Project ID: ST115

Achieving Hydrogen Storage Goals through High-Strength Fiber Glass

2015 U.S. DOE HYDROGEN and FUEL CELLS PROGRAM
ANNUAL MERIT REVIEW and PEER EVALUATION MEETING

Washington DC, June 8 - 12, 2015

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HEXAGON LINCOLN

Pacific Northwest NATIONAL LABORATORY
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Overview

Time Line

Project Starting Date: 09/01/14

Ending Date: 08/30/16

Barriers

- System Cost

Budget

- Total Project Budget: $1,400,006
  - Total Recipient Share: $200,006
  - Total FFRDC Share: $400,000
  - Total Federal Share: $800,000
  - Total DOE Funds Spent: ~$165,000

Partners

- **PPG Industries, Inc.**
- **Hexagon Lincoln**
- **Pacific Northwest National Lab**

Relevance: Project Goals

- **A Type IV composite overwrapped pressure vessel (COPV) reinforced exclusively with glass fiber**
- **Utilizing three major innovations**
  - A new glass fiber with strength matching with T-700 at less than half its cost
  - A novel glass fiber manufacturing process
    - Enable large scale direct draw production over existing S2-glass fiber parameter process limited to roving product
  - A study of the stress rupture behavior of composites made from the new fiber
- **The new tank will lower the composite contribution to system cost by nearly 50% with minimal impact on tank weight and capacity compared to tanks made with T-700 carbon fiber**

Relevance

Performance Assessment	Strength (KSI / MPa)	Safety Factor	Estimate Cost ($/kg)
Benchmarking [1] T700S CF (commercial)	710 / 4900[2]	2.25	28.67
Project Target	798 / 5500[2]	3.0 – 3.5	11 - 16
BP1 - Task 1: High strength glass fiber selections[4]	777 / 5357[3] 810 / 5583[3]		TBD

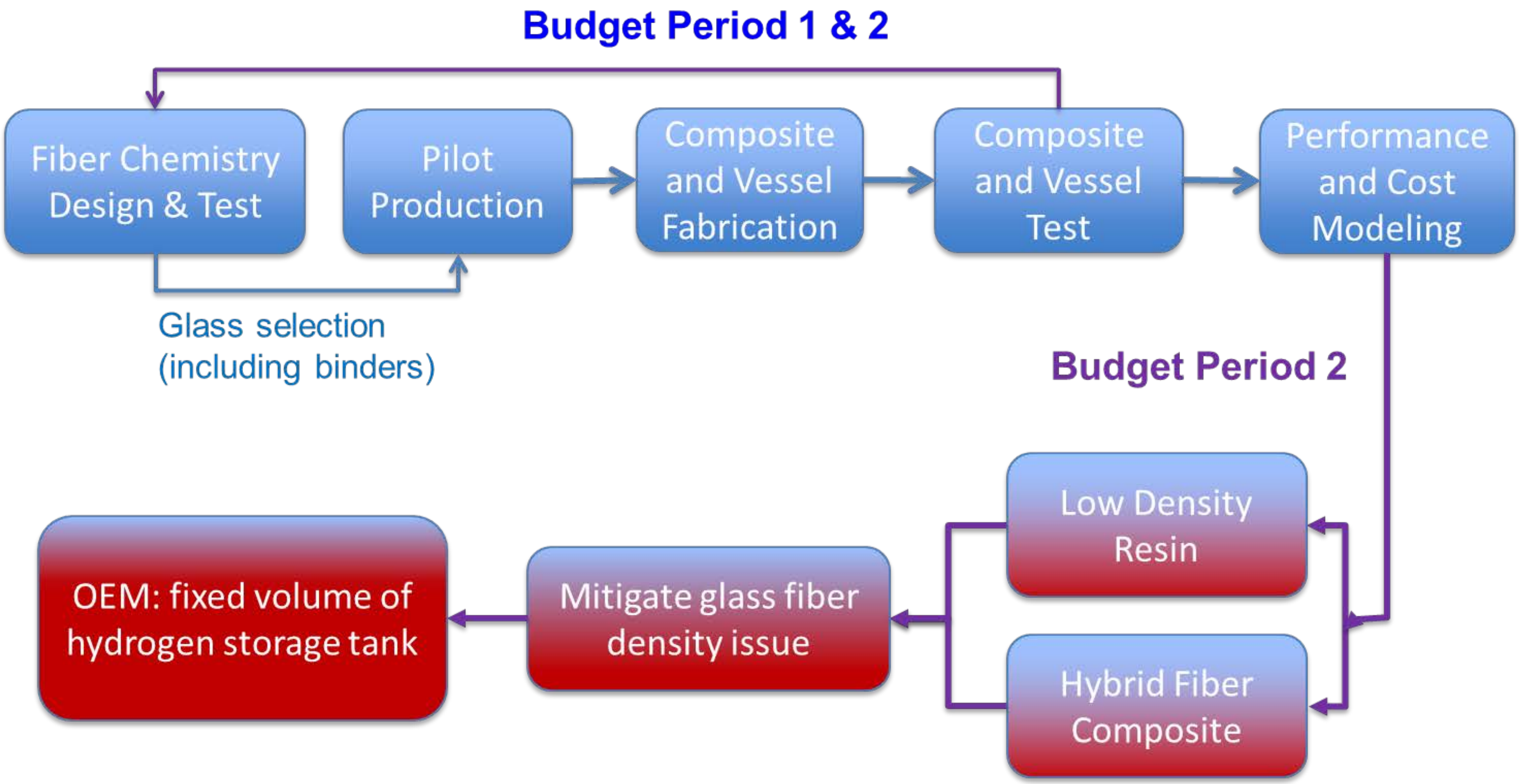
[1] DOE Fuel Cell Tech Office Record, 13010, Jun 11, 2013

[2] Tensile strength of strand

[3] Tensile strength of filament

[4] WO 2015009686A1, PPG Industries Ohio, Inc. (Jan 22, 2015)
US 20150018194A1, PPG Industries Ohio, Inc. (Jan 15, 2015)

Approach: Program Plan



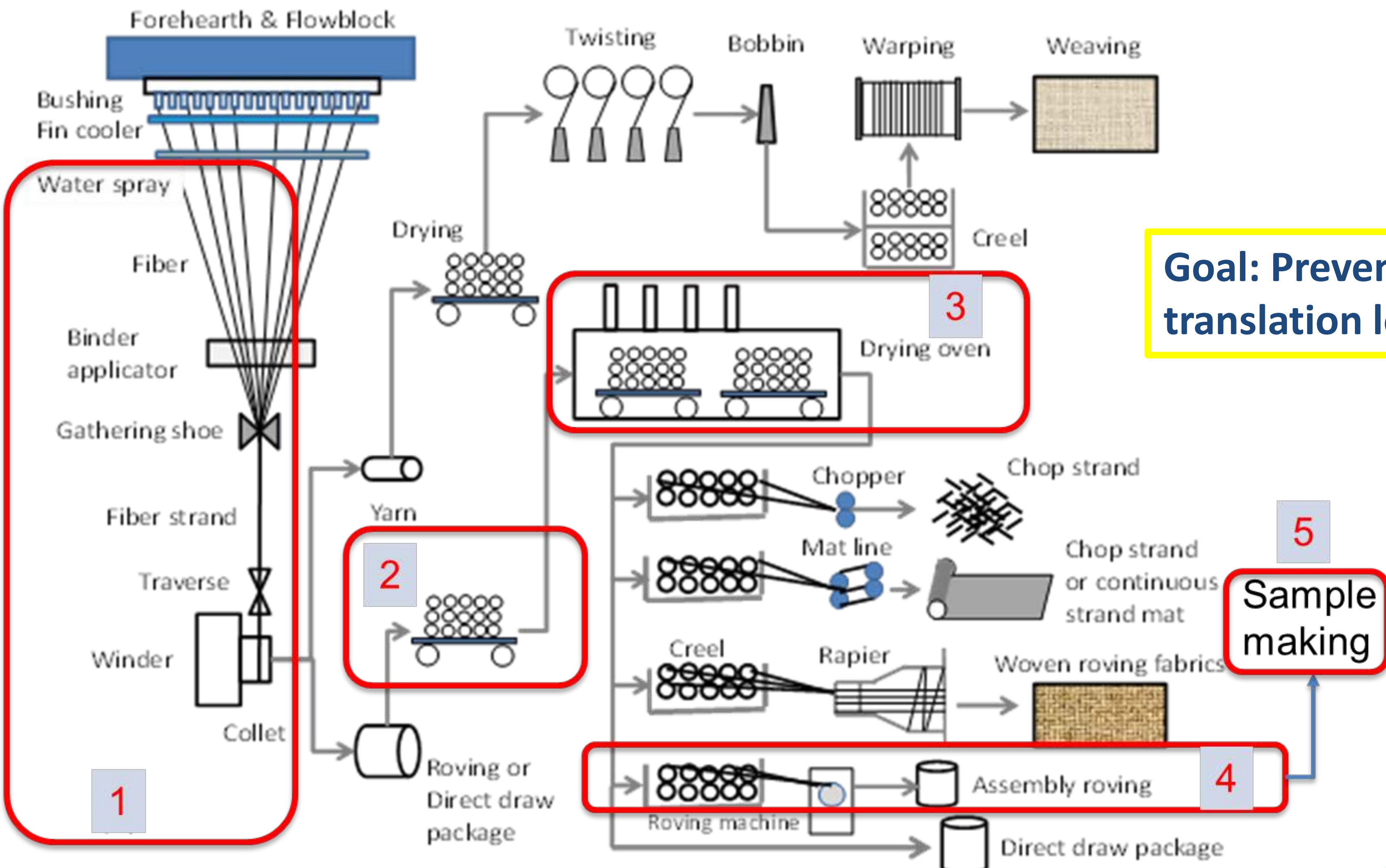
Approach: High Pressure Vessel Test Plan

- **Budget Period 1**
  - Control tanks
  - Prototype tanks
    - 4 combinations: 2 fiber glasses + 2 binder coatings

- **Budget Period 2**
  - Prototype tanks
    - 2 combinations: 1 fiber glass + 2 binders **OR** 2 fiber glasses + 1 binder
  - Full-size tanks
    - Baseline tank
    - Best fiber glass + best binder

Approach

Fiber Forming and Product Down Stream Processes



Goal: Prevent translation loss

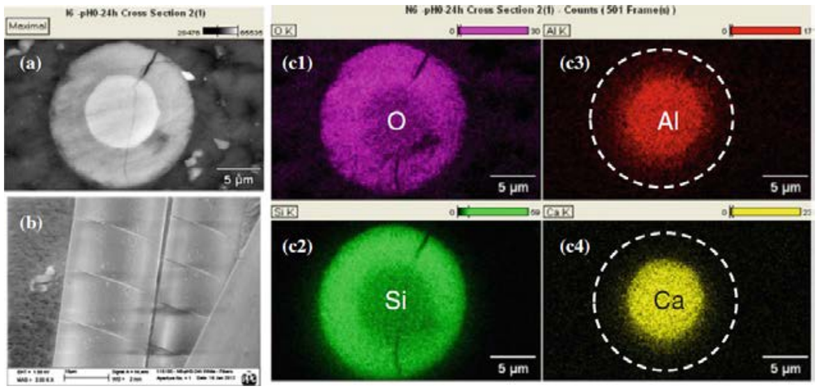
Approach / Milestones

Milestone	Plan * Updated - Actual Date	Status
1.1 Two glass compositions selected	02/13/15 * 04/23/15	Underway
1.2 Upgraded melter installed and operational	03/13/15 * 07/31/15	Underway
1.3 Produce 130 lbs. of 2 unique glass fibers	07/03/15 * 07/03/15	Underway
2.1 Tank design complete	10/28/14 * 03/20/15	Completed
2.2 Translation and ease of use determined	05/29/15 * 07/10/15	Underway
2.3 Performance gaps identified	07/03/15 * 07/24/15	
3.1 Identify stress rupture behavior differences	07/03/15 * 12/28/15	Delayed
4.1 Glass produced at 4x BP1 rate	02/15/16 * 04/18/16	
5.1 Alternate tank design complete	11/23/15 * 03/21/16	
5.2 Confirm predicted performance improvement	08/01/16	TBD
5.3 Confirm 700 bar tank performance	09/01/16	TBD
6.1 Confirm stress rupture performance	04/11/16	TBD
6.2 Identify impact of low-density resin	07/11/16	TBD
7.1 Subaward executed	10/01/14 – 02/27/15	Completed

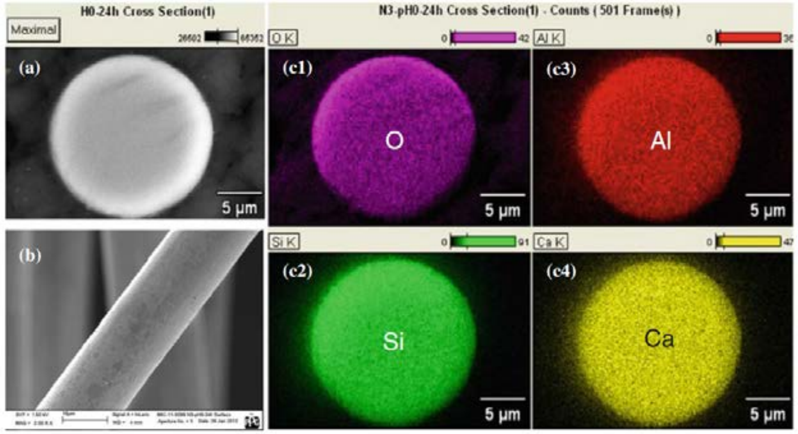
Accomplishments: Glass Composition

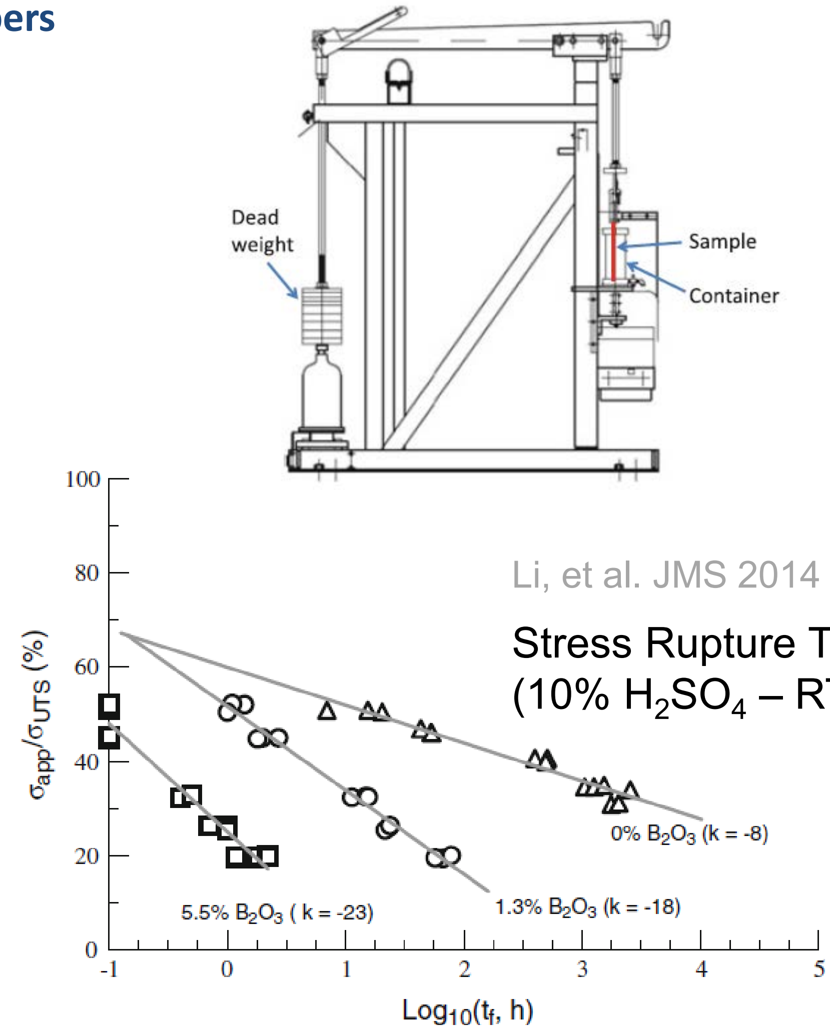
Basis of Choosing Reference Boron-Free Glass Fibers

Tradition E-glass (6% B_2O_3)



E-CR glass (0% B_2O_3)





Li, et al. JMS 2014 (PPG)

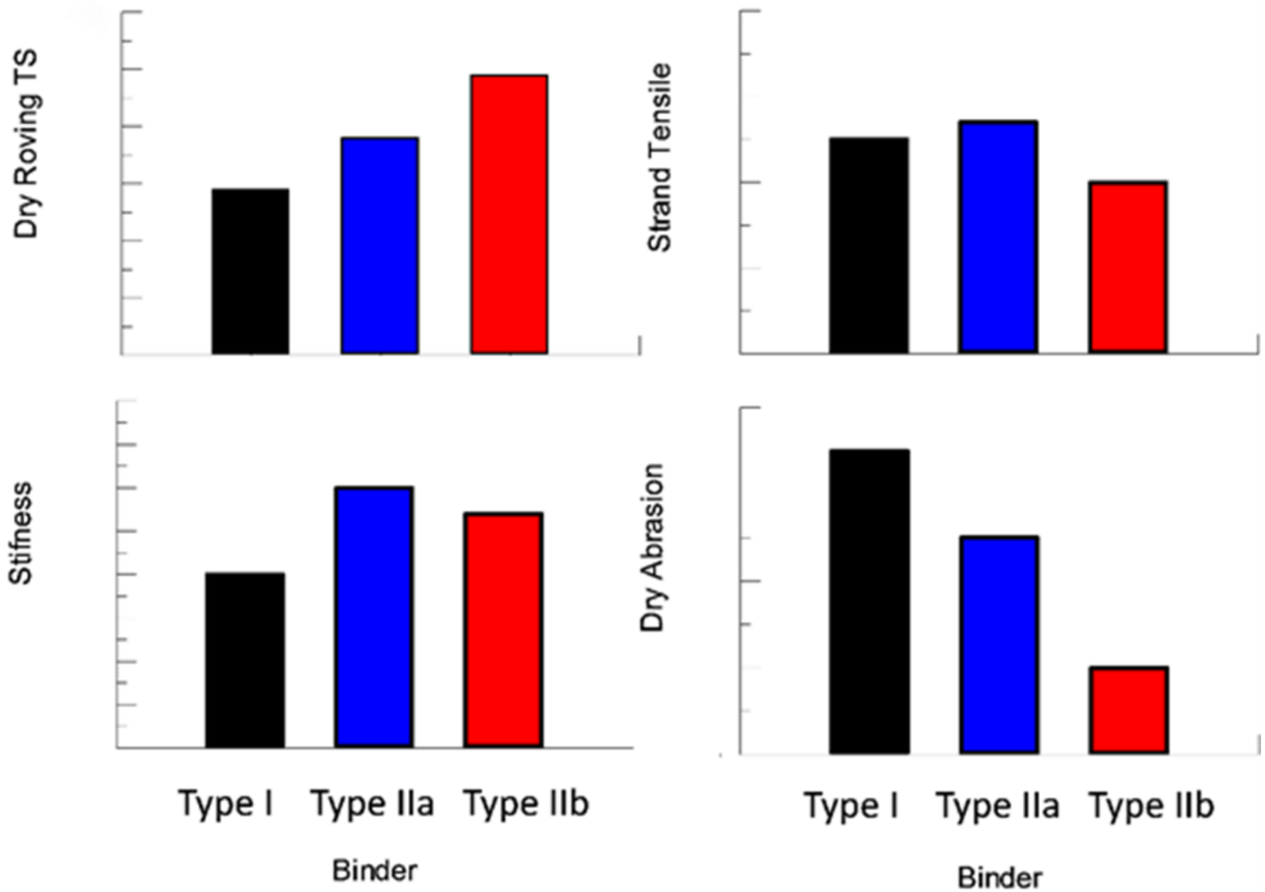
Stress Rupture Test
(10% H_2SO_4 – RT)

Boron-free shows improved performance

Accomplishments: Binder Selection

Basis to Binder Chemistry Selection

Glass Fiber Reference: INNOFIBER® CR Fiber Glass



New binder chemistries look promising

Accomplishments: Fiber Performance

Basis of Optimization of Fiber Forming Process:
Effects of Melting Temperature and Binder on Strand Tensile Strength

WO 2015009686A1, PPG Industries Ohio, Inc. (Jan 22, 2015)
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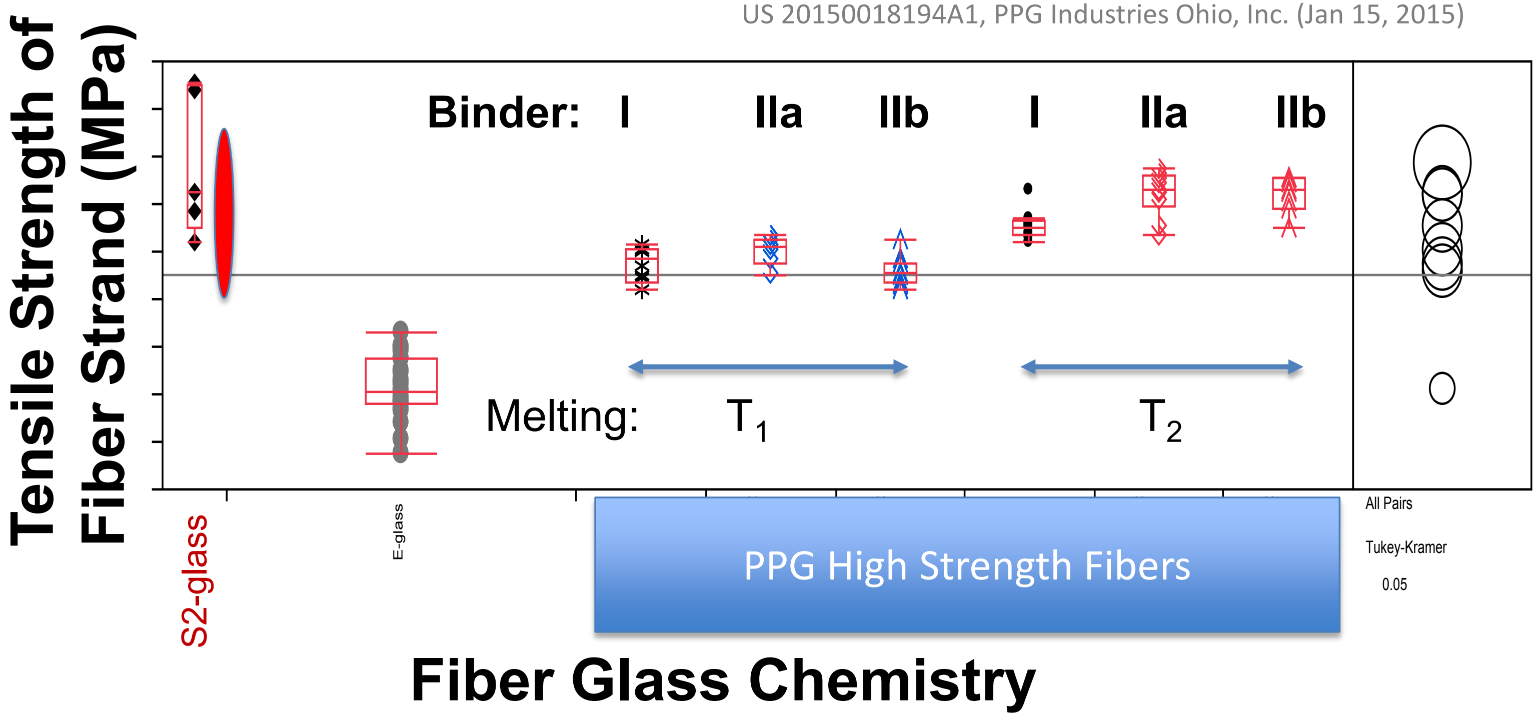
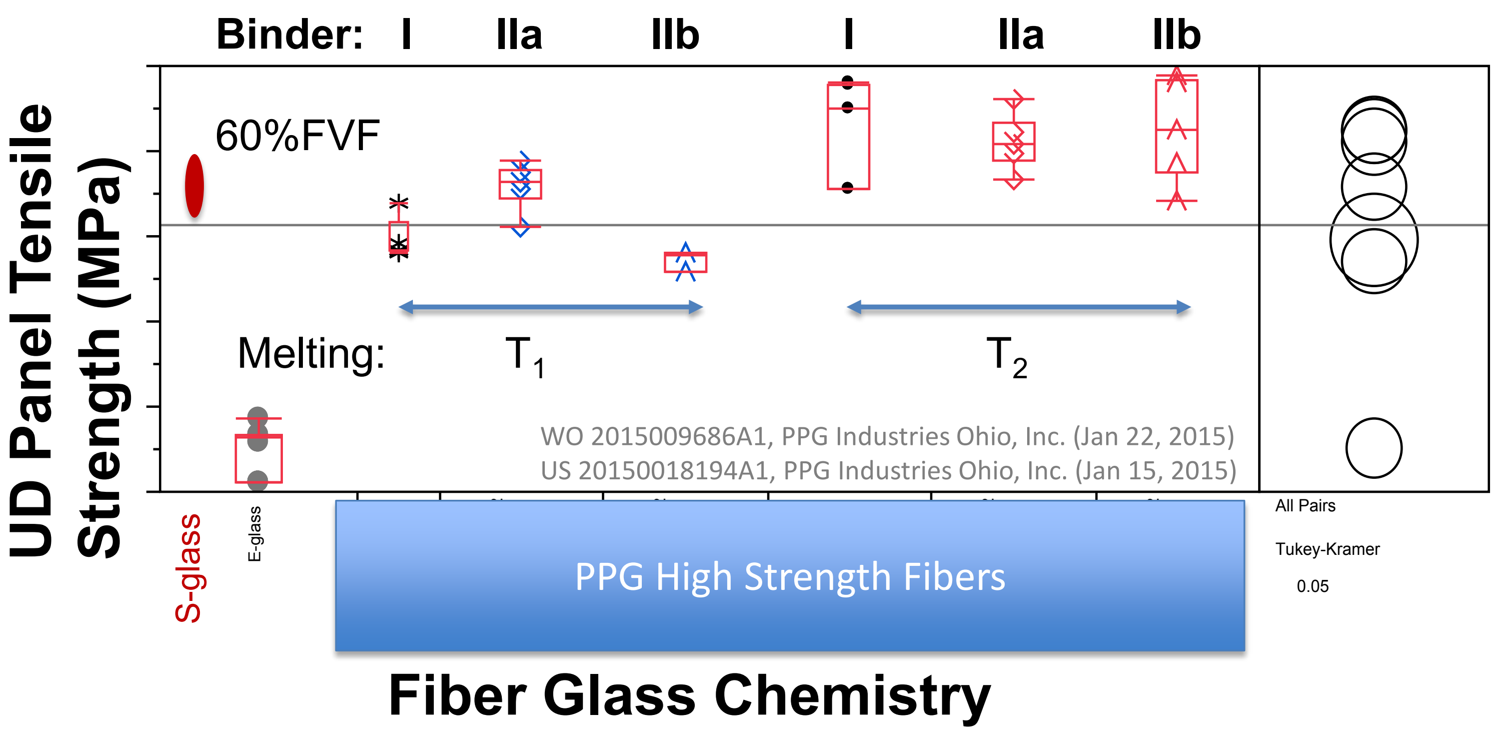


Melting temperature optimization improves strand tensile
Binder affects broken filament of strand, type II is better than type I

Fiber performance looks promising

Accomplishments: Composite Performance

Basis of Optimization of Fiber Forming Process (continued):
Effects of Melting Temperature and Binder on Laminate Tensile Strength



Melting temperature optimization improves UD laminate tensile strength
Binder affects broken filament of UD fabric, type II is better than type I

Composite performance looks promising

Accomplishments: Stress Rupture Test Design/Validation

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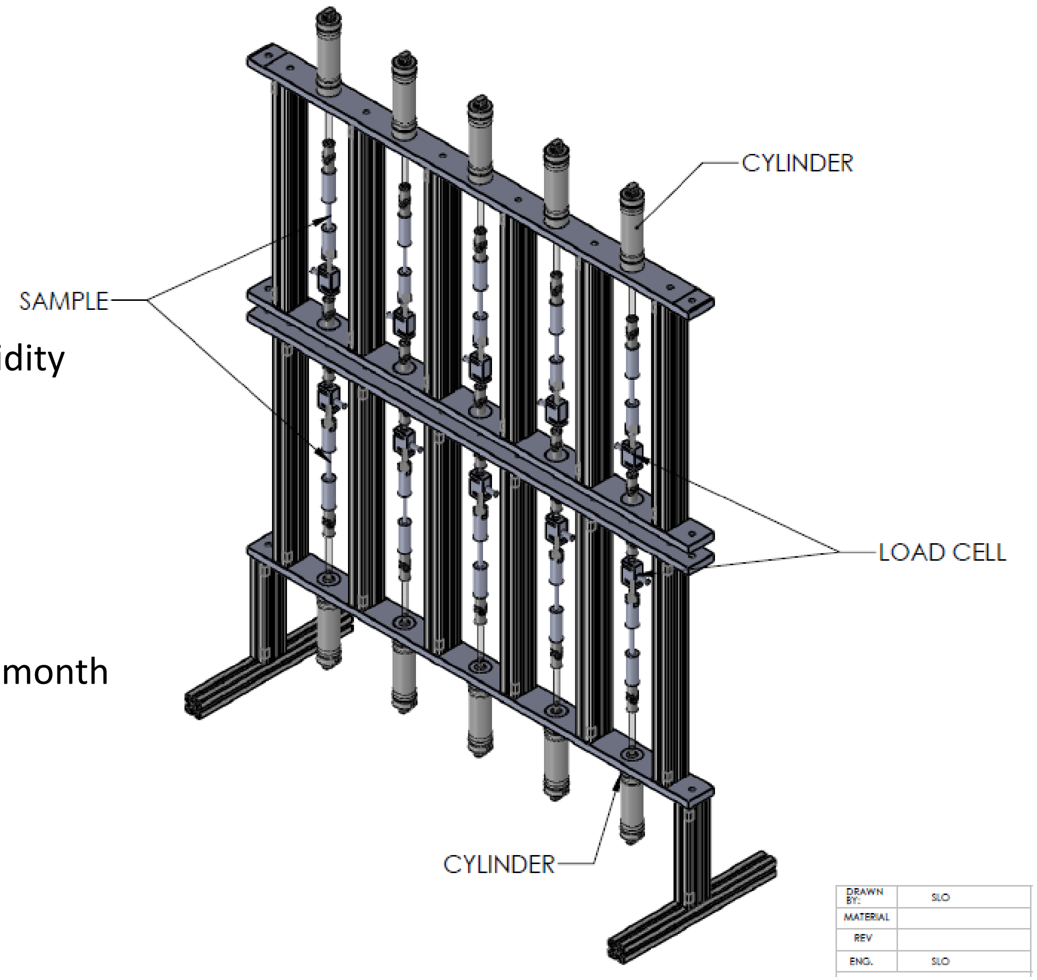
- **Stress Rupture Test**
  - Frame, Grip, Sample Tabbing
  - Design Constraints
    - 10 tensile test stands
    - Confined space of a temperature / humidity chamber.
    - Load isolation at rupture.
    - Grip methods and cost
  - Status
    - Air cylinder maintains constant load > 1 month
    - Frame meets design constraints
    - Identified available grips
    - Specimen geometry and grip methods evaluated



Fixture ready for new glass samples

Accomplishments: Stress Rupture Performance

Specimen Tabbing Methods

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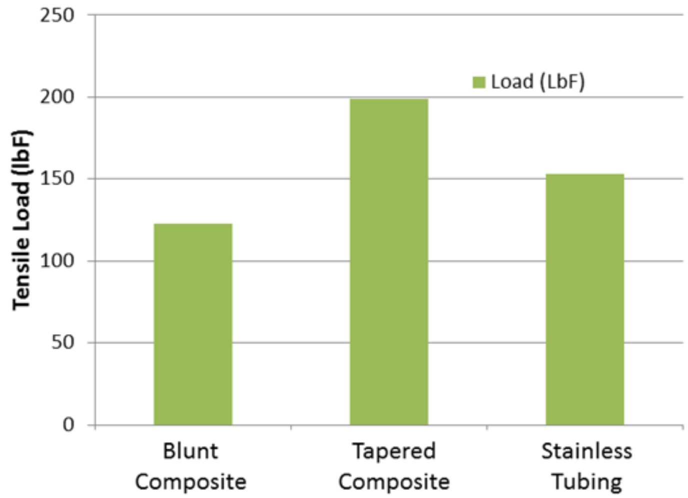
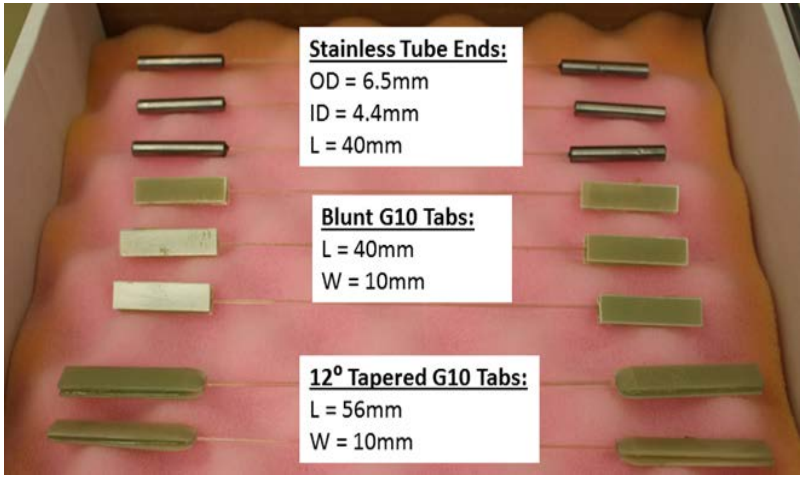
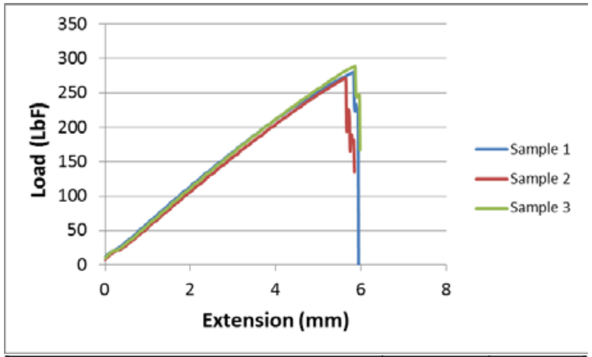
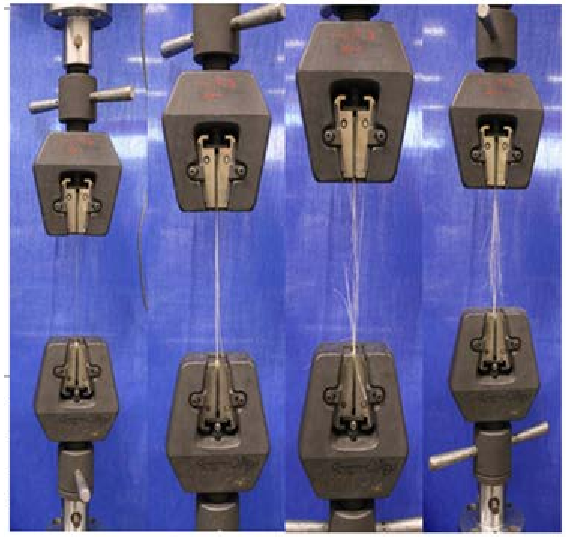






Blunt Cut G-10, 50mm*	Average	Stdev.
Peak Load (LbF)	280.23	8.70
Extension (mm)	5.93	0.07
Elongation at Break (%)	3.95	0.05



- Testing performed using self-tightening wedge grips
- Tapered samples provide the highest tensile load with the most consistent results

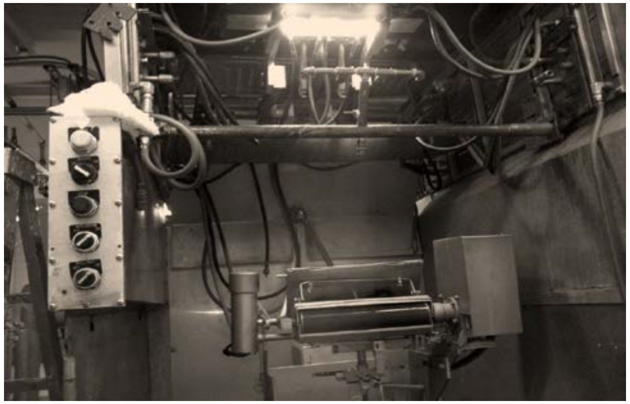
These methods provide important data for modeling activities

Collaborations

- **PPG**
  - New fiber glass and sizing development and characterization
  - New fiber production
- **Hexagon Lincoln**
  - Task modeling and design
  - Fiber evaluation
  - Task construction and tests
- **Pacific Northwest National Lab**
  - Stress rupture, performance and cost modeling
  - Sample coupon preparation and test
  - Low-density resin evaluation

Remaining Barriers and Challenges

- **Limited glass melting capacity and restriction on melting temperature**
  - Glass homogeneity
    - Uniformity of glass chemistry
    - Structure of glass network
    - Level of seeds (bubbles) in glass
  - **Resolution**: Produce fiber glass using low-risk, two-step process in BP1



- **Small bushing**
  - Down stream processes
    - Integrity of package after drying
    - Degree of contact damage in roving assembly process
  - **Resolution**: Binder chemistry production properties are considered foremost along with other performance properties
- **Ability to produce high-temperature fiber glass at scale**
  - Maximum temperature capability of large scale glass melting furnace exceeded
    - Fiber glass chemistry performance –vs- production tradeoff
  - **Resolution**: Investigation into production options planned

Proposed Future Work

- **Task 1 (PPG): Novel Fiber Development and Pilot Production**
  - Finish fiber glass and binder composition selections
  - Produce two fibers with two binders (2 x 2 combinations)
  - Complete melter capacity expansion
- **Task 2 (Hexagon Lincoln): Tank Modeling and Validation**
  - Build and test control tank
  - Build and test new tanks with 2 x 2 combinations
  - Refine tank design
- **Task 3 (PNNL): Stress Rupture Characterization and DOE Target Modeling**
  - Finish baseline stress rupture performance study
  - Complete stress rupture study with new 2 x 2 combinations
  - Complete performance and cost modeling
- **Task 7: Program Management**
  - Assess the impact of current delays on BP2 schedule

- **Continuation (Go/No-Go) Process for BP2**

Summary

- **Objective**
  - A Type IV composite overwrapped pressure vessel (COPV) reinforced exclusively with fiber glass having the composite strength matching with T700 carbon fiber composite
- **Relevance**
  - The tank will lower the composite contribution to system cost by nearly 50% with minimal impact on tank weight and capacity compared to tanks made with T-700 carbon fiber
- **Approach**
  - Develop and produce new fiber glass and binders
  - Model, build and test new tank designs with new glass/binder
  - Model performance, safety and cost of new tank
- **Accomplishments**
  - Two candidate glass fibers with pristine tensile strengths greater than T700 carbon fiber and two binder chemistry have been developed/selected for trial production
  - Tank design complete, baseline tank build with reference glass underway
  - Designed stress rupture test apparatus for controlled environment testing with preferred tabbing method and grip

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Back Up

Achieving Hydrogen Storage Goals through High-Strength Fiber Glass

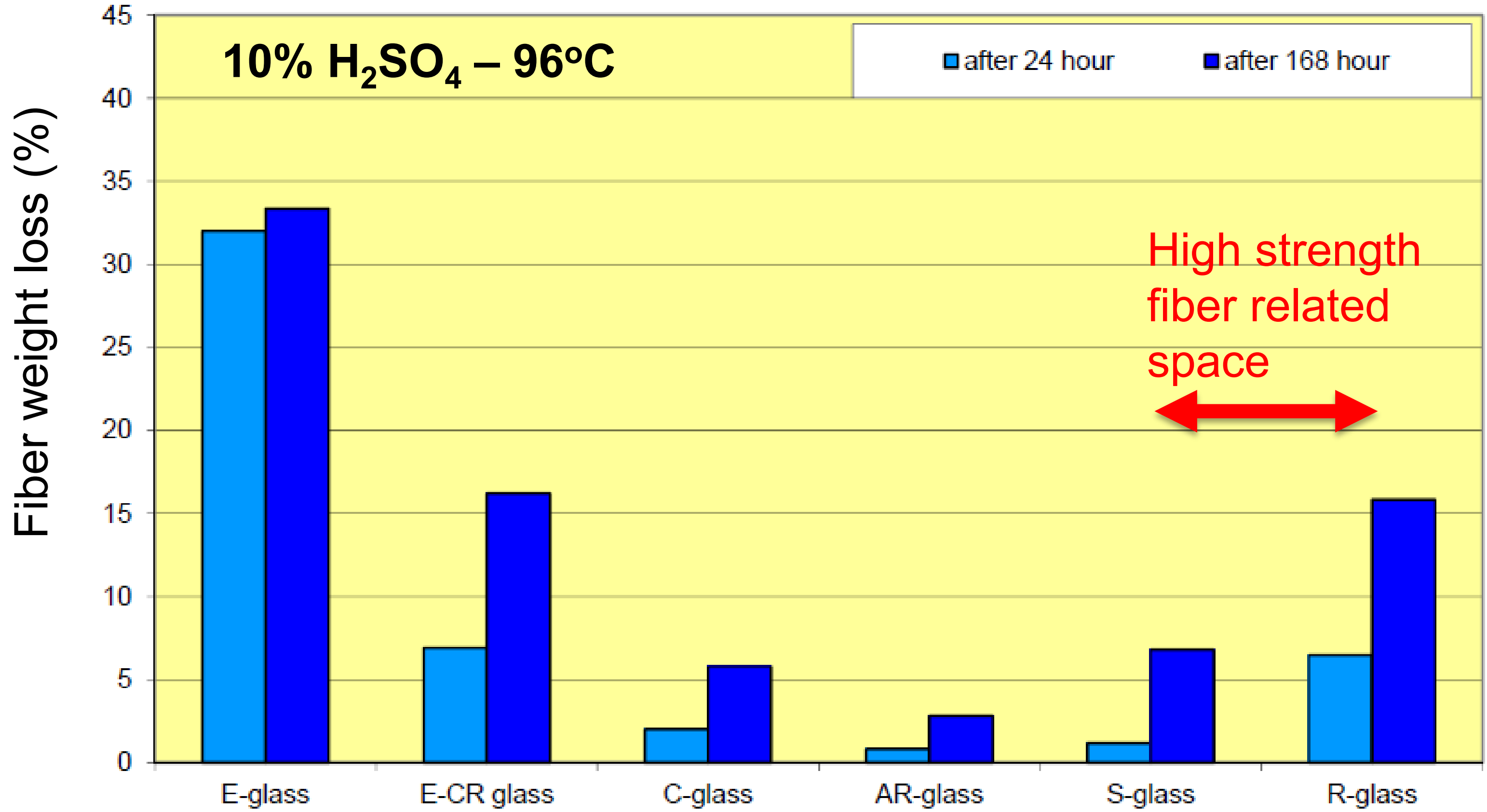
Project ID: ST115

Technology Transfer Activities

- There are no technology transfer activities to report at this time, due to the status of the project (6 months old)
- These activities are planned as part of the BP1 to BP2 Continuation process and are a major component of BP2

Composition Effect on Fiber Resistance to Sulfuric Acid

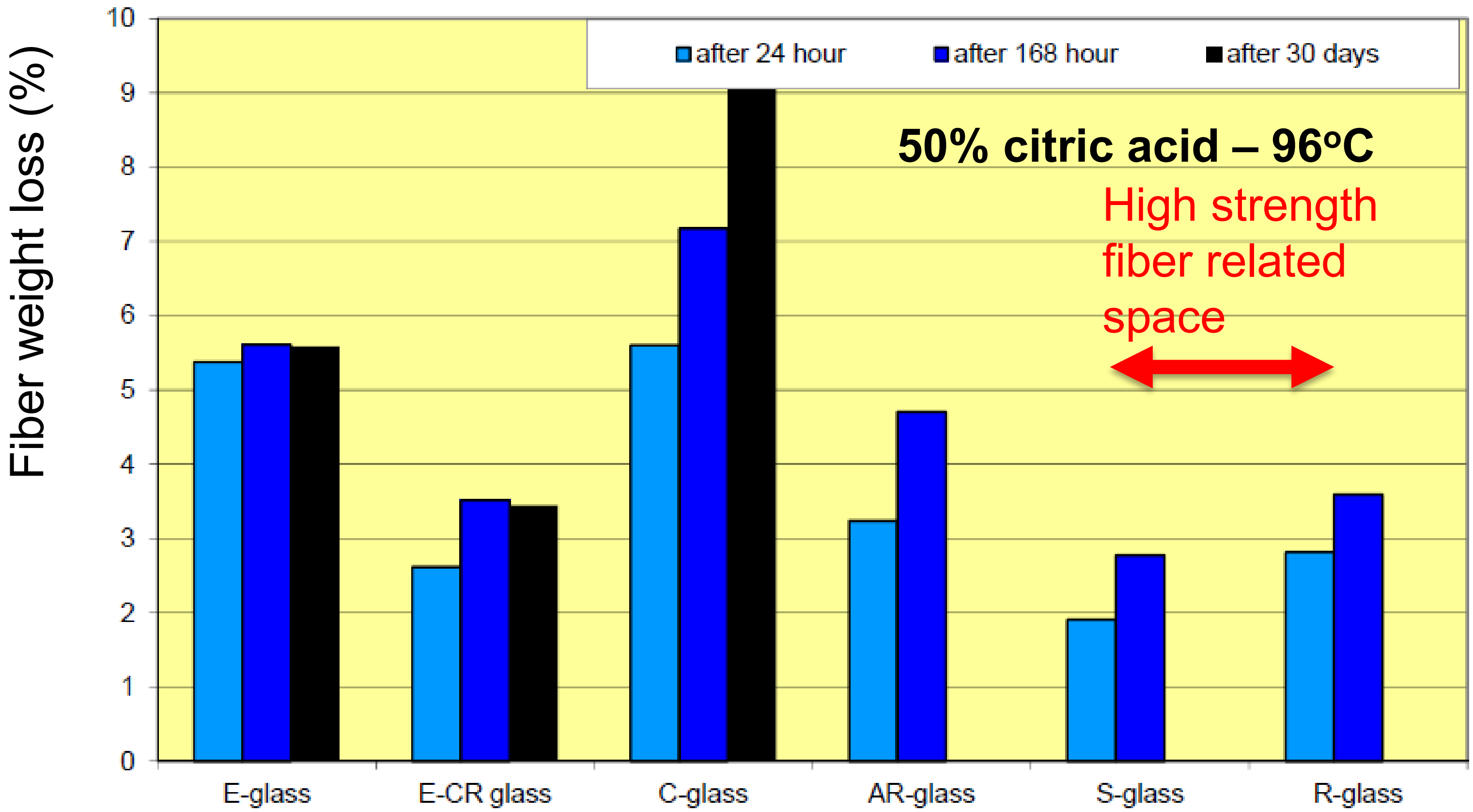
- **High strength glass fibers are expected to have good resistance to sulfuric acid as E-CR glass fibers** K. Spoo (*Chem. Proc. Symp. OCV,* 2012)



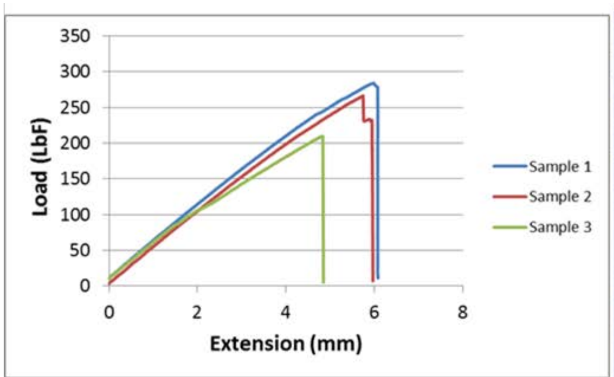
Composition Effect on Fiber Resistance to Citric Acid

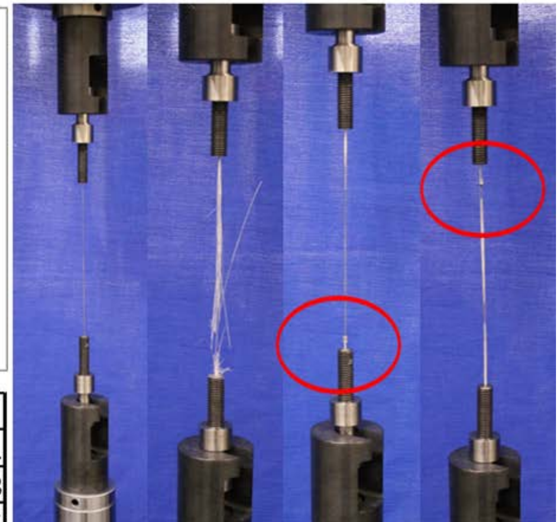
- **High strength glass fibers are expected to have good resistance to citric acid as E-CR glass fibers**

K. Spoo (*Chem. Proc. Symp. OCV*, 2012)

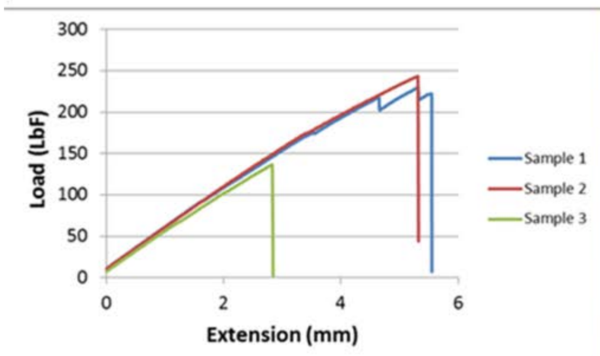


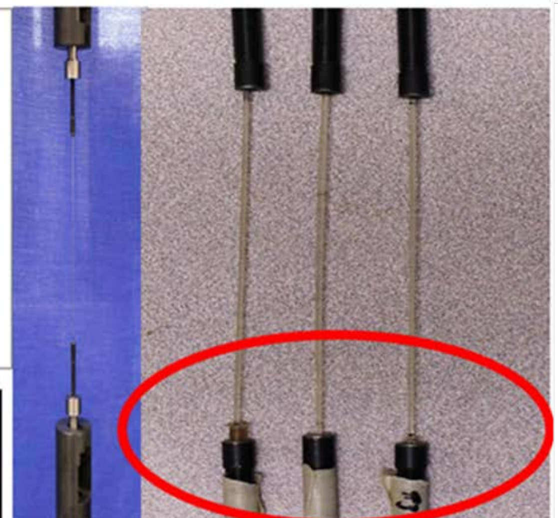
Other Specimen Tabbing Methods





7/16-14 Tapered Threaded Rod	Average	Stdev
Peak Load (LbF)	253.40	38.81
Extension (mm)	5.61	0.68
Elongation at Break (%)	3.76	0.45





1/4-20 Straight Threaded Rod	Average	Stdev
Peak Load (LbF)	203.19	58.22
Extension (mm)	4.56	1.51
Elongation at Break (%)	3.05	1.00