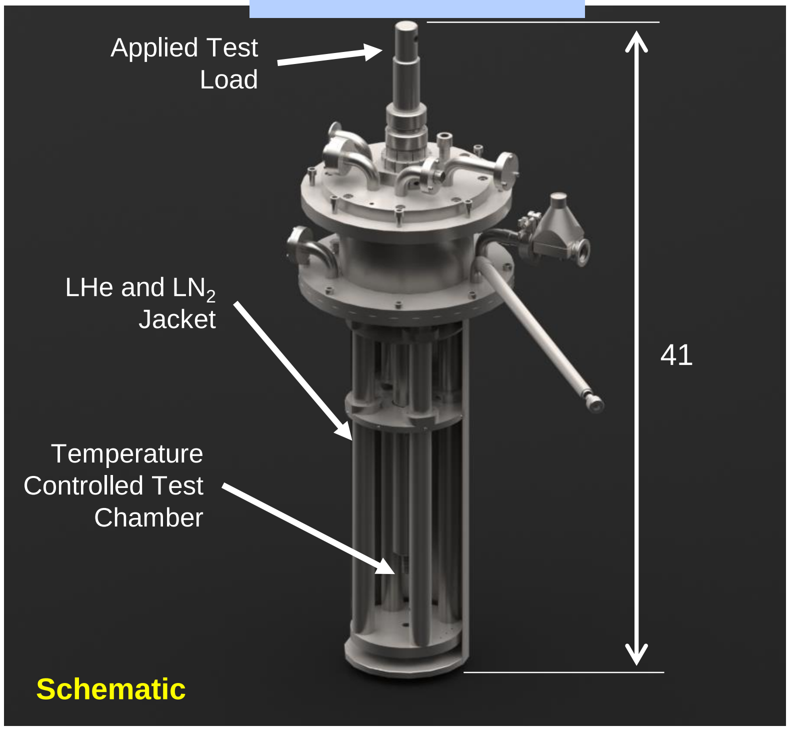
-130°C to +315°C





- Mechanical Test System (MTS) load frame with liquid nitrogen-cooled environmental chamber
- Strain measurement
  - Extensometer capabilities to -253˚C
  - Digital image correlation (DIC)

-253°C to +25°C



- Liquid helium-cooled Janis Research dewar for materials testing with controlled temperature chamber

Accomplishments and Progress

Thermomechanical Testing on Baseline Epoxy and Thermoplastic Liner Material

Tensile Properties

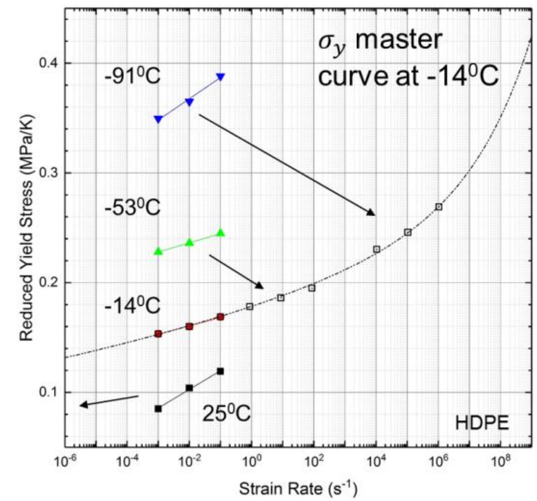
- Temperature-strain rate-dependent tensile properties measured at sub-ambient temperatures
  - Transformation will allow prediction of tensile properties at temperatures that are difficult to obtain in the laboratory

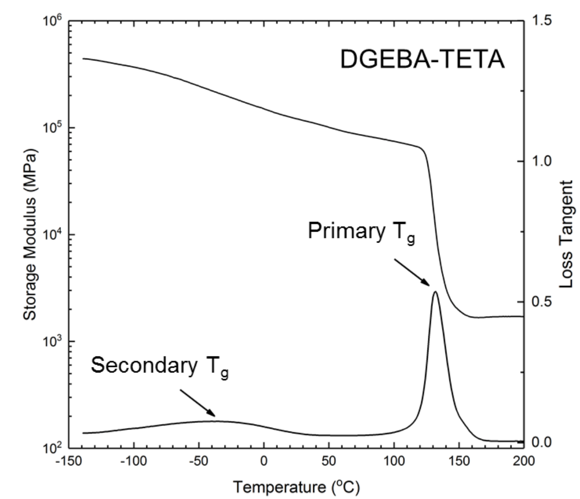
Dynamic Mechanical Properties

- Dynamic mechanical analysis shows primary and secondary glass transitions and crystalline melting temperatures defining thermal regimes of mechanical performance change

Thermal Contraction

- Temperature-dependent linear thermal contraction measured through -140˚C to +120˚C





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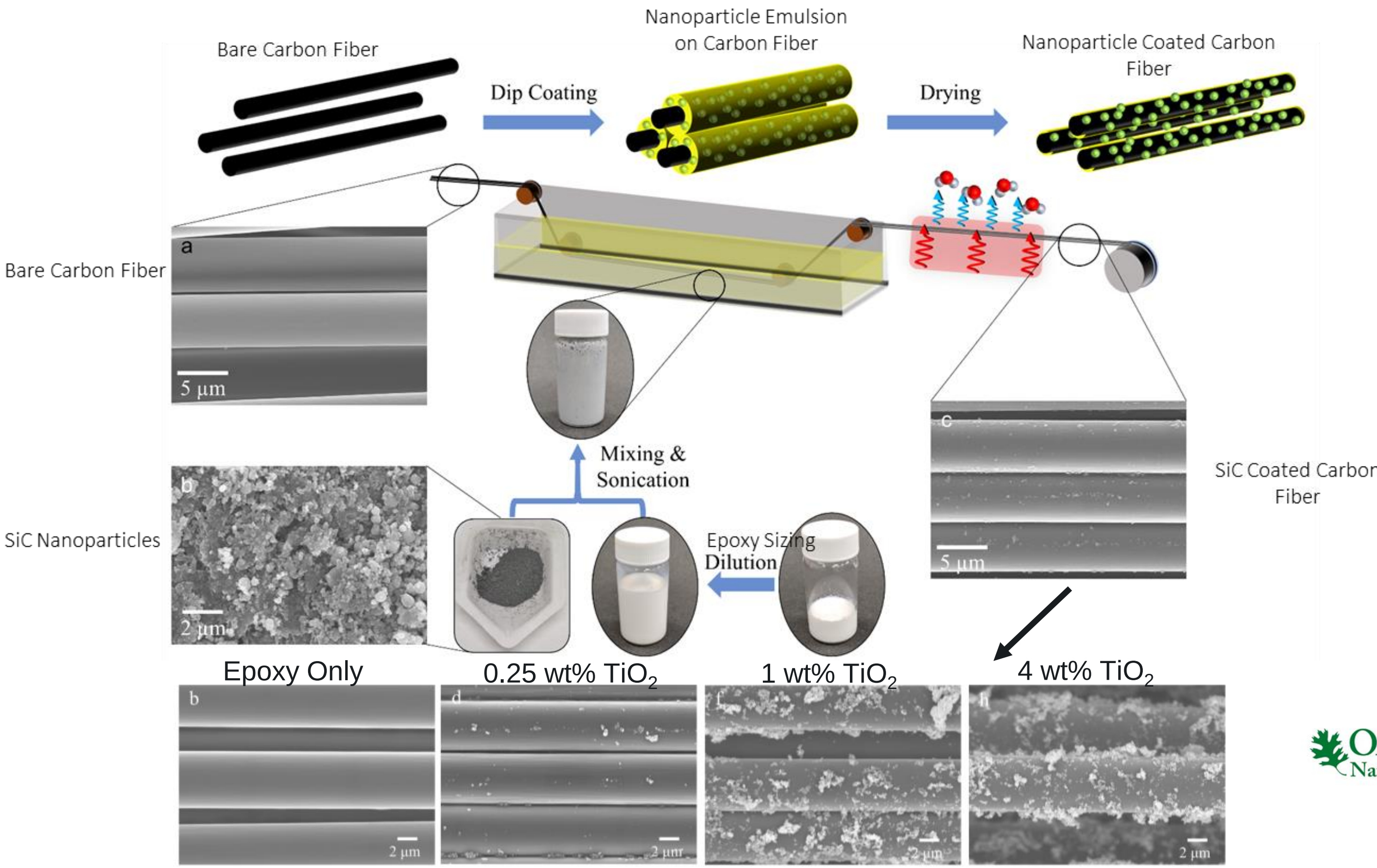
Approach

Sizing Modification to Improve Interfacial Adhesion

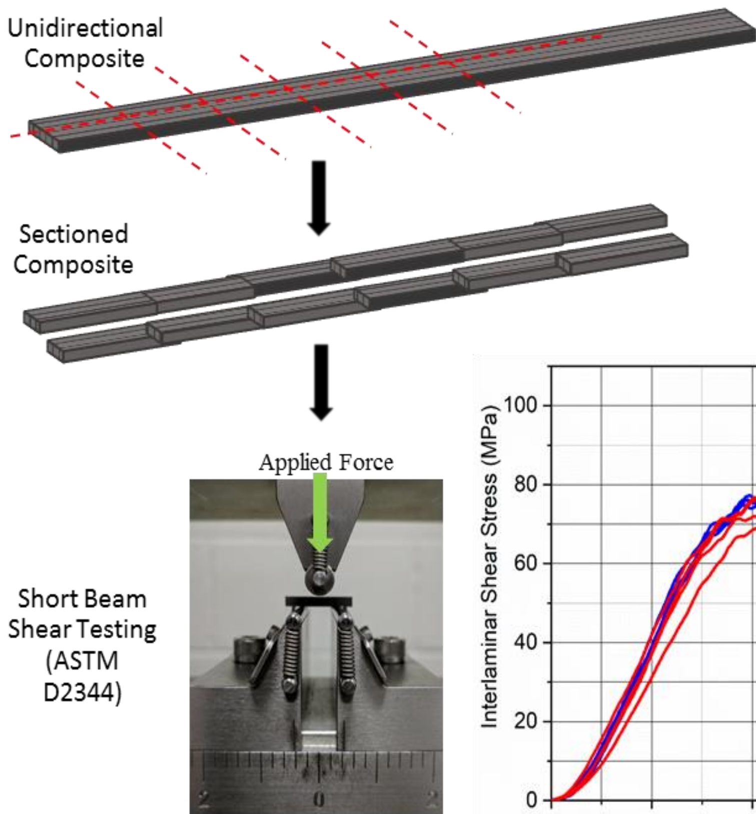


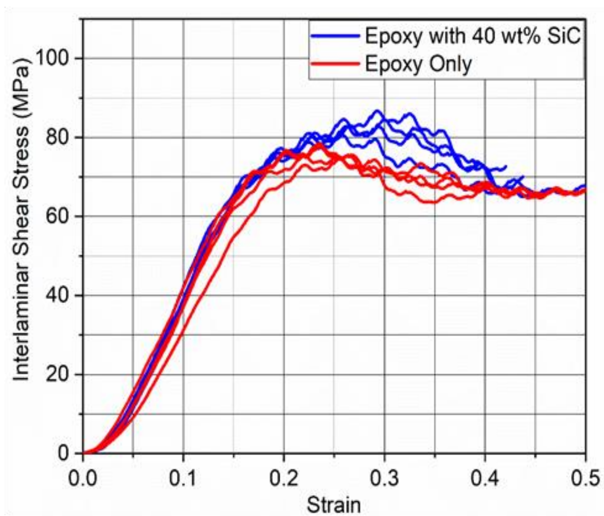
Concentration of nanoparticles in solution

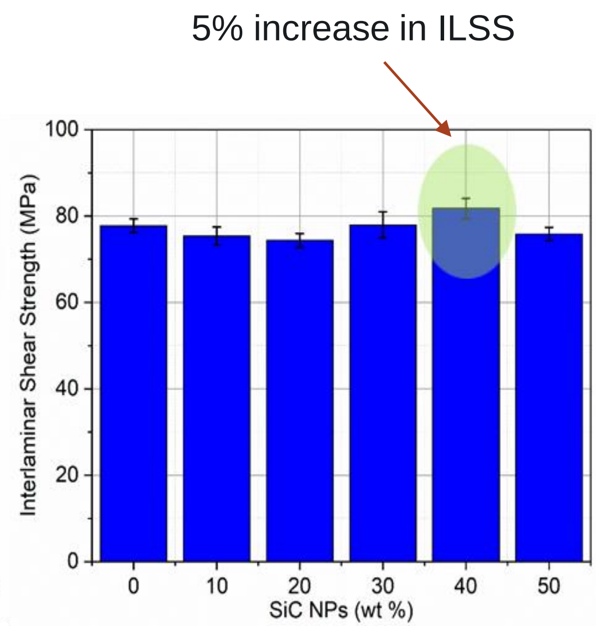
OAK RIDGE National Laboratory

Preliminary Short Beam Shear Results

- Short beam shear at room temperature for interlaminar shear strength (ILSS)
- 40 wt% SiC nanoparticles increased ILSS by 5%







Approach
Understanding Materials Behavior in Cryogenic Hydrogen Environments

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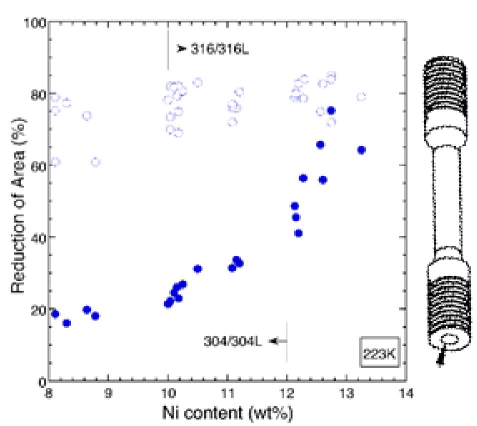
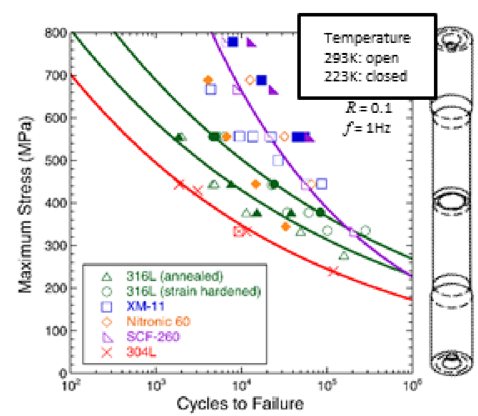
Sandia National Laboratories

- Establish baseline behavior of low-cost candidate materials in cryogenic hydrogen environments
  - Use H-precharging to overcome kinetics of hydrogen uptake
  - Evaluate high-performance welds and base materials of austenitic stainless steel: 304L
  - Evaluate performance of aluminum base material: AA2219

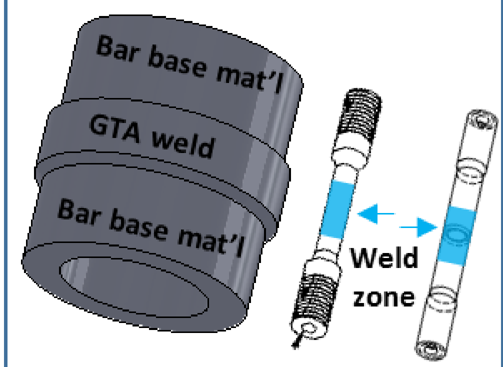
Slow strain rate tension



Notched fatigue life

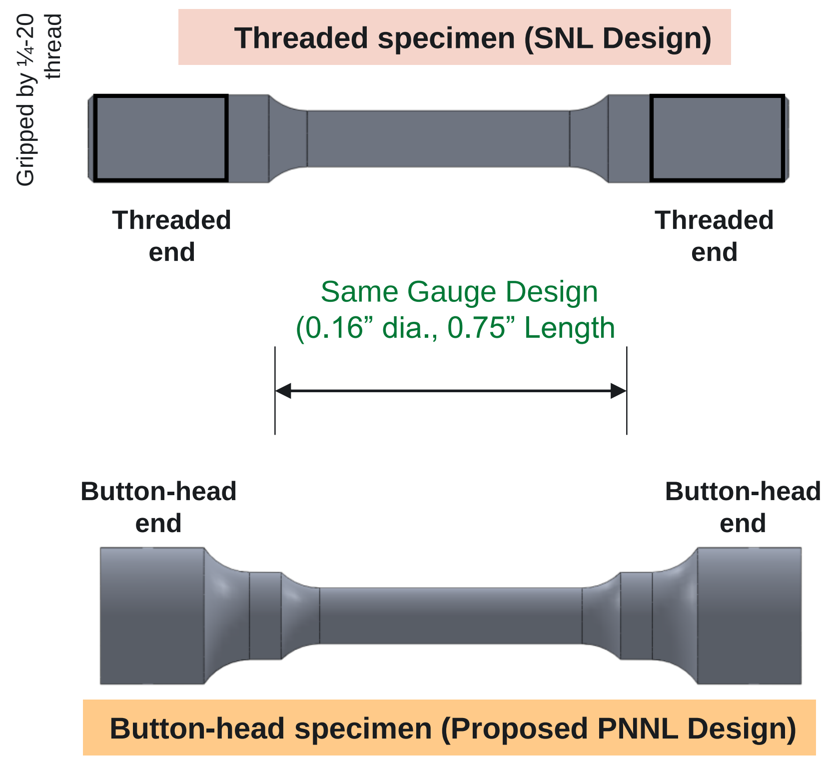


Welds



Approach
(some) Challenges in Low-temperature Testing Sample Design

- Sample handling → Limited maneuverability due to use of gloves/tongs etc. to handle samples at sub-zero temperature
- Sample gripping → Threaded ends can seize in the grips
  - Replace threaded end design with a button-head design for ease of installation and removal
- Load-frame capacity → Failure load of 304L (0.16” gauge dia.) <2,500 lbf @ 25˚C and <4000 lbf at -130°C. PNNL’s load frames can handle (upto 20,000 lbf)

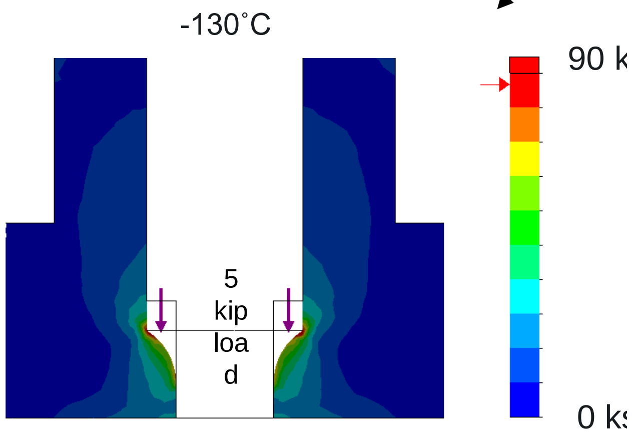


Approach

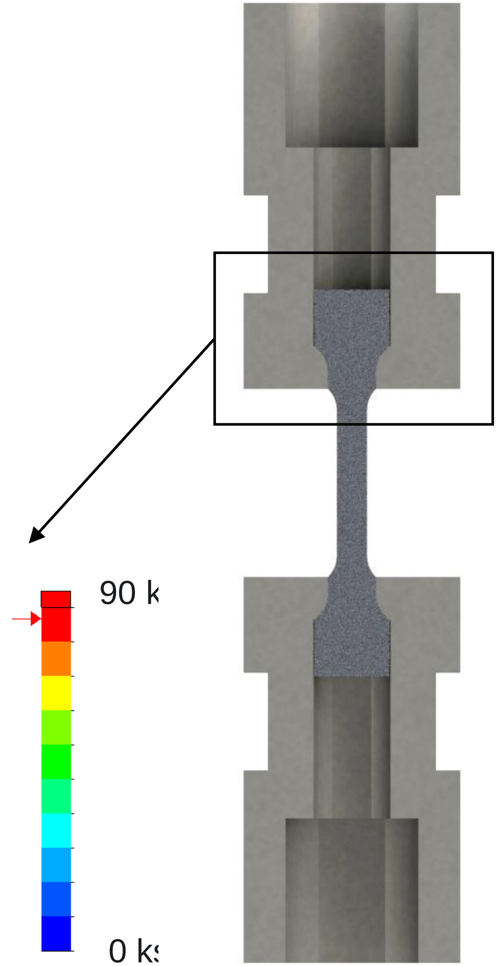
(some) Challenges in Low-temperature Testing: Grip design

- Button-head for rapid specimen fixturing between tests
- Grip material: Nitronic 60
  - $\sigma_y = 58\ \mathrm{ksi}$ at 25˚C
  - $\sigma_y = 85\ \mathrm{ksi}$ at -130˚C
  - <u>Maintains ductility </u>at -253˚C

Grip for Tension Testing



Stress Analysis of the Grip

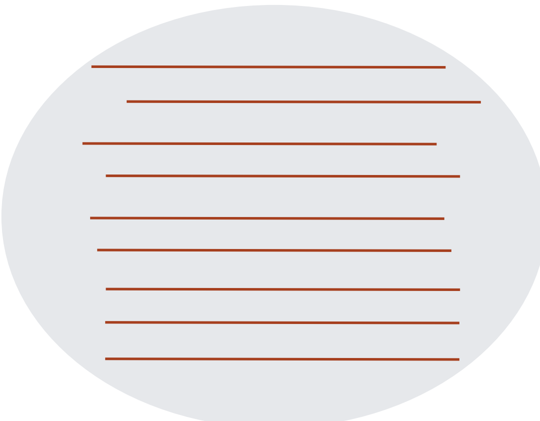


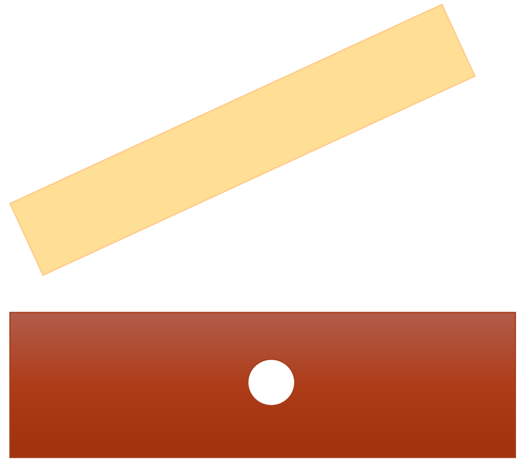
<u>Schedule</u>

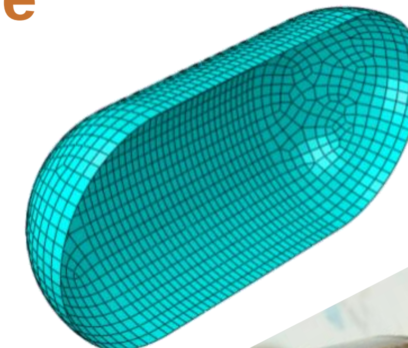
- Grip fabrication: end of March
- Trial tension testing (-130°C, 304L): March/April
- LHe dewar delivery: March/April

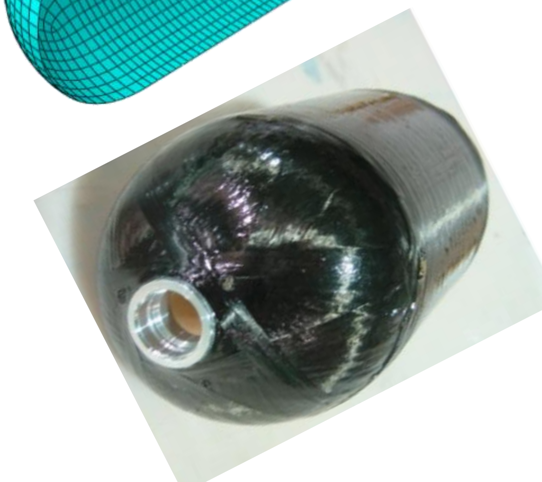
Approach:

Cryo-compressed H_2 Vessel Modeling from Constituents to Tank Structure









Micro and Meso scales

- CF & epoxy thermomechanical properties and stress/strain data as functions of temperature
- Homogenization & constitutive modeling (PNNL's EMTA & EMTA-NLA)

Macro scale 1

- Constitutive models validated on simple laminated specimens
- Predicted stress/strain responses and damage compared to experiments (EMTA-NLA/ABAQUS)

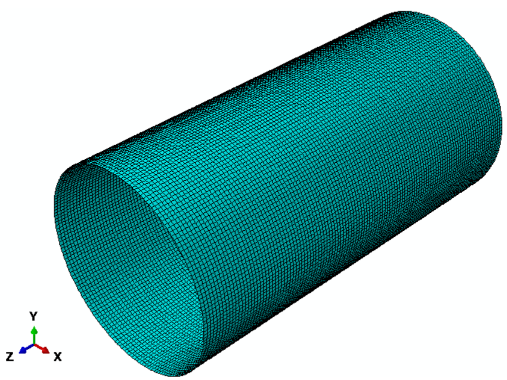
Macro scale 2

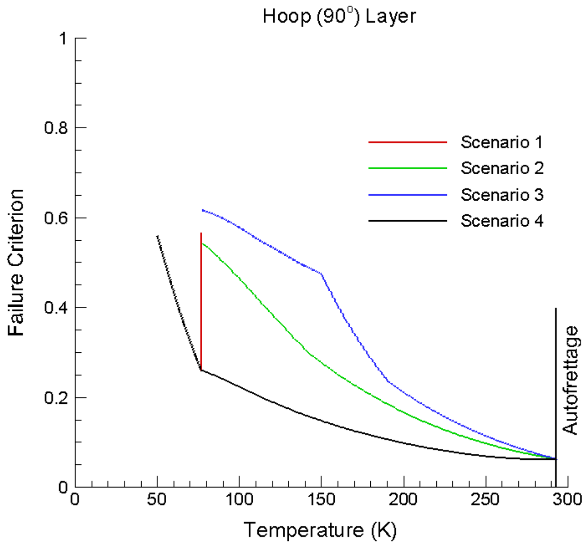
- Constitutive models validated on H_2 vessels
- Design vessel layup to sustain thermomechanical loadings (EMTA-NLA/ABAQUS)

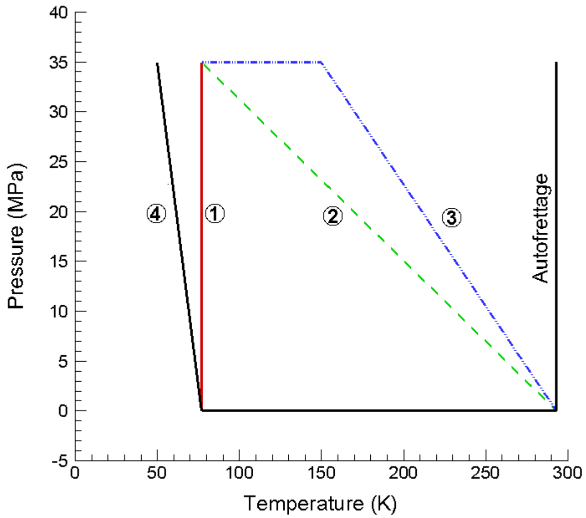
Accomplishments and Progress
Type 3 H_2 Pressure Vessel Analysis

ABAQUS/EMTA-NLA

- Composite cylinder with aluminum liner modeled with 4 pressurization scenarios mimicking tank filling
- Layup: Al liner/90º/+10º/-10º/ (with respect to the axial direction)
- Model accounts for CTE of fiber and matrix variation with temperature
- Tank is predicted not to exceed failure criterion for the loading scenarios considered

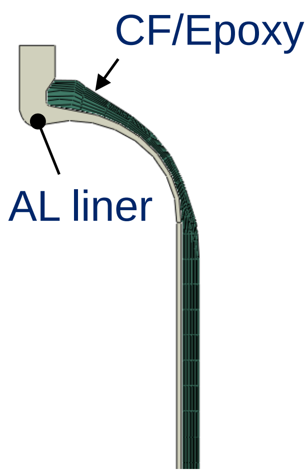


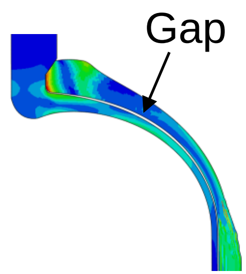




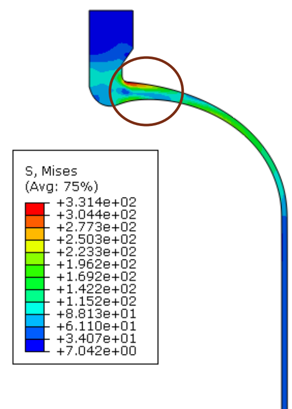
Accomplishments and Progress

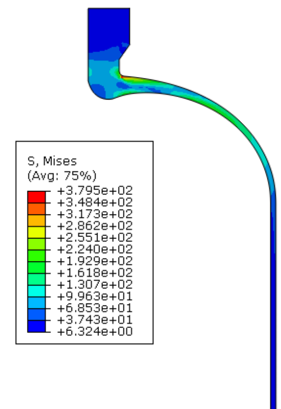
FE Analysis of RT to 80K 50 MPa Cryo Hydrogen Tank





CF/Epoxy and aluminum liner after cooling down (80K)





Aluminum liner (80K) w/ empty pressure of 0.5 MPa

Steel liner (80K) w/ empty pressure of 0.5 MPa

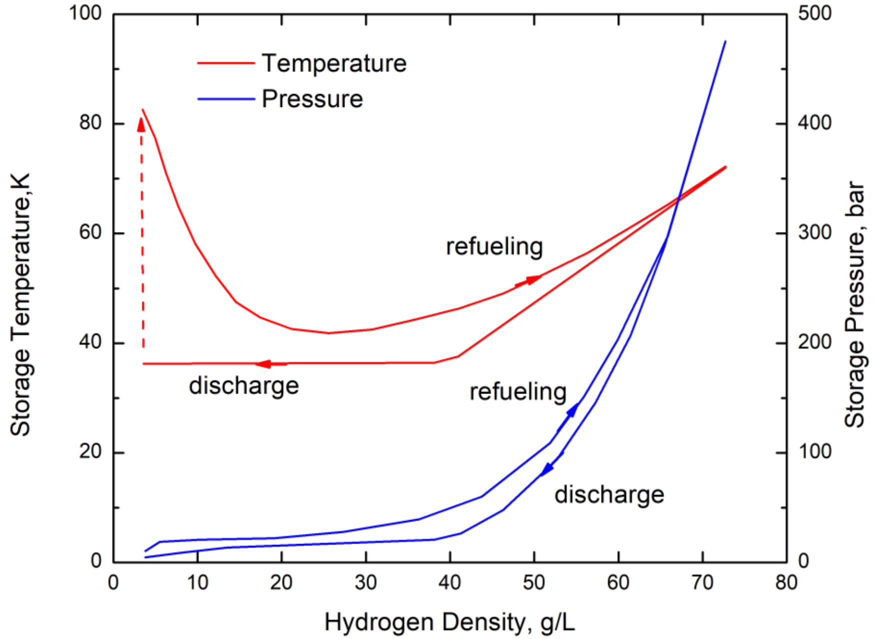
- FE model
  - 2D axisymmetric model
  - Assuming no bonding at the interface between AL liner and composite material
  - Applying burst pressure (2.25x 500 bar)
  - Compared maximum strains along fiber direction

- Sensitivity Analysis
  - The matrix-dominant properties are considered for sensitivity analysis
  - Found that any enhancement of shear stiffnesses has no effect on reducing the maximum fiber strain

- During cooling down step, cryo-tanks were empty. 0.5 MPa of empty pressure was applied. Both liner materials have the maximum stresses around the corner of the neck because composite layers resist to shrink.
- Plastic deformation did not occur in steel liner, but it occurred at the corner in aluminum liner.

Approach
Fatigue loading scenario

- Fatigue cyclic loads are imposed on the same condition as the refueling and discharging process. In this condition, temperature and internal pressure range from 35K to 80K and from 5 bar to 500 bar, respectively
- Also, temperature cycling from RT to 80K will be considered for fatigue analysis



Source: IJHE Vol 43 2018 pp10215-10231

Accomplishment Summary

- Thermomechanical techniques for temperatures down to -140˚C proven
- 5% increase in interlaminar shear strength by nanoparticle modifications
- Approach to testing H-charged steel and aluminum materials identified
- Pressure vessel modeling assuming literature values demonstrates tank survival
- Aluminum liner strain levels are high around the neck of the tank boss

Collaborative Activities

Partner	Project Roles
PNNL	Project Lead, resin, cryogenic mechanical testing, constituent material modeling, joining properties
ORNL	Interfacial surface modification of fibers and nanoparticulate resin modification for microcrack mitigation working with PNNL on cryo resins
ANL	FE full scale tank models integrated with PNNL constituent material models and cryogenic material data
SNL	Hydrogen compatibility of liner materials and joining properties at cryogenic temperatures with PNNL

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Proposed Future Work

- Remainder FY19
  - Cryogenic resin system testing
  - Transformation model for resin property prediction at cryogenic temperatures
  - Further modification of sizing and nanoparticle concentrations
  - Exploring various nanomaterials of different compositions and aspect ratios
  - Low temperature short beam shear strength testing
  - Resin modification to further enhance strength and improve gas permeability
  - Weld strength testing at ambient and cryogenic temperatures
  - Modeling of fatigue scenarios
- FY20
  - Cryogenic composite testing
  - Cryogenic testing of modified resins in composite materials
  - Multiaxial material properties testing at subambient and cryogenic temperatures
  - Modeling of multiaxial material test
  - Model validation of component level testing

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